CHAPTER 6

COLLECTING SEDIMENT SAMPLES

Sediment Samples

Sediment chemistry samples can give information both on trends in contaminant loading and on the potential for adverse effects on sediment and aquatic biota. The evaluation of sediment concerns depends on the site and the way in which the sediment is collected. In order to compare samples over time and from site to site, samples must be collected consistently and from representative sites.

Characteristics of Sediment

Many of the chemical constituents of concern are adsorbed onto fine particles. An objective in collecting a sediment sample is to obtain recently deposited fine sediment (see Figure 6.1A). Fine sediment generally consists of a mixture of silt, clay, and some sand. Avoid hard clay, bank deposits, gravel, sand, and disturbed or filled areas (see Figure 6.1B). Any sediment that resists being scooped with a dredge is probably not fine, recently deposited material.

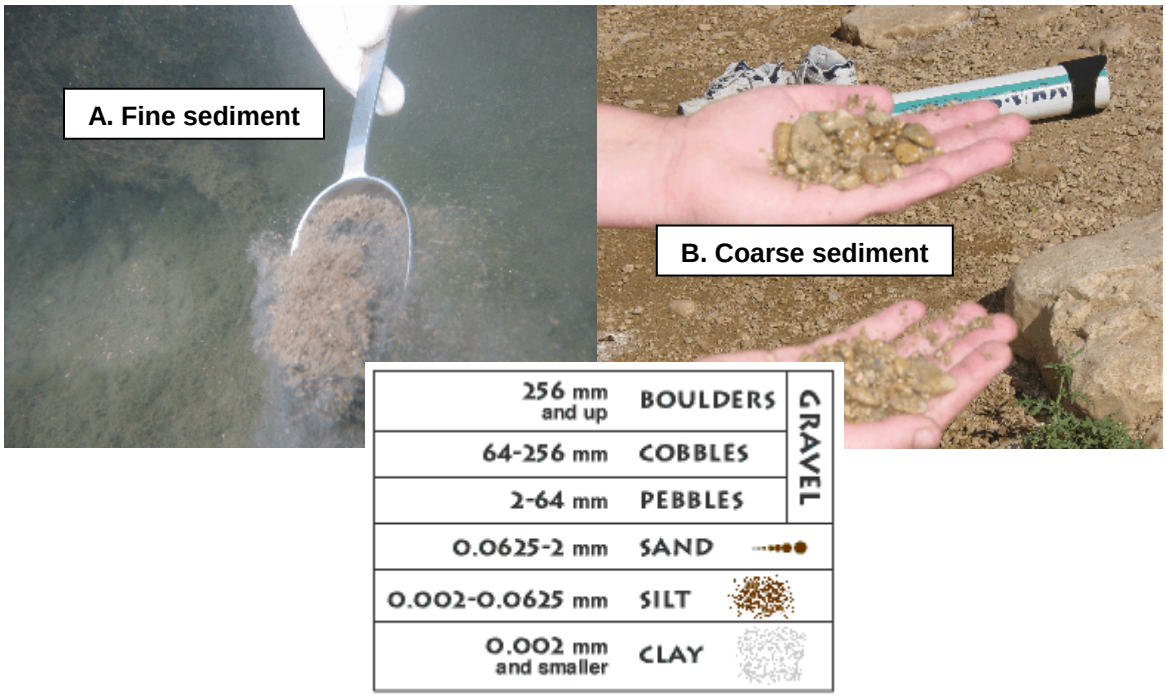

Figure 6.1. Sediment characteristics.

Characteristics of an Ideal Site

In streams and rivers, choose a sampling site with lower hydrologic energy, such as the inner (*depositional*) side of a bend or eddy where the water movement may be slower. Quiescent areas are conducive to the settling of finer materials. Reservoirs and estuaries are depositional environments where finding a suitable site is generally not a problem.

Sediment is often difficult to collect in streams with sandy, hard clay, and bedrock bottoms.

If a suitable site for collecting sediments cannot be found at a station, sampling personnel should consider collecting at a more representative location, or attempt to reschedule the sample collection. It is recommended that sites where samples are difficult to collect be eliminated from the routine sample schedule.

Selecting the Appropriate Sediment Type for Analysis

Sediment will vary from site to site and can vary between samplings at a particular site.

Streams and rivers. Sediment collection in flowing streams is often a challenge. In areas of frequent scouring, there may not be sufficient sediment for collection during or after periods of high flow. Sediment collection during these times may prove unsuccessful and may have to be rescheduled. **It is important to collect sediment from depositional areas.**

When the suspended load in rivers and streams precipitates due to reduction of velocity, most of the resulting sediment will be **fresh**. In such instances, the entire bite from the dredge subsample may be used. More often than not a dredge does not function very well in smaller streams. In these cases, sediment may have to be collected into a pan (or a jar for volatile organics) using a Teflon scoop or other suitable collection device, such as a Ponar dredge.

Reservoirs and estuaries. In areas where very little scouring normally occurs, such as in estuaries and reservoirs, the sediment will be vertically stratified. In reservoirs and estuaries, sediment is generally consolidated enough to be emptied into a flat, prerinsed plastic or Teflon pan as a cubical block of mud. Vertical stratification can then be observed. Typically, there may be light brown silt on top, followed by a gray aerobic zone overlying a typically black anaerobic layer. Because the thickness of these layers is variable, it is difficult to prescribe a certain thickness representing "recent deposits" to be sampled. Only the aerobic layer is sampled, because this zone represents more recent deposits and is where most of the benthic infauna live. If the sediment does not have an aerobic zone, collect the top 2 cm for analysis. If the aerobic zone is deeper than 5 cm, collect the sample for analysis from the top 5 cm to obtain the most recently deposited sediments (see Figure 6.2). Three or more grabs are composited for the sediment sample.

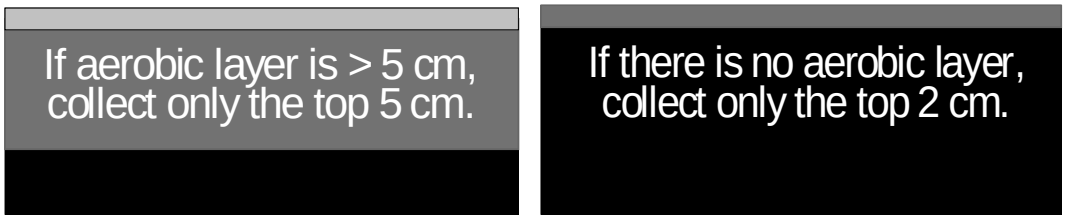

Figure 6.2. Typical sediment stratification.

Required Equipment

Basic sediment sampling equipment consists of either an Ekman or a Ponar dredge (see Figure 6.3), a plastic or Teflon pan or bucket, Teflon or stainless steel scoops, and 500 mL glass jars with Teflon lids for metals, organics, and conventionals (detergent washed and DI rinsed). See Chapter 9 for the list of basic SWQM equipment.

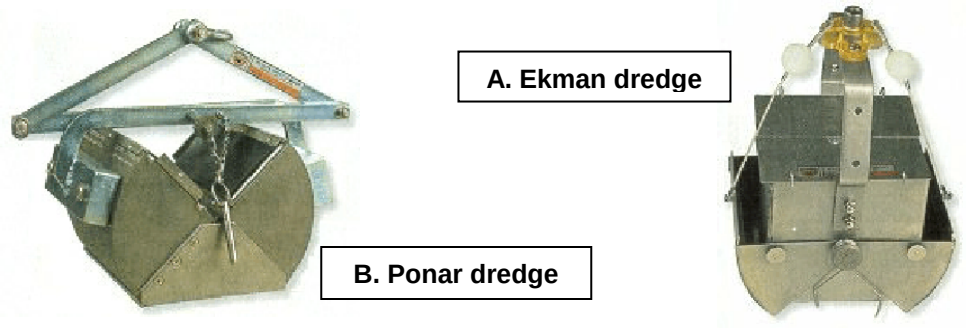

Figure 6.3. Sediment dredges.

General Collection Procedure

Collect sediment samples after the water samples. Submit sediment samples for metals and organics in separate pre-cleaned glass jars. Acid-rinsed jars (for metals) and solvent-rinsed jars (for organics) are not required.

After choosing an appropriate site, collect the sample using the following procedure: After setting the dredge in the open position, slowly lower it to the bottom, disturbing the substrate as little as possible. The idea is to lower the dredge at a rate that allows for an even grab of sediment without it coming out of the dredge. Adjust this method as necessary to accommodate the type of sediment at a site. Retrieve the closed dredge at a moderate speed (less than 2 ft/s). Upon retrieval, examine the grab to ensure that the sediment surface is undisturbed.

Things to consider when collecting sediment samples with a dredge:

- The mud surface must not be pressing out of the top of the sampler. If it is, lower the dredge more slowly.
- Overlying water must not be leaking out along the sides of the sediment in the dredge. This ensures that the surface sediment is not washed out.
- Sediment surface must be flat and level in the sampler. If it is not level, the dredge has tilted over before closing.
- The water overlying the sediment in the dredge must be very gently decanted by slightly tipping the dredge with the lid closed until the water runs out the top. Decanting should remove all of the overlying water but not the surface sediments. The laboratory reports the percentage of water for the sample. Overlying water is not included in the sample container. The sample should contain as little water as possible and must not have more water than sediment.
- Empty the sample into a pan (see Figure 6.4A). Examine the sediment for depth of penetration, color, thickness of top aerobic zone, and texture (see Figure 6.4B). Record these observations in the logbook.
- ***For estuarine and reservoir samples***—collect the top aerobic zone (up to 5 cm) from at least three subsamples and composite in a pan (see Figure 6.5).

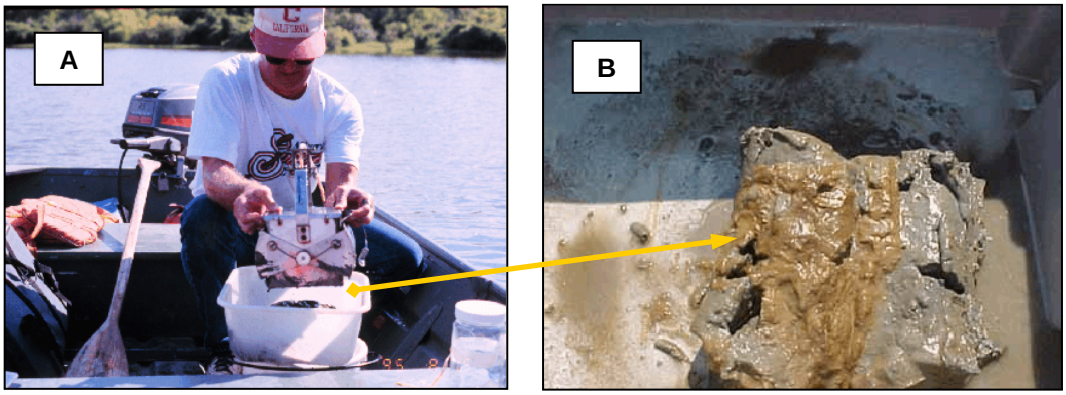

Figure 6.4. (A) Emptying the sample into a pan. (B) Sediment sample.

- ***For stream samples***—the entire grab may be composited. In streams with excessive bottom debris (rocks, sticks, leaves) where the use of an Ekman or a Ponar dredge is ineffective (the dredge does not close, causing loss of sediment), samples may be collected by hand, using a clean Teflon scoop or stainless steel spoon. Otherwise, exclude the bottom most layer and composite. Handle sediment as described in the following sections on metals and organics.

Figure 6.5. Compositing a sediment sample in a bucket or pan.

Note: Even if the volume of one dredge grab is sufficient to fill the required number of containers, a minimum of three separate dredge grabs are required for all sediment samples. This is required to get a representative sample for an area, rather than a single location.

Metals, Pesticides, and Semivolatile Organics in Sediment

For metals and semivolatile organics, a minimum of three grabs may be mixed in a clean pan or bucket, and distributed to the sample containers. Mixing is generally done with a Teflon or stainless steel scoop or spoon. Remember, the pan and dredge must be cleaned with native water between sites. Try not to transfer rocks, large leaves, or sticks into the container.

Collecting Metal, Semivolatile, or Pesticide Samples

Make sure the sample volume is adequate, but the containers do not need to be filled to the top. Seal the jars with the Teflon liner in the lid. There is no need to eliminate head space in the jar, as "no head space" applies only to volatile organics. If semivolatiles are requested, use only glass, Teflon, or metal items to collect samples; plastic equipment can contaminate a sample. See Table A7.3 of the SWQM Program QAPP for a list of methods used to analyze sediment (see Appendix A).

Sediment Conventionals

Sediment conventionals—including grain-size analysis, total organic carbon, and percent solids (moisture content)—are always requested with samples for both organics and metals samples. They are extremely important in determining the availability of metals and organics in sediment. The collection method is the same as for metals, semivolatile organics, and pesticides.

Volatile Organics in Sediment

For volatile organics, sediment collection slightly differs in order to reduce the release of volatiles. Composite the grabs by transferring an equal portion of successive grabs (at least three) to the sample containers with a clean Teflon or stainless steel scoop. Distribute each grab to the sample containers in a different sequence—rotating the order in which sediment is added to the jars.

First add to Jar 1, 2, then 3. Next start with Jar 2, 3, and 1, and so on until the jars are filled. This method is used when volatile organics are to be run on a sample. **Fill each container to the top, leaving no head space,** and seal the jars with the Teflon liner in the lid. "No head space" means that sediment must be to the lip of the jar. Compositing a sample in a pan or bucket may release volatiles from the sediment. Try not to transfer rocks, large leaves, or sticks into the container. To reduce handling of a volatile organic sample, sediment can be taken directly from the dredge (see Figure 6.6).

Figure 6.6. Collecting a sample directly from the dredge.

Sediment samples with high water content will settle leaving a layer of water on top. A sample with no head space in the field may arrive with a half inch or more of water on

top. This is not a problem for metals and conventional parameters but will cause headspace in a volatile organic sample.

Volatile organics are no longer collected routinely. If they are collected, it is generally for a special project.

Sample Containers

Place sediment samples in glass jars with Teflon liners. Jars for sediment samples require no special treatment. Acid and solvent rinses are no longer required for metals or organics in sediment.

Sample Size

Metals, organics (volatile, semivolatile, and pesticides), and conventionals in sediment each require 500 grams of sample. If you are collecting both organics and metals at the same station, fill three glass jars—one for metals, one for organics, and one for conventionals. When collecting only metals or organics at a station, fill two glass jars—one for metals (or organics), and the second for conventionals. Please note that the number of jars and sample size is a suggestion based on TCEQ laboratory protocols. The number, type of container, and sample size can be determined by the laboratory doing the analysis.

***Note*:** Even if you can get the entire sample volume in one grab, at least three grabs are required to get a more representative sample.

Labeling

Label the jars with the station ID and date of collection, as well as the type of analysis requested (metals, conventionals, organics, pesticides).

Preservation

Immediately place the labeled jar on ice, cool it to < 6°C (do not freeze it), and keep it in the dark until delivery to the laboratory. See Table 6.1.

Handling and Shipping Samples

Due to increased shipping restrictions, samples being sent via a freight carrier require additional packing. Even if care is taken to seal an ice chest, leaks do occur. To avoid leaks, place samples and ice in a large plastic bag inside the chest for shipping. The bag can be sealed by simply twisting it closed while removing excess air and taping the tail down. Leaking ice chests can cause samples to be returned or to arrive at the lab beyond the holding time. Some shipping companies, depending on the location, may require this extra step before shipping ice chests.

Place laboratory analytical request forms corresponding to samples in the ice chest in a Ziploc bag, taped to the inside of the lid. Secure the lid with tape. This is essential if samples and ice are not in a large plastic bag. This method of handling chain-of-custody forms should not override existing protocols of the TCEQ region or sampling organization.

Take special care when shipping glass. When shipping a combination of plastic and glass, plastic containers can be used to cushion glass containers. If most of the containers are

glass, extra care must be taken to prevent breakage during shipping. Suitable shipping materials include bubble wrap or plastic netting made for slipping over jars.

Field Notes

In the field logbook, record the depth at the location where the sample was taken. Record a gross description of the sample, including its color and texture, the number of grabs, the thickness of the grab sample, and the depth of the aerobic zone included in the composite sample. This information can be reported as comments with the sediment analytical results.

Field notes for each sample should include the following information:

Type of composite. Sediment composites are considered both time and space composites.

Number of grabs. Report the number of grab samples used in the composite. The minimum is three.

Start and end time and date. These are required fields for reporting composite-sample data.

Start and end depth. These are required fields for reporting composite-sample data. This number should reflect the depth of the water over the sediment. The depth is never reported as zero, since sediment must be covered by water at the time of collection. The start and end depth may be the same.

Data Reporting

Appropriate procedures and parameter codes necessary for submitting data are discussed in the *SWQM DMRG*.

Table 6.1. Quick reference guide—sediment-sample collection methods, preservation, storage, and handling**.**

Parameter	Recommended Containers	Sample Volume (grams)	Preservation	Hold Time
SEDIMENT				
Metals-All but mercury	1-pint glass jar with Teflon-lined lid; special treatment not required	500	Cool to < 6°C, dark; do not freeze	180 days
Metals-mercury				28 days
Organics (pesticides, semivolatiles, volatiles)	1-pint glass with Teflon-lined lid; special treatment not required	500	Cool to < 6°C, dark; do not freeze	14 days
Conventionals TOC, grain size, percent solids (moisture content)	1-pint glass jar with Teflon-lined lid	500	Cool to < 6°C, dark; do not freeze	28 days

Procedure for Collecting Sediment Samples

- Label containers with tag number, station location, date, and sample type.
- Wash dredge pan and bucket with ambient water before and after sample collection.
- Slowly lower the dredge into the sediment. Raise closed dredge at a rate of about 2 ft/s.
- Slowly decant overlying water. Empty sediment grab into a pan.
- **Composite a minimum of three grab samples.**

For **metals, semivolatile organics, and pesticides:**

- Composite sample may be collected in a pan or bucket. Put a minimum of three grab samples in a bucket, stir with Teflon scoop or spoon, and transfer to the container(s).
- Place samples into clean glass jars with Teflon lids. Put samples in dark and on ice. Containers do not have to be filled to the top.
- Record in field notebook the location and sediment description (color, texture, odor, and number of grabs).
- Put on ice and ship to lab.

For **volatile organics** (typically collected as part of a special project):

- Since compositing in a pan or bucket can release volatile compounds, care must be taken to limit disturbance of the sediment during collection and compositing.
- For first grab—put first scoop off top into Container 1; second scoop into Container 2; and third scoop into Container 3.
- Second grab—put first scoop in Container 3; second scoop in Container 2; and third in Container 1.
- Keep rotating until the jars are full.
- Place samples into clean glass jars with Teflon lids. Put samples in dark and on ice. **Fill containers to top with no head space.** No head space = sediment to the top of the jar.
- Record in field notebook the location, sediment description (color, texture, odor, and number of grabs).
- Put on ice, cool to < 6°C, and ship to lab.

Reject grab if: mud is coming out of top of dredge, overlying water is leaking out of dredge (removes surface sediment), or sediment is sloping in the dredge (surface of sediment bite in dredge should be relatively flat). This may be difficult for flowing-water samples. Entire bites from a flowing water site may be used.

For ***estuarine and reservoir samples***, collect the top aerobic zone (up to 5 cm) from at least three subsamples and composite a minimum of three grabs.

For ***stream samples***, the entire grab may be composited. Otherwise, exclude the bottommost layer and composite a minimum of three grabs.