



SEDIMENT COLLECTION GUIDANCE

SCOPE AND APPLICATION

1. This procedure describes general methods for the collection of sediment samples for use in toxicity and bioconcentration testing. **These instructions are provided as a courtesy to clients; project-specific sampling requirements may differ from the procedures described in this document.**
2. This is intended to provide a general overview of sediment collection methods. For detailed information on sediment collection procedures see EPA 2001b and ASTM 2006.

PROCEDURE SUMMARY

Sediment samples are collected in the field using appropriate sampling devices, composited and homogenized as necessary, transferred to high density polyethylene jars and then shipped or transported on ice to the lab.

INTERFERENCES

1. Insufficient chilling of samples during collection and shipment may result in toxicity being underestimated and/or tests being invalidated by the regulatory authority.
2. Delayed or improper shipping of samples may result in samples exceeding holding times.
3. Improper handling may adversely affect sample condition.
4. Exposure of sediment samples to air during collection and transfer should be minimized.
5. Contamination during sampling may result in toxicity being over or underestimated. Direct contact with the following materials should be avoided: PVC, natural or neoprene rubber, nylon, talcum powder, polystyrene, galvanized metal, brass, copper, lead or other (non-stainless steel) metal materials, soda glass, paper tissues, and painted surfaces.

SAFETY, WASTE MANAGEMENT AND POLLUTION PREVENTION

1. Collection of sediments may pose risks to personal safety and health. Standard laboratory and field safety procedures must be adhered to at all times. Gloves must be worn at all times when handling samples.
2. Solvents, cleaners and other potentially hazardous or toxic materials used for equipment cleaning must not be released into the environment during field collections. Collect such materials into appropriate labeled containers.

EQUIPMENT AND SUPPLIES

1. Sample containers (high density polyethylene (HDPE) wide-mouth jars, 500 ml to 4 L)*
2. Sampling device (e.g. Van Veen, Ponar, Petersen, Smith-Mac, gravity corer, vibracorer, etc.)
3. Thermometer
4. pH meter and calibration standards
5. D.O. meter
6. Refractometer or conductivity meter
7. Small beakers for subsample measurements
8. Powder-free latex or nitrile gloves
9. Cooler or insulated shipping container*
10. Ice packs or wet ice and heavy-duty plastic bags*
11. Chain-of-custody forms*
12. Electronic depth finder
13. Waste bucket or jar

*Items provided in Coastal Bioanalysts, Inc sample kit



REAGENTS AND STANDARDS (As needed for cleaning gear (see decontamination section below))

1. Acetone (ACS Reagent grade)
2. Ethanol (ACS Reagent grade)
3. Nitric acid, 10% (ACS Reagent grade)
4. Alconox or similar lab detergent
5. Distilled or deionized water (lab grade)

PROCEDURE

Sample volume

Sample volume depends on the species being tested; consult with laboratory personnel for project-specific volumes. Generally a 1 L sample from each site will suffice for *H. azteca* or *C. tentans* (2 L if both species tested). Saltwater amphipods require 4 L per site.

Gear Selection

Maintaining sample integrity during collection, storage and shipping is the primary concern because disruption of sample structure may alter physicochemical and biological characteristics. Selection of appropriate gear is important in this respect and depends on the site location, accessibility, sediment structure, volume of sediment needed and whether the primary concern is historical contamination (deeper sediment layers) or recent contamination (surficial layers). Study-specific data quality objectives (DQO) or instructions may dictate these decisions. For detailed guidance refer to sections 3.1 and 3.2 of EPA 2001b.

Related equipment used in the collection of samples, such as spatulas, mixing bowls, scoops, etc. should be constructed of materials appropriate for the contaminants of concern (e.g. stainless steel or Teflon for organics, polyethylene or polycarbonate for metals). Pre-cleaning of sampling gear is detailed in ASTM 2006 and EPA 2001b.

Decontamination of Sampling Gear

For most applications rinsing of the sampling device with site water between collections is sufficient. If highly contaminated sites are suspected or known to be present, additional cleaning between collections may be necessary to prevent cross-contamination. ASTM recommends 1) soap and water, 2) distilled/deionized water rinse, 3) acetone or ethanol rinse, and finally 4) site water rinse. Waste rinse solvents should be collected in labeled hazardous waste containers. Acetone or nitric acid (10%) can be used as specific rinses for organics or metals respectively.

Sample acceptability

Samples should be inspected as soon as the sampling device is retrieved. If the sample fails to meet acceptability criteria the rejected sample should be discarded in a manner that will not affect subsequent samples at that site and a new sample should be collected as close as possible to the original sampling station but slightly upstream of any stream or tidal current. Table 1 below outlines sample acceptability criteria. Additional site-specific criteria or DQOs may also exist.



Table 1. Sediment Sample Acceptability Criteria (from EPA 2001b)

Grab Samples:	
√	The sampler is not overfilled so that sediment surface is touching the top of the sampler
√	Overlying water is present (indicating minimal leakage). The overlying water is removed before sediment is retrieved from device.
√	The overlying water is relatively clear, not overly turbid.
√	The sediment-water interface is intact and relatively flat with little evidence of channeling or washout.
√	The desired depth of penetration was achieved.
√	There is no evidence of sediment loss (e.g. incomplete sampler closure, penetration at an angle, tilting upon retrieval)
Core Samples:	
√	The core sampler was not inserted at an angle or tilted upon retrieval.
√	The core was collected to the required sediment depth of study objectives with no loss of sediment.

Field measurements and observations

Study-specific requirements and DQOs for field measurements and observations may exist. In addition to collection information such as sample date and time, ambient weather conditions, tide, etc., any information regarding sample integrity should be noted. Table 2 outlines field information typically documented:

Table 2. Typical field collection data

Measurement or Observation	Matrix		Notes
	Sediment	Overlying Water	
PH	√	√	
Temperature	√	√	
Salinity or Conductivity		√	Conductivity in freshwater systems, salinity in estuarine/marine
Dissolved oxygen		√	
Depth	√	√	Sediment penetration depth as well as water column depth.
Visual	√	√	Photographs may be helpful, especially if needed to document stratification of core samples. Also note sediment texture (e.g. sand, mud) color, presence of biota, sheens or debris.

Sample compositing and homogenization

Samples collected from sequential grabs at the same site or different sites may need to be composited to meet volume or study requirements. Transfer subsamples as needed to a clean mixing bowl or bucket and mix sample thoroughly with a spoon or paddle for several minutes. Use clean glass, polyethylene, or high grade (e.g. 316) stainless steel mixing implements (see above). Perform mixing as quickly and efficiently as possible to reduce sample oxidation and loss of volatile components. Special procedures (e.g. glove box with inert atmosphere for metals, minimal UV exposure for PAHs) may need to be used depending on the study objectives (refer to EPA 2001b). Collection and testing of replicate subsamples from within the homogenate can be used to assess the effectiveness of the homogenization process.



Sample Transport and Storage - General

After samples or homogenized sample composites are transferred to appropriate containers, the containers of sample are chilled immediately. Chilled samples are then placed in insulated shipping containers, packed with ice or ice packs, and then delivered (by courier, UPS or FedEx) to the testing laboratory as soon as possible. Samples must be delivered to the lab with sufficient time to allow for sample processing and test setup while meeting the maximum sample holding time (2-8 weeks, depending on method or regulatory requirements). Note that if samples to be used in the same study are collected over two or more days, the sample hold time begins upon collection of the first sample.

1. Because samples may need to be used within 2 weeks of collection of the first sample, time is important. Coordination of sample and shipping dates with the laboratory is critical.
2. Samples must arrive chilled (>0 to 6°C). Tests initiated with samples exceeding these shipping/holding temperatures may be invalidated by the regulatory authority.
3. **Sediment samples must be immediately placed in a refrigerator or on ice to chill to < 6° C prior to packing in a shipping container. Ice and/or frozen ice packs used for shipping cannot both chill down a warm sample and hold the sample at ≤ 6° C during transit.**
4. All applicable portions of the chain-of-custody form provided must be completely filled out by the person collecting samples. Common mistakes are: no sampling date or time, test requirements not indicated, incorrect date and incorrect station-lab ID numbers recorded.

Filling Sample Containers

1. Pre-label sample container(s) prior to going out in the field as dry bottles are much easier to label.
2. Collect sample as described above and transfer to provided sample container(s).
3. Fill the container(s) as full as possible with sample and cap; headspace in the sample container should be minimal.
4. Check that all labeling on sample bottle is intact and correct. If necessary, make corrections using a permanent marker (e.g. Sharpie), writing directly on the bottle and lid.
5. Immediately place the grab sample in a refrigerator or on ice to chill to < 6° C prior to packing in shipping container (see below).

Shipping

1. Place chilled samples and frozen icepacks in the shipping containers provided. The frozen ice packs will maintain the temperature of a previously chilled sample but will not both cool down a warm sample and maintain it at ≤ 6°C.
2. IF WET ICE IS USED: Leaking boxes will be returned to the sender by FedEx and UPS (they treat leaking boxes as hazardous waste). Be sure to double-bag ice (using heavy trash bags) prior to placing it in with samples.
3. Place lid on shipping container and tape shut using 2" packaging tape, duct tape or other suitable material.
4. Ship samples by overnight carrier (UPS or FedEx) or courier. Samples must be received in the lab no later than the day following shipment (i.e. use Priority Overnight service) to insure holding temperature is maintained. DO NOT use the US Postal Service or Airborne Express as these carriers do not provide reliable delivery service to our location.
5. FRIDAY SAMPLES (SATURDAY DELIVERY):
 - a. Carefully follow UPS or FedEx Priority/Saturday Delivery instructions on the airbill.
 - b. Indicating only Priority Overnight will result in the sample arriving at the lab the next business day (i.e. Monday), invalidating the samples due to temperature. The Saturday Delivery box must be checked.
 - c. Be sure that a Saturday Delivery sticker is attached to the box next to the address label.
 - d. Fax (804-695-1129), call (804-694-8285) or email (webcontact@[coastalbio.com](mailto:webcontact@coastalbio.com)) the shipping number for all Friday samples. This will enable us to track the sample on Saturday if there are delivery problems.

Ship to: **Sample Receiving**
 Coastal Bioanalysts, Inc.
 6400 Enterprise Ct.
 Gloucester, VA 23061



QUALITY CONTROL/DATA ASSESSMENT & ACCEPTANCE CRITERIA

1. Only properly collected sediments (see Table 1 for criteria) should be used.
2. All supporting activities, such as field measurements (e.g. Table 2), field replicates, etc. should be conducted in accordance with any study specific requirements or DQOs.
3. Shipping temperature and sample hold times (see general section above) should be within method specific criteria.
4. Samples may be deemed conditionally acceptable if there are minor deviations from specified conditions; determination of conditional acceptance shall be made by the client, project officer and/or permitting authority in charge of sampling.

CALIBRATION AND STANDARDIZATION

All field instruments should be calibrated with appropriate NIST-traceable standards where available.

REFERENCES

ASTM 2006a. Standard Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing and for Selection of Samplers Used to Collect Benthic Invertebrates. E 1391-03. In: *Annual Book of Standards. Vol. 11.05 Biological Effects and Environmental Fate; Biotechnology; Pesticides*. American Society for Testing and Materials, West Conshohocken, PA

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EPA 2000. *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates, 2nd Edition*. EPA/600/R-99/064, Office of Research and Development, Washington, DC.

EPA 2001a. *Methods for Assessing the Chronic Toxicity of Marine and Estuarine Sediment-associated Contaminants with Amphipod *Leptocheirus plumulosus**. EPA/600/R-01/020, Office of Research and Development, Washington, DC.

EPA 2001b. *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual*. October 2001. EPA-823-B-01-002.