

Field Sampling Plan for the 2013 Sediment and Floodplain-Soil Sampling Program for the Anniston PCB Site

Version 2.0

Prepared for:

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Prepared – October, 2013 – by:

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Field Sampling Plan for the 2013 Sediment and Floodplain-Soil Sampling Program for the Anniston PCB Site - Approval Page

On behalf of the Natural Resource Trustees for the Anniston PCB Site, the following representatives have reviewed the Field Sampling Plan for the 2013 Sediment and Floodplain-Soil Sampling Program for the Anniston PCB Site and find that the procedures outlined in this document will result in data that can be used for evaluating the chemistry and toxicity of environmental samples collected at the Anniston PCB Site, Alabama.

Karen W Marlowe 12-3-2013
Karen Marlowe Date
United States Fish and Wildlife Service

Carl Orazio 8/12/2015
Carl Orazio Date
United States Geological Survey

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1.0 Introduction

The Anniston PCB Site is located in the north-eastern portion of Alabama in the vicinity of the municipality of Anniston in Calhoun County. Although there are a variety of land use activities within the Choccolocco Creek and Coosa River watersheds, environmental concerns in the area have focused primarily on releases of polychlorinated biphenyls (PCBs), which were manufactured by Monsanto, Inc. at the Anniston facility from 1935 to 1971. During production, PCBs may have been released from the facility in production waste, effluent discharges, uncontrolled releases from landfills, accidental spills, stormwater runoff, and other sources. The total mass of PCBs released from the Anniston facility is uncertain, however.

In response to public concerns, a remedial investigation/feasibility study (RI/FS) is being conducted by the United States Environmental Protection Agency (USEPA) and Solutia/Pharmacia (S/P) to assess risks to human health and ecological receptors and to evaluate remedial options for addressing environmental contamination at the Anniston PCB Site. The Anniston PCB site consists of the entire area in and around Anniston where PCBs have come to be located (i.e., the vicinity of the Anniston facility and within the water courses and floodplains of Snow Creek, Choccolocco Creek, and Lake Logan Martin to the Logan Martin Dam and potentially beyond). Based on the available data, environmental media collected in the Anniston PCB Site show elevated levels of PCBs and/or other contaminants. Hence, there are potential risks to ecological receptors exposed to contaminated environmental media.

In addition to the RI/FS, the Natural Resource Trustees (NRTs; including United States Fish and Wildlife Service, Geological Survey of Alabama, and Alabama Department of Conservation and Natural Resources) are conducting a Natural Resource Damage Assessment (NRDA) to evaluate injuries to, loss of, or destruction of natural resources associated with releases of hazardous substances from the Anniston PCB Site. As indicated in the Stage 1 Assessment Plan, the NRTs will use data and information from the RI, from Trustee-led studies, and from other sources to determine if injuries to natural resources have resulted from releases of hazardous substances in the vicinity

of the Anniston PCB Site. The NRTs will then evaluate the magnitude and extent (both spatially and temporally) of any natural resource injuries that are identified to support damage determination and restoration planning.

As part of the overall natural resource injury determination and quantification process, the NRTs recently (i.e., May, 2012) conducted a preliminary evaluation of ecological service losses (ESLs) to surface water, geologic, and biological resources associated with releases of PCBs in the vicinity of the Anniston PCB site. To support the preliminary evaluation of ESLs, the available data on PCBs and other contaminants in environmental media were acquired, evaluated, and compiled. Other data and information, potentially relevant to the NRDA, were also compiled at that time (e.g., whole-sediment toxicity data). Evaluation of the available information revealed that there were a number of data gaps that reduced the confidence in the results of the preliminary evaluation of ESLs. More specifically, limitations on the quality, quantity, and/or spatial coverage of the available sediment chemistry, soil chemistry, and tissue chemistry (e.g., invertebrates, fish, bird eggs) data were considered to be important sources of uncertainty in the preliminary evaluation of ESLs. Hence, addressing these critical data gaps was identified as a priority by the NRTs.

The 2013 sediment and floodplain-soil sampling program is intended to address uncertainties and data gaps associated with the spatial characterization of sediments and floodplain soils in the vicinity of the Anniston PCB Site. This program will provide the data needed to characterize un-sampled and under-sampled areas within the study area, determine if other COPCs (i.e., in addition to PCBs) occur at concentrations sufficient to injure resources in the study area, and confirm that the data collected by S/P provide a reliable basis for evaluating injury to natural resources in the study area (see MacDonald *et al.* 2012 for more information). In addition, this sampling program will provide the information needed to determine if additional sampling is required to fully evaluate the nature and extent of contamination at the Anniston PCB Site. The supplemental sediment and floodplain-soil chemistry data collected during this sampling program will be used to refine the estimates of ESLs

associated with injuries to surface water, geologic, and biological resources in the study area.

In order to integrate all technical and quality aspects of this project, including planning, implementation, and assessment, it is essential that a Sampling and Analysis Plan (SAP) is developed and that the associated quality assurance/quality control (QA/QC) requirements will be established and adhered to. A SAP consists of three primary elements, including:

- Quality Assurance Project Plan (QAPP; see the site-wide QAPP for the NRDA of the Anniston PCB Site [MacDonald *et al.* 2013]);
- Field Sampling Plan (FSP; this document); and,
- Health and Safety Plan (HSP; see the site-wide HSP for the NRDA of the Anniston PCB Site [Wainwright *et al.* 2013]).

The QAPP describes the policy, organization, functional activities, and QA/QC protocols necessary to achieve project data quality objectives (DQOs) dictated by the intended use of the data. This document, the FSP, provides guidance for all fieldwork by defining in detail the sampling and data-gathering methods to be used during the project. More specifically, this FSP describes the methods and procedures that will be applied during the collection, handling, and preparation of the environmental samples that are to be obtained during the sampling program. The HSP provides an overview of the various considerations required to ensure the health and safety of all personnel during the implementation of the sampling programs.

2.0 Study Purpose and Objectives

The purpose of the 2013 sediment and floodplain-soil sampling program is to address uncertainties and data gaps associated with the spatial characterization of the concentrations of PCBs and other contaminants in sediments and floodplain soils in

the vicinity of the Anniston PCB Site (i.e., the study area; Figure 1). The sediment and floodplain-soil chemistry data collected during this sampling program are intended to address the limitations of the available data (i.e., incomplete spatial coverage, limited suite of analytes) and to confirm the reliability of data collected by S/P. In addition, sediment and floodplain-soil chemistry data are required to refine the estimates of ESLs associated with injuries to surface water, geologic, and biological resources in the study area. More specifically, the objectives of the 2013 sediment and floodplain-soil sampling program are to:

- **Determine the concentrations of PCBs in sediments and floodplain soils in un-sampled and under-sampled areas within the study area:** This sampling program will provide the data needed to characterize un-sampled and under-sampled areas within the study area in order to address critical data gaps identified in the preliminary evaluation of ESLs. Collection and chemical analysis of sediment and soil samples will provide supplemental data on the concentrations of PCBs and other contaminants, providing a basis for determining the nature, magnitude, and areal extent of contamination in surficial sediments and soils.
- **Determine if chemicals of potential concern other than PCBs occur at concentrations sufficient to injure natural resources in the study area:** Existing sediment and floodplain-soil data available for the study area provide a limited suite of analytes for many of the samples. This sampling program will include analysis of other contaminants (i.e., in addition to PCBs) including metals and dioxins/furans. The resultant data will be used to determine if these chemicals occur in concentrations sufficient to injure surface water, geologic, and biological resources in the study area.
- **Confirm that the data collected by the responsible party provide a reliable basis for evaluating injury to natural resources in the study area:** This sampling program will provide the data needed to confirm the reliability of data collected by S/P, thereby reducing uncertainty in the evaluation of injury to surface water, geologic, and biological resources in the study area.

- **Determine whether additional sampling is required to determine the nature and extent of contamination of sediments and/or floodplain soils in the study area:** The supplemental sediment and floodplain-soil data collected in this field sampling program are intended to address the limitations of the available data and to confirm the reliability of data collected by S/P. Analysis of the resultant data will reveal whether these objectives are satisfied by this field sampling program, or if additional sampling is required beyond the 2013 sampling effort .

3.0 Sampling Program Design

Based on the review of existing data and other information, a number of candidate sampling areas for sediment and soil were identified in the study area. Most of the selected sampling areas for sediments represent un-sampled or under-sampled areas within Choccolocco Creek and nearby contiguous or off-channel wetland habitats (i.e., Reaches CC02 to CC10), with a limited number of locations also identified within the floodplain of Lake Logan Martin (Reach CR02). For floodplain soils, most of the selected sampling areas are riparian habitats located adjacent to PCB-contaminated portions of Snow Creek and Choccolocco Creek, historic oxbows, and agricultural lands that were under-sampled during the RI.

Given the budget available for sample collection, laboratory analysis, and other costs involved in the field sampling program, the 2013 sediment and floodplain-soil sampling program will target the collection and analysis of about 100 primary samples (i.e., samples that will be analyzed as part of this program). In addition, about 70 secondary sediment and floodplain-soil sampling locations were identified throughout the sampling area; these samples may be collected and archived for future analysis. Decisions on the collection of samples at each secondary sampling location will be made in the field by the sampling team leader, based on time and budget considerations). A number of alternate sampling locations were also identified in the event that the field sampling team cannot safely or effectively obtain a sample from the

target coordinates (i.e., primary or secondary sampling locations), or if the station is deemed to be inappropriate.

Primary, secondary, and alternate sampling locations for sediment and soil are provided in Tables 1 and 2 respectively, and are illustrated in Figures 2 to 16 (sediment) and Figures 17 to 35 (soil). Up to 16 QA/QC samples will also be prepared and submitted for analysis as part of the program (i.e., about 10% of the overall sampling program analytical resources will be allotted to QA/QC). The general approach that was used to design the sampling program is described in the following sections, while the details of each sampling program element are provided in the subsequent sections of this document.

3.1 Project Tasks

Fulfilment of the objectives of the FSP will necessitate successful completion of a number of project tasks, including:

- Identifying landowners and obtaining permission to collect sediment and soil samples on private properties in the study area;
- Collection and processing of sediment and soil samples from the study area;
- Storing and transporting sediment and soil samples to the analytical laboratory;
- Implementation of laboratory analyses to determine the physical and chemical characteristics of sediment and floodplain-soil samples; and,
- Data compilation, evaluation, analysis, and reporting.

Each of these project tasks are described in this FSP and/or the QAPP (MacDonald *et al.* 2013), which comprise the key technical components of the SAP.

3.2 Identification of Landowners

Following determination of sampling locations, the landowners within the areas targeted for sample collection will be identified for Talladega and Calhoun Counties. Each of the landowners whose land may need to be accessed during the course of the study will be contacted to obtain permission to access the property. Written access agreements will be prepared to formalize the approvals.

3.3 Collection and Processing of Sediment and Soil Samples

A total of about 45 sediment samples and about 55 soil samples will be collected during the field sampling program from the identified primary and, if needed, alternate locations. In addition, 45 sediment and 24 soil locations have been identified for the collection of secondary samples, which will be archived for future analysis. The decision to collect secondary samples will be made in the field at the discretion of the field sampling team leader. The locations selected for sediment collection are described in Table 1 and plotted in Figures 2 to 16. Soil sampling locations are listed in Table 2 and shown in Figures 17 to 35. Figure 36 shows the location of two supplementary sampling stations located outside the study area.

In the field, each sampling location will be located using maps and a GPS unit. Upon arrival at each sampling station, a cursory reconnaissance will be conducted to determine if it is feasible and safe to collect a sample. If the primary or secondary sampling location is deemed unacceptable for sample collection, an alternate sampling location will be selected. If conditions are appropriate for sample collection, a total of three to five grab sediment samples or five grab soil samples will be collected and composited to support chemical characterization. These grab samples will all be collected within a 25 m radius of the designated sampling coordinates, focusing on those areas that appear to be the most depositional (for sediments; i.e., areas with the most fine-grained material) or the lowest in elevation (for soils; i.e., areas that are more likely to be inundated during flooding events).

Sediment samples will be collected at each station by deploying and retrieving a hand-held core sampler (i.e., the modified Besser sampler) or Ekman sampler, targeting the top 15 cm of surficial material. For soil, samples will be collected from the top 10 to 15 cm with a soil coring device (i.e., stainless steel bulb transplanter). In some cases, it may not be possible to penetrate the sediment/soil matrix to the desired depth (i.e., due to the presence of coarse substrate, root masses, debris, etc.). In such cases, the field sampling team may choose to accept the sample as collected (recording depth of penetration), or to sample from an alternate sampling location. All samples will be sieved in the field to a diameter of 2 mm. Material that exceeds this size will be discarded.

Samples will be collected in 1.25 gallon high-density polyethylene (HDPE) buckets. Each container will be filled with at least 2.5 liters (L) of sieved material to facilitate analysis. These samples will be carefully sealed and labeled following collection and stored in coolers on ice prior to being transported to the field laboratory. At the field laboratory, all samples will be logged, clearly labeled, packaged for transport, and stored in the temporary cold storage facility. All samples will be accompanied by a Field Chain-of-Custody (COC) Form. Detailed information on the sampling and processing methods for sediment and soil are provided in the subsequent sections of this document.

3.4 Sample Holding and Transporting

Upon conclusion of field sampling activities each day, all samples collected and processed at the field laboratory will be deposited in the cold storage facility (e.g., refrigerated trailer) and held at 4°C prior to being shipped to the analytical laboratory. At the conclusion of the sampling program, samples will be shipped to the Engineer Research Development Center (ERDC) in Vicksburg, Mississippi for chemical analysis. Samples will be packed in ice and shipped in coolers to ensure the temperature is maintained during transport. Each cooler will include a Laboratory COC Form and will be properly labeled and sealed prior to pick-up for transport.

Samples will be shipped at a time that ensures samples are delivered to the ERDC laboratory on a weekday.

3.5 Chemical Analysis of Sediment and Soil Samples

The following analyses will be conducted on samples from each primary (or alternate, if required) sampling locations:

- Total metals;
- Total organic carbon (TOC);
- Percent moisture;
- Grain size; and,
- PCBs (Aroclors).

In addition, the following analytes will be measured in a subset of the sediment and soil samples that are collected during the 2013 sampling program:

- PCBs (congeners and homologs);
- Dioxins/furans; and/or,
- Mercury.

Completion of all of these analyses will require about 2.5 L of sieved material for each sample. The methods that will be used to determine the concentration of each analyte in the sediment and soil samples are documented in the site-wide QAPP (MacDonald *et al.* 2013). In addition, the procedures that will be applied to ensure data quality are documented in the QAPP.

3.6 Data Compilation, Evaluation, Analysis, and Reporting

All of the sediment and soil chemistry data generated during this investigation will be compiled in the project database and evaluated to determine data usability. The procedures that will be used to evaluate data quality are specified in the site-wide QAPP (MacDonald *et al.* 2013). All of the field data that are collected during the investigation will be compiled in a data report for the project.

4.0 Timing of Field Activities

The sediment and soil sampling program will be conducted in the fall of 2013, with the exact timing depending on the schedule for finalizing the QAPP/FSP and the coordination of personnel schedules and logistical details. The sampling program will be conducted over a relatively short period of time, so there is no need to randomize the schedule for collecting the sediment and soil samples.

5.0 Roles and Responsibilities of the Sampling Team

Samples to support the sediment and floodplain-soil investigation will be systematically collected within the study area. The sampling team will consist of:

- Sample collection crews (each consisting of four technical staff, with one designated as a “clean hands” person); and,
- One sample preparation and shipping crew (consisting of four technical staff).

The sample collection crew will be responsible for ensuring that all necessary sampling equipment and associated supplies are loaded onto the sampling vessel each day, verifying the locations of the stations that are to be sampled (using GPS [NAD83 - UTM Zone16N]), collecting sufficient volumes of sieved sediment/soil to support chemical analyses, preparing and labeling samples for transport to the field laboratory,

and decontaminating the sampling equipment following the completion of activities at each sampling location. It is anticipated that two people will work together to deploy and retrieve the sediment/soil samplers, retrieve the sediment/soil from the sampler, and decontaminate the samplers prior to redeployment. The other two crew members will sieve the samples, take field notes, and prepare and label each sample for transport to the field laboratory. One of these crew members will be designated as the “clean-hands” person and will be responsible for filling out the Sample Data Collection Form and Field COC Forms. All members of the sample collection crew will be required to wear personal flotation devices at all times while on the water.

The sample preparation and shipping crew will consist of four people and will be stationed at the field laboratory. This crew will be responsible for receiving sediment and soil samples from the sample collection crews, ensuring that all sub-samples are appropriately labeled, cleaning the sample containers, preparing sample inventories and COC forms, preparing the samples for shipment, storing/shipping the samples, and verifying that samples have arrived at their destinations on time and in good condition. One of the crew members will be responsible for providing GIS technical assistance, downloading the GPS coordinates (NAD83 - UTM Zone 16N), verifying the station locations with each sampling crew, compiling, collating, and copying the field collection forms, and assisting with other tasks as needed. The sample preparation and shipping crew will also ensure that the sampling crews are provided with sufficient quantities of refreshments during the day. The sample preparation and shipping crew will include the Project Health and Safety Officer, who will be accessible by mobile telephone at all times.

The contact information for field crews and an example personnel schedule for the field sampling program are included in Addenda 1 and 2, respectively.

6.0 Sample Designation

A sample designation system was developed to facilitate numbering of sediment and soil samples collected during the sampling program. The sample identification numbers are comprised of pre-determined station numbers and the sample type (i.e., primary, secondary, alternate, or replicate). Using this system, sample numbers will consist of 11 or 12 characters. The first two characters will identify the media type that was sampled (i.e., SD = sediment and SL = soil). The next two numbers identify the year of sample collection (i.e., 13 = 2013). The next six characters identify the unique sample numbers, which consist of the reach (e.g., SC01, CC01, etc.) and sequential identifier (e.g., 01, 02, etc.). The last one or two characters identify whether it is a primary, secondary, or alternate sample, and if it is a replicate sample (i.e., P = primary sample, S = secondary sample, A = alternate sample, PR = primary replicate sample, SR = secondary replicate sample, and AR = alternate replicate sample). For example:

- SD-13-CC02-07-PR

This is a field replicate of sediment sample number 07 collected from a primary station in reach CC02 in 2013. The numbering system that was developed to identify sediment and soil stations and samples is presented in Tables 1 and 2, respectively.

7.0 Sampling Equipment and Methods

Equipment and materials required to support the 2013 sediment and floodplain-soil sampling program are listed in Table 3. Methods are described in the following sections of this document.

7.1 Information to be Collected at All Sampling Stations

At each sampling location, the following basic information will be collected and recorded on the Sample Data Collection Form (see Addendum 3) by the sample collection crew:

- Names of sampling personnel;
- Sampling date and time;
- Weather conditions;
- Station number and location information (i.e., county and land parcel information);
- Coordinates of the sampling location (actual; UTM Zone 16N); and,
- Hydrologic condition (i.e., wet or dry).

7.2 Sampling Procedures

Maps and a hand-held GPS unit will be used to locate the sampling locations, which will be accessed over land or by waterways in small vessels such as canoes or kayaks. For sampling locations accessed via waterways, vessels will be brought to a safe location on the shore (i.e., where the crew is able to safely exit the boat) upstream of the sampling location; the sampling crew will then exit the vessel and proceed with all sampling and equipment decontamination procedures from shore. Upon arrival at each sampling station, a cursory reconnaissance will be conducted to determine if it is feasible and safe to collect a sample. Examples of situations where the location may be deemed unsuitable for sampling include:

- The location is unsafe to access and/or collect samples; and,
- The location is inaccessible (e.g., the location is in a thickly vegetated area or, in the case of soil sampling locations, is underwater).

If necessary, the sampling location will be re-located to one of the alternate sampling locations, which will be determined by the Field Supervisor. The spatial coordinates will be taken from the centroid of each sampling location.

Two types of samples will be collected during the field sampling program, including surficial sediment samples and surficial floodplain-soil samples. Based on the sample volumes needed to support the various chemical analyses (Tables 9 and 10 of the QAPP; MacDonald *et al.* 2013), at least 2.5 L of sediment/soil will need to be collected at each station. Detailed procedures for collecting each type of sample are described in the following sections.

7.2.1 Collection of Sediment Samples

In shallow waters, sediment samples will generally be collected using a modified Besser sampler (3-inch diameter schedule 40 polyvinyl chloride [PVC] scoop). In some cases, it may be more practical to collect the sample by excavation using a stainless steel trowel, shovel, or post-hole digger to obtain a suitable sample (i.e., if the sediment is not covered by water, or in marsh areas with root wads). In deep water areas (i.e., for some sampling locations in Reach CC10 and CR02), the sample will be collected using an Ekman sampler. At each sampling station, three to five grab samples will be collected within a 25 m radius of the designated sampling coordinates. Samples will preferentially be collected from areas that appear to be the most depositional (i.e., areas with the most fine-grained sediments).

At each sampling location, new or cleaned personal equipment (e.g., gloves) will be worn and pre-cleaned sampling equipment will be rinsed with water from the site. At shallow water stations, the modified Besser sampler will be slowly pushed through the sediment to a depth of about 15 cm; the scoop will then be rotated and brought out of the sediment while attempting to minimize loss of fine-grain particles. At deep water locations, the Ekman sampler will be deployed and retrieved according to device specifications. Once the sampler has been retrieved from the water, the sample will be inspected to determine if it is acceptable for retention (see Addendum 4 for criteria for identifying acceptable and unacceptable sediment grab samples). Any sediment

samples that are not intact following retrieval (i.e., low volume, partially washed out) should be discarded in a manner that does not affect subsequent samples collected at the station. For sediment samples deemed to be acceptable, any excess water that is retained on the sediment surface (i.e., overlying water) will be siphoned off using a polyethylene siphon hose and/or hand pump.

Following visual examination of the sample and removal of overlying water, the material will be passed through a 2.00 mm sieve into a pre-cleaned 1.25 gallon pre-cleaned HDPE bucket to remove coarse material, sticks, and/or other debris. Sediments that have large amounts of materials >2 mm may first be sieved with a 5 mm sieve. The material will be gently pressed through the screen using a stainless steel spoon or gloved hands (i.e., press sieved). Material that is retained on the screen will be described and discarded. Grab samples from a minimum of three and maximum of five locations within a 25 m radius of the station will be collected and sieved until at least 2.5 L of sieved material is obtained. The sample will be labeled with the sample identification number and date on the container and lid in indelible ink prior to sediment transfer. The sample collection container will remain covered at all times, except when sediment is being added. The following information will be recorded for each sample (see Addendum 3):

- Sample identification number;
- Sample depth (sediment surface to bottom of sampler);
- Type of sampler used;
- Target and estimate of the sample volume collected;
- Number and volume of grabs collected;
- Size of mesh screen used, sieving method, and description of matter retained on the screen after sieving;
- Description of substrate (e.g., silt, sand, clay, mud, shells, detritus);

- Description of sample color (e.g., black, brown; soil samples will be described using a Munsell Soil Color Book);
- Description of sample odor, if readily apparent (i.e., sulfur, oily, sewage, none; Note: do not intentionally smell sample to evaluate odor);
- Description of surface biology (e.g., molluscs, crustacea, worms, etc.); and,
- Notes (e.g., problems encountered and unusual events during sampling).

Samples will be sealed and stored in coolers packed with ice in the sampling vessels until they are delivered to the field laboratory. Table 9 in the QAPP (MacDonald *et al.* 2013) lists the container material, container volume, preservation techniques, and holding times that will be used to handle, store, and transport the samples that have been collected.

7.2.2 Collection of Floodplain-Soil Samples

Soil samples will be generally be collected by deploying a hand-held core sampler (i.e., stainless steel bulb transplanter). At each sampling station, five grab samples will be collected within a 25 m radius of the designated sampling coordinates. Samples will preferentially be collected from low elevation areas (i.e., areas that would be more likely to be inundated during high flow events).

At each sampling location, new or cleaned personal equipment (e.g., gloves) will be worn and pre-cleaned sampling equipment will be rinsed. The area to be sampled will first be cleared of surface debris (such as twigs, rocks, and litter) that can be easily removed by hand. The pre-cleaned sampler will be pushed through the soil to a depth of about 10 to 15 cm. Following visual examination of the excavated sample, the material will be passed through a 2.00 mm sieve into a pre-cleaned 20 stainless steel bowl to remove coarse material, sticks, and/or other debris. Samples that have large amounts of materials >2.00 mm may first be sieved with a 5.00 mm sieve. If necessary, the material will be gently pressed through the screen using a stainless steel spoon or gloved hands (i.e., press sieved). Material that is retained on the screen will

be described and discarded. At each station, samples will be collected and sieved until at least 2.5 L of sieved material is obtained. The sieved material will be homogenized and then transferred into a 1.25 gallon HDPE bucket using a clean stainless steel spoon. The sample will be labeled with the sample identification number, date, and names of sampling personnel on the container and lid in indelible ink prior to sample transfer. The sample collection container will remain covered at all times, except when soil is being added. The information listed in Addendum 3 and summarized in Section 7.2.1 will be recorded for each sample. Munsell color charts will be used to describe soil color.

Samples will be sealed, placed in coolers packed with ice, and stored in the sampling vessels or vehicles until they are shipped and/or delivered to the field laboratory. Table 10 in the QAPP (MacDonald *et al.* 2013) lists the container material, container volume, preservation techniques, and holding times that will be used to handle, store, and transport the soil samples that have been collected. See Addendum 5 for additional guidance on the collection of soil samples.

7.3 Decontamination Procedures

Following the collection of each sample from a sampling location, sampling equipment will be submerged in the water at the site to release the remaining sediment from the sampler. If necessary, a stainless steel or Teflon spatula will be used to scrape the remaining sediment from the equipment. Next, the equipment will be scrubbed with a long bristle scrub brush and rinsed with site water to remove any remaining sediment. Then, the sampling equipment will be squirted with Liquinox solution, scrubbed, and rinsed. Waste water created from the Liquinox cleanse will be contained in a 20 L bucket and will be properly disposed of when convenient. Finally, the sampling equipment will be rinsed with site water to complete the decontamination process. This decontamination process will be applied to all sampling equipment including the sampling device, stainless steel or Teflon spatula, stainless steel spoon, brush, sieve, the bucket into which samples were sieved, and any other equipment that was used during sampling. All sampling equipment will then be placed in clean plastic bags to

avoid contamination between sampling locations. The waste sediment should be returned to the water after all sampling has been completed at the sampling station (i.e., the excess sediment should be returned to the water before leaving the sampling station).

Following the collection of soil samples, excess soil will be removed from the sampling equipment and the equipment will be rinsed with tap water. If necessary, the sampling equipment will be scrubbed with a scrub brush to remove any remaining soil. Then, the equipment will be squirted with Liquinox solution, scrubbed, and rinsed. Waste water created from the Liquinox cleanse will be contained in a 20 L bucket and will be properly disposed of when convenient. Finally, the sampling equipment will be rinsed with distilled water to complete the decontamination process. This decontamination process will be applied to all sampling equipment included the sampling device, sieve, the bucket into which samples were sieved, and any other equipment that was used during sampling. All sampling equipment will then be placed in clean plastic bags to avoid contamination between sampling locations. The waste soil should be returned to the site after all sampling has been completed at the sampling station (i.e., the excess soil should be returned to the site before leaving the sampling station).

7.4 Procedures for Avoiding Sample Contamination

Generation of high quality, reliable data on sediment and soil quality conditions in the study area is a primary objective of the sampling program. As such, all reasonable efforts should be made to minimize the potential for sample contamination during sample collection, handling, and processing. Some of the steps that should be taken to avoid sample contamination include:

- Approaching the sampling locations from the downwind direction (to the extent possible), and in a manner that will not disturb the samples (i.e., do not step or place equipment on the sampling location as the area is approached);

- Ensuring that sediment and soil samples do not come in contact with any item that has not undergone the approved decontamination process;
- Designating a clean-hands person to label sample containers, take notes, and seal sample containers;
- Ensuring that any utensils that are used in the sediment and soil sampling process do not come in contact with any item that has not undergone the approved decontamination process;
- Placing the sampler and other sampling equipment in clean plastic bags during transit between sampling stations;
- Covering the sample collection container after each grab sample has been taken, and covering the sieved sample container when material is not being transferred in or out of the container;
- Fully decontaminating all sampling equipment after sampling has been completed at a sampling location; and,
- Prohibiting any activity during sampling that could result in sample contamination (e.g., smoking, applying sunscreen, and consuming food or drinks during the sampling process; Note: there will be a cooler provided with food and drinks that are to be consumed at appropriate times).

7.5 Procedures for Avoiding Exposure to Contaminated Sediment and Soil

It is anticipated that contaminated sediment and soil will be routinely encountered during sampling throughout much of the study area. As such, sampling crews should take precautions to minimize exposure to potentially toxic and/or bioaccumulative substances. Some of the steps that should be taken include:

- Handling sampling equipment and sediment/soil samples carefully;
- Avoiding direct dermal contact with sediments and soils; and,

- Wearing protective equipment, such as gloves, safety glasses, long-sleeved shirts, long pants, and rubber boots/waders.

The need for respirators or Tyvek suits is not anticipated to ensure the health and safety of the sampling team members. The Project Health and Safety Officer will review the available data, examine conditions at the site, and determine if additional safety equipment is needed to protect field crews at highly contaminated locations in the study area. Nevertheless, sampling team members are required to report unsafe or hazardous conditions to the Project Health and Safety Officer immediately.

8.0 Sample Handling and Preparation

Following collection, sediment/soil samples will be transported to the field laboratory. Upon arrival at the field laboratory, samples will be logged in (Sample Inventory Form; Addendum 6) and stored in coolers on ice (i.e., to cool samples to 4°C). Samples will be delivered to the field laboratory in clearly marked sample containers (i.e., with the date and sample identification number written on the container and lid in indelible ink). Field COC forms will document transfer of the samples from the sampling crews to the sample processing crew (Addendum 7).

Following sample preparation, samples will be stored in coolers packed with ice until they are deposited in the secure, project-designated cooled storage facility (e.g., a refrigerated trailer) and held at 4°C until the end of the field program. At the conclusion of the sampling program, samples will be transported on ice to the ERDC laboratory. Samples will be accompanied by a sample manifest (e.g., copies of the Sample Inventory Forms; Addendum 6) and Laboratory COC Forms (Addendum 8).

9.0 Chemicals of Potential Concern

The results of the investigations conducted to date provide a basis for identifying the toxic and bioaccumulative COPCs at the site (see the site-wide QAPP; MacDonald *et al.* 2013). This list of COPCs has been used, in conjunction with integrating the priorities and budget considerations of the project, to develop the list of analytes that will be quantified in sediments and soils. The priority analytes include total metals, methylmercury, PCBs (Aroclors, congeners, and homologs), PCDDs/PCDFs, TOC, percent moisture, and grain size. The data quality objectives (DQOs) for the project and the performance criteria for measurement data are specified in the project QAPP (MacDonald *et al.* 2013).

10.0 References Cited

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Tables

Table 1. List of the proposed sediment sampling locations for the 2013 sampling program for the Anniston PCB Site.

Reach / Station ID	Station Type	UTM Coordinates (Zone 16N)		Geographic Coordinates		Figure No.
		Northing	Easting	Latitude	Longitude	
CC02						
UF/EE24/23	Ugeqpf ct{	593: : 5; 089:	82; 697037	5508258: ; 3	/: 70 4223; 6	4
UF/EE24/24	Ugeqpf ct{	593: : 70 99	82; 97: 0643	5508262: 33	/: 70 38; 827	4
UF/EE24/25	Rtko ct{	593: 328022:	82; 8740792	5507; 927; 6	/: 70 3: 3; 94	4
UF/EE24/26	Ugeqpf ct{	593: 45205:	82; 667075;	5507; : 4259	/: 70 42634:	4
UF/EE24/27	Rtko ct{	593: 4550873	82; 56; 024:	5507; : 4639	/: 70 436746	4
UF/EE24/28	Rtko ct{	593: 93502; 3	82; 2; 40: 7	5508247; 36	/: 70 463836	4
UF/EE24/29	Rtko ct{	593: 9970578	82; 594059:	5508253464	/: 70 433565	4
UF/EE24/2:	Rtko ct{	593: 922085:	82; 7930848	55082464; ;	/: 70 3: ; ; 85	4
UF/EE24/2:	Ugeqpf ct{	593: : ; 0 : 8	82; 83; 0 : 3	5508264439	/: 70 3: 673:	4
UF/EE24/32	Ugeqpf ct{	593: 9860682	82; 83: 0546	5508252228	/: 70 3: 6: 74	4
UF/EE24/33	Ugeqpf ct{	593: : 590843	82; : 470578	55082585; 2	/: 70 384674	4
UF/EE24/34	Ugeqpf ct{	593: 6; ; 0 56	82; : 480 34	5508227; 4:	/: 70 384922	4
UF/EE24/35	Ugeqpf ct{	593: 58304; 6	82; 9970766	5507; ; 56: :	/: 70 38: 628	4
UF/EE24/36	Cngtpcvg	593: : 7708; :	82; 823073	550825: 427	/: 70 3: 8749	4
UF/EE24/37	Cngtpcvg	593: 7; 30 44	83226206: 7	5508236234	/: 70 35; 794	4
UF/EE24/38	Cngtpcvg	593: 9570 63	82; 4470 88	5508249: 4;	/: 70 449392	4
UF/EE24/39	Cngtpcvg	593: 4: 3083;	82; 5420 ; 3	5507; : 8948	/: 70 4396; :	4
UF/EE24/3:	Uwr r ngo gpcn	593: 7: ; 0124	82; 2580427	5508236: 22	/: 70 469: 22	4
CC04						
UF/EE26/23	Rtko ct{	5938267088;	82778; 0454	55079: : ; 46	/: 70 846646	5
UF/EE26/24	Ugeqpf ct{	59383690752	82779; 0858	55079: ; 2; ;	/: 70 8453: 4	5
UF/EE26/25	Rtko ct{	59384570578	8277: 80758	5507: 28234	/: 70 844557	5
UF/EE26/26	Ugeqpf ct{	5937; 58053:	8278470 6:	550799; 228	/: 70 83: 665	5
UF/EE26/27	Rtko ct{	5937; 750788	82796: 0547	55079: 2662	/: 70 82745:	5
UF/EE26/28	Ugeqpf ct{	5937; 630468	827: : 706: 7	550799; 3; 5	/: 70 7; 2698	5
UF/EE26/29	Rtko ct{	5937: : 30285	8276470432	5507996444	/: 70 862357	5
UF/EE26/2:	Rtko ct{	5937: 640499	8275: ; 0 4;	5507992982	/: 70 865; : 3	5
UF/EE26/2:	Rtko ct{	5937; 7; 0874	8275790536	55079: 3598	/: 70 869579	5
UF/EE26/32	Rtko ct{	5937: : 80 2;	8274670 66	5507996; 3:	/: 70 87; 663	5
UF/EE26/33	Rtko ct{	593796208: 7	8243960465	55079868: 6	/: 70 ; ; 2762	6
UF/EE26/34	Ugeqpf ct{	5937866084;	8243480672	5507978289	/: 70 ; ; 79; :	6
UF/EE26/35	Rtko ct{	5937897084	8243750473	550797: : 62	/: 70 ; ; 4: 97	6
UF/EE26/36	Rtko ct{	593822: 0287	8244780 53	55079: : 983	/: 70 ; ; 3546	6
UF/EE26/37	Rtko ct{	5937; 5; 0873	8244: 90 86	55079: 4784	/: 70 ; 9: 27;	6
UF/EE26/38	Ugeqpf ct{	5937: 740 ; :	8245340872	5507996937	/: 70 ; 976; ;	6
UF/EE26/39	Ugeqpf ct{	5937; 8305:	82747: 0 ; 5	55079: 3848	/: 70 879; 69	5
UF/EE26/3:	Ugeqpf ct{	5937: 78028	82757307; ;	55079942; 2	/: 70 86: 2; 6	5
UF/EE26/3:	Ugeqpf ct{	59382730; 6	8244760 37	55079: 4929	/: 70 ; ; 3724	6
UF/EE26/42	Ugeqpf ct{	5937; 5: 0462	8245690 37	55079: 4599	/: 70 ; 93824	6
UF/EE26/43	Ugeqpf ct{	5937: 89027	8245680726	5507997; 77	/: 70 ; 93: 58	6
UF/EE26/44	Cngtpcvg	5937; 22028	8276680 32	5507997; 85	/: 70 859996	5
UF/EE26/45	Cngtpcvg	5937: ; 30 6:	8274; 50663	5507997547	/: 70 87653:	5
UF/EE26/46	Cngtpcvg	5938245076:	8244: ; 0864	55079; 2348	/: 70 ; 999: 4	6
UF/EE26/47	Cngtpcvg	5937; 350894	8244: ; 0 3;	55079: 2385	/: 70 ; 98: 34	6
UF/EE26/48	Cngtpcvg	5937: ; 209;	82455; 073	550799: 265	/: 70 ; 947: 2	6

Table 1. List of the proposed sediment sampling locations for the 2013 sampling program for the Anniston PCB Site.

Reach / Station ID	Station Type	UTM Coordinates (Zone 16N)		Geographic Coordinates		Figure No.
		Northing	Easting	Latitude	Longitude	
CC05						
UF/EE27/23	Rtko ct{	593728; 0525	7; ; 2; ; 08; 9	55079292; 8	/: 70 544743	7
UF/EE27/24	Rtko ct{	5936: ; 80457	7; : : ; 60892	55078; 388;	/: 70 566: 22	7
UF/EE27/25	Ugeqpf ct{	59362390892	7; : : 5; (86;	55078346; 3	/: 70 573922	8
UF/EE27/26	Rtko ct{	593629: 0238	7; : 9580928	550783: 24;	/: 70 584944	8
UF/EE27/27	Rtko ct{	593624609: 9	7; : 7; 50 79	5507835592	/: 70 59; 468	8
UF/EE27/28	Ugeqpf ct{	59372440474	7; : : 980 43	5507925272	/: 70 568795	7
UF/EE27/29	Ugeqpf ct{	59369840522	7; : : ; 50756	550789; 7; 4	/: 70 567293	7
UF/EE27/2:	Ugeqpf ct{	59362: 50 : 8	7; : 9220842	550783: 823	/: 70 588879	8
UF/EE27/2:	Cngtpcvg	59372930 98	7; : ; 7; 0745	5507929679	/: 70 55983;	7
UF/EE27/32	Cngtpcvg	59362720873	7; : 99204: 6	55078376: 7	/: 70 57; 358	8
UF/EE27/33	Cngtpcvg	593628: 0 : 3	7; : 8720987	55078394: 6	/: 70 593; ; 2	8
CC06						
UF/EE28/23	Rtko ct{	59356450857	7; 72; 60425	55077844: ;	/: 70 978: 84	9
UF/EE28/24	Rtko ct{	59353540953	7; 653; 027:	550775899;	/: 70 : 5; 7: 6	9
UF/EE28/25	Ugeqpf ct{	5934; 23072;	7; 59530599	5507738667	/: 70 ; 25347	:
UF/EE28/26	Rtko ct{	5933: 3: 0 28	7; 58590685	550763: : ; 9	/: 70 ; 36596	:
UF/EE28/27	Ugeqpf ct{	5934: ; 40437	7; 6; 770 92	5507744952	/: 70 9: 3; 35	9
UF/EE28/28	Ugeqpf ct{	5935239029:	7; 5: 6: 079;	5507748987	/: 70 : ; 25: 2	:
UF/EE28/29	Cngtpcvg	5934; 75055:	7; 6; 9: 029	5507742239	/: 70 98: 989	9
UF/EE28/2:	Cngtpcvg	59352770469	7; 689: 06; 7	550774; 696	/: 70 : 22; 73	9
UF/EE28/2:	Cngtpcvg	593533508; 9	7; 64560 89	5507757359	/: 70 : 6: 883	9
CC07						
UF/EE29/23	Rtko ct{	5935: ; 3087	7; 376; 05: 4	5507829829	/: 802359349	:
UF/EE29/24	Rtko ct{	5935: 2307; 4	7; 3636045	55077; ; 849	/: 802373946	:
UF/EE29/25	Ugeqpf ct{	5935: 790 9:	7; 37530792	5507826825	/: 80235; 2: 2	:
UF/EE29/26	Ugeqpf ct{	59359: ; 0 33	7; 368; 0 62	55077; ; 63;	/: 80236799:	:
UF/EE29/27	Ugeqpf ct{	59359; 30 95	7; 3596033	55077; ; 9; 6	/: 80237832;	:
CC09						
UF/EE2; /23	Ugeqpf ct{	5933; 350; 9	7: 6; 2406: :	5507656928	/: 802: 77235	32
UF/EE2; /24	Rtko ct{	59342440545	7: 69520899	55076668: 6	/: 802: 95689	32
UF/EE2; /25	Ugeqpf ct{	59342; 30254	7: 687506; 8	5507672; 63	/: 802: : 3883	32
UF/EE2; /26	Rtko ct{	59345: 7089	7: 62: ; 0677	5507699; 8:	/: 802; 64354	32
UF/EE2; /27	Rtko ct{	59352340756	7: 52350 57	5507757557	/: 802795: ;	33
UF/EE2; /28	Rtko ct{	593482806: 6	7: 49640737	55076: ; : 48	/: 802: 9222	33
UF/EE2; /29	Rtko ct{	5934; 75032	7: 45: 3027	5507752733	/: 8034783:	33
UF/EE2; /2:	Ugeqpf ct{	59357; 4043	7: 3: ; 5076:	55077: : 693	/: 8039975:	33
UF/EE2; /2:	Rtko ct{	59358780774	7: 39630 55	55077; 65; :	/: 803; 5: 33	33
UF/EE2; /32	Ugeqpf ct{	59352; 408: 3	7: 44360 23	55077653: 3	/: 803655: 3	33
UF/EE2; /33	Ugeqpf ct{	59357690476	7: 3: 5708; 3	55077: 668;	/: 803; 5: 33	33
UF/EE2; /34	Cngtpcvg	59353770452	7: 43: 40792	550776: : 69	/: 80368: 28	33
UF/EE2; /35	Cngtpcvg	593585; 0792	7: 3: 45068:	55077; 4: 26	/: 803: 7265	33

Table 1. List of the proposed sediment sampling locations for the 2013 sampling program for the Anniston PCB Site.

Reach / Station ID	Station Type	UTM Coordinates (Zone 16N)		Geographic Coordinates		Figure No.
		Northing	Easting	Latitude	Longitude	
CC10						
UF/EE32/23	Rtko ct{	59367650233	7: 27550 94	5507897484	/: 808545356	34
UF/EE32/24	Rtko ct{	59372390 78	7: 24370; :	550793: 557	/: 80857926;	34
UF/EE32/25	Ugeqpf ct{	5936: 4: 0445	7: 2869086	5507922: ; :	/: 8085328: 4	34
UF/EE32/26	Ugeqpf ct{	59373430834	7: 25; 60 22	5507949725	/: 808559828	34
UF/EE32/27	Rtko ct{	5936759084;	79; 4460 88	5507897979	/: 808686392	35
UF/EE32/28	Rtko ct{	5935; 47056	79; 6320838	550784262:	/: 808666979	35
UF/EE32/29	Ugeqpf ct{	59357570 : 8	79; 57402; 3	55077: 7542	/: 808673576	35
UF/EE32/2:	Rtko ct{	59352; 50299	79; 4: 2093	550776765:	/: 80867; 662	35
UF/EE32/2:	Rtko ct{	593576: 086;	79; 2280; 6	55077: 8949	/: 8086: : 75:	35
UF/EE32/32	Ugeqpf ct{	5935: 2: 0643	79; 27: 02; :	5507832339	/: 8086: 49: 4	35
UF/EE32/33	Rtko ct{	5936934055;	79: 7; 702;	55078; 3; 9;	/: 808753: 83	35
UF/EE32/34	Ugeqpf ct{	593685908: 8	798: 58025	55078: 86: 5	/: 808943587	36
UF/EE32/35	Rtko ct{	59367620736	798848062	5507899; 38	/: 808966366	36
UF/EE32/36	Rtko ct{	59364: 208;	798; 4: 0 47	5507876432	/: 80893395:	36
UF/EE32/37	Ugeqpf ct{	59372; ; 0 4:	7: 28580834	5507947632	/: 808533849	34
UF/EE32/38	Cngtpcvg	5936: ; ; 0 : 9	7: 29940: 9	5507929497	/: 8084; 9283	34
UF/EE32/39	Cngtpcvg	593643807; ;	79; 64; 0893	5507868875	/: 8086645; 4	35
UF/EE32/3:	Cngtpcvg	593548: 02; 5	79; 675075	55077832; 5	/: 808662928	35
CR02						
UF/ET24/23	Rtko ct{	59357470898	7979270437	55077: 9226	/: 808: 6642:	37
UF/ET24/24	Rtko ct{	59353950 89	798569086:	5507776: 94	/: 808997526	37
UF/ET24/25	Ugeqpf ct{	59352430544	7975380 4;	5507763: 5:	/: 808: : 8692	37
UF/ET24/26	Rtko ct{	593487208: 8	7985390786	5507729923	/: 80899: ; ; 4	37
UF/ET24/27	Rtko ct{	59339490 32	7977: 30 ; 5	5507646; ; 5	/: 808: 7; 224	38
UF/ET24/28	Ugeqpf ct{	5932; 87095	797725024	5507578546	/: 808: 8: 364	38
UF/ET24/29	Rtko ct{	5932: 6: 02:	796; 4604: 5	5507568369	/: 808; 52797	38
UF/ET24/2:	Ugeqpf ct{	593546; 06:	79777902: 8	55077843; 4	/: 808: 7: 465	37
UF/ET24/2:	Ugeqpf ct{	593438: 065	79833; 032	550768659:	/: 808: 22978	37
UF/ET24/32	Ugeqpf ct{	59335: 2056	7978490574	55075; 5855	/: 808: 76632	38
UF/ET24/33	Cngtpcvg	5934: 24022	798439052:	55077436: 4	/: 8089: ; 883	37
UF/ET24/34	Cngtpcvg	593476: 067	798: 930 2:	55076: ; 32;	/: 80893; 5: 3	37
UF/ET24/35	Cngtpcvg	59332260788	797454083;	5507582235	/: 808: ; 94; 4	38
Outside the study area						
UF/TGH/23	Uwr rgo gpvn	593: 4670893	83549404;	5507; 9; 622	/: 7099; 3922	58

Table 2. List of the proposed soil sampling locations for the 2013 sampling program for the Anniston PCB Site.

Reach / Station ID	Station Type	UTM Coordinates (Zone 16N)		Geographic Coordinates		Figure No.
		Northing	Easting	Latitude	Longitude	
SC01						
UN/UE23/23	Rtko ct{	594554; 025	8286850852	55086669; 6	/: 70 73; 583	39
UN/UE23/24	Rtko ct{	59456; 3063	8288990266	550867; 468	/: 70 6; 8378	39
UN/UE23/25	Rtko ct{	594596; 056	828; ; 30 9:	55086; 4544	/: 70 694893	39
UN/UE23/26	Rtko ct{	59462450 87	828; 930782	5508729294	/: 70 696765	39
UN/UE23/27	Ugeqpf ct{	594559608; 2	8288340 64	550866; 9; 4	/: 70 72543:	39
UN/UE23/28	Ugeqpf ct{	594622; 068	8292720744	550872774;	/: 70 677487	39
UN/UE23/29	Cngtpcvg	59456230 88	828; 890 38	5508672; : 8	/: 70 6978; 6	39
UN/UE23/2:	Cngtpcvg	59458940 54	82934705; 8	5508697393	/: 70 6697; 8	39
CC01						
UN/EE23/23	Rtko ct{	593; 66906; 3	82; 2330 98	55082; 4444	/: 70 46; 582	3:
UN/EE23/24	Rtko ct{	593; 6290277	82; 2820722	55082; : 747	/: 70 4663; 2	3:
UN/EE23/25	Rtko ct{	593; 5840528	82; 27; 065	55082; 66; 4	/: 70 466689	3:
UN/EE23/26	Rtko ct{	593; 5930693	82; 2460 38	55082; 7575	/: 70 46; 27:	3:
UN/EE23/27	Rtko ct{	593; 5240332	82; ; ; 50 8;	550829; 352	/: 70 4736; :	3:
UN/EE23/28	Rtko ct{	593; 4330 : 5	82; 2580999	5508292; 72	/: 70 468; 97	3:
UN/EE23/29	Ugeqpf ct{	593; 67407; 6	82; 297083;	55082; 4839	/: 70 4646; 6	3:
UN/EE23/2:	Ugeqpf ct{	593; 63204; 2	82; 33; 04:	55082; : 97:	/: 70 459; 22	3:
UN/EE23/2;	Ugeqpf ct{	593; 6220522	82; ; ; 70 84	55082; 9; : 5	/: 70 473376	3:
UN/EE23/32	Ugeqpf ct{	593; 48; 0 69	82; ; 850; 8	5508298483	/: 70 4769; 2	3:
UN/EE23/33	Ugeqpf ct{	593; 48; 06; 6	82; 2870 ; 3	5508298247	/: 70 46598:	3:
UN/EE23/34	Ugeqpf ct{	593; 4330566	82; 2; 508; 5	5508292; 75	/: 70 463; 43	3:
UN/EE23/35	Cngtpcvg	593; 5750 6;	82; 3520254	55082; 5887	/: 70 458973	3:
CC02						
UN/EE24/23	Rtko ct{	593; 8; 208; 5	82; 34508; 4	5508245; 38	/: 70 45; 469	3;
UN/EE24/24	Rtko ct{	593; 9250436	82; 2730894	5508247288	/: 70 468268	3;
UN/EE24/25	Rtko ct{	593; 8890 59	82; ; 93069	5508243; 79	/: 70 4768; 4	3;
UN/EE24/26	Rtko ct{	593; 7; 40 ; ;	82; : 96028	5508237529	/: 70 4873; :	3;
UN/EE24/27	Rtko ct{	593; 82; 086	82; 2450246	5508238835	/: 70 46; 3; 7	3;
UN/EE24/28	Rtko ct{	593; 7; 30254	82; 3770; 6	5508235; 62	/: 70 456; 45	3;
UN/EE24/29	Rtko ct{	593; 6; 709;	82; 35; 065	5508227526	/: 70 45898;	3;
UN/EE24/2:	Ugeqpf ct{	593; 94; 042:	82; 4270 ; 6	5508249473	/: 70 44; 552	3;
UN/EE24/2;	Cngtpcvg	593; 8260273	82; 2830993	5508238335	/: 70 467247	3;
UN/EE24/32	Cngtpcvg	593; 6830844	82; 28; 0858	5508225484	/: 70 466682	3;
CC03						
UN/EE25/23	Ugeqpf ct{	59386; 70458	8295380 33	5507; 48; 39	/: 70 65782:	42
UN/EE25/24	Ugeqpf ct{	593875905:	8292; ; 0; 3	5507; 53966	/: 70 67; 267	42
UN/EE25/25	Ugeqpf ct{	593864; 0 ; 2	82928; 06; 8	5507; 442; 6	/: 70 684553	42
UN/EE25/26	Ugeqpf ct{	59384980 39	8293520 85	5507; 2; 43;	/: 70 677; 25	42
UN/EE25/27	Ugeqpf ct{	59387450894	827; ; ; 034	5507; 53824	/: 70 799764	43
UN/EE25/28	Ugeqpf ct{	59388570638	827; 470 6:	5507; 63974	/: 70 7; 7525	43
UN/EE25/29	Ugeqpf ct{	593875907; 7	8292680 68	5507; 53; 2;	/: 70 686864	42
UN/EE25/2:	Ugeqpf ct{	59386430473	828; ; ; 068	5507; 43588	/: 70 68; :::	42

Table 2. List of the proposed soil sampling locations for the 2013 sampling program for the Anniston PCB Site.

Reach / Station ID	Station Type	UTM Coordinates (Zone 16N)		Geographic Coordinates		Figure No.
		Northing	Easting	Latitude	Longitude	
CC03 (cont.)						
UN/EE25/2;	Ugeqpf ct {	5938923044	8283350493	55° 696: 3	/: 70 787254	43
UN/EE25/32	Ugeqpf ct {	5938946079	827: 6; 0748	55° 6; : 6:	/: 70 7; 5643	43
UN/EE25/33	Cngtpcvg	593874308; 2	82938: 0 ; ;	55° 5242:	/: 70 673733	42
CC04						
UN/EE26/23	Ugeqpf ct {	59386520474	827: 390555	55° 4557:	/: 70 7; 945:	43
UN/EE26/24	Ugeqpf ct {	59387: 30 : 8	8278; 508: 4	55° 59377	/: 70 8325: 3	43
UN/EE26/25	Rtko ct {	59377: 90 6:	8274440688	55° 969; : 3	/: 70 884545	44
UN/EE26/26	Rtko ct {	59379880 6;	8273470574	55° 98644:	/: 70 894796	44
UN/EE26/27	Rtko ct {	593799; 073	82727506: 6	55° 987667	/: 70 8: 2567	44
UN/EE26/28	Rtko ct {	59379290 38	826; 7; 057	55° 97; 28:	/: 70 8; 2773	44
UN/EE26/29	Rtko ct {	59377: 3034	825833072;	55° 96; 228	/: 70 : 57: : 3	45
UN/EE26/2:	Rtko ct {	59377760623	8256; : 073	55° 968875	/: 70 : 6: 2; 5	45
UN/EE26/2:	Rtko ct {	59387920829	8243860 59	55° 5; 756	/: 70 ; ; 2825	46
UN/EE26/32	Rtko ct {	59387580829	82447205; 7	55° 58653	/: 70 ; ; 3645	46
UN/EE26/33	Ugeqpf ct {	5937885068:	8272930668	55° 976; 6;	/: 70 89: 725	44
UN/EE26/34	Ugeqpf ct {	593783; 045	826: 6; 076;	55° 9733: 9	/: 70 924683	44
UN/EE26/35	Ugeqpf ct {	5937; 45045	8259; 505;	55° 99; 893	/: 70 : 37; 38	45
UN/EE26/36	Ugeqpf ct {	593828; 087	825962086	55° 9; 4: 78	/: 70 : 435: ;	45
UN/EE26/37	Ugeqpf ct {	59377; : 066	8258580692	55° 972739	/: 70 : 55394	45
UN/EE26/38	Ugeqpf ct {	5937833093	825673094	55° 973: 58	/: 70 : 75342	45
UN/EE26/39	Ugeqpf ct {	5938837026;	8243650 7;	55° 65829	/: 70 ; ; 4: 23	46
UN/EE26/3:	Ugeqpf ct {	59388340 6;	82463: 08: 7	55° 65376	/: 70 ; 85479	46
UN/EE26/3:	Ugeqpf ct {	593866: 0 56	824779068	55° 4: 452	/: 70 ; 6: 673	46
UN/EE26/42	Cngtpcvg	593778; 0 3	825768086	55° 969; ; 6	/: 70 : 64; 57	45
UN/EE26/43	Cngtpcvg	593783: 0 22	8257580 42	55° 974654	/: 70 : 65: 95	45
CC07						
UN/EE29/23	Rtko ct {	5935; 520 ; 4	7; 37990 77	55° 833379	/: 80235623;	47
UN/EE29/24	Rtko ct {	5935: 330 ; 7	7; 359904: 4	55° 8227: :	/: 802377969	47
UN/EE29/25	Rtko ct {	5935: 620: 6	7; 34950; 2	55° 8254: 4	/: 802388: 87	47
UN/EE29/26	Rtko ct {	59363780695	7; 35; 90 74	55° 853867	/: 80237538:	47
UN/EE29/27	Rtko ct {	5935: 450 37	7; 33280 : 4	55° 823; 25	/: 8023: 6: 73	47
UN/EE29/28	Rtko ct {	5935: ; 70 ; 8	7; 2; 95022	55° 838932	/: 80242; : 69	47
UN/EE29/29	Rtko ct {	59366720 78	7; 2; ; ; 0234	55° 87: 757	/: 8023; 7: 64	47
UN/EE29/2:	Rtko ct {	5936: 650 89	7; 2; 840: 2	55° 8; 6239	/: 8023; ; 567	47
UN/EE29/2:	Rtko ct {	59338980658	7: : 736082	55° 63263;	/: 802688432	48
UN/EE29/32	Ugeqpf ct {	5935: 7: 0235	7; 36720 95	55° 8268: 6	/: 802369984	47
UN/EE29/33	Ugeqpf ct {	5935: 730 63	7; 32760844	55° 826697	/: 8023; 2684	47
UN/EE29/34	Ugeqpf ct {	59367; 408; 4	7; 334506: :	55° 893396	/: 8023: 44: ;	47
UN/EE29/35	Ugeqpf ct {	5936; 68025:	7; 344206: :	55° 925224	/: 802393698	47
UN/EE29/36	Ugeqpf ct {	59336; 70 ; :	7: : 832087	55° 75; 6289	/: 8026782; 4	48
UN/EE29/37	Cngtpcvg	5935; 330695	7; 37670643	55° 78; 646	/: 802359755	47
UN/EE29/38	Cngtpcvg	5935: 860853	7; 32; 907: 2	55° 8277: 5	/: 8023: 7: 44	47
UN/EE29/39	Cngtpcvg	593377: 0 5:	7: : 63; 084:	55° 75; ; ; 5	/: 802698847	48

Table 2. List of the proposed soil sampling locations for the 2013 sampling program for the Anniston PCB Site.

Reach / Station ID	Station Type	UTM Coordinates (Zone 16N)		Geographic Coordinates		Figure No.
		Northing	Easting	Latitude	Longitude	
CC08						
UN/EE2:/23	Rtko ct{	5933655074	7: : 34: 0428	55075: :: 57	/: 80272: 29;	48
UN/EE2:/24	Rtko ct{	59335: 30593	7: 9: : 60 : 6	55075: 6469	/: 802745787	48
UN/EE2:/25	Rtko ct{	59344650252	7: 8: 57077:	5507684: ; 8	/: 802868724	49
UN/EE2:/26	Rtko ct{	593436902; 2	7: 8: 68078	5507676376	/: 8028568: 6	49
UN/EE2:/27	Rtko ct{	5933; 7508; 5	7: 8: 730476	5507658967	/: 8028672; 5	49
UN/EE2:/28	Ugeqpf ct{	59336950743	7: : 544084;	55075: 449;	/: 8026: 9378	48
UN/EE2:/29	Ugeqpf ct{	5933; 5; 0; 4	7: 8: 8902: 6	5507657664	/: 802854854	49
UN/EE2:/2:	Ugeqpf ct{	5934; 9: 027	7: 7: 340556	5507752229	/: 802978222	4:
UN/EE2:/2:	Ugeqpf ct{	593527708; :	7: 7: 680534	5507758: 82	/: 8029636; 7	4:
UN/EE2:/32	Ugeqpf ct{	593526408: 8	7: 7: 2: 0 36	5507757: 64	/: 802978432	4:
UN/EE2:/33	Ugeqpf ct{	59352: 506: ;	7: 7: ; 506; 3	550775; 676	/: 802969379	4:
UN/EE2:/34	Cngtpcvg	59352; : 084:	7: 8277084;	5507762866	/: 80294; 955	4:
UN/EE2:/35	Cngtpcvg	59357; ; 05: :	7: 96720492	55077: 6937	/: 80279: ; 8;	4:
UN/EE2:/36	Cngtpcvg	59359: 70 3;	7: 96: ; 0 48	5507823727	/: 802796736	4:
UN/EE2:/37	Cngtpcvg	59362230469	7: 9993027;	55078428; 5	/: 80276623;	4;
UN/EE2:/38	Cngtpcvg	593622; 082	7: 9; 880297	5507843486	/: 802745226	4;
CC09						
UN/EE2;/23	Rtko ct{	593462304: 8	7: 63: 30483	550769; 4; 7	/: 802; 5444;	52
UN/EE2;/24	Rtko ct{	5934697037	7: 62: : 05; 4	55076: 82: 2	/: 802; 64384	52
UN/EE2;/25	Rtko ct{	593466: 024:	7: 5; 780 9:	55076: 58: 9	/: 802; 78564	52
UN/EE2;/26	Ugeqpf ct{	593477407; 5	7: 634808: 5	55076: 4; : 6	/: 802; 59; 87	52
UN/EE2;/27	Ugeqpf ct{	59348; ; 0472	7: 4; : 2049	5507729329	/: 802834: ;	52
UN/EE2;/28	Cngtpcvg	59349760 7;	7: 52330439	55077342: :	/: 80279; 43	52
UN/EE2;/29	Cngtpcvg	59348640469	7: 4; 660597	5507723; ; 6	/: 80287447	52
CC10						
UN/EE32/23	Rtko ct{	5936; 24025;	7: 268808; 3	55079298; 4	/: 80255227:	53
UN/EE32/24	Rtko ct{	59368830 56	7: 25840245	55078: 8339	/: 802563773	53
UN/EE32/25	Rtko ct{	59366: 20736	7: 24470794	550788; : 7:	/: 802578636	53
UN/EE32/26	Rtko ct{	5936; 4; 0862	79; : 8: 044;	5507932853	/: 8025; 672;	53
UN/EE32/27	Rtko ct{	593689205;	7: 22780 28	55078: 92: 8	/: 802596636	53
UN/EE32/28	Rtko ct{	593655; 0245	79; 8840 46	5507879742	/: 802639387	53
UN/EE32/29	Rtko ct{	59352430 32	79; 5320794	550775: ; ::	/: 8026784: 5	54
UN/EE32/2:	Rtko ct{	5934; 740443	79; 4520 86	5507745975	/: 80268723;	54
UN/EE32/2:	Rtko ct{	5935422023	798756073	550777936;	/: 802977349	56
UN/EE32/32	Ugeqpf ct{	593494805: ;	79; 5230755	5507734574	/: 80267973;	54
UN/EE32/33	Ugeqpf ct{	593689; 023	79884; 046;	55078; 2635	/: 80296589;	55
UN/EE32/34	Cngtpcvg	5936662079	7: 265; 059	5507888279	/: 8025555: ;	53
UN/EE32/35	Cngtpcvg	59368960 84	7: 276; 069	55078: 9372	/: 8025435: 2	53
UN/EE32/36	Cngtpcvg	593594708: :	79; 68: 034	5507824528	/: 80265: 8; ;	54
UN/EE32/37	Cngtpcvg	5936944054	7985: 902: 5	55078; 66: 7	/: 80298; 954	55
UN/EE32/38	Cngtpcvg	59368890 89	7983: 902: 7	55078: ; 947	/: 8029; 3547	55
UN/EE32/39	Cngtpcvg	5936999056	7983690 28	55078: ; 848	/: 8029; 7685	55
UN/EE32/3:	Cngtpcvg	5935843036	79852; 0 29	55077; 7493	/: 80299: ; ;:	56

Table 2. List of the proposed soil sampling locations for the 2013 sampling program for the Anniston PCB Site.

Reach / Station ID	Station Type	UTM Coordinates (Zone 16N)		Geographic Coordinates		Figure No.
		Northing	Easting	Latitude	Longitude	
CR02						
UN/ET24/23	Rtko ct{	5935577089;	797924073	5507793897	/: 808: 66839	56
UN/ET24/24	Rtko ct{	59326220645	7986660645	5507526889	/: 808989474	57
UN/ET24/25	Rtko ct{	593269: 064:	7986820 36	55075338; 2	/: 80898762;	57
UN/ET24/26	Ugeqpf ct{	59349420; 6	7985270 25	5507736254	/: 8089: 23: :	56
UN/ET24/27	Ugeqpf ct{	5934978099	7988560535	5507739264	/: 8089669: 5	56
UN/ET24/28	Ugeqpf ct{	59324; 3057	79874902: 3	55074; 69: 9	/: 80897: 666	57
UN/ET24/29	Cngtpcvg	593577504; 8	7979540252	55077: ; 743	/: 808: 634; 7	56
UN/ET24/2:	Cngtpcvg	59352340827	79746402; 2	5507763327	/: 808: ; 674:	56
UN/ET24/2:	Cngtpcvg	59336820873	7982290424	5507622774	/: 808: 35656	57
UN/ET24/32	Cngtpcvg	59334: 40 ; 7	7979; 60 82	55075: 6939	/: 808: 58664	57
Outside the study area						
UN/TGH/23	Uwr rgo gpvn	593: 5; 70 44	8353: 40845	5507; ; 5259	/: 70: 23495	58

Table 3. Sampling equipment required to support the 2013 sampling program for the Anniston PCB Site.

Item	Volume / Number	Responsibility for Acquiring and Bringing to the Field
Cleaning Supplies		
É Cegwqpg	6'i cmqpu	GTF E
É Cnepqz "engcpgt" "Nks wkpqz +	4'i cmqpu	GTF E IO GUN
É Ectdq { 'hwppgn	4	GTF E
É F kur qucdng "tci u	4'dqzgu	GTF E IO GUN
É F kunknfg "y cvgt." ectdq {	5"z "7'i cmqp	GTF E
É Nqpi "dtlkvng "uetwd "dt wuj "	8	GTF E
É Nqpi "dtlkvng "uetwd "dt wuj . "e { nlpf tlecn/uj cr gf "	4	O GUN
É Rqn{ gj { npgp "y cuj "dqwrgu "hqt "cegqpg	4"z "3'N	GTF E
É Rqn{ gj { npgp "y cuj "dqwrgu "hqt "f kunknfg "y cvgt	4"z "3'N	GTF E
É Rqn{ gj { npgp "y cuj "dqwrgu "hqt "Nks wkpqz	4"z "3'N	GTF E
É Vtcuj "dci u	3'dqz	GTF E
É Y cuug "ectdq {	5"z "7'i cmqp	GTF E
General Sampling Supplies		
É Dcem cem "hqt "ectt { lpi "uco r nki "uwr r nkgu +	4	GTF E IO GUN
É Dcwgtkgu "ur ctg "hqt "I RU "cmgu "y q "CCu +	:	GTF E
É Dguugt "uco r ngt "o qf lkqf +	3	O GUN
É Dwpi gg "Eqtf u	ugxgtn	GTF E
É Ecpqgu "qt "Mc { cmu "y kj "r cf f ngulqctu	4	O GUN
É Ej guv "y cf gtu	ugxgtn	O GUNletgy
É Erkr dqctf u "hqt "hknkpi "qwwhkqf "hqt o u	6	GTF E IO GUN
É F ki kcn "eco gtc "y kj "ceeguuqtkgu	4	GTF E IO GUN
É Gno cp IRqpc "uco r ngt . "8"z "8"z "8\$ "y kj "j cpf ng "cpf "	4	
o guugpi gt		
É Hkqf "F cv "Eqmge vqp "Hqto u "qp "y cvgr tqqh "r cr gt +	o cp {	O GUN
É Hrci i lpi "vcr g ."xctlkqwu "eqmqtu	42'tqmu	GTF E
É I npxgu "nqpi "urggxf . "j gcx { "f wqf . "xctlkqwu "f r gu +	5	GTF E
É J kr "y cf gtu	ugxgtn	O GUNletgy
É Ncdgn "hqt "uco r ng "eqpvckpgtu	VDF	GTF E IO GUN
É Ncvz "i npxgu " "pqp / r qy f gtgf	8'dqzgu	GTF E IO GUN
É Nkq "lcmengu	6	GTF E
É O cej gvg "dt wuj "engctkpi +	3	GTF E
É O cr u "qh "y g "uwf { "ctgc	o cp {	GTF E IO GUN
É O gcuwtkpi "Vcr g ."52'o	3	GTF E
É P { nqp "Tqr g "3 16\$ +	522'hgv	GTF E IO GUN
É Rckrlqr gpqt	3	GTF E IO GUN
É Rrcuvle "nqgu "y kj "hkf u " "q "j qrf "gs wkr o gpvt	ugxgtn	GTF E IO GUN
É Twddgt "o cmgu	4	GTF E IO GUN
É Uchgv "i ncuugu	8	GTF E IO GUN
É Uj ctr kg "r gpv " "tgi wrct "r qkpv +	3'dqz	GTF E
É Ukr j qp lr wo r "hqt "tgo qxkpi "gzeguu "y cvgt "hqt o "uco r ngt " "r qn{ gj { npgp +	4	GTF E IO GUN
É Uco r ng "eqpvckpgtu " "3047 "i cr J F RG "dwengv "y kj "hkf +	VDF	GTF E IO GUN
É Uckprguu "uqgn "o guj "ukgxgu " "4 "o o . " : \$f kco gvt +	4	GTF E

Table 3. Sampling equipment required to support the 2013 sampling program for the Anniston PCB Site.

Item	Volume / Number	Responsibility for Acquiring and Bringing to the Field
General Sampling Supplies (cont.)		
É Uckprguu'uggn'l'o guj 'ulgxgu'*7'o o .: \$'f kco gvgt+	4	GTF E
É Uckprguu'uggn'qt "ghmp'ur cwr	6	GTF E IO GUN
É Uckprguu'uggn'lj qxgnu	4	GTF E
É Uckprguu'uggn'uqkn'eqtg"uco r ngt"*dwd"tcpur ncpvgt+	37	GTF E
É Uckprguu'uggn'tqy gnu	4	GTF E
É Wkkw' r ckn'r qnq gnu { nppg"36N+	6	GTF E IO GUN
É Y tkw/lp/tclp'hkrf 'pqgdqqmu	8	GTF E IO GUN
É Y tkw/lp'tclp'hkrf "r cr gt"*gzvtc+	nqw	GTF E IO GUN
Sample Processing Supplies		
É Engct'r cemki "cr g	32'tqmu	GTF E
É Grgevlecn'r cr g	8'tqmu	GTF E IO GUN
É O wpgm'Uqkl'Eqmjt'Dqqm	3	GTF E
É Rcr gt"qy gnu	34'tqmu	GTF E IO GUN
É Uckprguu'uggn'dqy nu	7	GTF E
É Uckprguu'uggn'lur qqp"/'ncti g	7	GTF E
É Uckprguu'uggn'ueqqr	7	GTF E
Measurement Supplies		
É I RU*ij cpf j grf +	4	GTF E IO GUN
Shipping and Storage Supplies		
É Dwddrg'y tcr "Tr cemki 'o cvgtlcn	Cu'pggf gf	GTF E IO GUN
É Ej ckp'qh'e wuqf { 'hqto u"qp'y cvgr tqqh'r cr gt+	Nqw	GTF E IO GUN
É Keg"q'o ckpckp"uco r ngu'cv'6Å	Cu'pggf gf	GTF E IO GUN
É O cuqgt'rqemj'hqj"tckngt	4	O GUN
É Rrcwle'eqqrgtu"/'6: /N	32	GTF E
É Tghtki gtcvgf "tckngt	3	O GUNIWI U
É Uco r ng"cpf "twd/uco r ng'lpqxpvt { 'hqto	3	O GUN
É Vgo r gtcwtg'f cvc"nji i gt	3	O GUNIWI U
É Y tcr 'hko	3'tqm	GTF E IO GUN
É \ kr mje"dcu u"/'3'i cmpp	4"dqzgu	GTF E
Personal Supplies		
É Eqqrgtu'hqj"qpukg'hqjf "cpf "dgxgtci gu"	4"z"6: 'N	GTF E IO GUN
É HktuvCkf 'Mkv	4	GTF E IO GUN
É Y cvgt'eqqrgt	3	GTF E
É Dwi 'tgr gmcpv	cu'pggf gf	Rgtuqpcn
É I nqxgu	cu'pggf gf	Rgtuqpcn
É J cv	cu'pggf gf	Rgtuqpcn
É Mpff'r cf u	cu'pggf gf	Rgtuqpcn
É Nqpi 'r cpwu	cu'pggf gf	Rgtuqpcn
É Nqpi /uggxgf 'uj ktu	cu'pggf gf	Rgtuqpcn
É Tckp'i gct	cu'pggf gf	Rgtuqpcn

Table 3. Sampling equipment required to support the 2013 sampling program for the Anniston PCB Site.

Item	Volume / Number	Responsibility for Acquiring and Bringing to the Field
Personal Supplies (cont.)		
É Uwpuet ggp'hqvkqp	cu'pggf gf	Rgtuqpcn
É Y cvgrt tqqh'hgif "dqqu	cu'pggf gf	Rgtuqpcn
Miscellaneous Supplies for the Field Command Post / Sample Processing Station		
É F wev'Vcr g	4	GTFE
É Gz vgpukqp'eqtf u	6	GTFE IO GUN
É Hkgf "Uco r rkpi 'Rrcp" *qp "y cvgrt tqqh'r cr gt+	5	O GUN
É Hkg'hqrf gtu'cpf "r ncuule lectf dqctf 'hkdpi "ecdpgv	3	GTFE
É J gcnj "cpf "Uchgv{ 'Rrcp	4	O GUN
É Ncr vqr "eqo r wgtu	4	GTFE IO GUN
É Rcenkpi "cr g"l kur gpugt	3	GTFE
É Rgpeku	3"dqz	GTFE
É Rqtvcdrig'Rqy gt 'Rcem	3	GTFE
É Rtkpvt 'hqt'hgif "eqo o cpf "r quv"	3	GTFE IO GUN
É S wrkv{ 'Cuuwtceg'Rtqlgev'Rrcp	3	O GUN
É Twrgt	3	GTFE IO GUN
É Ueknuqtu	4	GTFE IO GUN
É Vqqri'nlv	3	GTFE
É WUD'y kjj "cm't grgxcpv'hkgu	3	O GUN
É \ cr 'utcr u	O cp{	GTFE IO GUN

Figures

Figure 1. Map of the study area for the 2013 sediment and soil sampling program for the Anniston PCB Site.

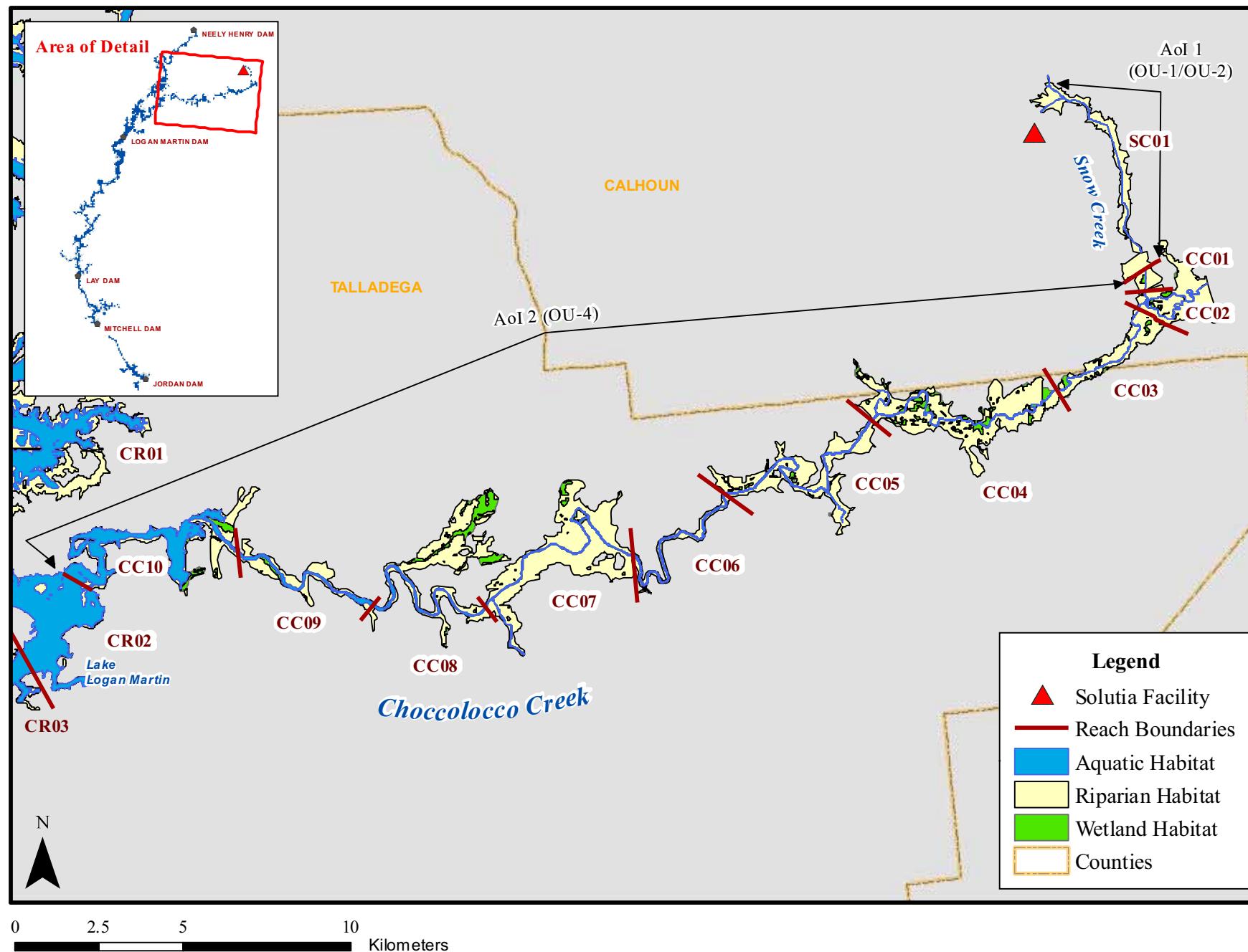


Figure 2. Locations of sediment sampling stations in Reach CC02 of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

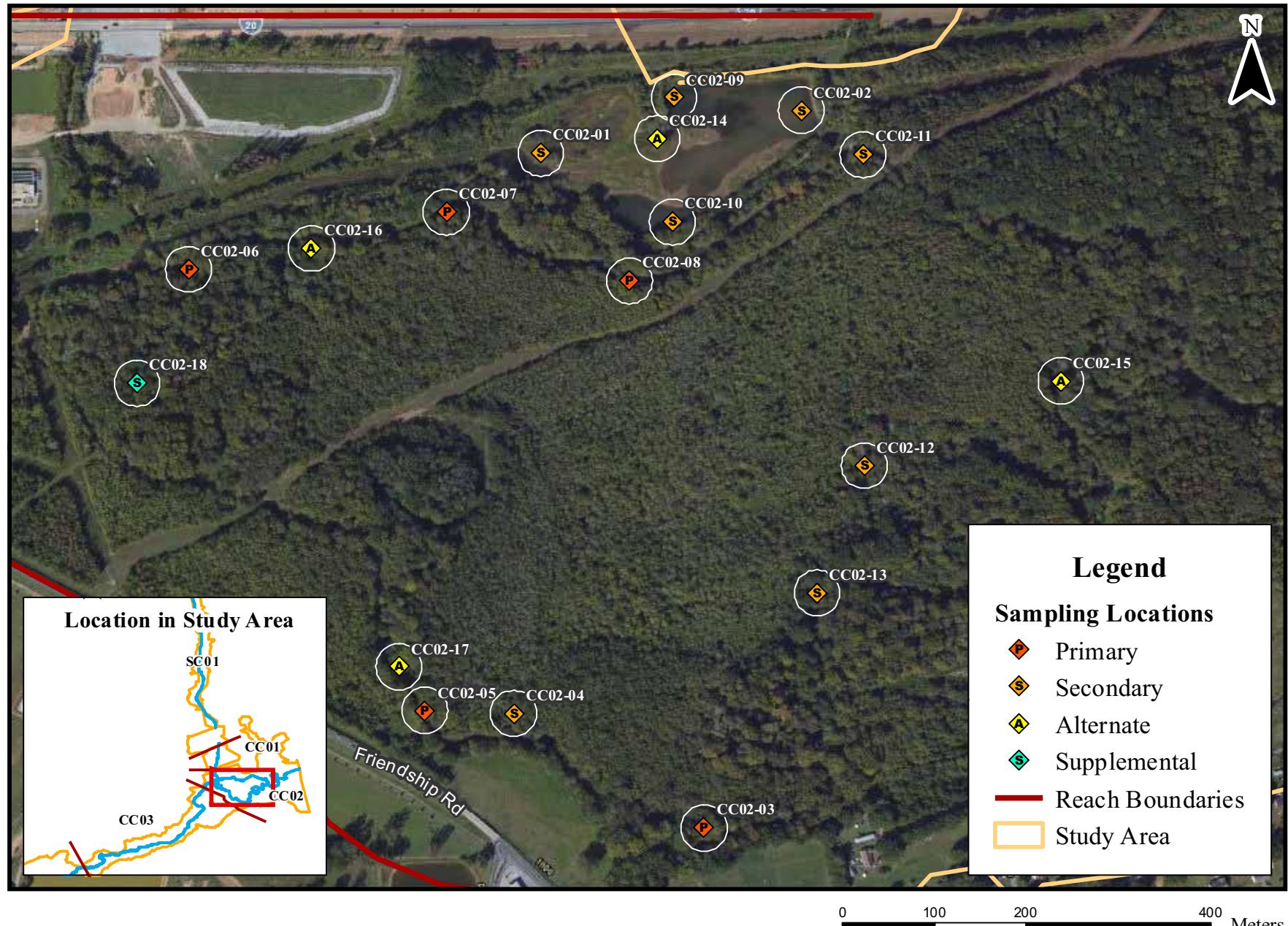


Figure 3. Locations of sediment sampling stations in Reach CC04 (upstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

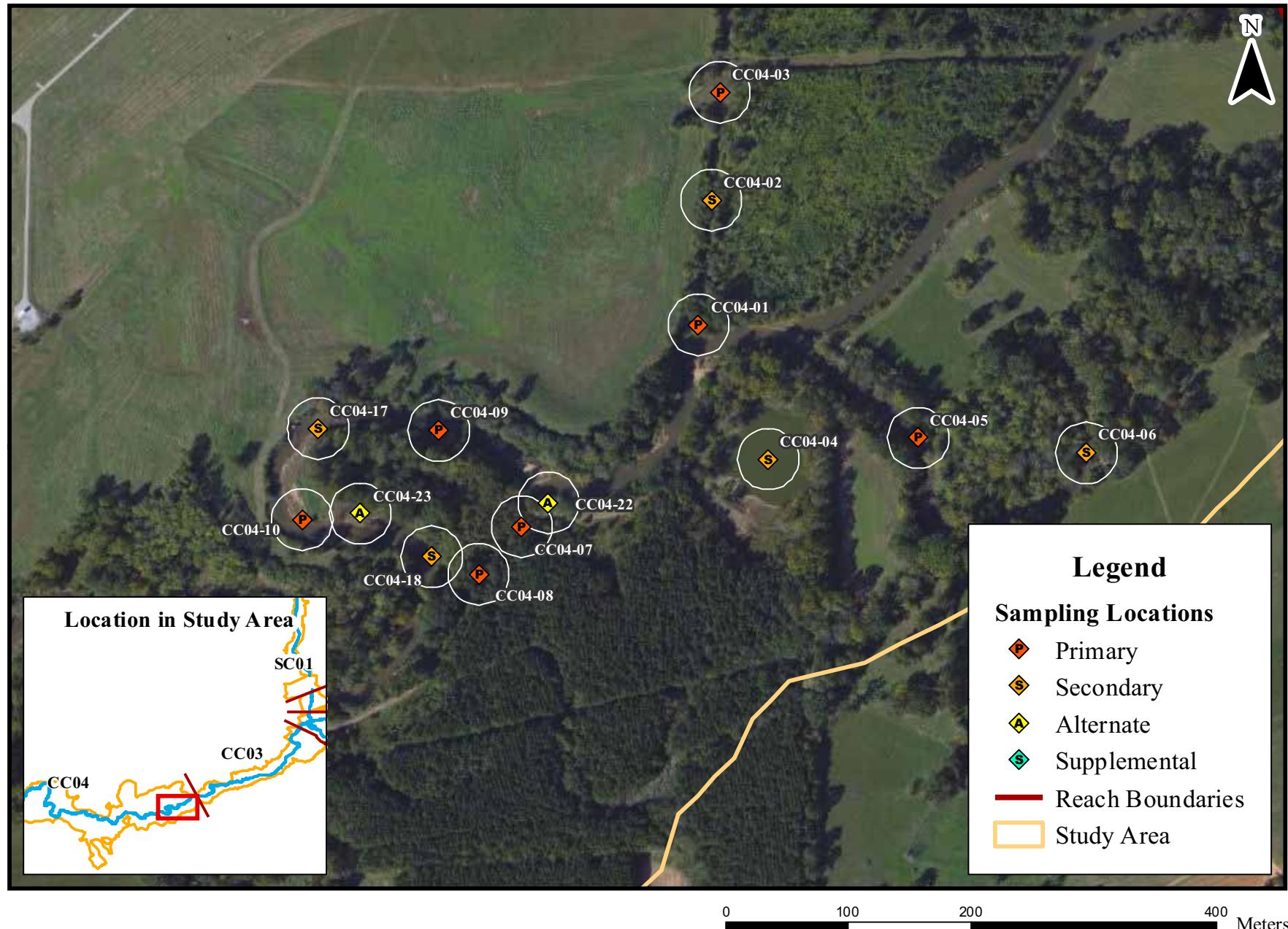


Figure 4. Locations of sediment sampling stations in Reach CC04 (downstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

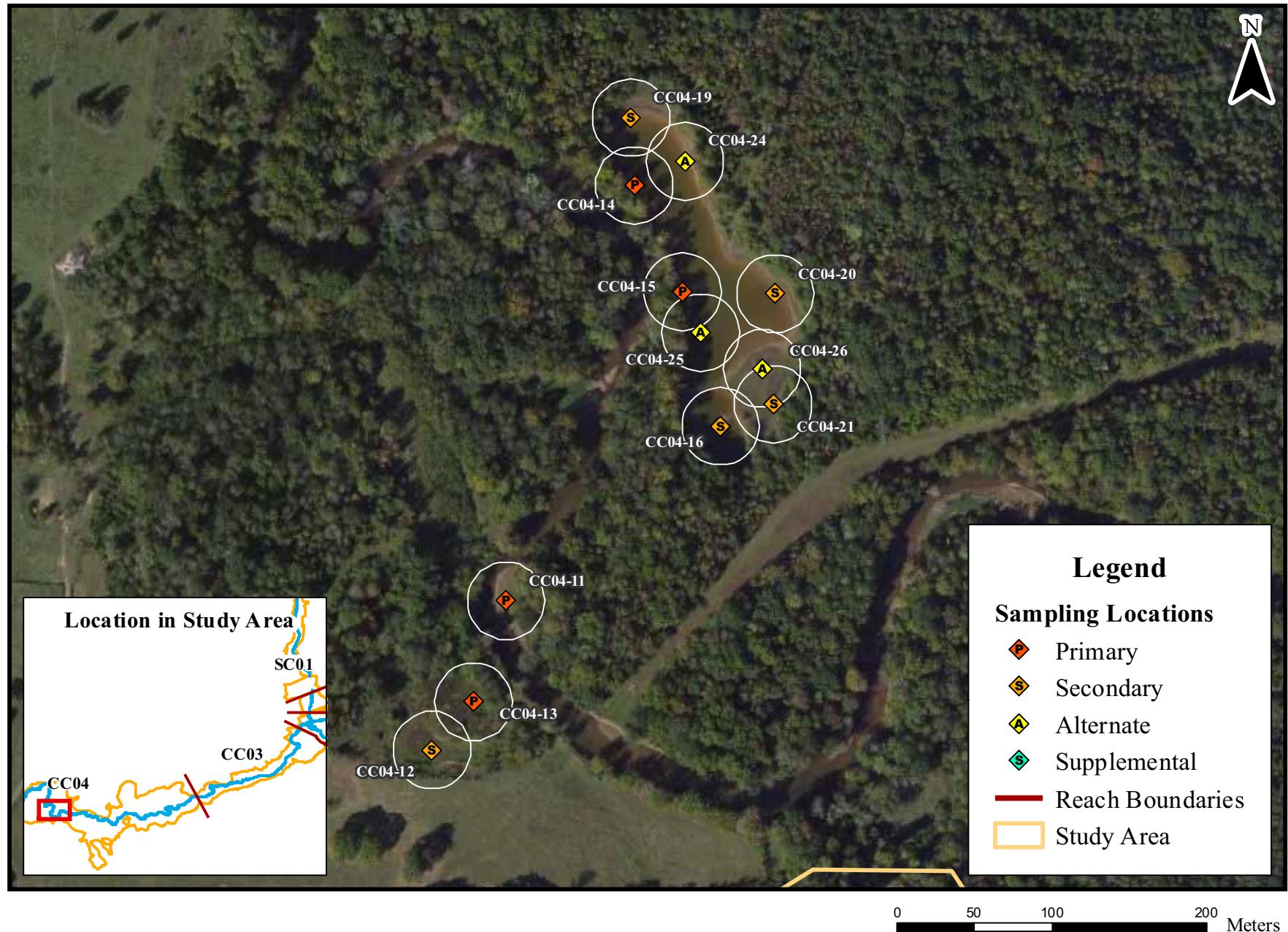


Figure 5. Locations of sediment sampling stations in Reach CC05 (upstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

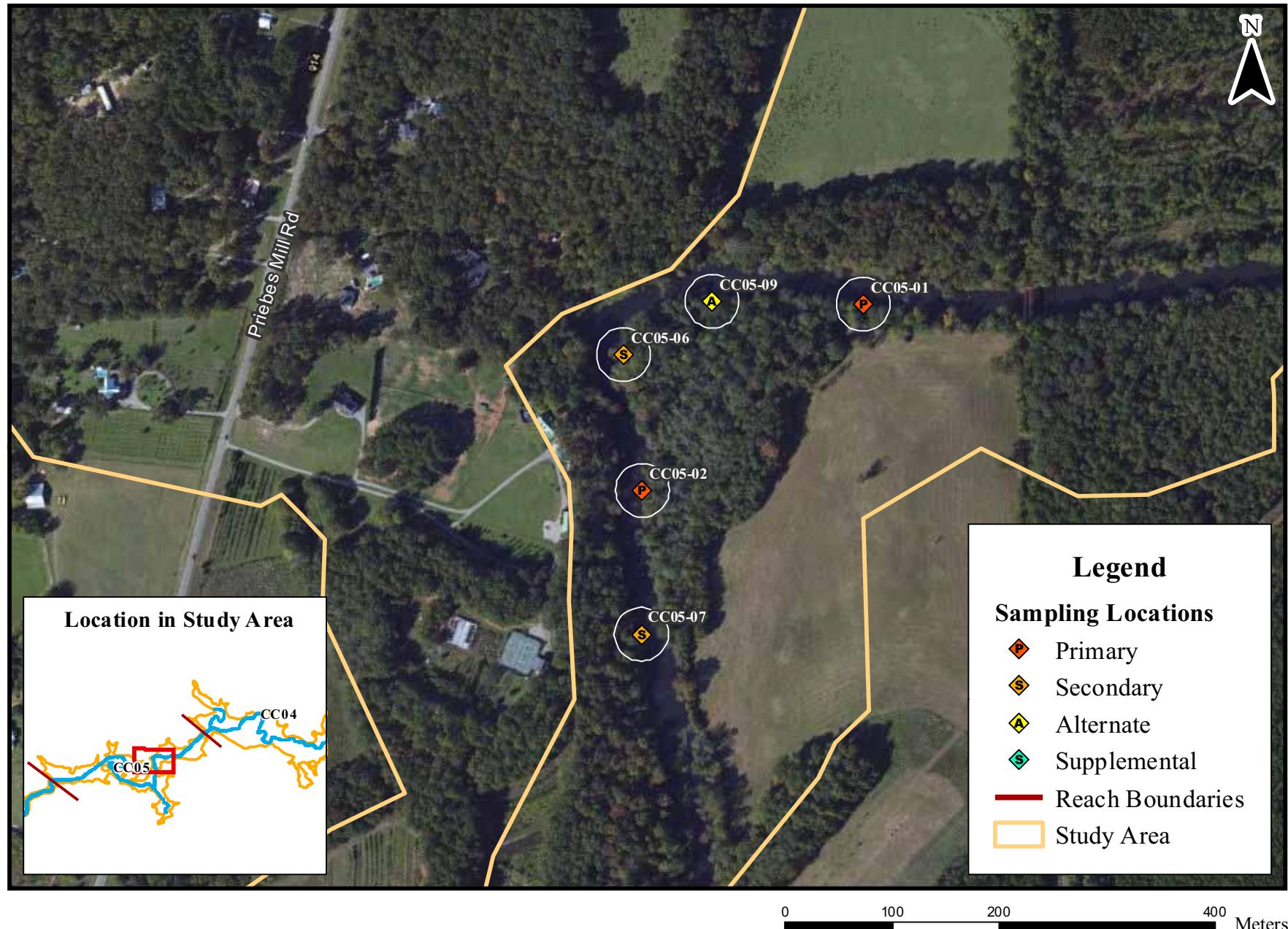


Figure 6. Locations of sediment sampling stations in Reach CC05 (downstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

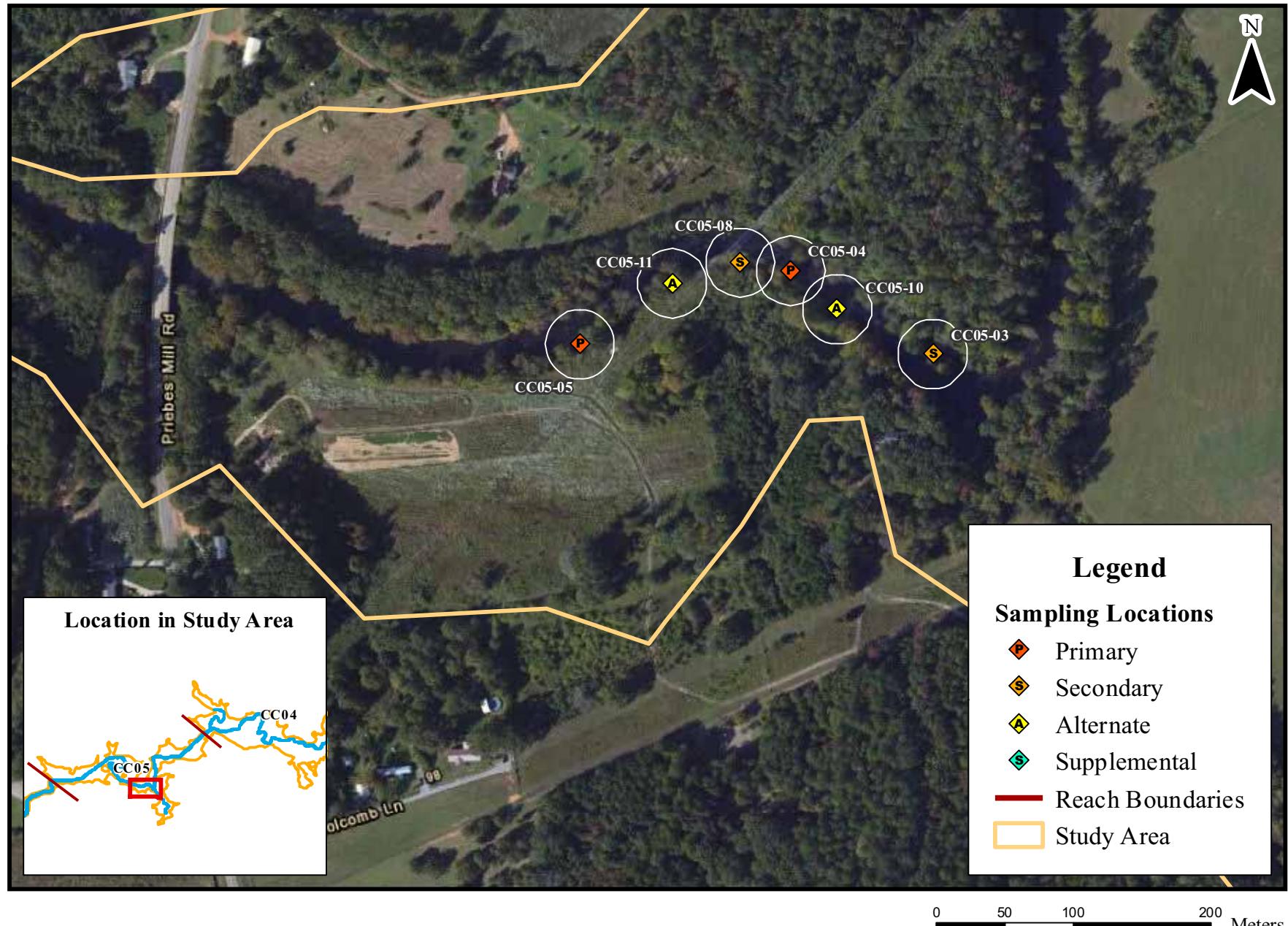


Figure 7. Locations of sediment sampling stations in Reach CC06 (upstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

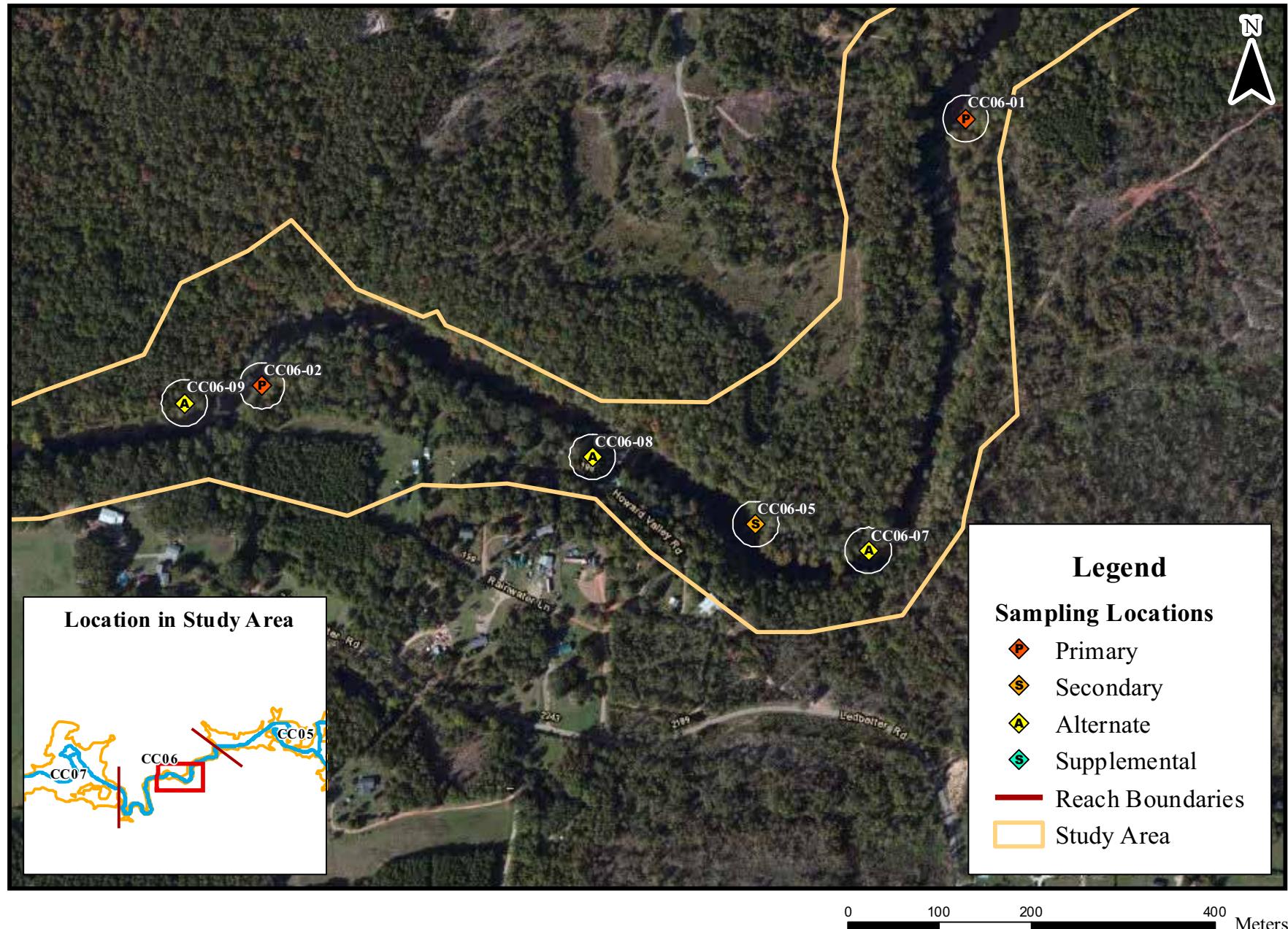


Figure 8. Locations of sediment sampling stations in Reach CC06 (downstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

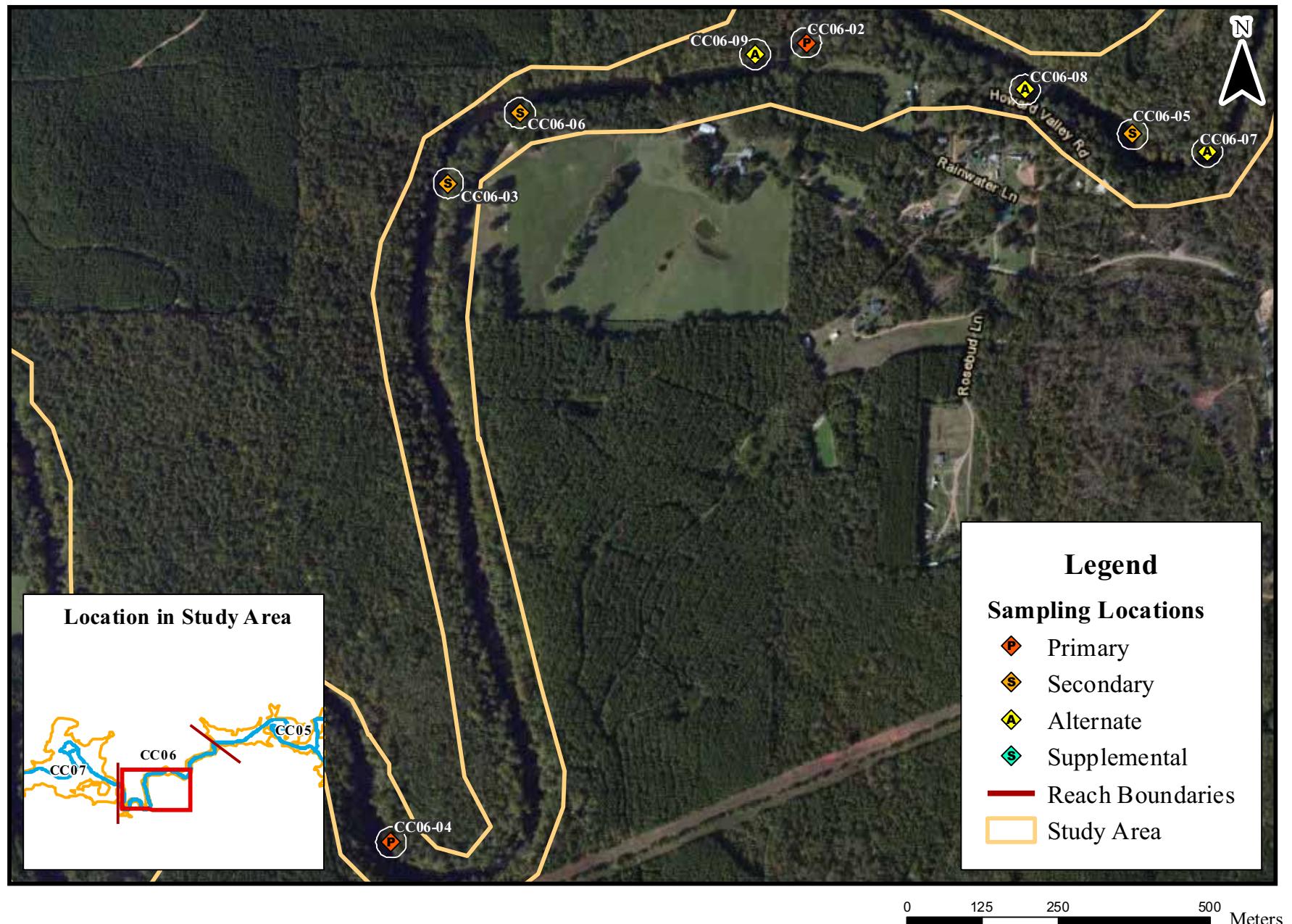


Figure 9. Locations of sediment sampling stations in Reach CC07 of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

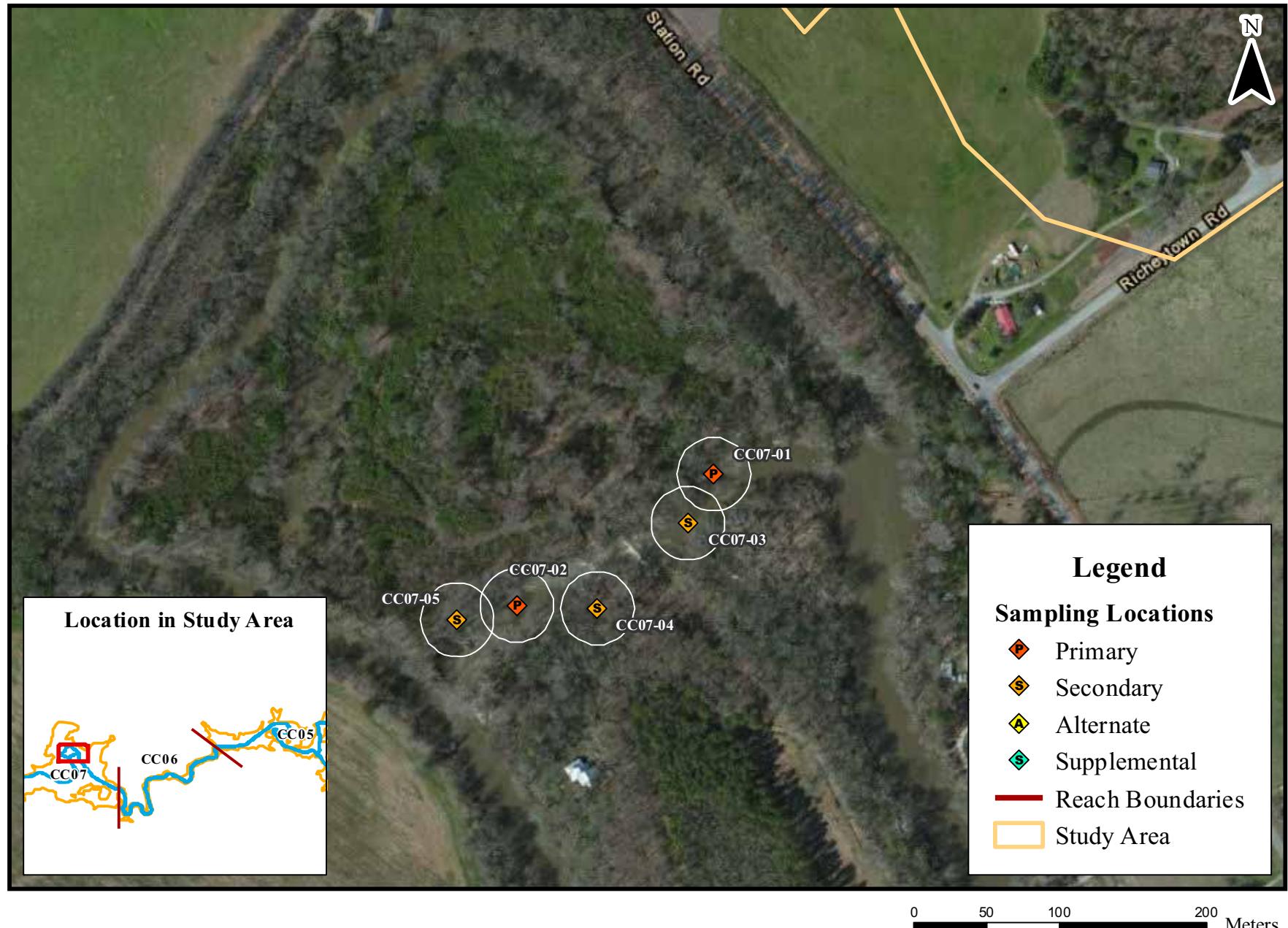


Figure 10. Locations of sediment sampling stations in Reach CC09 (upstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

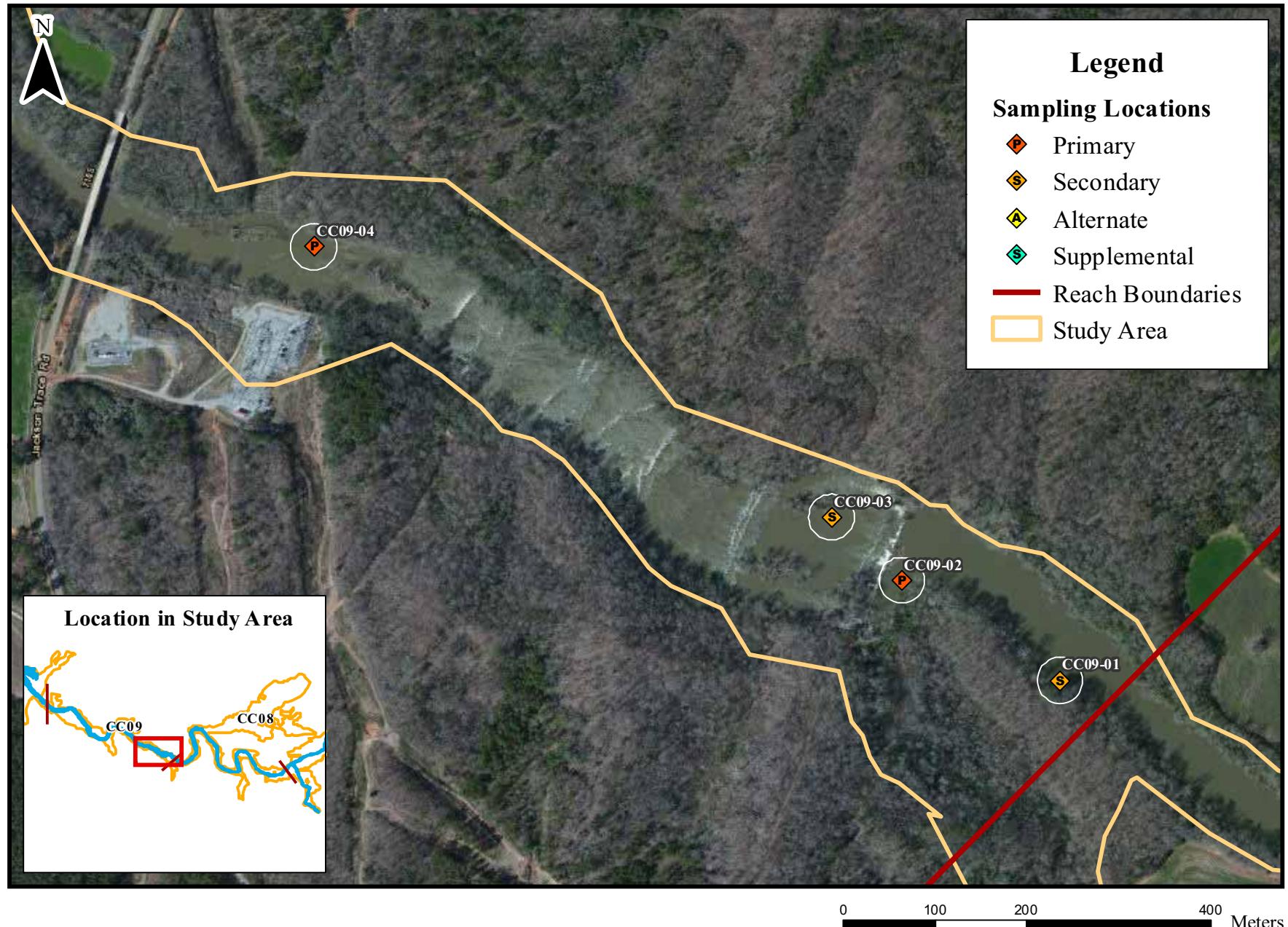


Figure 11. Locations of sediment sampling stations in Reach CC09 (downstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

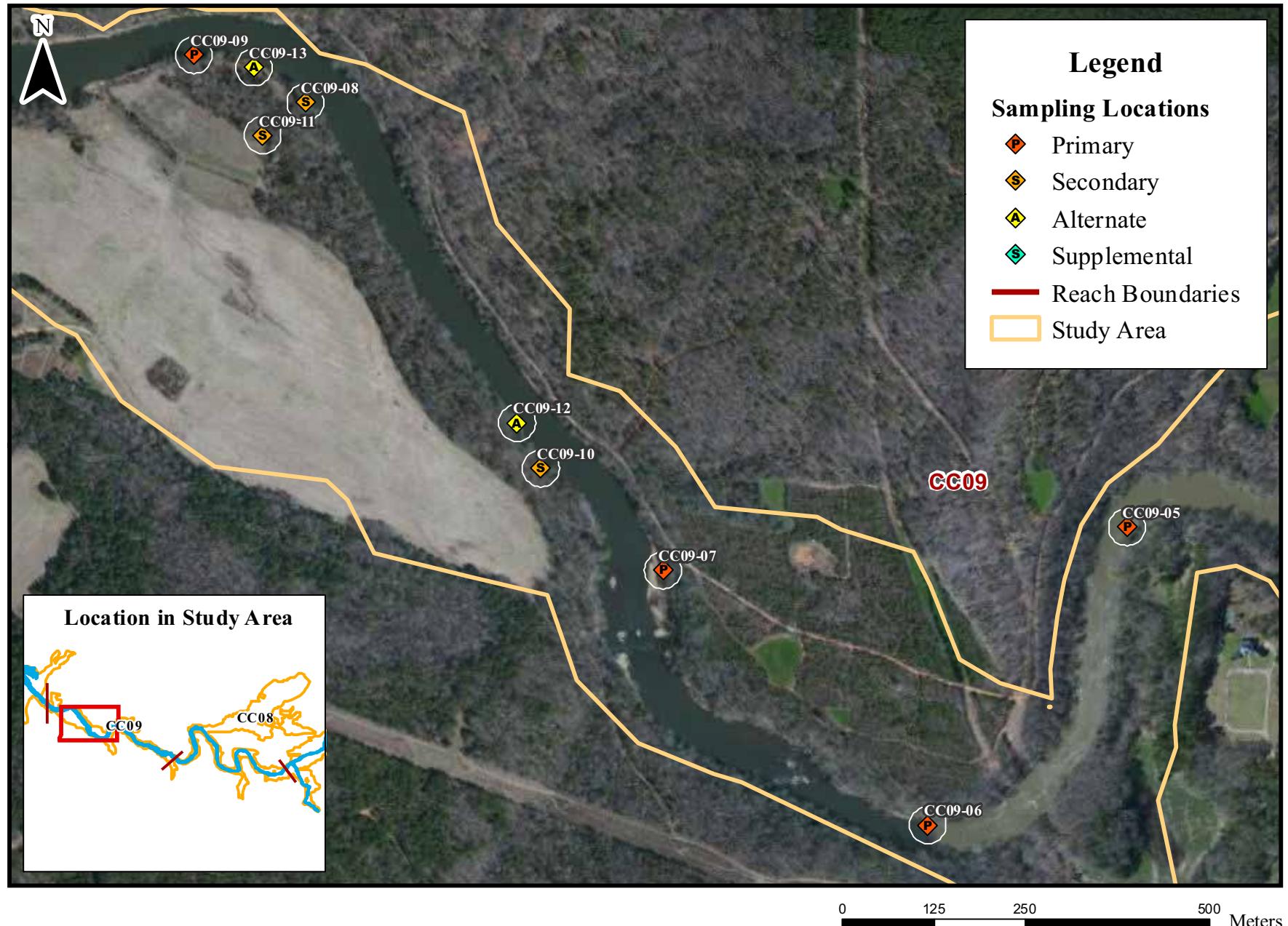


Figure 12. Locations of sediment sampling stations in Reach CC10 (upstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

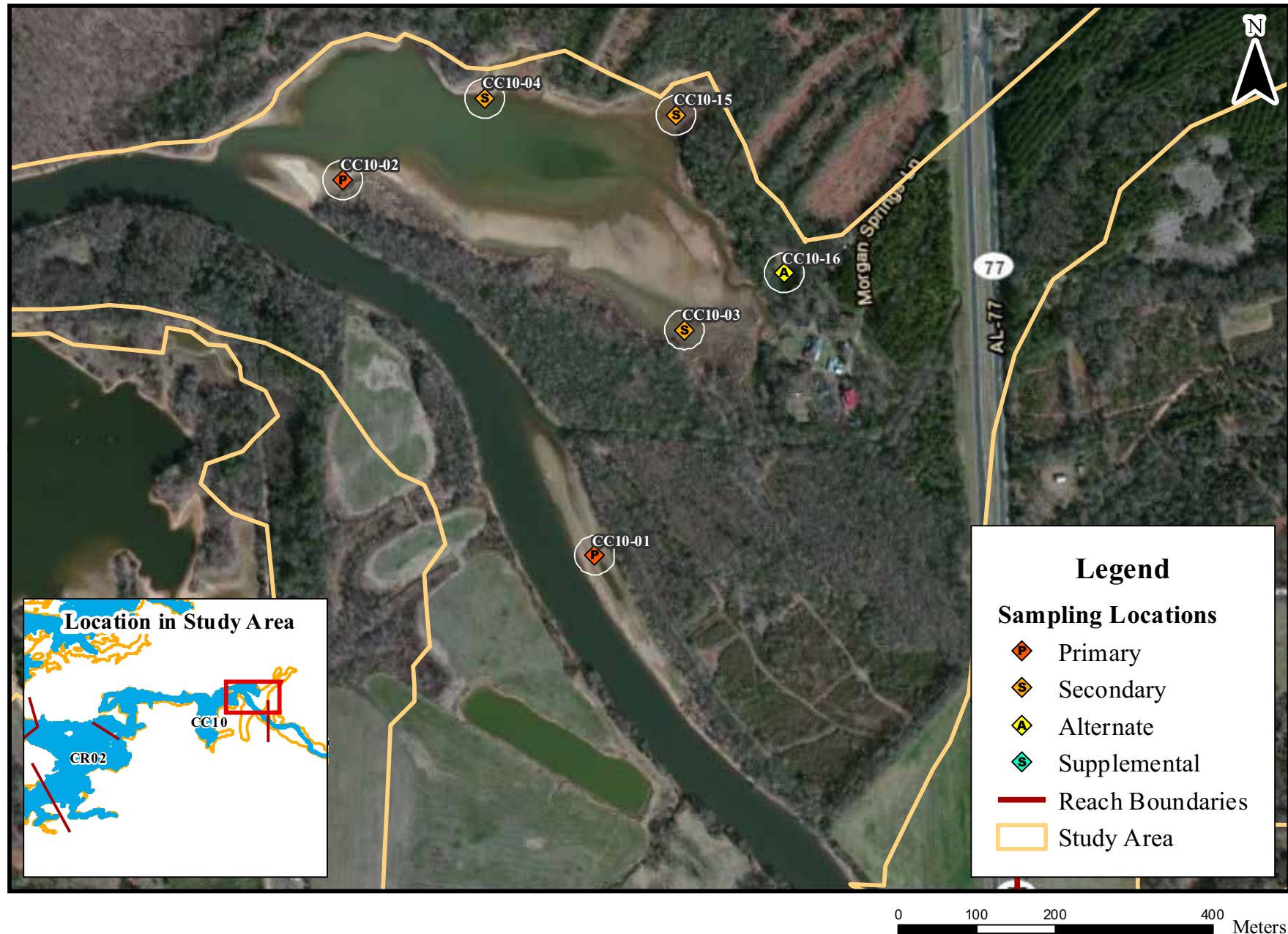


Figure 13. Locations of sediment sampling stations in Reach CC10 (middle portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

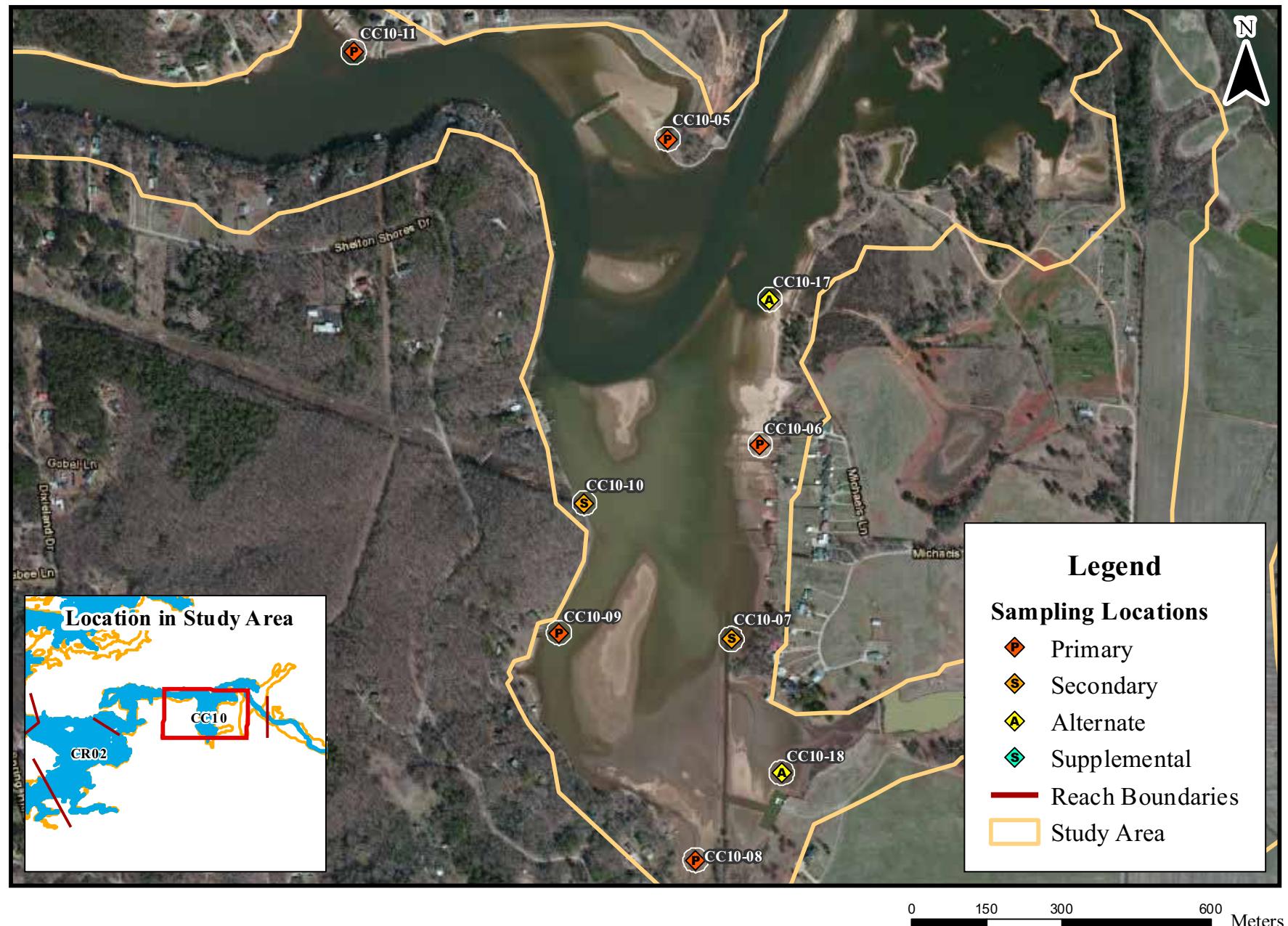


Figure 14. Locations of sediment sampling stations in Reach CC10 (upstream portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

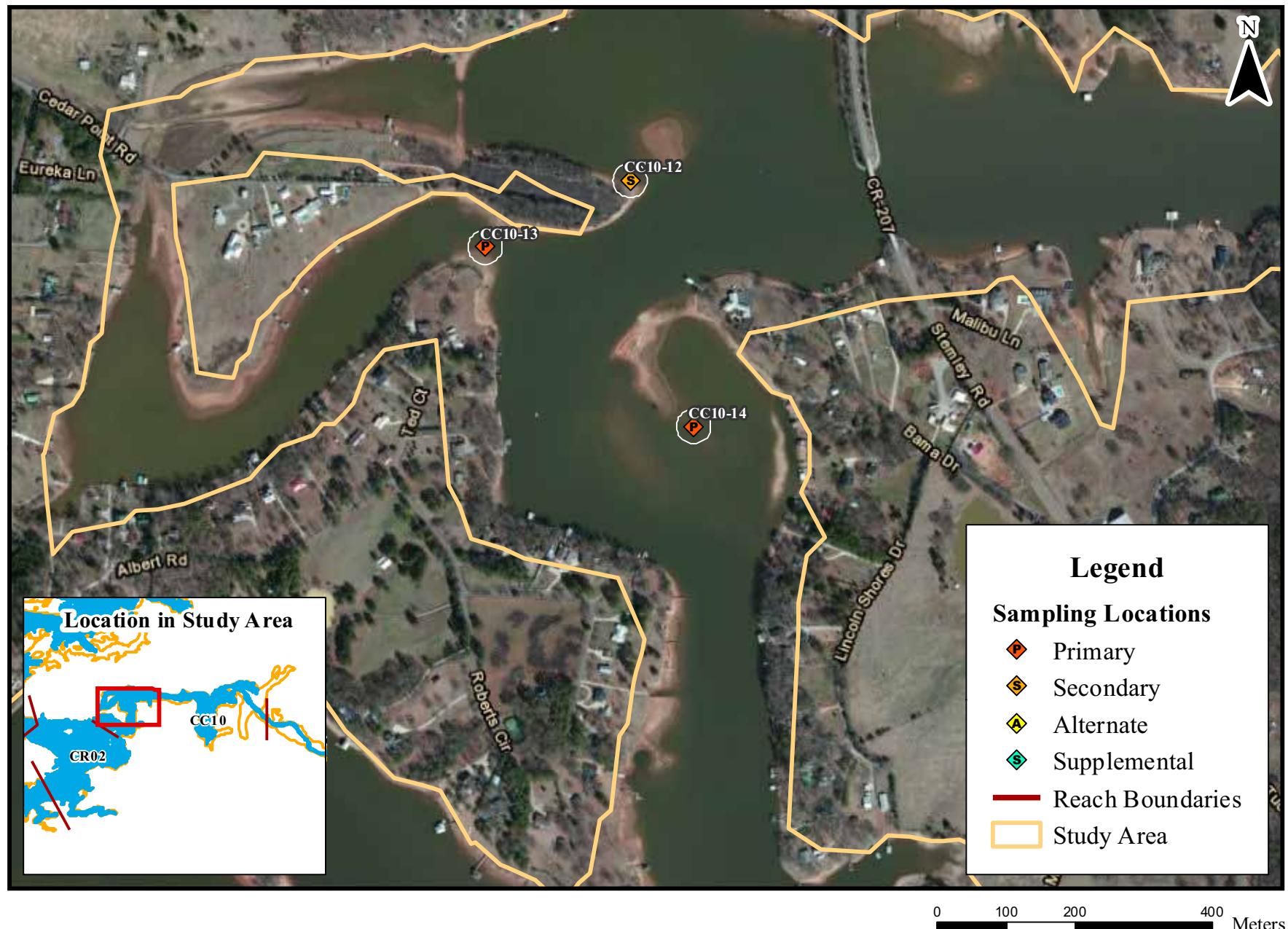


Figure 15. Locations of sediment sampling stations in Reach CR02 (northern portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).



Figure 16. Locations of sediment sampling stations in Reach CR02 (southern portion) of the study area (the 25 meter buffer within which sediment samples will be collected during the 2013 sampling program is also shown).

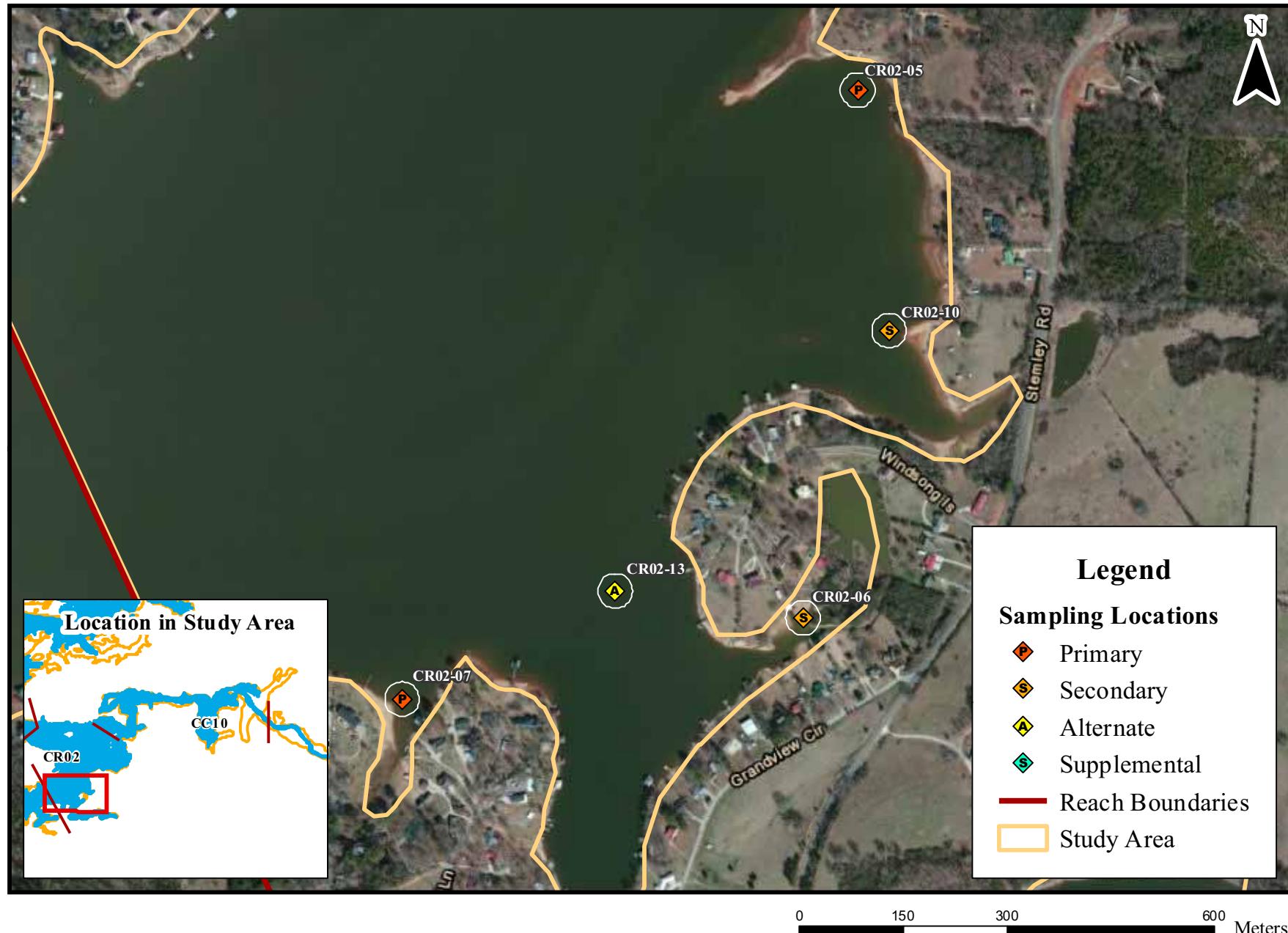


Figure 17. Locations of soil sampling stations near Reach SC01 of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

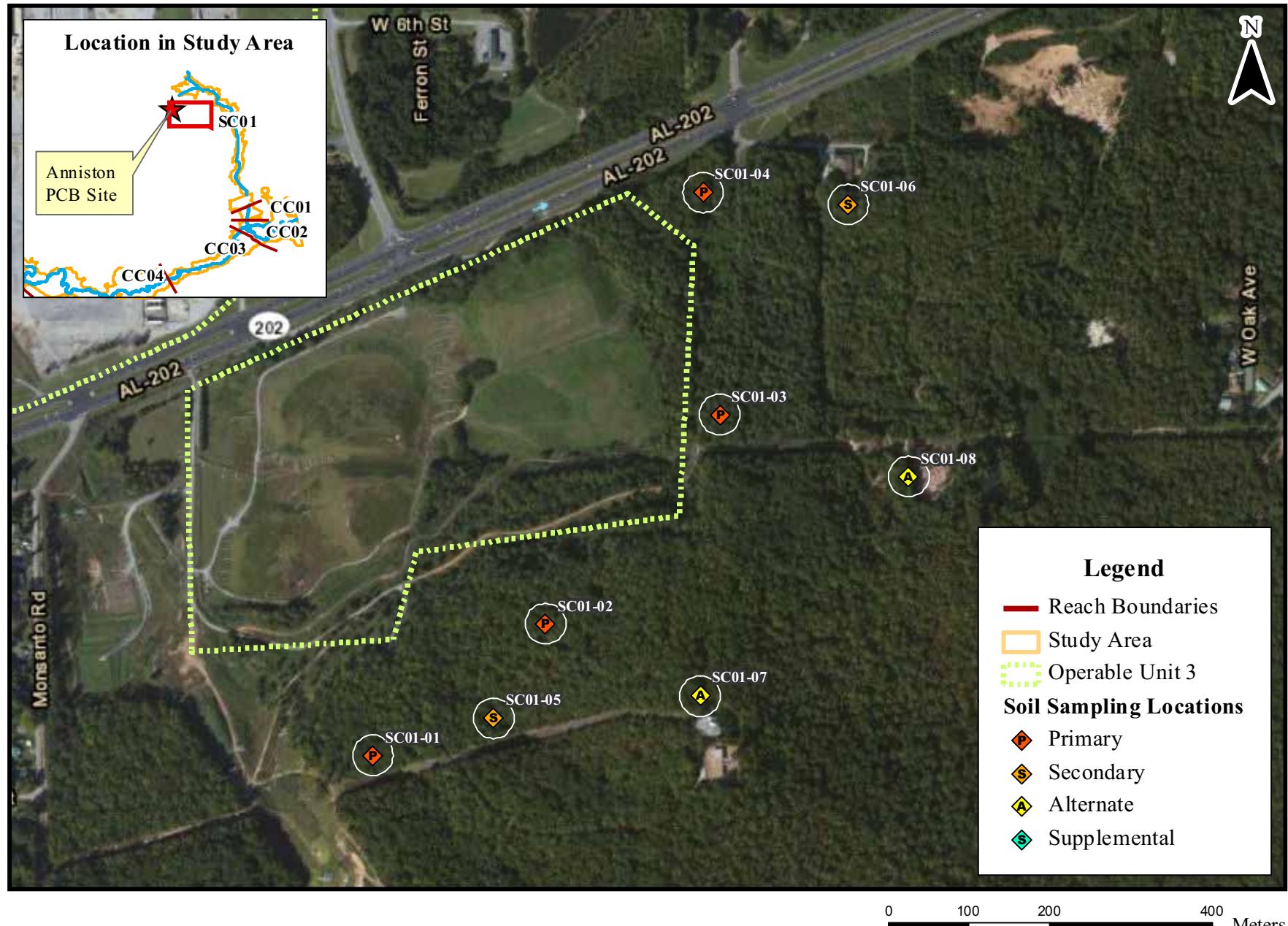


Figure 18. Locations of soil sampling stations in Reach CC01 of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

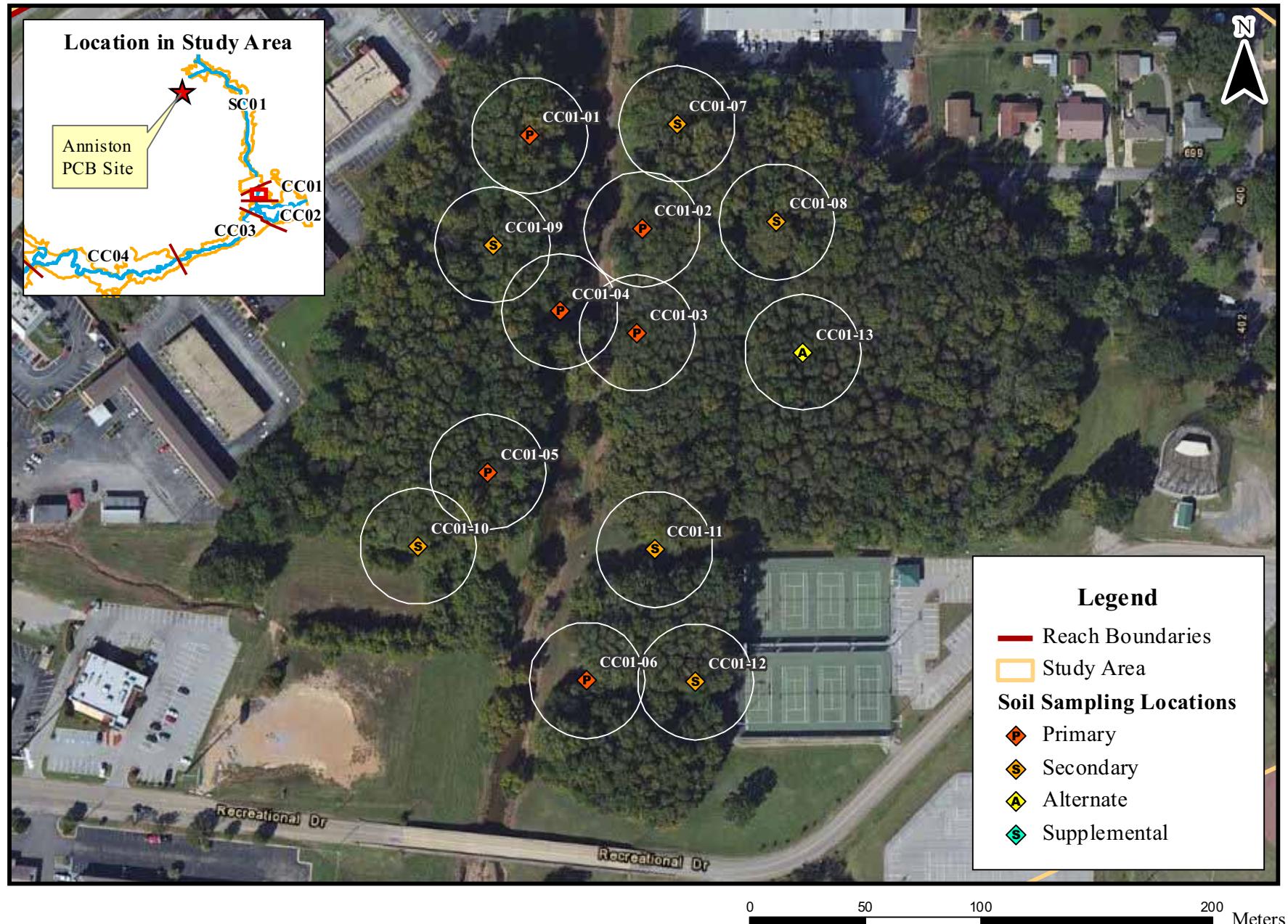


Figure 19. Locations of soil sampling stations in Reach CC02 of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

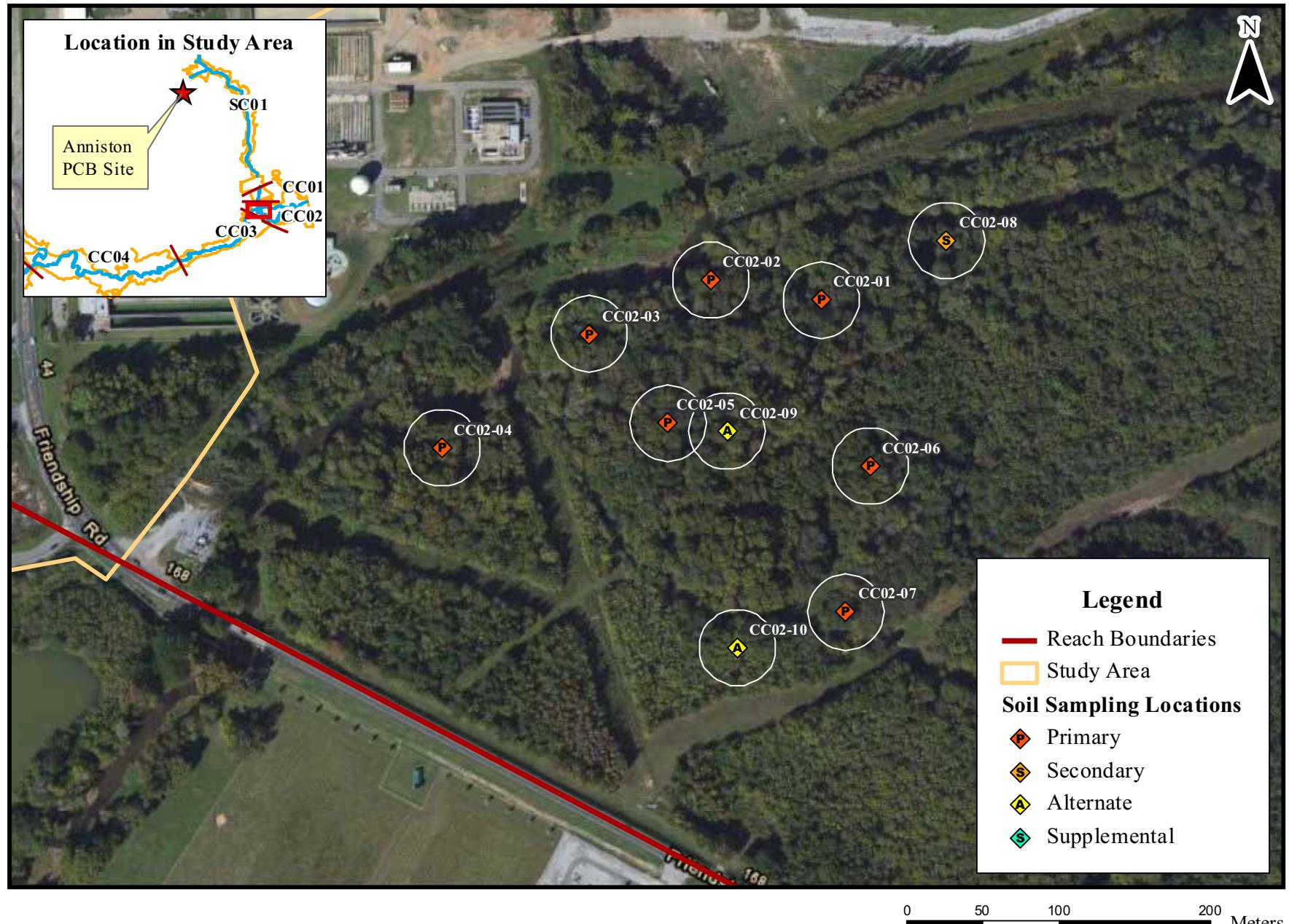


Figure 20. Locations of soil sampling stations in Reach CC03 of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

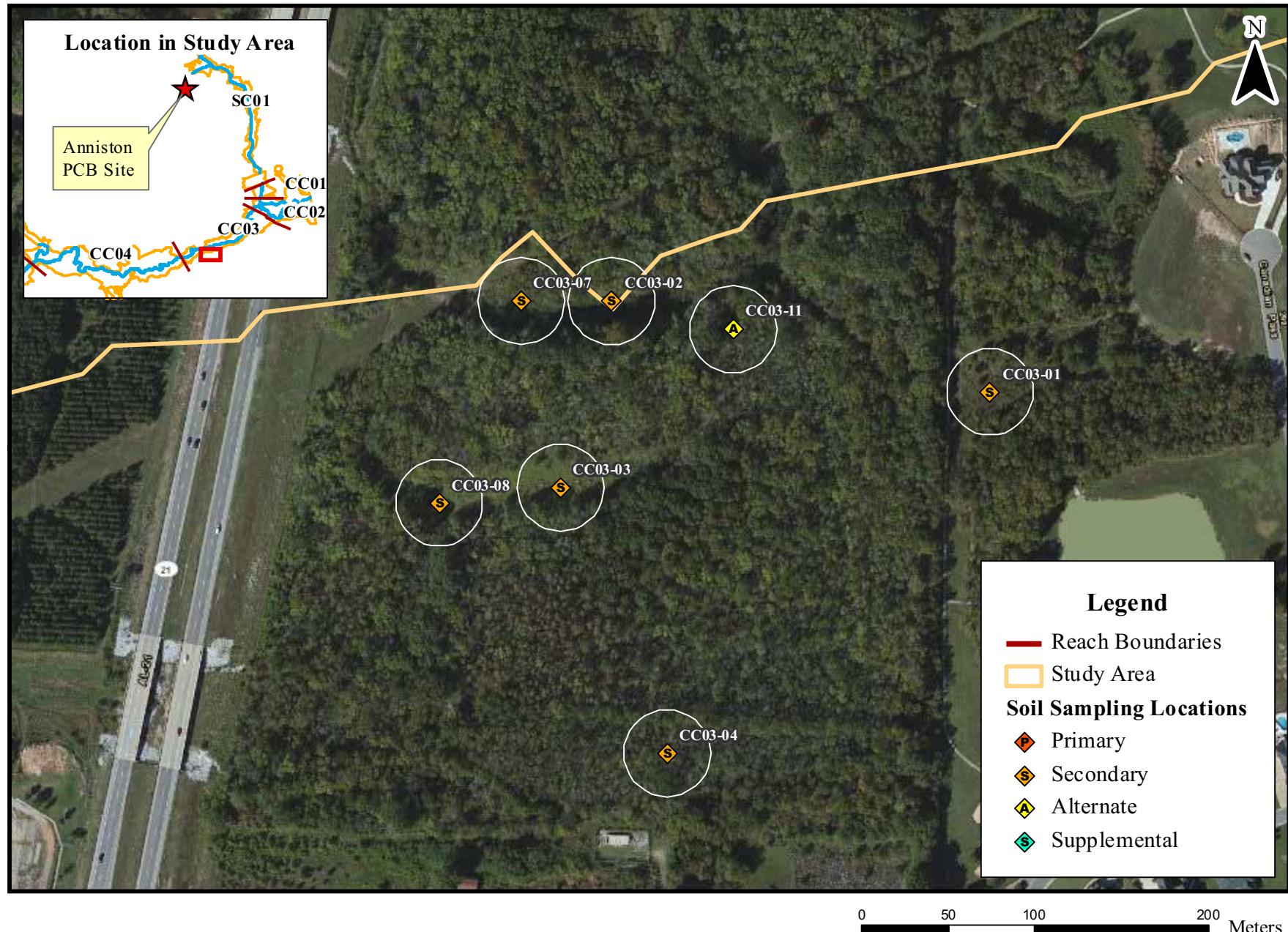


Figure 21. Locations of soil sampling stations in Reaches CC03 and CC04 of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

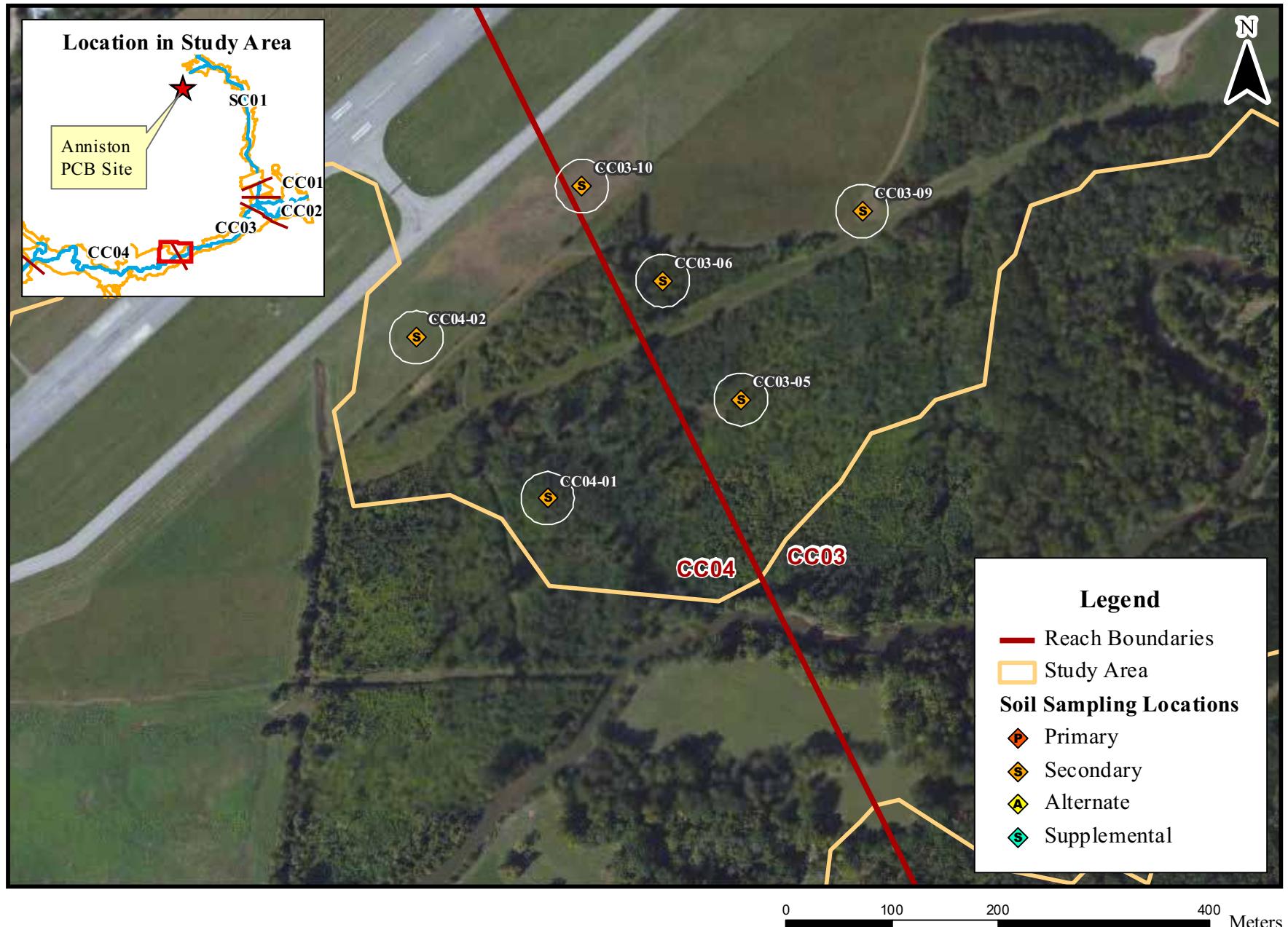


Figure 22. Locations of soil sampling stations in Reach CC04 (upstream portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

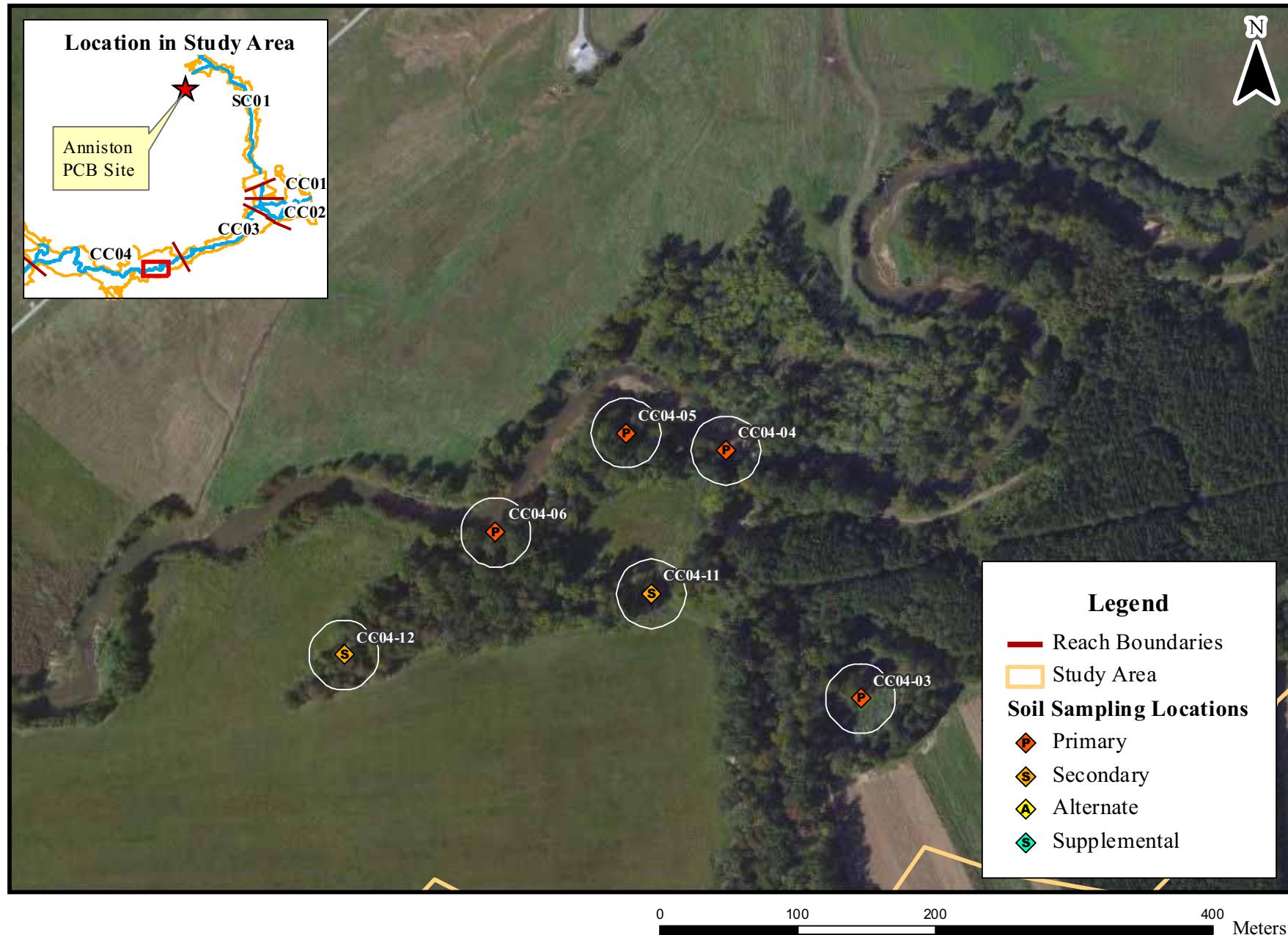


Figure 23. Locations of soil sampling stations in Reach CC04 (middle portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).



Figure 24. Locations of soil sampling stations in Reach CC04 (downstream portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

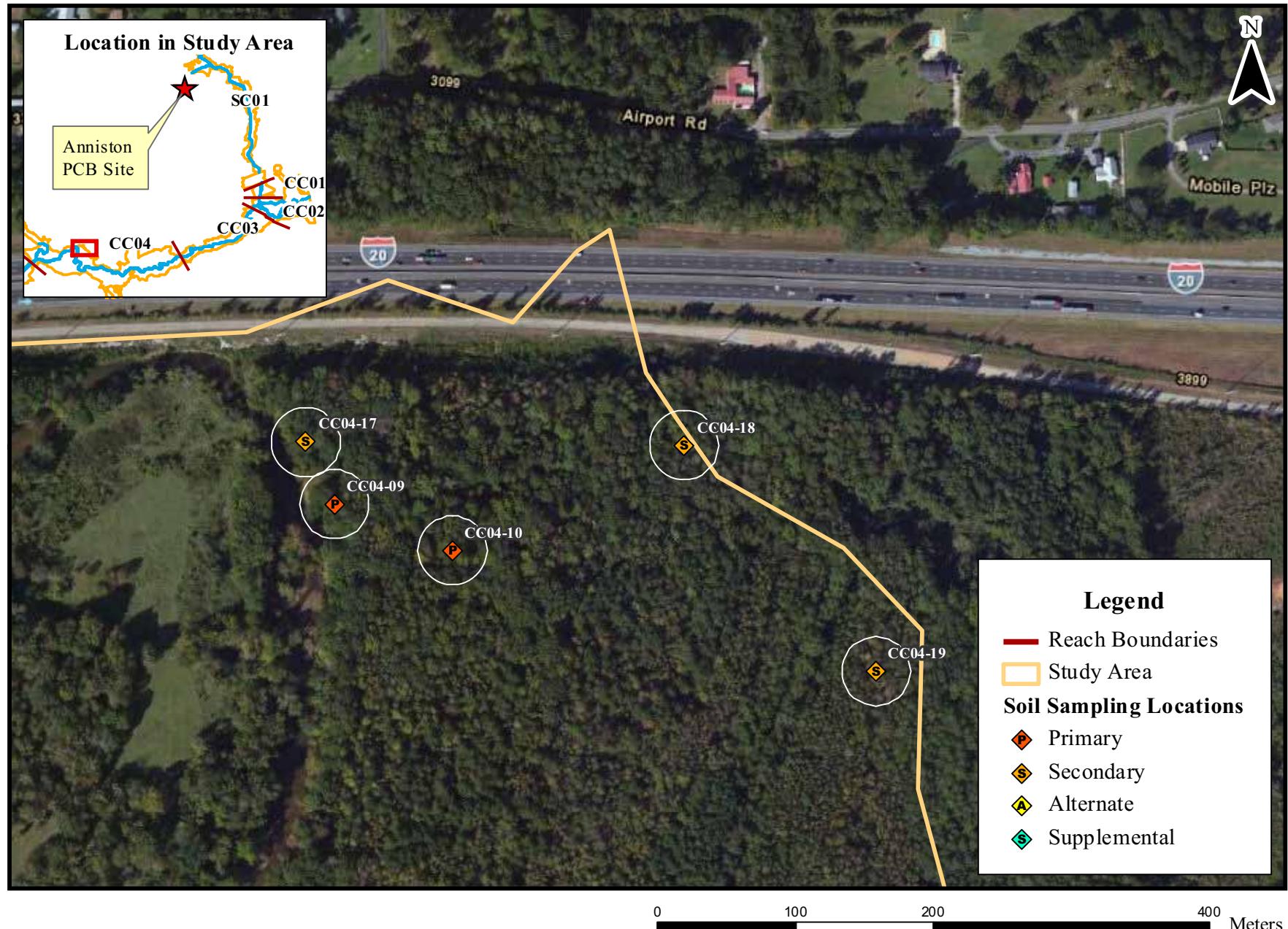


Figure 25. Locations of soil sampling stations in Reach CC07 (upstream portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

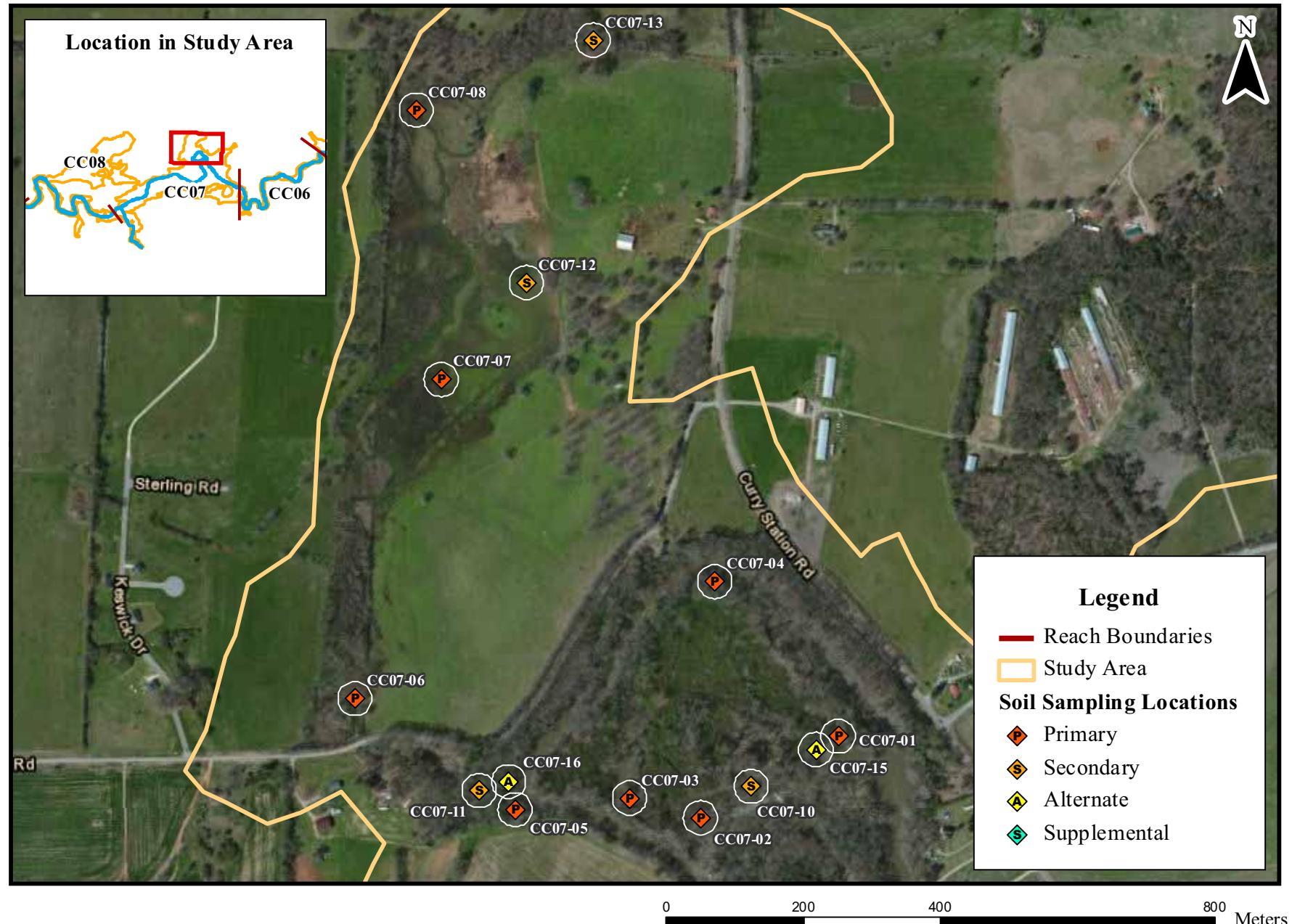


Figure 26. Locations of soil sampling stations in Reaches CC07 and CC08 of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

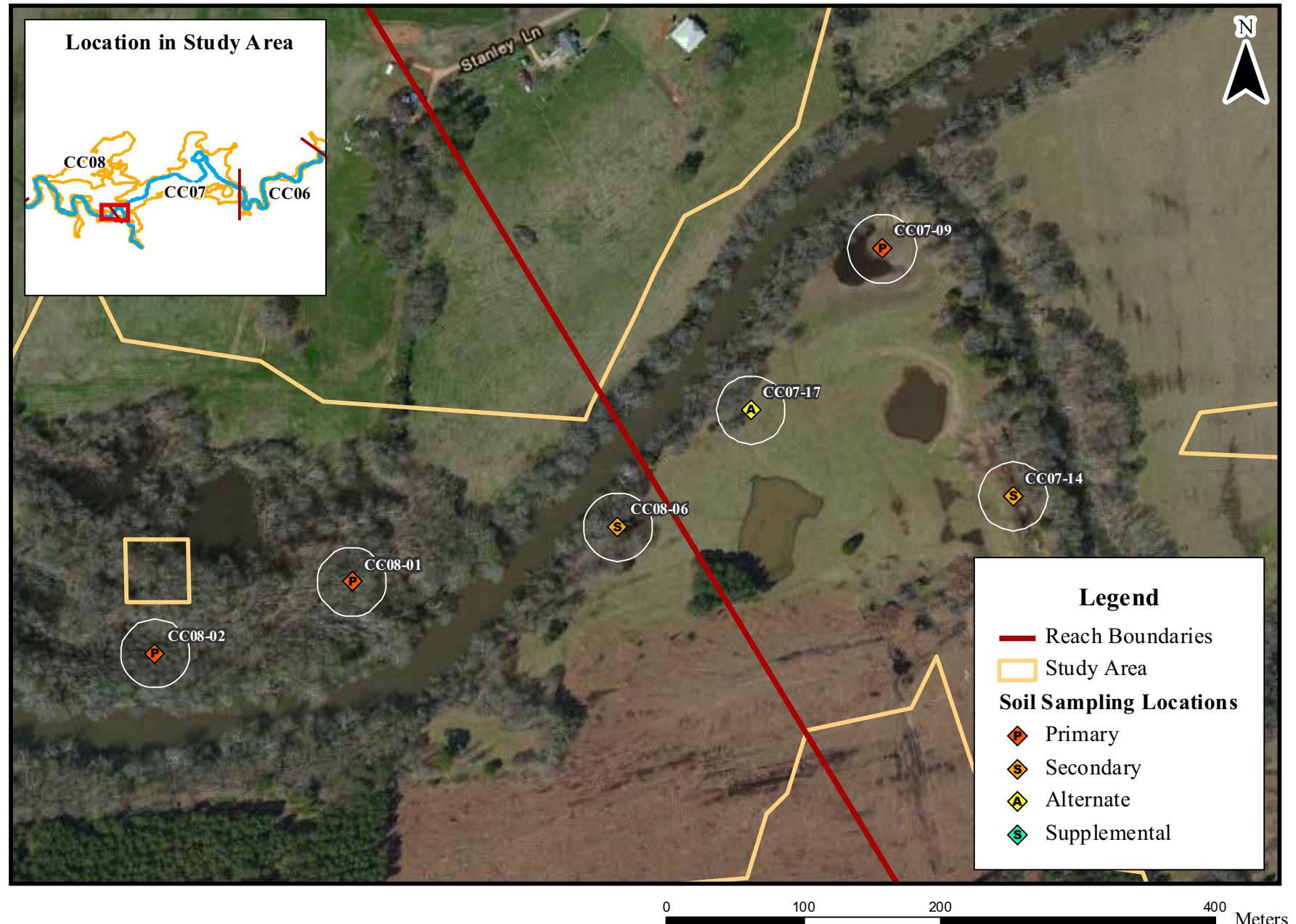


Figure 27. Locations of soil sampling stations in Reach CC08 (middle portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

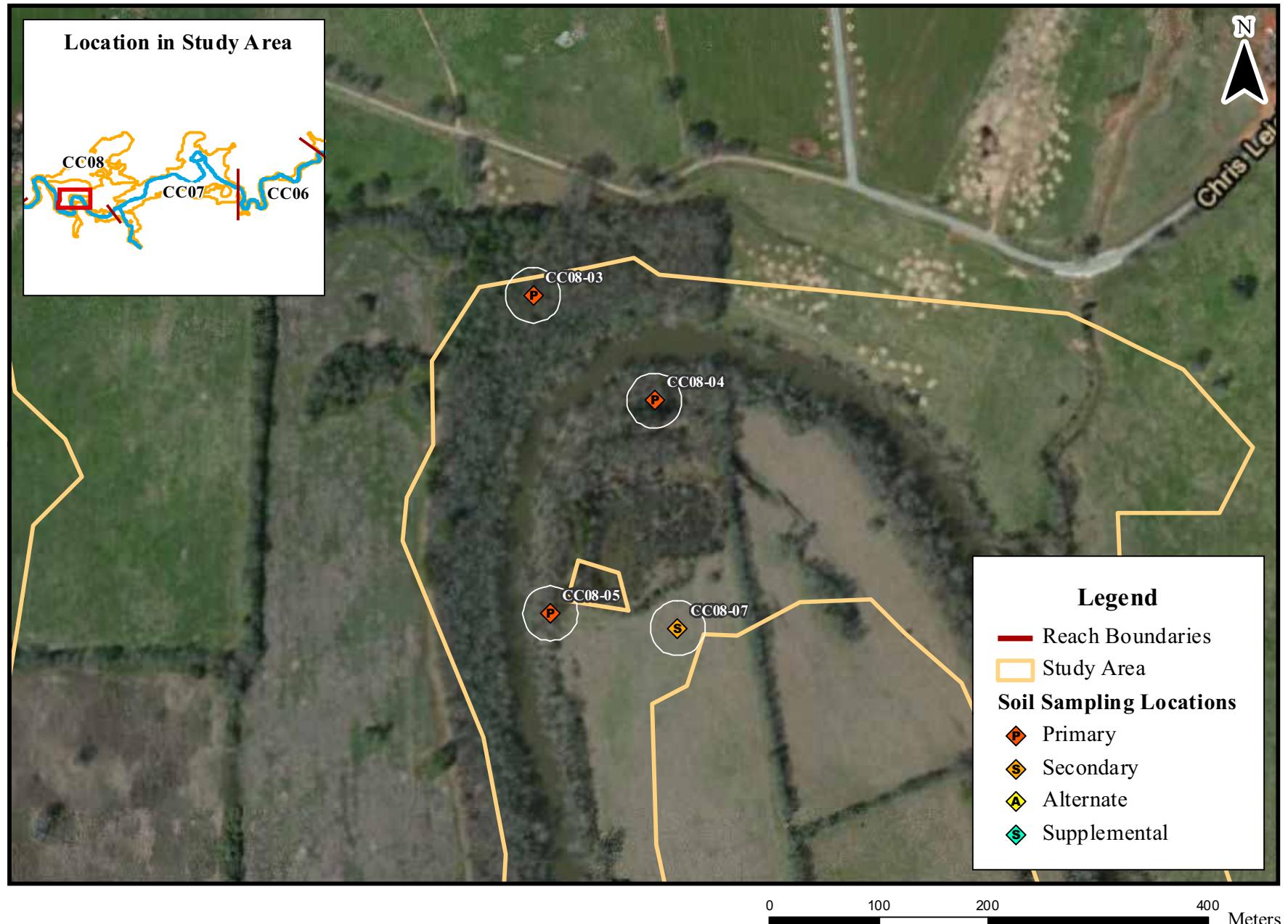


Figure 28. Locations of soil sampling stations in Reach CC08 (upstream portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

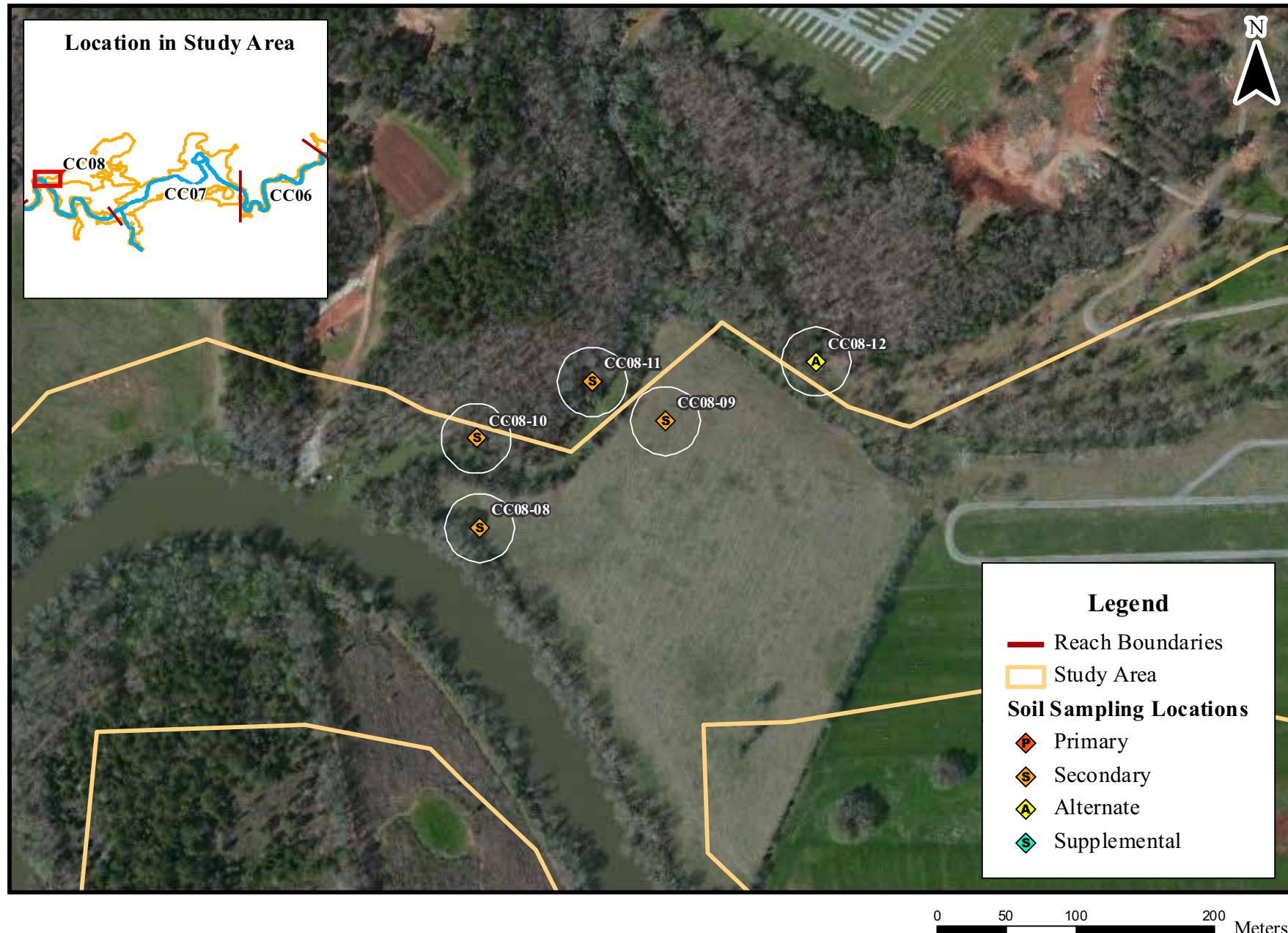


Figure 29. Locations of soil sampling stations in Reach CC08 (northern portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

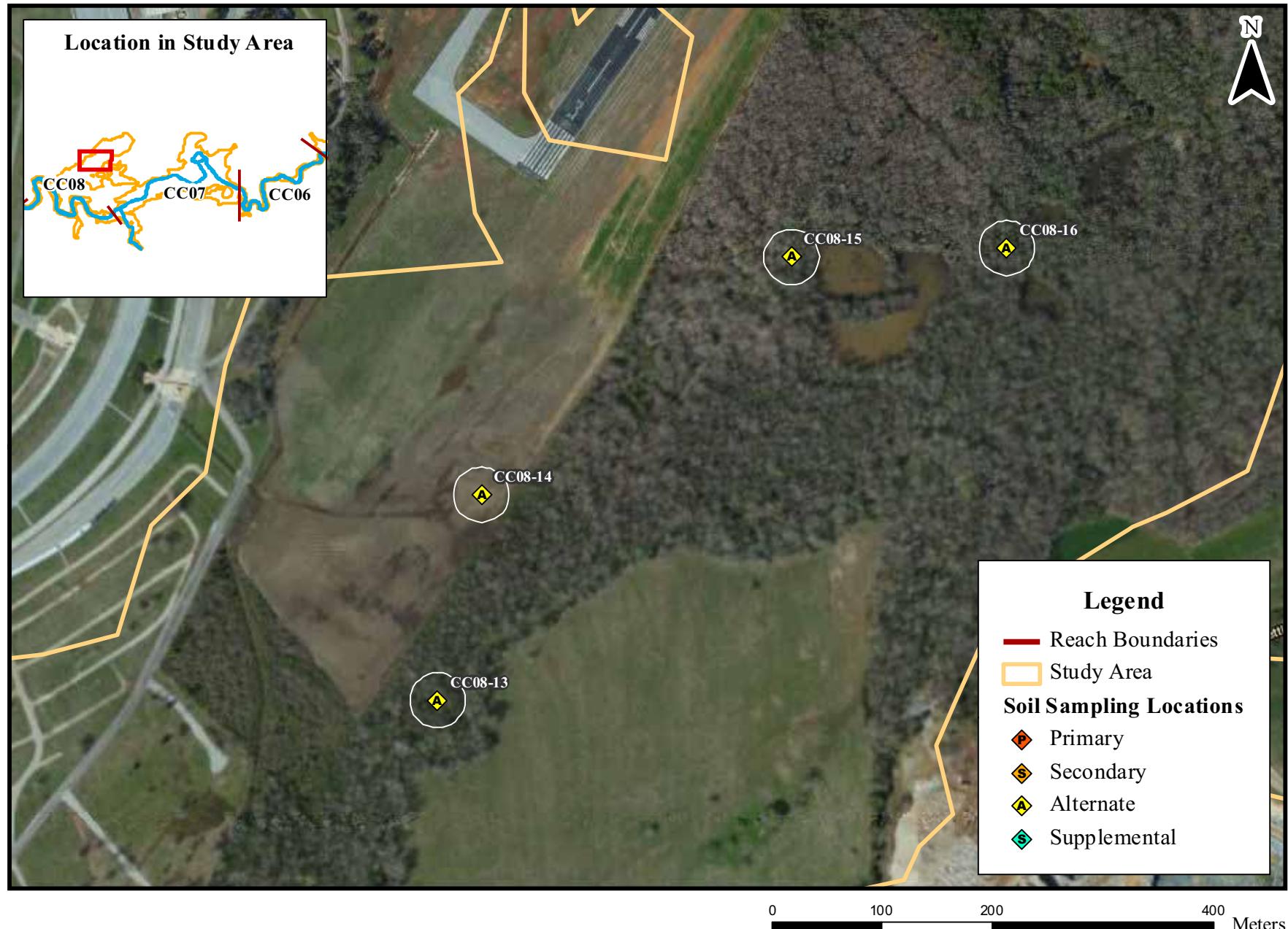


Figure 30. Locations of soil sampling stations in Reach CC09 of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

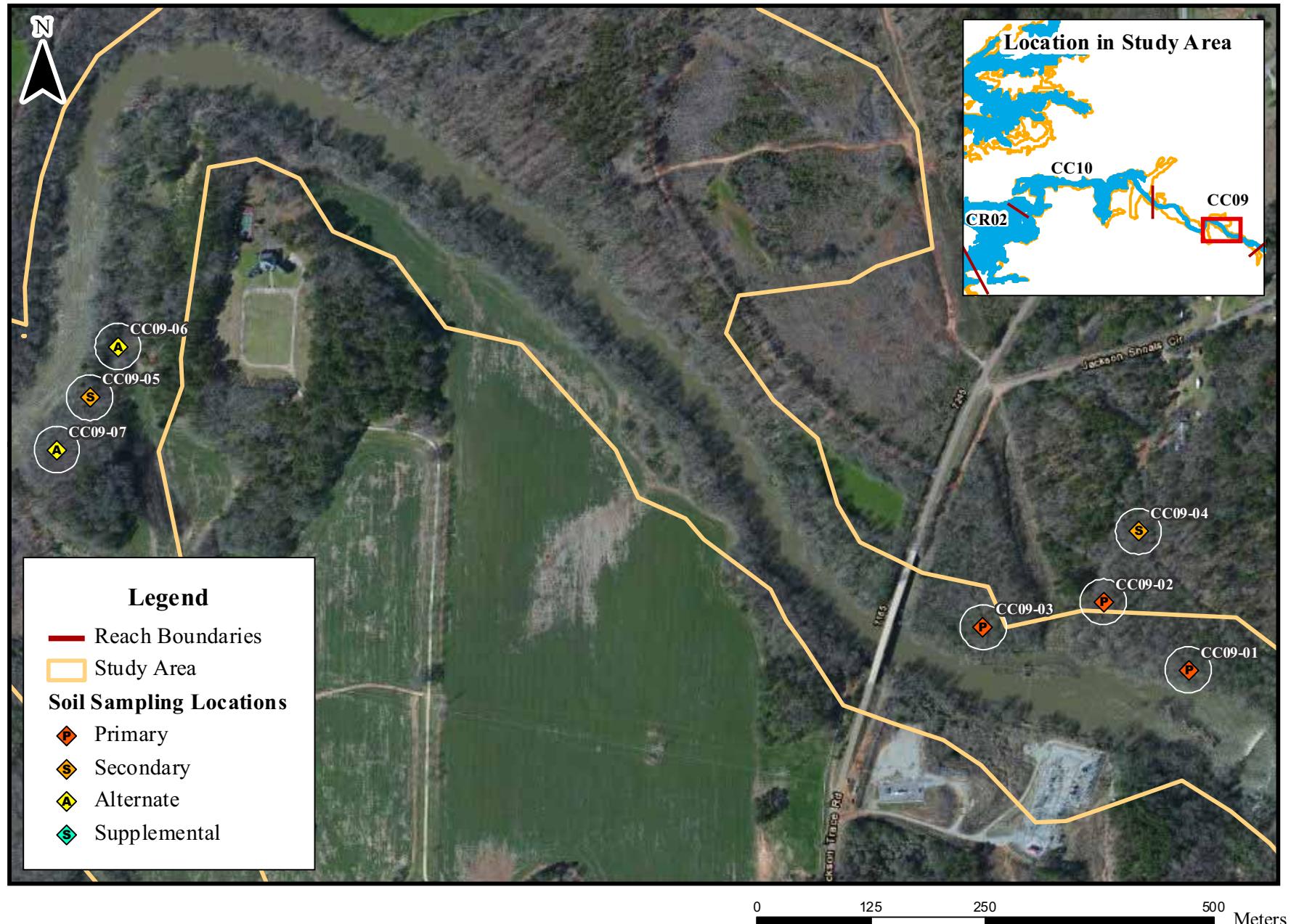


Figure 31. Locations of soil sampling stations in Reach CC10 (upstream portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

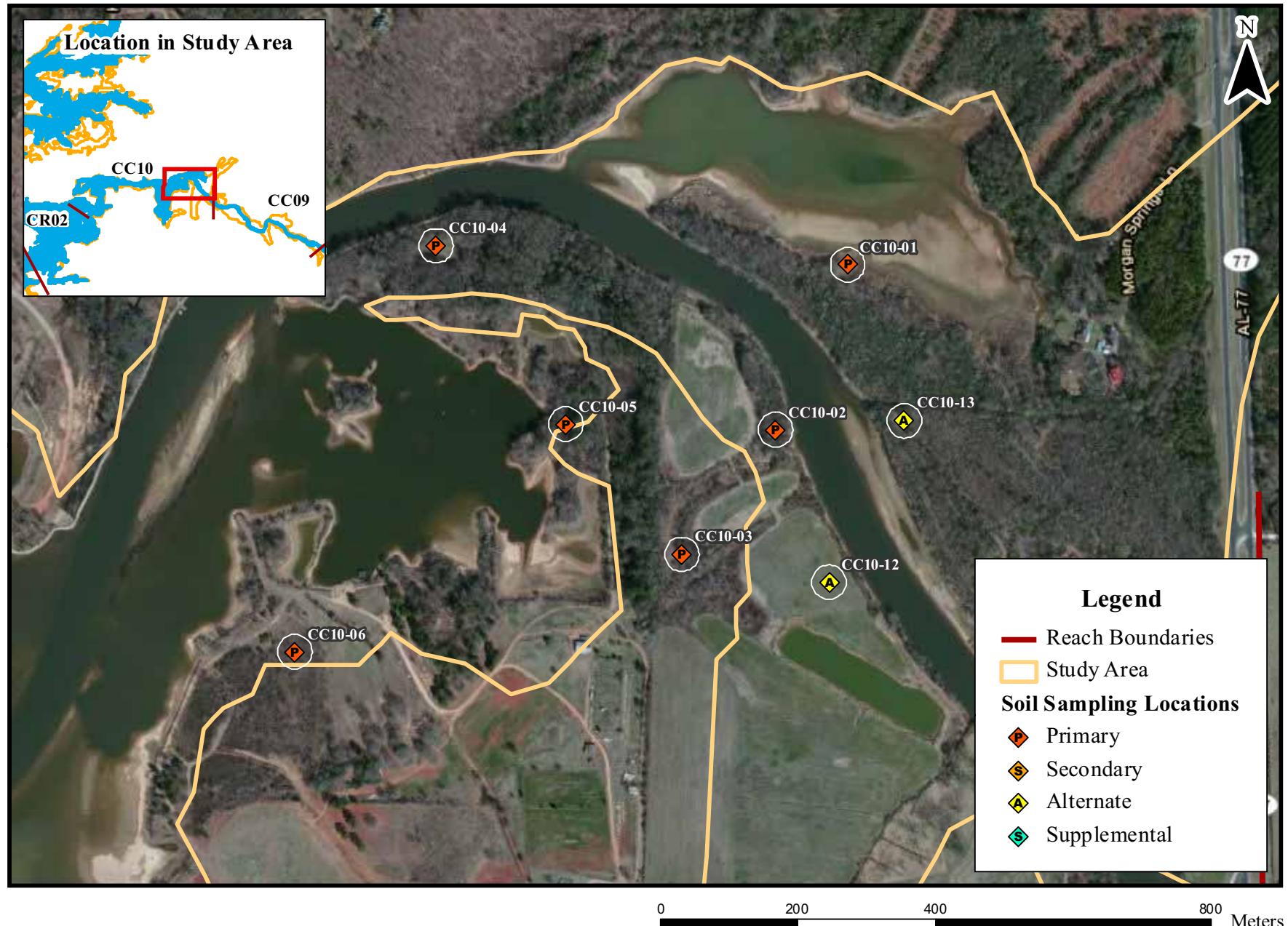


Figure 32. Locations of soil sampling stations in Reach CC10 (southern portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

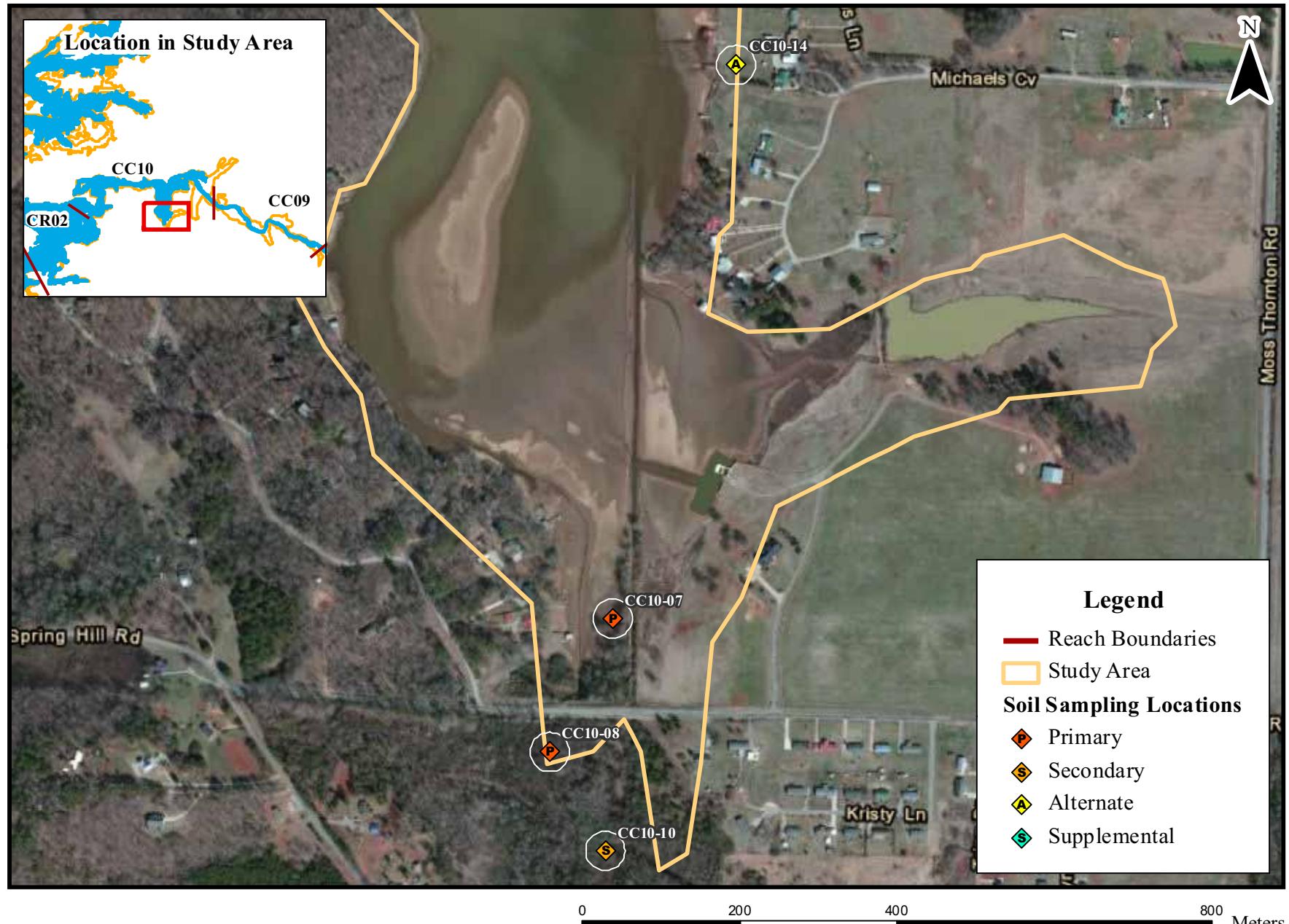


Figure 33. Locations of soil sampling stations in Reach CC10 (downstream portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

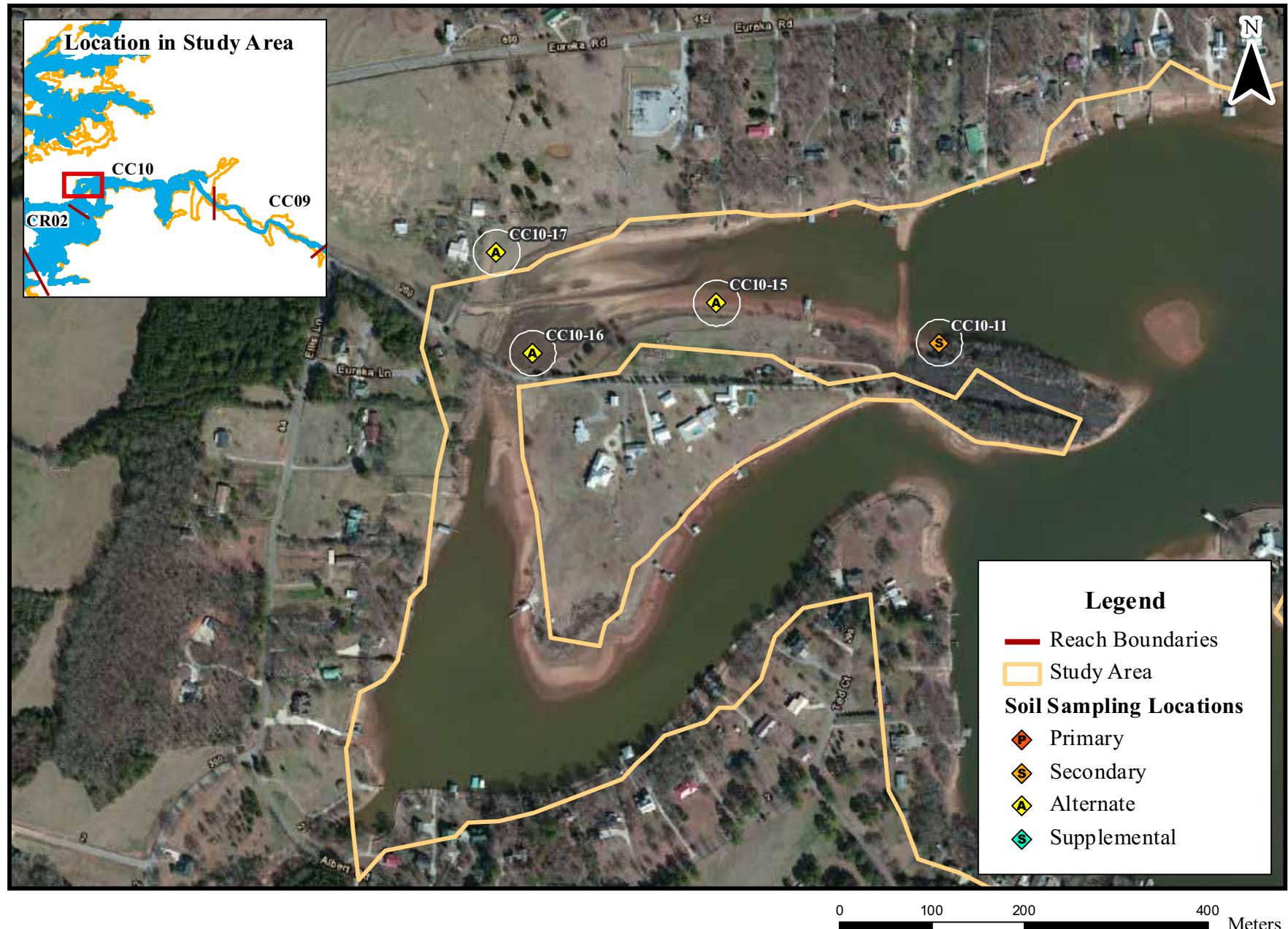


Figure 34. Locations of soil sampling stations in Reaches CC10 and CR02 of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).



Figure 35. Locations of soil sampling stations in Reach CR 02 (southern portion) of the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).

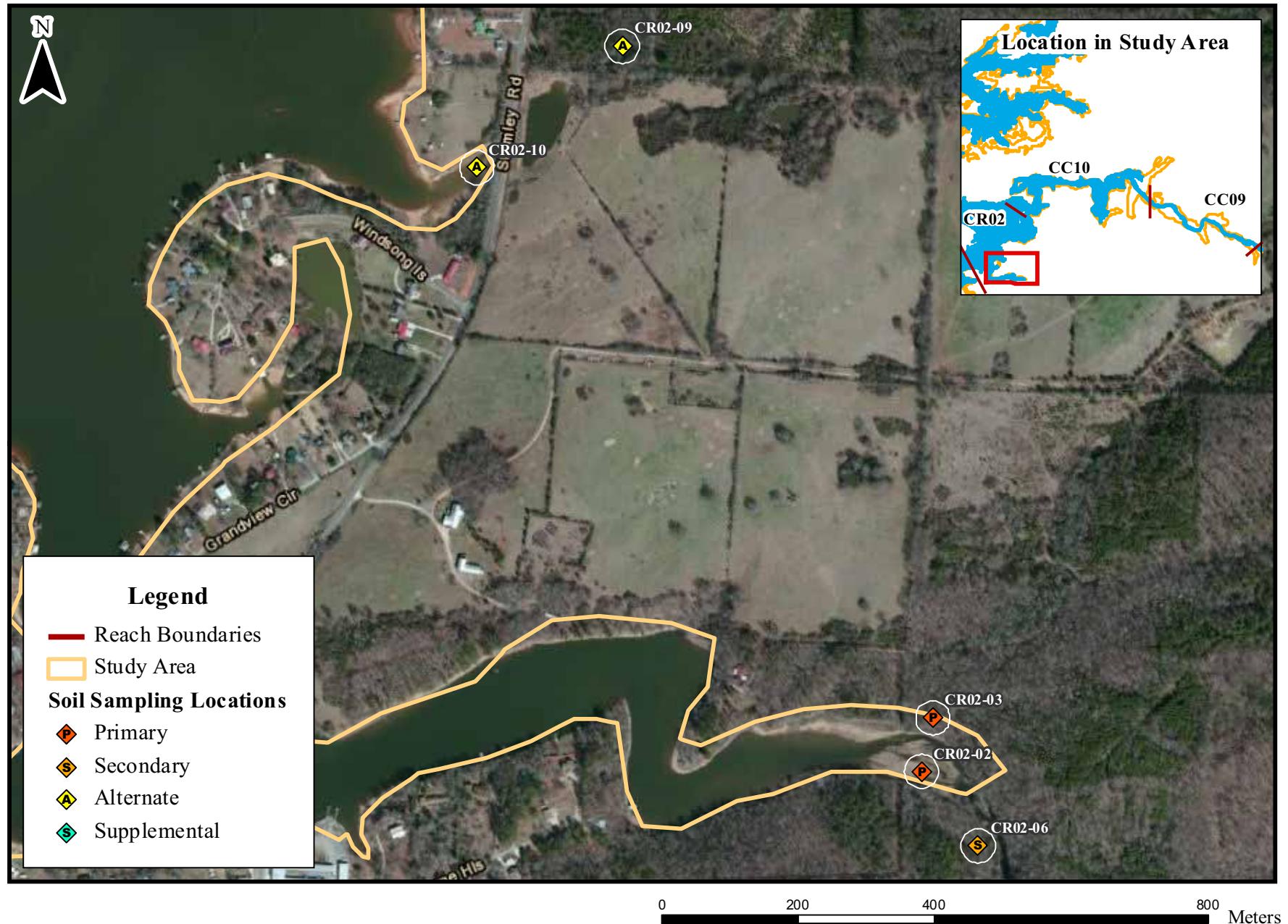
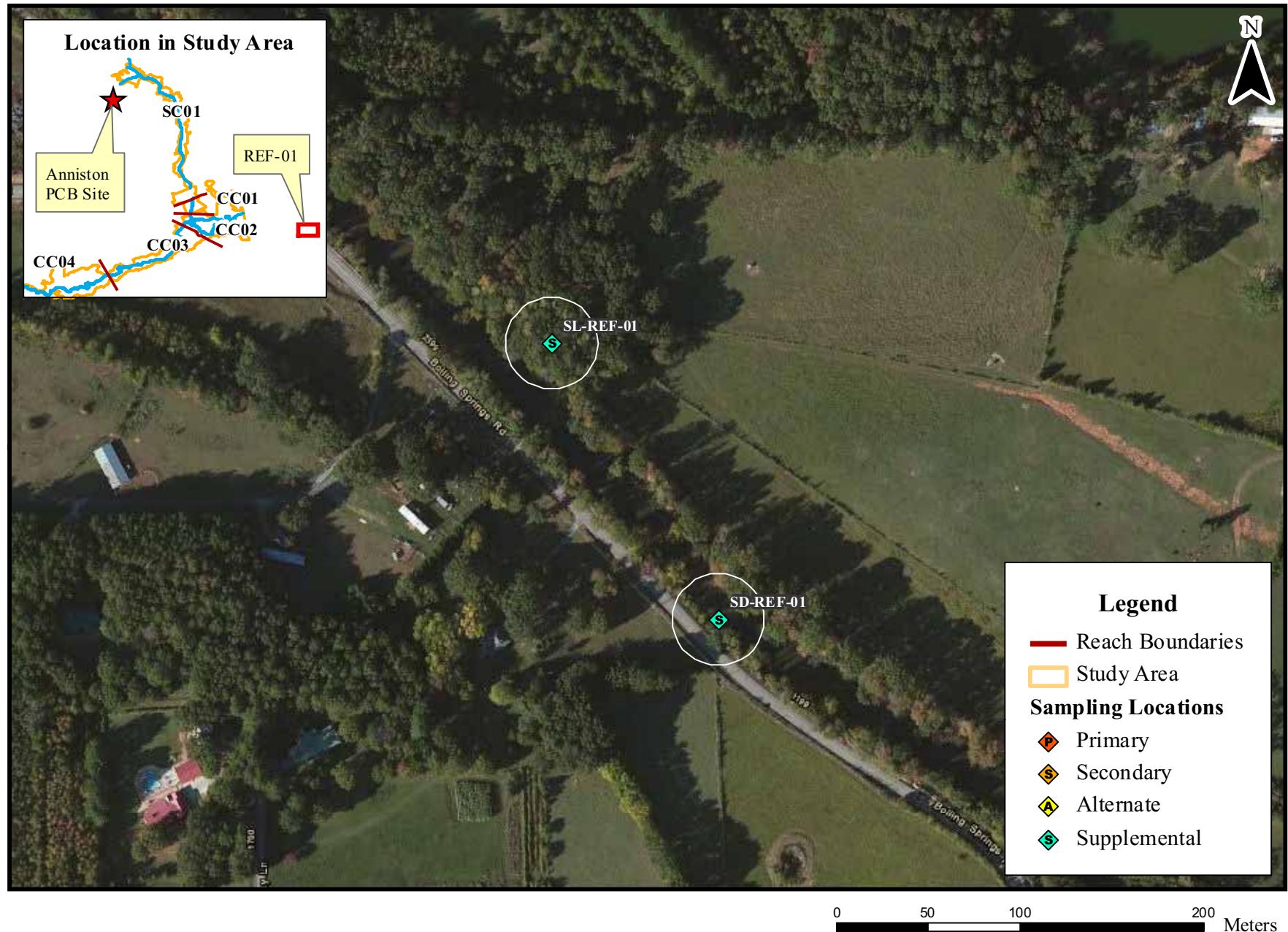


Figure 36. Locations of supplementary sediment and soil sampling stations located outside the study area (the 25 meter buffer within which soil samples will be collected during the 2013 sampling program is also shown).



Addenda

Addendum 1. Contact List for Field Personnel¹

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Addendum 2. Example Personnel Schedule for the Field Program

Team	Name	Mon	Tue	Wed	Thu	Fri	Sat	Sun	Mon	Tue	Wed	Thu	Fri	Sat
<i>Sediment Sampling Crew</i>														
Ngcf gt'3	F qp'O ceF qpcrf **O GUN+													
Ngcf gt'4														
Etgy "3														
Etgy "4														
Cngtpcvg"3														
Cngtpcvg"4														
<i>Soil Sampling Crew</i>														
Ngcf gt'3	Y cttgp"Nqtgpv\ **WUCEG+													
Ngcf gt'4														
Etgy "3														
Etgy "4														
Cngtpcvg"3														
Cngtpcvg"4														
<i>Sample Processing / Logistics Team</i>														
Ngcf gt'3														
Ngcf gt'4														
Etgy "3	*J gcmj "cpf "Uchgv\ "Qhlkgt+													
Etgy "4	*S C"Qhlkgt+													
Cngtpcvg"3														
Cngtpcvg"4														

Z"? "lwmnf c{=" "? "j cihf c{0

Addendum 3**Sample Data Collection Form for the Anniston PCB Site****Station:****General Information**

Names of sampling personnel			
Sampling date and time	Date:	Start Time:	End Time:
Weather conditions			
Precipitation			
Wind speed and direction			
Notes			

Location Information

Station ID	<input type="checkbox"/> Primary <input type="checkbox"/> Secondary <input type="checkbox"/> Alternate <input type="checkbox"/> Reference		
County / Land Parcel	<input type="checkbox"/> Calhoun	<input type="checkbox"/> Talladega	PPIN: _____ Landowner: _____
Target coordinates (UTM Zone 16N)	Easting:	Northing:	
Target coordinates (Decimal Degrees)	Longitude:	Latitude:	
Actual coordinates	Easting/Long:	Northing/Lat:	GPS Error (+/- m):
Hydrologic Condition	<input type="checkbox"/> Dry	<input type="checkbox"/> Saturated	<input type="checkbox"/> Overlying Water (Depth: _____ Velocity: _____)
Site location photo(s)	<input type="checkbox"/> Photo(s) taken Photo name(s): _____		
Location notes (including fauna observed in the vicinity of the station)			

Sample Information

Sample ID	<input type="checkbox"/> Sediment Sample <input type="checkbox"/> Soil Sample <input type="checkbox"/> Field Replicate				
Sample depth (surface to bottom of sampler)	Target:	Actual:	<input type="checkbox"/> cm <input type="checkbox"/> Inches		
Type of sampler used	<input type="checkbox"/> Bulb Transplanter	<input type="checkbox"/> Besser Sampler	<input type="checkbox"/> Ekman/Ponar	<input type="checkbox"/> Other (specify): _____	
Sample volume collected	Target:	Actual (est.):	<input type="checkbox"/> Liter <input type="checkbox"/> Gallon		
Number of grabs collected	Number of Grabs:	<input type="checkbox"/> Composite Sample Prepared			
Sieve size, method, and description of matter retained on the screen					
Sieve material photo(s)	<input type="checkbox"/> Photo(s) taken Photo name(s): _____				
Substrate type (Surface)	<input type="checkbox"/> Sand (< 0.1")	<input type="checkbox"/> Silt	<input type="checkbox"/> Bedrock	<input type="checkbox"/> Gravel (0.1" - 2.5")	<input type="checkbox"/> Cobble
	<input type="checkbox"/> Clay (Slick)	<input type="checkbox"/> Boulders	<input type="checkbox"/> Detritus	<input type="checkbox"/> Other: _____	
Sample color					
Sample odor (if readily apparent)	<input type="checkbox"/> None	<input type="checkbox"/> Petroleum	<input type="checkbox"/> Hydrogen Sulphide	<input type="checkbox"/> Sewage	<input type="checkbox"/> Other: _____
Description of surface biology					
Sample photo(s)	<input type="checkbox"/> Photo(s) taken Photo name(s): _____				
Notes (including problems encountered and unusual events during sampling)	<input type="checkbox"/> Completion of COC paperwork				

Additional Notes



Designation: E 1391 – 03

Standard Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing and for Selection of Samplers Used to Collect Benthic Invertebrates¹

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*

1.1 This guide covers procedures for obtaining, storing, characterizing, and manipulating marine, estuarine, and freshwater sediments, for use in laboratory sediment toxicity evaluations and describes samplers that can be used to collect sediment and benthic invertebrates (Annex A1). This standard is not meant to provide detailed guidance for all aspects of sediment assessments, such as chemical analyses or monitoring, geophysical characterization, or extractable phase and fractionation analyses. However, some of this information might have applications for some of these activities. A variety of methods are reviewed in this guide. A statement on the consensus approach then follows this review of the methods. This consensus approach has been included in order to foster consistency among studies. It is anticipated that recommended methods and this guide will be updated routinely to reflect progress in our understanding of sediments and how to best study them. This version of the standard is based primarily on a document developed by USEPA (2001 (1))² and by Environment Canada (1994 (2)) as well as an earlier version of this standard.

1.2 Protecting sediment quality is an important part of restoring and maintaining the biological integrity of our natural resources as well as protecting aquatic life, wildlife, and human health. Sediment is an integral component of aquatic ecosystems, providing habitat, feeding, spawning, and rearing areas for many aquatic organisms (MacDonald and Ingersoll 2002a,b (3)(4)). Sediment also serves as a reservoir for contaminants in sediment and therefore a potential source of contaminants to the water column, organisms, and ultimately human consumers of those organisms. These contaminants can arise from a number of sources, including municipal and industrial discharges, urban and agricultural runoff, atmospheric deposition, and port operations.

¹ This guide is under the jurisdiction of ASTM Committee E47 on Biological Effects and Environmental Fate and is the direct responsibility of Subcommittee E47.03 on Sediment Toxicology.

Current edition approved Nov. 10, 2003. Published January 2004. Originally published as E 1391 – 90. Last previous edition E 1391 – 02.

² The boldface numbers in parentheses refer to the list of references at the end of this standard.

1.3 Contaminated sediment can cause lethal and sublethal effects in benthic (sediment-dwelling) and other sediment-associated organisms. In addition, natural and human disturbances can release contaminants to the overlying water, where pelagic (water column) organisms can be exposed. Sediment-associated contaminants can reduce or eliminate species of recreational, commercial, or ecological importance, either through direct effects or by affecting the food supply that sustainable populations require. Furthermore, some contaminants in sediment can bioaccumulate through the food chain and pose health risks to wildlife and human consumers even when sediment-dwelling organisms are not themselves impacted (Test Method 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:

	Section
Scope	1
Referenced Documents	2
Terminology	3
Summary of Guide	4
Significance and Use	5
Interferences	6
Apparatus	7
Safety Hazards	8
Sediment Monitoring and Assessment Plans	9
Collection of Whole Sediment Samples	10
Field Sample Processing, Transport, and Storage of Sediments	11
Sample Manipulations	12
Collection of Interstitial Water	13
Physico-chemical Characterization of Sediment Samples	14
Quality Assurance	15
Report	16
Keywords	17
Description of Samplers Used to Collect Sediment or Benthic Invertebrates	Annex A1

1.6 Field-collected sediments might contain potentially toxic materials and should thus be treated with caution to minimize occupational exposure to workers. Worker safety must also be considered when working with spiked sediments

*A Summary of Changes section appears at the end of this standard.

containing various organic, inorganic, or radiolabeled contaminants, or some combination thereof. Careful consideration should be given to those chemicals that might biodegrade, volatilize, oxidize, or photolyze during the exposure.

1.7 The values stated in either SI or inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.* Specific hazards statements are given in Section 8.

2. Referenced Documents

2.1 ASTM Standards:³

- D 1067 Test Methods for Acidity And Alkalinity of Water
- D 1126 Test Method for Hardness in Water
- D 1129 Terminology Relating to Water
- D 1426 Test Methods for Ammonia Nitrogen in Water
- D 3976 Practice for Preparation of Sediment Samples for Chemical Analysis
- D 4387 Classification of 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 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)
- E 729 Guide for Conducting Acute Toxicity Tests 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 1367 Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Invertebrates
- E 1525 Guide for Designing Biological Tests with Sediment
- E 1611 Guide for Conducting Sediment Toxicity Tests with Marine And Estuarine Polychaetous Annelids
- E 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

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 Practice E 380.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *site, n*—a study area comprised of multiple sampling station.

3.2.2 *station, n*—a location within a site where physical, chemical, or biological sampling or testing is performed.

4. Summary of Guide

4.1 This guide provides a review of widely used methods for collecting, storing, characterizing, and manipulating sediments for toxicity or bioaccumulation testing and also describes samplers that can be used to collect benthic invertebrates. Where the science permits, recommendations are provided on which procedures are appropriate, while identifying their limitations. This guide addresses the following general topics: (1) Sediment monitoring and assessment plans (including developing a study plan and a sampling plan), (2) Collection of whole sediment samples (including a description of various sampling equipment), (3) Processing, transport and storage of sediments, (4) Sample manipulations (including sieving, formulated sediments, spiking, sediment dilutions, and preparation of elutriate samples), (5) Collection of interstitial water (including sampling sediments in situ and ex situ), (6) Physico-chemical characterizations of sediment samples, (7) Quality assurance, and (8) Samplers that can be used to collect sediment or benthic invertebrates.

5. Significance and Use

5.1 Sediment toxicity evaluations are a critical component of environmental quality and ecosystem impact assessments, and are used to meet a variety of research and regulatory 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.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

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 physico-chemical 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 physico-chemical characterization and biological testing, using procedures that are most likely to maintain in situ conditions, most accurately represent the sediment in question, or satisfy particular needs, to help generate consistent, high quality data collection.

5.5 This standard is intended to provide technical support to those who design or perform sediment quality studies under a variety of regulatory and non-regulatory programs. Information is provided concerning general sampling design considerations, field and laboratory facilities needed, safety, sampling equipment, sample storage and transport procedures, and sample manipulation issues common to chemical or toxicological analyses. Information contained in this standard reflects the knowledge and experience of several internationally-known sources including the Puget Sound Estuary Program (PSEP), Washington State Department of Ecology (WDE), United States Environmental Protection Agency (USEPA), US Army Corps of Engineers (USACE), National Oceanic and Atmospheric Administration (NOAA), and Environment Canada. This standard attempts to present a coherent set of recommendations on field sampling techniques and sediment or interstitial water sample processing based on the above sources, as well as extensive information in the peer-reviewed literature.

5.6 As the scope of this standard is broad, it is impossible to adequately present detailed information on every aspect of sediment sampling and processing for all situations. Nor is such detailed guidance warranted because much of this information (for example, how to operate a particular sampling device or how to use a Geographical Positioning System (GPS) device) already exists in other published materials referenced in this standard.

5.7 Given the above constraints, this standard: (1) presents a discussion of activities involved in sediment sampling and sample processing; (2) alerts the user to important issues that should be considered within each activity; and (3) gives recommendations on how to best address the issues raised such that appropriate samples are collected and analyzed. An attempt is made to alert the user to different considerations pertaining to sampling and sample processing depending on the objectives of the study (for example, remediation, dredged material evaluations or status and trends monitoring).

5.8 The organization of this standard reflects the desire to give field personnel and managers a useful tool for choosing

appropriate sampling locations, characterize those locations, collect and store samples, and manipulate those samples for analyses. Each section of this standard is written so that the reader can obtain information on only one activity or set of activities (for example, subsampling or sample processing), if desired, without necessarily reading the entire standard. Many sections are cross-referenced so that the reader is alerted to relevant issues that might be covered elsewhere in the standard. This is particularly important for certain chemical or toxicological applications in which appropriate sample processing or laboratory procedures are associated with specific field sampling procedures.

5.9 The methods contained in this standard are widely applicable to any entity wishing to collect consistent, high quality sediment data. This standard does not provide guidance on how to implement any specific regulatory requirement, or design a particular sediment quality assessment, but rather it is a compilation of technical methods on how to best collect environmental samples that most appropriately address common sampling objectives.

5.10 The information presented in this standard should not be viewed as the final statement on all the recommended procedures. Many of the topics addressed in this standard (for example, sediment holding time, formulated sediment composition, interstitial water collection and processing) are the subject of ongoing research. As data from sediment monitoring and research becomes available in the future, this standard will be updated as necessary.

6. Interferences

6.1 Maintaining the integrity of a sediment sample relative to ambient environmental conditions during its removal, transport, and testing in the laboratory is extremely difficult. The sediment environment is composed of a myriad of microenvironments, redox gradients, and other interacting physicochemical and biological processes. Many of these characteristics influence sediment toxicity and bioavailability to benthic and planktonic organisms, microbial degradation, and chemical sorption. Any disruption of this environment complicates interpretations of treatment effects, causative factors, and in situ comparisons. Individual sections address specific interferences.

7. Apparatus

7.1 A variety of sampling, characterization, and manipulation methods exist using different equipment. These are reviewed in Sections 10-14.

7.2 *Cleaning*—Equipment used to collect and store sediment samples, equipment used to collect benthic invertebrate samples, equipment used to prepare and store water and stock solutions, and equipment used to expose test organisms should be cleaned before use. All non-disposable sample containers, test chambers, and other equipment that have come in contact with sediment should be washed after use in the manner described as follows to remove surface contaminants (Test Method 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) the appointment of a laboratory health and safety officer with the responsibility and authority to develop and maintain a safety program, (2) the preparation of a formal, written health and safety plan, which is provided to each laboratory staff member, (3) an ongoing training program on laboratory safety, and (4) regular safety inspections.

8.1.2 Collection and use of sediments may involve substantial risks to personal safety and health. Chemicals in field-collected sediment may include carcinogens, mutagens, and other potentially toxic compounds. Inasmuch as sediment testing is often started before chemical analyses can be completed, worker contact with sediment needs to be minimized by: (1) using gloves, laboratory coats, safety glasses, face shields, and respirators as appropriate, (2) manipulating sediments under a ventilated hood or in an enclosed glove box, and (3) enclosing and ventilating the exposure system. Personnel collecting sediment samples and conducting tests should take all safety precautions necessary for the prevention of bodily injury and illness that might result from ingestion or invasion of infectious agents, inhalation or absorption of corrosive or toxic substances through skin contact, and asphyxiation because of lack of oxygen or presence of noxious gases.

8.1.3 Before beginning sample collection and laboratory work, personnel should determine that all required safety equipment and materials have been obtained and are in good condition.

8.2 Safety Equipment:

8.2.1 *Personal Safety Gear*—Personnel should use safety equipment, such as rubber aprons, laboratory coats, respirators, gloves, safety glasses, face shields, hard hats, safety shoes, water-proof clothing, personal floatation devices, and safety harnesses.

8.2.2 *Laboratory Safety Equipment*—Each laboratory should be provided with safety equipment such as first-aid kits, fire extinguishers, fire blankets, emergency showers, and eye wash stations. Mobile laboratories should be equipped with a telephone to enable personnel to summon help in case of emergency.

8.3 General Laboratory and Field Operations:

8.3.1 Special handling and precautionary guidance in Material Safety Data Sheets (MSDS) should be followed for reagents and other chemicals purchased from supply houses.

8.3.2 Work with some sediments may require compliance with rules pertaining to the handling of hazardous materials. Personnel collecting samples and performing tests should not work alone.

8.3.3 It is advisable to wash exposed parts of the body with bactericidal soap and water immediately after collecting or manipulating sediment samples.

8.3.4 Strong acids and volatile organic solvents should be used in a fume hood or under an exhaust canopy over the work area.

8.3.5 An acidic solution should not be mixed with a hypochlorite solution because hazardous fumes might be produced.

8.3.6 To prepare dilute acid solutions, concentrated acid should be added to water, not vice versa. Opening a bottle of concentrated acid and adding concentrated acid to water should be performed only under a fume hood.

8.3.7 Use of ground-fault systems and leak detectors is strongly recommended to help prevent electrical shocks. Electrical equipment or extension cords not bearing the approval of Underwriter Laboratories should not be used. Ground-fault interrupters should be installed in all "wet" laboratories where electrical equipment is used.

8.3.8 All containers should be adequately labeled to indicate their contents.

8.3.9 A clean and well-organized work place contributes to safety and reliable results.

8.4 *Disease Prevention*—Personnel handling samples which are known or suspected to contain human wastes should be immunized against hepatitis B, tetanus, typhoid fever, and polio. Thorough washing of exposed skin with bacterial soap should follow handling of samples collected from the field.

8.5 *Safety Manuals*—For further guidance on safe practices when handling sediment samples and conducting toxicity tests, check with the permittee and consult general industrial safety manuals including (7),(8).

8.6 *Pollution Prevention, Waste Management, and Sample Disposal*—Guidelines for the handling and disposal of hazardous materials should be strictly followed (Guide 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); 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

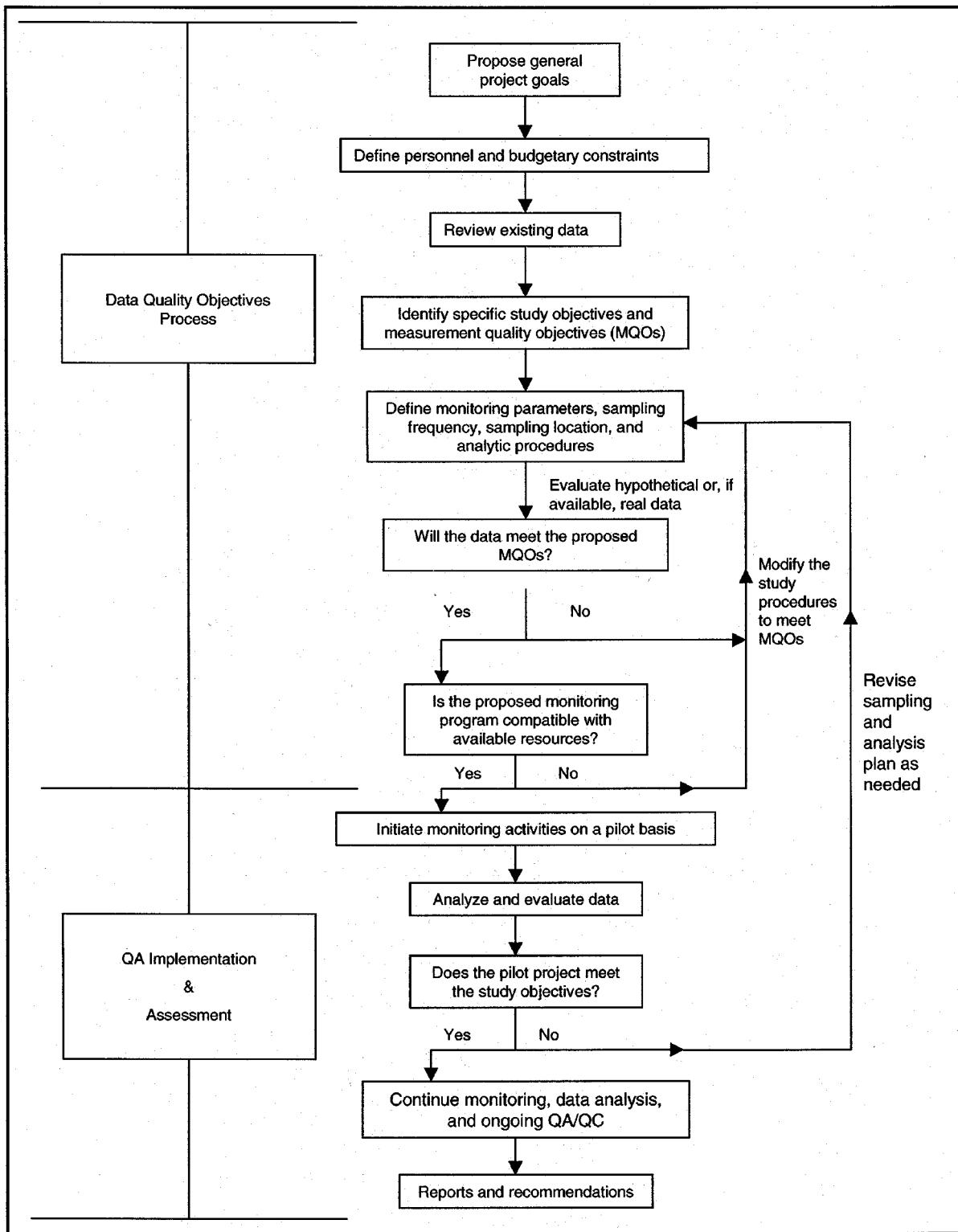


FIG. 1 Flow Chart Summarizing the Process that Should Be Implemented in Designing and Performing a Monitoring Study
 (modified from MacDonald et al. (1991 (10)); USEPA 2001 (1))

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 com-

piled in the DQO process is used to develop a project-specific Quality Assurance Project Plan (QAPP; Section 10, USEPA 2000a (12)) that should be used to plan the majority of sediment quality monitoring or assessment studies. In some

instances, a QAPP may be prepared, as necessary, on a project-by-project basis.

9.3.2 The DQO process addresses the uses of the data (most importantly, the decision(s) to be made) and other factors that will influence the type and amount of data to be collected (for example, the problem being addressed, existing information, information needed before a decision can be made, and available resources). From these factors the qualitative and quantitative data needs are determined Fig. 2. DQOs are qualitative and quantitative statements that clarify the purpose of the monitoring study, define the most appropriate type of data to collect, and determine the most appropriate methods and conditions under which to collect them. The products of the DQO process are criteria for data quality, and a data collection design to ensure that data will meet the criteria.

9.3.3 For most instances, a Sampling and Analysis Plan (SAP) is developed before sampling that describes the study objectives, sampling design and procedures, and other aspects of the DQO process outlined above (USEPA 2001(1)). The following sections provide guidance on many of the primary issues that should be addressed in a study plan.

9.4 Study Plan Considerations:

9.4.1 Definition of the Study Area and Study Site:

9.4.1.1 Monitoring and assessment studies are performed for a variety of reasons (ITFM, 1995 (13)) and sediment assessment studies can serve many different purposes. Developing an appropriate sampling plan is one of the most important steps in monitoring and assessment studies. The sampling plan, including definition of the site (a study area that can be comprised of multiple sampling stations) and sampling

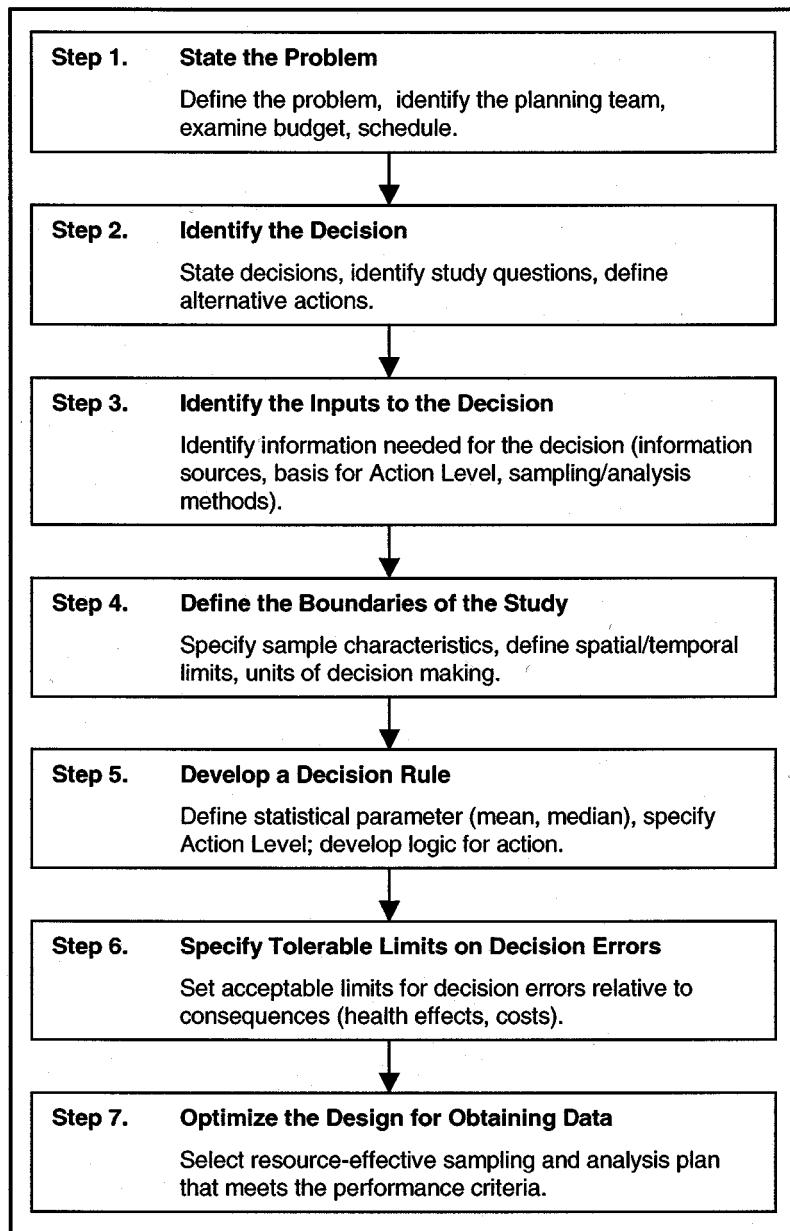


FIG. 2 Flow Chart Summarizing the Data Quality Objectives Process (after USEPA 2000a (12); 2001 (1))

design, will be a product of the general study objectives Fig. 1. Station location, selection, and sampling methods will necessarily follow from the study design. Ultimately, the study plan should control extraneous sources of variability or error to the extent possible so that data are appropriately representative of the sediment quality, and fulfill the study objectives.

9.4.1.2 The study area refers to the body of water that contains the study sampling stations(s) to be monitored or assessed, as well as adjacent areas (land or water) that might affect or influence the conditions of the study site. The study site refers to the body of water and associated sediments to be monitored or assessed.

9.4.1.3 The size of the study area will influence the type of sampling design (see 9.5) and site positioning methods that are appropriate (see 9.8). The boundaries of the study area need to be clearly defined at the outset and should be outlined on a hydrographic chart or topographic map.

9.4.2 Controlling Sources of Variability:

9.4.2.1 A key factor in effectively designing a sediment quality study is controlling those sources of variability in which one is not interested (USEPA 2000a,b (12),(14)). There are two major sources of variability that, with proper planning, can be minimized, or at least accounted for, in the design process. In statistical terms, the two sources of variability are sampling error and measurement error (USEPA 2000b (14); Solomon et al. 1997 (15)).

9.4.2.2 Sampling error is the error attributable to selecting a certain sampling station that might not be representative of the site or population of sample units. Sampling error is controlled by either: (1) using unbiased methods to select stations if one is performing general monitoring of a given site (USEPA, 2000b (14)) or (2) selecting several stations along a spatial gradient if a specific location is being targeted (see 9.5).

9.4.2.3 Measurement error is the degree to which the investigator accurately characterizes the sampling unit or station. Thus, measurement error includes components of natural spatial and temporal variability within the sample unit as well as actual errors of omission or commission by the investigator. Measurement error is controlled by using consistent and comparable methods. To help minimize measurement error, each station should be sampled in the same way within a site, using a consistent set of procedures and in the same time frame to minimize confounding sources of variability (see 9.4.3). In analytical laboratory or toxicity procedures, measurement error is estimated by duplicate determinations on some subset of samples (but not necessarily all). Similarly, in field investigations, some subset of sample units (for example, 10 % of the stations) should be measured more than once to estimate measurement error (see Replicate and Composite Samples, 9.6.7). Measurement error can be reduced by analyzing multiple observations at each station (for example, multiple grab samples at each sampling station, multiple observations during a season), or by collecting depth-integrated, or spatially integrated (composite) samples (see 9.6.7).

9.4.2.4 Optimizing the sampling design requires consideration of tradeoffs among the procedures used to analyze data. These include, the effect that is considered meaningful, desired power, desired confidence, and resources available for the

sampling program (Test Method 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); Barbour et al. 1999 (19)). This type of sampling might be especially advantageous for characterizing sediment toxicity, sediment chemistry, and benthic macroinvertebrate and other biological assemblages (USEPA, 2000c (20)). In addition, this approach is useful if sediment contamination is related to, or being separated from, high flow events or if influenced by tidal cycles. By sampling overlying waters during both low and high flow conditions or tidal cycles, the relative contribution of each to contaminant can be better assessed, thereby better directing remedial activities, or other watershed improvements.

9.4.3.2 Projects that sample the same station over multiple years are interested in obtaining comparable data with which they can assess changes over time, or following remediation (GLNPO, 1994 (11)). In these cases, index period sampling is especially useful because hydrological regime (and therefore biological processes) is likely to be more similar between similar seasons than among different seasons.

9.5 Sampling Designs:

9.5.1 As mentioned in earlier sections, the type of sampling design used is a function of the study DQOs and more specifically, the types of questions to be answered by the study. A summary of various sampling designs is presented in Fig. 3. Generally, sampling designs fall into two major categories: random (or probabilistic) and targeted (USEPA, 2000b (14)). USEPA (2000b,c (14),(20)) Gilbert (1987 (21)), and Wolfe et al. (1993 (22)) present discussions of sampling design issues and information on different sampling designs. Appendix A in USEPA (2001, (1)) presents hypothetical examples of sediment quality monitoring designs given different objectives or regulatory applications.

9.5.2 Probabilistic and Random Sampling:

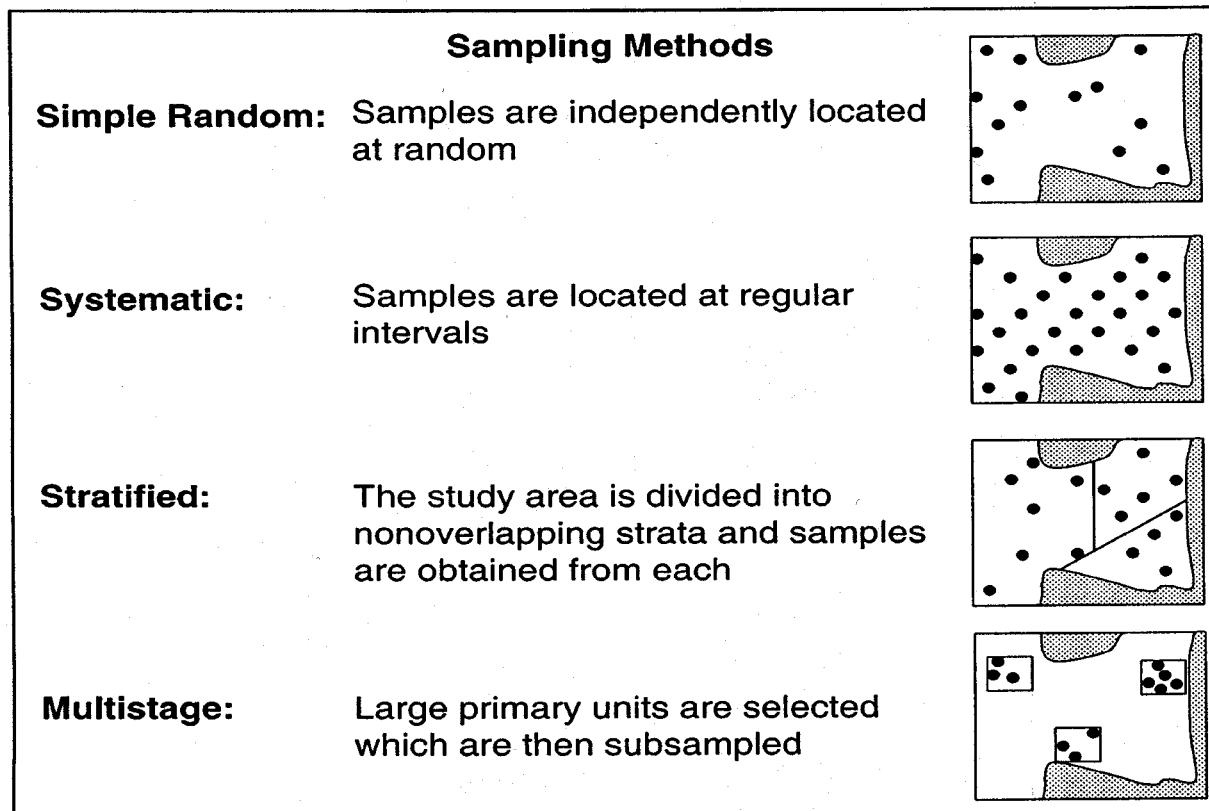


FIG. 3 Description of Various Sampling Methods (adapted from USEPA 2000c (20); 2001(1))

9.5.2.1 Probability-based or random sampling designs avoid bias in the sample results by randomly assigning and selecting sampling locations. A probability design requires that all sampling units have a known probability of being selected. Both the USPEA Environmental Monitoring Assessment Program and the NOAA National Status and Trends Program use a probabilistic sampling design to infer regional and national patterns with respect to contamination or biological effects.

9.5.2.2 Stations can be selected on the basis of a truly random scheme or in a systematic way (for example, sample every 10 m along a randomly chosen transect). In simple random sampling, all sampling units have an equal probability of selection. This design is appropriate for estimating means and totals of environmental variables if the population is homogeneous. To apply simple random sampling, it is necessary to identify all potential sampling times or locations, then randomly select individual times or locations for sampling.

9.5.2.3 In grid or systematic sampling, the first sampling location is chosen randomly and all subsequent stations are placed at regular intervals (for example, 50 m apart) throughout the study area. Clearly, the number of sampling locations could be large if the study area is large and one desires "fine-grained" contaminant or toxicological information. Thus, depending on the types of analyses desired, such sampling might become expensive unless the study area is relatively small, or the density of stations (that is, how closely spaced are the stations) is relatively low. Grid sampling might be effective for detecting previously unknown "hot spots" in a limited study area.

9.5.2.4 In stratified designs, the selection probabilities might differ among strata. Stratified random sampling consists of dividing the target population into non-overlapping parts or subregions (for example, ecoregions, watersheds, or specific dredging or remediation sites) termed strata to obtain a better estimate of the mean or total for the entire population. The information required to delineate the strata and to estimate sampling frequency should either be known before sampling using historic data variability, available information and knowledge of ecological function, or obtained in a pilot study. Sampling locations are randomly selected from within each of the strata. Stratified random sampling is often used in sediment quality monitoring because certain environmental variables can vary by time of day, season, hydrodynamics, or other factors. One disadvantage of using random designs is the possibility of encountering unsampleable stations that were randomly selected by the computer. Such problems result in the need to reposition the vessel to an alternate location (Heimbuch et al. 1995 (23), Strobel et al. 1995 (24)). Furthermore, if one is sampling to determine the percent spatial extent of degradation, it might be important to sample beyond the boundaries of the study area to better evaluate the limits of the impacted area.

9.5.2.5 A related design is multistage sampling in which large subareas within the study area are first selected (usually on the basis of professional knowledge or previously collected information). Stations are then randomly located within each subarea to yield average or pooled estimates of the variables of interest (for example, concentration of a particular contaminant or acute toxicity to the amphipod *Hyalella azteca*) for each

subarea. This type of sampling is especially useful for statistically comparing variables among specific parts of a study area.

9.5.2.6 Use of random sampling designs might also miss relationships among variables, especially if there is a relationship between an explanatory and a response variable. As an example, estimation of benthic response or contaminant concentration, in relation to a discharge or landfill leachate stream, requires sampling targeted locations or stations around the potential contaminant source, including stations presumably unaffected by the source (for example, Warwick and Clarke, 1991 (25)). A simple random selection of stations is not likely to capture the entire range needed because most stations would likely be relatively removed from the location of interest.

9.5.3 Targeted Sampling Designs:

9.5.3.1 In targeted (also referred to as judgmental, or model-based) designs, stations are selected based on prior knowledge of other factors, such as salinity, substrate type, and construction or engineering considerations (for example, dredging). The sediment studies conducted in the Clark Fork River (Pascoe and DalSoglio, 1994 (26); Brumbaugh et al. 1994 (27)), in which contaminated areas were a focus, used a targeted sampling design.

9.5.3.2 Targeted designs are useful if the objective of the investigation is to screen an area(s) for the presence or absence of contamination at levels of concern, such as risk-based screening levels, or to compare specific sediment quality against reference conditions or biological guidelines. In general, targeted sampling is appropriate for situations in which any of the following apply (USEPA, 2000b (14)):

(1) The site boundaries are well defined or the site physically distinct (for example, USEPA Superfund or CER-CLA 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.

TABLE 1 Checklist for the DQO Process (USEPA 2001(1))

Clearly state the problem: purpose and objectives, available resources, members of the project team: For example, the purpose might be to evaluate current sediment quality conditions, historical conditions, evaluate remediation effects, or validate a sediment model. It is important to review and evaluate available historical data relevant to the study at this point in the process.

Identify the decision; the question(s) the study attempts to address: For example, is site A more toxic than site B?; Are sediments in Lake Y less toxic now than they used to be?; Does the sediment at site D need to be remediated? What point or nonpoint sources are contributing to sediment contamination?

Identify inputs to the decision: information and measurements that need to be obtained: For example, analyses of specific contaminants, toxicity test results, biological assessments, bioaccumulation data, habitat assessments, hydrology, and water quality characterization.

Define the study boundaries (spatial and temporal): Identify potential sources of contamination; determine the location of sediment deposition zones; determine the frequency of sampling and need for a seasonal sampling and/or sampling during a specific index period; consider areas of previous dredged or fill material discharges/disposal. Consideration of hydraulic patterns, flow event frequency, and/or sedimentation rates could be critical for determining sampling frequency and locations.

Develop a decision rule: define parameters of interest and determine the value of a parameter that would cause follow-up action of some kind: For example., exceedance of Sediment Quality Guidelines (Wenning and Ingersoll 2002 (6)) or toxicity effect results in some action. For example, in the Great Lakes Assessment and Remediation of Contaminated Sediments (ARCS) Program, one decision rule was: if total PCB concentration exceeds a particular action level, then the sediments will be classified as toxic and considered for remediation (GLNPO, 1994 (11)).

Specify limits on decision errors: Establish the measurement quality objectives (MQOs) which include determining the level of confidence required from the data; precision, bids, representativeness, and completeness of data; the sample size (weight or volume) required to satisfy the analytical methods and QA/QC program for all analytical tests; the number of samples required, to be within limits on decision errors, and compositing needed, if any.

Optimize the design: Choose appropriate sampling and processing methods; select appropriate method for determining the location of sampling stations; select an appropriate positioning method for the site and study. Consult historical data and a statistician before the study begins regarding the sampling design (i.e., the frequency, number, and location of field-collected samples) that will best satisfy study objectives.

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 Section 11). 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

TABLE 2 Conventional Sediment Variables and Their Use in Sediment Investigations (adapted from WDE, 1995(29) and USEPA 2001(1))

Conventional Sediment Variable	Use
Total organic carbon (TOC)	Normalization of the concentrations of nonionizable organic compounds Identification of appropriate reference sediments for biological tests
Acid Volatile Sulfide (AVS)	Normalization of the concentrations of divalent metals in anoxic sediments
Sediment grain size	Identification of appropriate reference sediments for biological tests Interpretation of sediment toxicity test data and benthic macroinvertebrate abundance data Evaluation of sediment transport and deposition Evaluation of remedial alternatives
Total solids	Expression of chemical concentrations on a dry-weight basis Interpretation of sediment toxicity test data
Ammonia	Interpretation of sediment toxicity test data
Total sulfides	Interpretation of sediment toxicity test data

TABLE 3 Typical Sediment Volume Requirements for Various Analyses per Sample (USEPA 2001(1))

Sediment Analysis	Minimum Sample Volume
Inorganic chemicals	90 mL
Non-petroleum organic chemicals	230 mL
Other chemical parameters (for example, total organic carbon, moisture content)	300 mL
Particle size	230 mL
Petroleum hydrocarbons ^A	250 to 1000 mL
Acute and chronic whole sediment toxicity tests ^B	1 to 2 L
Bioaccumulation tests ^C	15 L
Benthic macroinvertebrate assessments	8 to 16 L
Pore water extraction	2 L
Elutriate preparation	1 L

^A The maximum volume (1000 mL) is required only for oil and grease analysis; otherwise, 250 mL is sufficient.

^B Amount needed per whole sediment test (that is, one species) assuming 8 replicates per sample and test volumes specified in USEPA, 2000d (35).

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

TABLE 4 Recommendations on Determining How Many Samples and How Much Sample Volume Should Be Collected (USEPA 2001(1))

The testing laboratory should be consulted to confirm the amount of sediment required for all desired analyses.

The amount of sediment needed from a given site will depend on the number and types of analyses to be performed. If biological, toxicological, and chemical analyses are required (sediment triad approach), then at least 10 L of sediment might be required from each station.

Since sampling events might be expensive and/or difficult to replicate, it is useful to collect extra samples if possible, in the event of problems encountered by the analytical laboratories, failure of performance criteria in assays, or need to verify/validate results.

Consider compositing samples from a given station or across similar station types to reduce the number of samples needed.

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); 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 independent testing of *a posteriori* hypotheses that might arise from screening the initial data. However, storage of sediments might result in changes in bioavailability of chemical contaminants (see 11.6) or in exceeding analytical holding times. Therefore, follow-up testing of archived samples should be done cautiously.

9.6.7 Replicate and Composite Samples:

9.6.7.1 *Replicate samples:* As mentioned in the previous section, the number of samples collected and analyzed will always be a compromise between the desire of obtaining high quality data that fully addresses the overall project objectives (MQOs), and the constraints imposed by analytical costs, sampling effort, and study logistics. Therefore, each study needs to find a balance between obtaining information to satisfy the stated DQOs or study goals in a cost-effective manner, and yet have enough confidence in the data to make appropriate decisions (for example, remediation, dredging; Step 3 in the DQO process, Fig. 2). Two different concepts are used to satisfy this challenge: replication and sample compositing.

9.6.7.2 Replication is used to assess precision of a particular measure and can take many forms depending on the type of precision desired. For most studies, analytical replicates are the most frequently used form of replication because most MQOs

are concerned with analytical data quality (USEPA 2001(1)). The extent of analytical replication (duplicates) varies with the study DQOs. Performing duplicate analyses on at least 10 % of the samples collected is considered satisfactory for most studies (GLNPO, 1994 (11); USEPA/USACE, 1991(32); PSEP, 1997a (33); USEPA/USACE, 1998 (34)). An MQO of less than 20 to 30 % relative percent difference (RPD) is commonly used for analytical replicates depending on the analyte.

9.6.7.3 Field replicates can provide useful information on the spatial distribution of contaminants at a station and the heterogeneity of sediment quality within a site. Furthermore, field replicates provide true replication at a station (analytical replicates and split samples at a station provide a measure of precision for a given sample, not the station) and therefore can be used to statistically compare analyses (for example, toxicity, tissue concentration, whole sediment concentration) across stations.

9.6.7.4 Results of field replicate analysis yield the overall variability or precision of both the field and laboratory operations (as well as the variability between the replicate samples themselves, apart from any procedural error). Because field replicate analyses integrate a number of different sources of variability, they might be difficult to interpret. As a result, failure to meet a precision MQO for field replicates might or might not be a cause of concern in terms of the overall study objectives, but would suggest some uncertainty in the data. Many monitoring programs perform field replicates at 10 % of the stations sampled in the study as a quality control procedure. An MQO of less than 30 to 50 % relative percent difference (RPD) is typically used for field replicates depending on the analyte (USEPA 2001(1)). Many regulatory programs (for example, Dredged Disposal Management within the Puget Sound Estuary Program) routinely use 3 to 5 field replicates per station. Appendix C of USEPA (2001 (1)) summarizes statistical considerations in determining the appropriate number of replicate samples given different sampling objectives.

9.6.7.5 Split sample replication is less commonly performed in the field because many investigators find it more useful to quantify data precision through the use of analytical and field replicates described above. However, split sample replication is frequently used in the laboratory in toxicity and bioaccumulation analyses (USEPA, 2000d (35)) and to verify homogeneity of test material in spiked sediment tests (see 12.4). In the field, samples are commonly split for different types of analyses (for example, toxicity, chemistry, benthos) or for inter-laboratory comparisons rather than to replicate a given sample. This type of sample splitting or subsampling is further discussed in 11.3.

9.6.7.6 *Composite Samples*—A composite sample is one that is formed by combining material from more than one sample or subsample. Because a composite sample is a combination of individual aliquots, it represents an “average” of the characteristics making up the sample. Compositing, therefore, results in a less detailed description of the variability within the site as compared to taking field replicates at each station. However, for characterizing a single station, compositing is generally considered a good way to provide quality data

with relatively low uncertainty. Furthermore, many investigators find it useful to average the naturally heterogeneous physicochemical conditions that often exist within a station (or dredging unit, for example), even within a relatively small area (GLNPO, 1994 (11); PSEP, 1997a (33)). Some investigations have composited 3 to 5 samples from a given location or depth strata (GLNPO, 1994 (11)).

9.6.7.7 Compositing is also a practical way to control analytical costs while providing information from a large number of stations. For example, with relatively little more sampling effort, five analyses can be performed to characterize a project segment or site by collecting 15 samples and combining sets of three into five composite samples. The increased coverage afforded by taking composite samples might justify the increased time and cost of collecting the extra 10 samples in this case (USEPA/USACE, 1998 (34)). Compositing is also an important way to provide the large sample volumes required for some biological tests and for multiple types of analyses (for example, physical, chemical, toxicity, and benthos). However, compositing is not recommended where combining samples could serve to “dilute” a highly toxic but localized sediment “hot spot” (WDE, 1995 (29); USEPA/USACE, 1998 (34)). Also, samples from stations with very different grain size characteristics or different stratigraphic layers of core samples should not be composited (see 11.4).

9.7 Site-Specific Considerations for Selecting Sediment Sampling Stations:

9.7.1 Several site-specific factors might ultimately influence the appropriate location of sampling stations, both for large-scale monitoring studies, in which general sediment quality status is desired, and for smaller, targeted studies. If a targeted or stratified random sampling design is chosen, it might be important to locate sediment depositional and erosional areas to properly identify contaminant distributions. Tables 5 and 6 presents a summary of site-specific factors that should be considered when developing a sampling plan. A more detailed review of such considerations is provided by Mudroch and MacKnight (1994 (36)).

9.7.2 Review Available Data—Review of available historical and physical data is important in the sample selection process and subsequent data interpretation. Local experts should be consulted to obtain information on site conditions and the origin, nature, and degree of contamination. Other potential sources of information include government agency records, municipal archives, harbor commission records, past geochemical analyses, hydrographic surveys, bathymetric maps, and dredging or disposal history. Potential sources of contamination should be identified and their locations noted on a map or chart of the proposed study area. It is important that recent hydrographic or bathymetric data be used in identifying representative sampling locations, especially for dredging or other sediment removal projects. The map or chart should also note adjacent land and water uses (for example, fuel docks, storm drains). The quality and age of the available data should be considered, as well as the variability of the data.

9.7.3 Site Inspection:

9.7.3.1 A physical inspection of the site should be performed when developing a study plan in order to assess the

TABLE 5 Practical Considerations for Selection of Sampling Stations in Developing a Sampling Plan (USEPA 2001(1))

Activity	Consideration
Determination of areas where sediment contamination might occur	Hydrologic information: quality and quantity of runoff potential depositional inputs of total suspended solids up-wellings seepage patterns
Determination of depositional and erosional areas	Bathymetric maps and hydrographic charts: water depth zones of erosion, transport, and deposition bathymetry distribution, thickness, and type of sediment velocity and direction of currents sedimentation rates Climatic conditions: prevailing winds seasonal changes in temperature, precipitation, solar radiation, etc. tides, seiches seasonal changes in anthropogenic and natural loadings
Determination of potential sources of contamination	Anthropogenic considerations: location of urban lefts historical changes in land use types, densities, and size of industries location of waste disposal sites location of sewage treatment facilities location of stormwater outfalls and combined sewer overflows location, quantity, and quality of effluents previous monitoring and assessment or geochemical surveys location of dredging and open-water dredged material disposal sites location of historical waste spills
Factors affecting contaminant bioavailability	Geochemical considerations: type of bedrock and soil/sediment chemistry physical and chemical properties of overlying water
Determination of representativeness of samples	area to be characterized volume to be characterized depth to be characterized possible stratification of the deposit to be characterized

completeness and validity of the collected historical data, and to identify any significant changes that might have occurred at the site or study area (Mudroch and MacKnight, 1994 (36)). A site inspection of the immediate drainage area and upstream watershed might also identify potential stressors (such as erosion), and help determine appropriate sampling gear (such as corer vs. grab samplers and boat type), and sampling logistics.

9.7.3.2 If resources allow, it is useful to perform some screening or pilot sampling and analyses at this stage to further refine the actual sampling design needed. Pilot sampling is particularly helpful in defining appropriate station locations for targeted sampling, or to identify appropriate strata or subareas in stratified or multistage sampling.

9.7.4 Identify Sediment Deposition and Erosional Zones:

9.7.4.1 When study DQOs target sampling to the highest contamination levels or specific subareas of a site, it might be

**TABLE 6 Recommendations for Positioning of Sampling Stations
(USEPA 2001 (1))**

Depending on level of accuracy needed, regular calibration of the positioning system by at least two methods might be required to ensure accuracy.
For monitoring and assessment studies of large areas (for example, large lakes or offshore marine environments), where an accuracy of \pm 100 m typically is sufficient, either the Long Range Navigation (LORAN) or Global Positioning System (GPS) system is recommended.
For near-shore areas, or areas where the sampling stations are numerous or located relatively close together, GPS or a microwave system should be used if the required position accuracy is less than 10 m. Where visible or suitable and permanent targets are available, RADAR can be used if the required position accuracy is between 10 and 100 m.
For small water bodies and urban waterfronts, GPS is often capable of giving precise location information. Alternatively, visual angular measurements (for example, sextant) by an experienced operator, a distance line, or taut wire could also provide accurate and precise positioning data.

important to consider sediment deposition and sediment erosional zones, since grain size and related physicochemical characteristics (including conventional parameters, such as total organic carbon and acid volatile sulfide, as well as other contaminants), are likely to vary between these two types of zones. Depositional zones typically contain fine-grained sediment deposits which are targeted in some sampling programs because fine-grained sediments tend to have higher organic carbon content (and are therefore a more likely repository for contaminants) relative to larger sediment particle size fractions (for example, sand and gravel; Environment Canada 1994(2), USEPA 2001(1)). However, for some studies such as remediation dredging evaluations or USEPA Superfund sites, eroding sediment beds and non-depositional zones might be of most concern as these could be a major source of contaminants in the water column and in organisms USEPA/USACE,(1991 (32)).

9.7.4.2 Various non-disruptive technologies are available to assist in the location of fine-grained sediments ranging from simplistic to more advanced. For example, use of a steel rod or PVC pipe can be used in many shallow areas to quickly and easily probe the sediment surface to find coarse (sand, gravel) vs. fine sediments (silt, clay). This technique can not, however, determine sediment grain size at depth. Other more advance methods, including acoustic survey techniques (for example, low frequency echo sounding, seismic reflections) and side-scan sonar used with a sub-bottom profiler (Wright et al. 1987 (37)), can provide useful information on surficial as well as deeper sediment profiles. However, these techniques are often limited in their accuracy and have high equipment costs (Guigné et al. 1991 (38)). Sediment Profile Imaging (SPI) or REMOTS can also assist in the identification of grain size and substrate type in advance of field-sampling activities (Germano 1989 (39); Rhoads and Germano 1982 (40), 1986 (41)).

9.7.4.3 Aerial reconnaissance, with or without satellite imagery, might assist in visually identifying depositional zones where clear water conditions exist. However, these methods are not reliable if the water is turbid. Other methods that can be used to locate sediment deposition zones include grab sam-

pling, inspection by divers, or photography using an underwater television camera or remotely operated vehicle (Burton, 1992 (42)).

9.8 *Positioning Methods for Locating Sampling Stations:*

9.8.1 The most important function of positioning technology is to determine the location of the sampling station (for example, latitude and longitude), so that the user can later re-sample to the same position (USEPA, 1987 (43)). Knowing the precise location of sampling stations is also important to determine if the area(s) of interest have been sampled. There are a variety of navigation or position-fixing systems available, including optical or line-of-site techniques, electronic positioning systems, and satellite positioning systems. Global Positioning System (GPS) is generally regarded as the positioning technique of choice as it is accurate, readily available, and often less expensive than many other comparably sophisticated systems. Given the removal of selective availability of satellite data by the U.S. military, GPS is now capable of high accuracy positioning (1 to 10 m).

9.8.2 Regardless of the type of system selected, calibration of the system should be done using at least two of these methods to determine accuracy, particularly for stations that may be resampled. At each sampling station, a fathometer or meter wheel can be used to determine the sampling depth. This will help to establish that the water is the desired depth and the bottom is sufficiently horizontal for proper operation of sampling equipment. Ideally, it is best to print out a copy of the ship's location from the GPS monitor navigation chart, as well as the latitude and longitude, so the sampling station can be placed in a spatial context. Tidal or subsurface currents may push either the vessel or its suspended sampler away from the intended location which can lead to inaccurate sampling location.

9.9 *Preparations for Field Sampling:*

9.9.1 Proper preparation for any field sampling study is an essential part of Quality Assurance is important to the successful project outcome and adherence to the objectives specified in the QAPP. Section 15 further discusses related Quality Assurance/Quality Control procedures that should be used in sediment quality studies.

9.9.2 Before performing field work, characteristics of the site and accessibility of the individual sampling stations should be determined. Pictures of sampling stations both before as well as during sampling are often useful to document that the correct stations were sampled, and to document weather and water conditions during sampling. Adequate reconnaissance of stations before sampling is also valuable for preparing against potential sampling hazards or unforeseen difficulties. Such a reconnaissance can also help determine the necessary time needed to perform the desired sampling (that is, time to get from one station to the next).

9.9.3 The appropriate vessel or sampling platform is one of the most important considerations in preparing for field sampling. The vessel should be appropriate for the water body type, and should provide sufficient space and facilities to allow collection, any on-board manipulation, and storage of samples. Ice chests or refrigeration might be required for sample storage, depending on the time course of the operation. The

vessel should provide space for storage of decontamination materials, as well as clean sampling gear and containers to minimize contamination associated with normal vessel operations. Space for personal safety equipment is also required.

9.9.4 Additionally, the vessel should be equipped with sufficient winch power and cable strength to handle the weight of the sampling equipment, taking into account the additional suction pressure associated with extraction of the sediments. Large sampling devices typically weigh between 50 and 400 kg empty, and when filled with wet sediment might weigh from 125 to over 500 kg.

9.9.5 Care should be taken in operating the vessel to minimize disturbances of the sediment to be sampled as well as sampling equipment. This would include physical disturbance through propeller action and chemical contamination from engines or stack emissions. For example, Page et al. (1995 a,b (44),(45)) reported that they positioned the ships' stern into the wind to prevent stack gases from blowing onto sampling equipment during deployment, recovery, and subsampling of sediments in Prince William Sound, Alaska.

9.9.6 The sampling plan and projected time schedule should be posted for view by all personnel. The names, addresses, and telephone numbers of all participants involved with the preparation and execution of the sampling program should be available to all participants, and the duties and responsibilities of each participant clearly documented. The study supervisor should determine that the appropriate personnel clearly understand their role and are capable of carrying out their assigned responsibilities and duties. Contingency planning should address the need for backup personnel in the event of accident or illness.

9.9.7 A variety of sampling and sample handling equipment and supplies are often needed in sediment monitoring studies. Besides the actual samplers themselves (for example, grab or core device to be used), equipment is needed to remove and process the samples such as spatulas, scoops, pans or buckets, and gloves. If it is important to maintain anoxic conditions of the sample, a glove box and inert gas source (for example, nitrogen) is needed. Sample storage and transport equipment and supplies need to be available as well. These include refrigeration, ice chests, dry ice or ice, insulation material to stabilize samples in transport, custody seals, and shipping air bills.

9.9.8 The reagents for cleaning, operating, or calibrating equipment, or for collecting, preserving or processing samples should be handled by appropriately qualified personnel and the appropriate data for health and safety (for example, Material Safety Data Sheets) should be available. Standard operating procedures (including QA/QC requirements) should be readily accessible at all times, to facilitate the proper and safe operation of equipment. Data forms and log books should be prepared in advance so that field notes and data can be quickly and efficiently recorded. Extra forms should be available in the event of a mishap or loss. These forms and books should be waterproof and tear resistant. Under certain circumstances, audio or audio/video recordings might prove valuable.

9.9.9 All equipment used to collect and handle samples should be cleaned and all parts examined to facilitate proper

functioning before going into the field. A repair kit should accompany each major piece of equipment in case of equipment failure or loss of removable parts. Backup equipment and sampling gear should be available.

9.9.10 Storage, transport, and sample containers, including extra containers, should be available in the event of loss or breakage (see 11.2 for more information on appropriate containers). These containers should be pre-cleaned and labeled appropriately (that is, with a waterproof adhesive label to which the appropriate data can be added, using an indelible ink pen capable of writing on wet surfaces). The containers should have lids that are fastened securely, and if the samples are collected for legal purposes, they should be transported to and from the field in a locked container with custody seals secured on the lids. Samples to be frozen before analyses should not be filled to the very top of the container. Leave at least 10 % headspace to accommodate expansion during freezing (laying glass jars on their side during freezing may help to reduce the chance of the container breaking during freezing). Whether for legal purposes or not, all samples should be accompanied by a chain-of-custody form that documents field samples to be submitted for analyses (see Section 15). Transport supplies also include shipping air bills and addresses. Whole-sediment sediment samples should never be frozen for toxicity or bioaccumulation testing (Test Method E 1706 and Guide E 1688).

9.9.11 A sample-inventory log and a sample-tracking log should be prepared in advance of sampling. A single person should be responsible for these logs who will track the samples from the time they are collected until they are analyzed and disposed of or archived.

10. Collection of Whole Sediment Samples

10.1 General Procedures:

10.1.1 Most sediment collection devices are designed to isolate and retrieve a specified volume and surface area of sediment, from a required depth below the sediment surface, with minimal disruption of the integrity of the sample and no contamination of the sample. Maintaining the integrity of the collected sediment, for the purposes of the measurements intended, is a primary concern in most studies because any disruption of the sediment structure changes its physicochemical and biological characteristics, thereby influencing the bioavailability of contaminants and the potential toxicity of the sediment. This section discusses the factors to be considered in selecting a sediment collection device and minimizing disruption of sediment samples. A variety of samplers are described (Annex A1), and recommendations are made regarding their use in different situations.

10.1.2 Figs. 4 and 5 provide suggested grab and core samplers based on site factors (such as depth and particle size), and sampling requirements (such as sample depth and volume of sample needed).

10.1.3 The planned mode of access to the sampling area (for example, by water, over land or ice, or from the air) plays an important role in the selection of sampling gear. If the sampling gear needs to be transported to a remote area or shipped by air, its weight and volume might should be taken into account. It is often the case that a specific vessel, having a fixed lifting

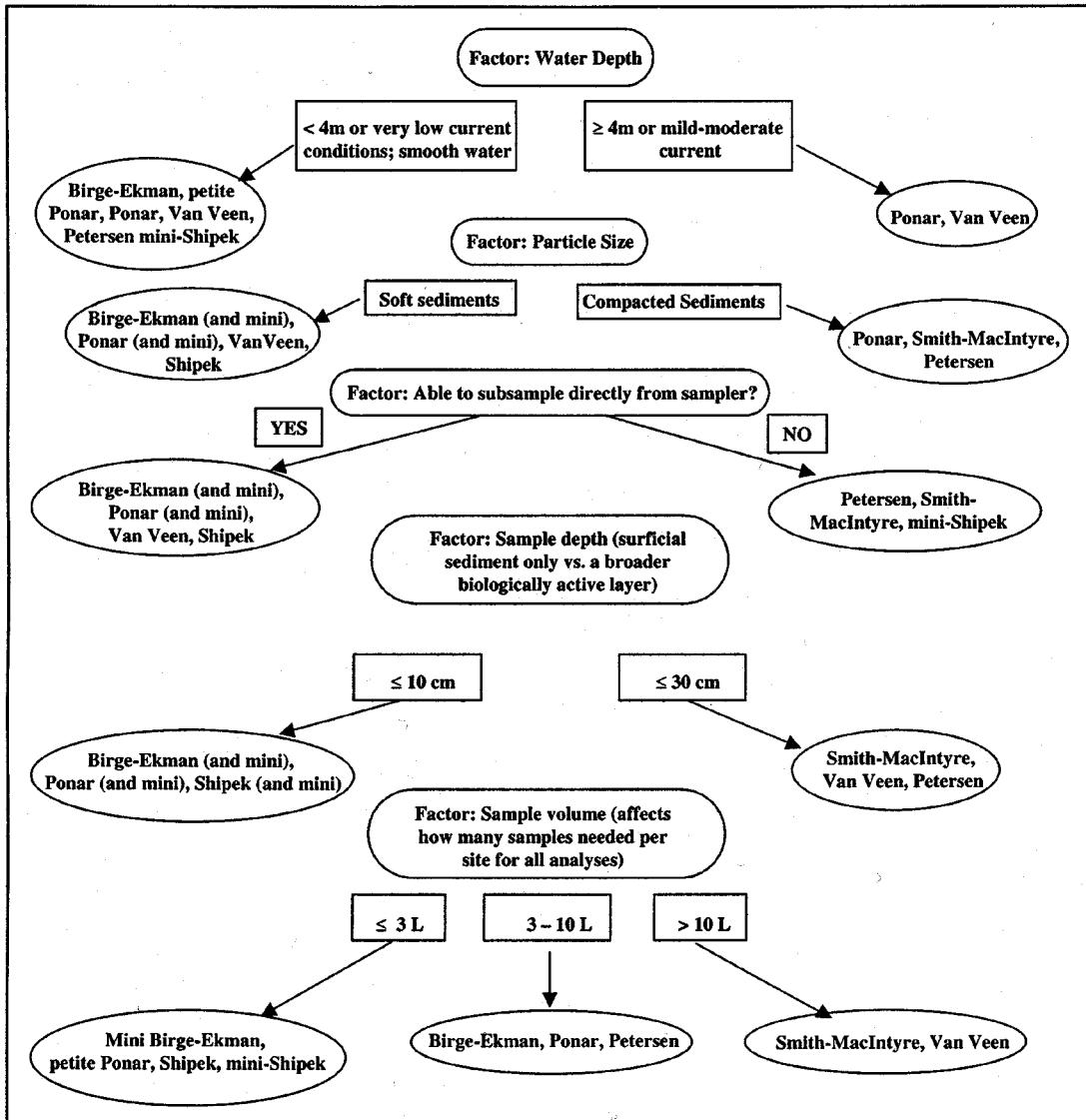


FIG. 4 Flowchart for Selecting Appropriate Grab Samplers Based on Site Specific or Design Factors (USEPA 2001 (1))

capacity based on the configuration of its winch, crane, boom, A-frame, or other support equipment, is the only one available for use. This will affect the type of sampling equipment that can be safely operated from that vessel.

10.1.4 Many samplers are capable of recovering a relatively undisturbed sample in soft, fine-grained sediments, but fewer are suitable for sampling harder sediments containing significant quantities of sand, gravel, firm clay, or till (Mudroch and Azcue, 1995 (46)). One of the most important factors in determining the appropriate sampling device for the study are DQOs. Many monitoring programs, such as the USEPA Environmental Monitoring and Assessment Program (EMAP) and the NOAA National Status and Trends program, are primarily interested in characterizing recent environmental impacts in lakes, estuaries, and coastal waters, and therefore sample surface sediments (for example, Long et al. 1996 (31)). Other programs (for example, dredged material characteriza-

tion studies conducted for USEPA and the US Army Corps of Engineers), are concerned with the vertical distribution of contaminants in sediment to be dredged and therefore seek to characterize a sediment column (USEPA/USACE, 1991(32), 1998 (34)). Each of these applications might use different sampling devices.

10.1.5 Related to study objectives, another important factor in selecting a sampler is desired depth of sediment penetration. For monitoring and assessment studies where historical contamination is not the focus, the upper 10 to 15 cm is typically the horizon of interest. For example, Test Method E 1706 states sediment should be collected from a depth that will represent expected exposure. Generally, these are the most recently deposited sediments, and most epifaunal and infaunal organisms are found in this horizon. To minimize disturbance of the upper layer during sampling, a minimum penetration depth of 6 to 8 cm is suggested, with a penetration depth of 10 to 15 cm

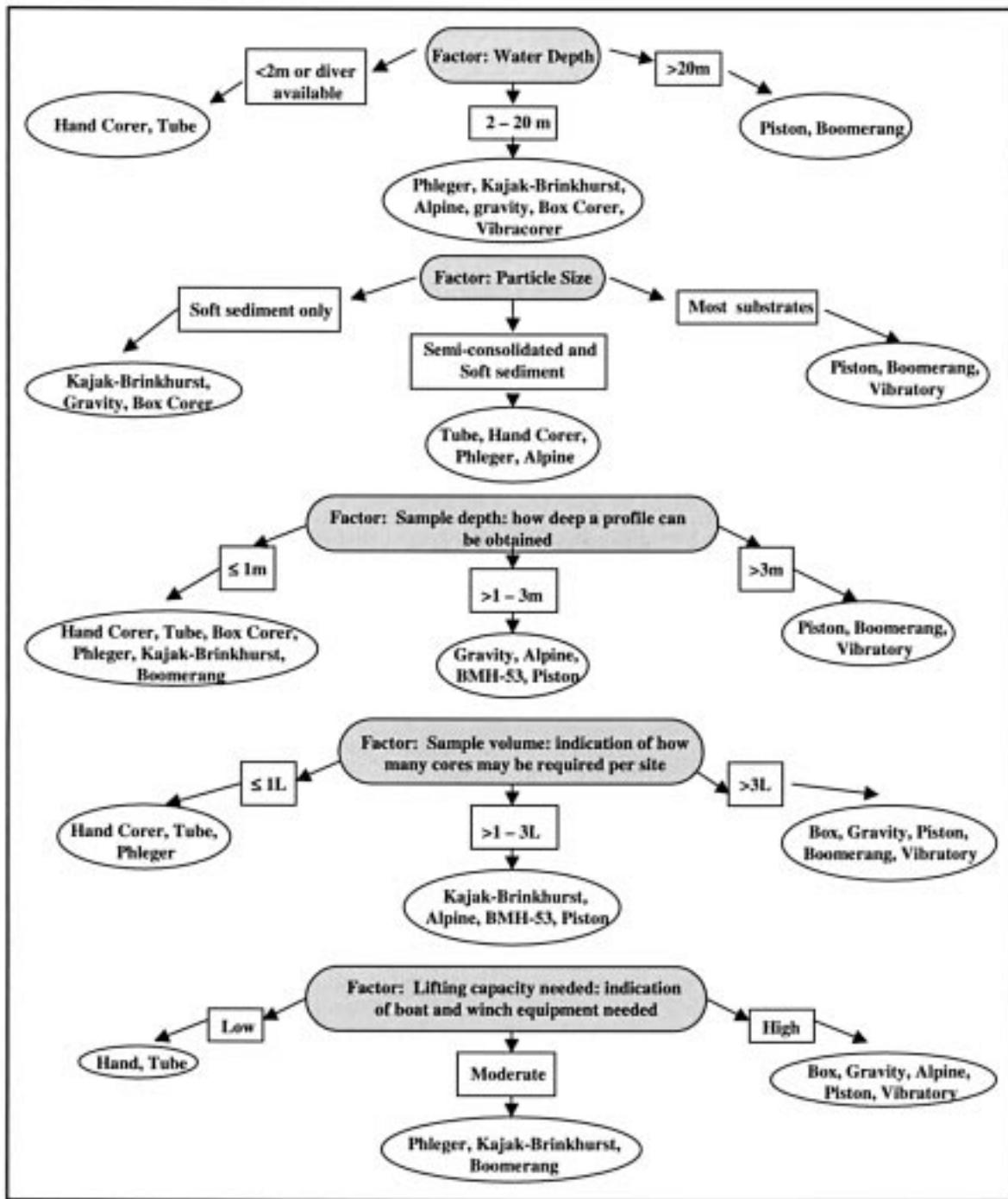


FIG. 5 Flowchart for Selecting Appropriate Core Samplers Based on Site-specific Factors (USEPA 2001 (1))

being preferred. However, if sediment contamination is being related to organism exposures (for example, benthic macroinvertebrates or fish) then more precise sampling of sediment depths might be needed, such as with a core sampler. The life history and feeding habits of the organisms (receptors) of concern should be considered. For example, some organisms (for example, shrimp, rotifers) might be epibenthic and are only exposed to surficial sediments (for example, 0 to 1 cm), while others (for example, amphipods, polychaetes) that are

infaunal irrigators might receive their primary exposure from sediments that are several centimeters in depth. Relating contaminant levels that occur in sediment layers where resident organisms are not exposed might produce incorrect conclusions (Lee 1991(17)).

10.1.6 Sampling of the surface layer provides information on the horizontal distribution of parameters or properties of interest for the most recently deposited material. Information obtained from analysis of surface sediments can be used, for

example, to map the distribution of a chemical contaminant in sediments across a specific body of water (for example, lake, embayment, estuary). A sediment column, including both the surface sediment layer and the sediment underneath this layer, is collected to study historical changes in parameters of interest (as revealed through changes in their vertical distribution), and to characterize sediment quality with depth.

10.1.7 Once study objectives and the general type of sampler have been identified, a specific sampler is selected based on knowledge of the bathymetry and areal distribution of physically different sediment types at the sampling site. Therefore, this information should be gathered during the initial planning stage of the sample collection effort (see 9.7.2).

10.1.8 The quantity of sediment to be collected at each sampling station may also be an important consideration in the selection of a sampling device (see also 9.6.6). The required quantity of sediment typically depends on the number and type of physicochemical and biological tests to be carried out. Table 3 provides a summary of typical sediment volumes needed for different analyses.

10.1.9 Regardless of the type of sampler used, it is important to follow the standard operating procedures specific to each device. Before retrieving the sample, the outside of the sampling device should be carefully rinsed with water from the sampling station. Between each sampling event, the sampling device should be cleaned, inside and out, by dipping the sampler into and out of the water rapidly or by washing with water from the location being sampled. More rigorous between-sample cleaning of the sampler (for example, chemical decontamination or washing with soap) might be required, depending on the nature of the investigation (see 10.5).

10.1.10 To minimize cross-contamination of samples and to reduce the amount of equipment decontamination required, it might be prudent to sample reference stations (that is, relatively clean stations) first, followed by test stations. If certain stations are known to be heavily contaminated, it might be prudent to sample those stations last when sampling many locations at one time.

10.2 Types of Sediment Samplers:

10.2.1 There are three main types of sediment sampling devices: grab samplers, core samplers, and dredge samplers. Grab samplers (Annex A1) are typically used to collect surficial sediments for the assessment of the horizontal distribution of sediment characteristics. Core samplers (Annex A1) are typically used to sample thick sediment deposits, or to collect sediment profiles for the determination of the vertical distribution of sediment characteristics or to characterize the entire sediment column. Dredge samplers are used primarily to collect benthos (Annex A1). Dredges cause disruption of sediment and pore water integrity, as well as loss of fine-grained sediments. For these reasons, only grab and core samplers are recommended for sediment physicochemistry or toxicity evaluations. Since many grab samplers are appropriate for collecting benthos as well (Klemm et al. 1990 (30) and Guide D 4387), grab samplers are likely to be more useful than dredges in sediment quality assessments. Therefore, dredges are not considered further in the following sections.

10.2.2 Advantages and disadvantages of various grab and core samplers are summarized in Tables A1.1-A1.4 in Annex A1 and are discussed briefly in the following sections. Figs. 4 and 5 and Table 7 provide recommendations regarding the type of sampler that would be appropriate given different study objectives. For many study objectives either cores or grab samplers can be used, however, in practice, one will often be preferred over the other depending on other constraints such as amount of sample required for analyses and equipment availability.

10.2.3 Grab Samplers:

10.2.3.1 Grab samplers consist either of a set of jaws that shut when lowered into the surface of the bottom sediment, or a bucket that rotates into the sediment when it reaches the bottom (Annex A1). Grab samplers have the advantages of being relatively easy to handle and operate, readily available, moderately priced, and versatile in terms of the range of substrate types they can effectively sample.

10.2.3.2 Of the grab samplers, the Van Veen, Ponar, and Petersen are the most commonly used. These samplers are effective in most types of surface sediments and in a variety of environments (for example, lakes, rivers, estuaries, and marine waters). In shallow, quiescent water, the Birge-Ekman sampler also provides acceptable samples and allows for relatively nondisruptive sampling. However, this sampler is typically limited to soft sediments. The Van Veen sampler, or the

TABLE 7 Recommendations for Selecting Appropriate Sediment Sampling Devices Based on the Study Objectives (USEPA 2001 (1))

Grab or core samplers are preferred over dredges for collecting surficial sediments for physicochemical or toxicity analyses. Dredges might be acceptable for collecting macroinvertebrates.

Grab samplers are recommended for surficial sediment analyses where accurate resolution of surficial sediment depths is not necessary. Core samplers are recommended for: (a) assessments requiring accurate surficial sediment depth resolution, (b) historical sediment analyses, (c) detailed sediment quality studies of vertical sediment profiles, to characterize sediment quality at depth, (d) when characterizing thick sediment deposits (such as shoals to be excavated), and/or (e) where it is important to maintain an oxygen-free environment.

In sand, gravel, firm clay, or till sediments, grab samplers might be preferred over core samplers (when only surface material needs to be collected and samples at depth are not necessary) because the latter are often less efficient in these sediment types.

Ponar, VanVeen, or Ekman samplers are commonly used and generally preferred for grab sampling. Ekman samplers, however, are less efficient in deep waters.

The Kajak-Brinkhurst corer is a common core sampler for soft, fine grained sediments where large volumes or deep cores are not needed. The Phleger corer is commonly used for a variety of sediments including peat and plant roots but is not appropriate where large volumes or deep cores are needed.

Box corers are especially recommended for: (a) studies of the sediment-water interface; (b) collecting larger volumes of sediment from a given depth (generally less than one meter depth, however); (c) for in-situ studies involving interstitial water characterization; and (d) collecting subsamples for different analyses from the same station.

Vibracorers are recommended for studies requiring deep cores (> 1 m), or where sediment consists of very compacted or large grained material (for example, gravel).

modified Van-Veen (Ted Young), is used in several national and regional estuarine monitoring programs, including the NOAA National Status and Trends Program, the USEPA Environmental Monitoring and Assessment Program (EMAP), and the USEPA National Estuary Program, because it can sample most types of sediment, is less subject to blockage and loss of sample than the Petersen and Ponar samplers, is less susceptible to forming a bow wave during descent, and provides generally high sample integrity (Klemm et al. 1990 (30)). The support frame further enhances the versatility of the VanVeen sampler by allowing the addition of either weights (to increase penetration in compact sediments) or pads (to provide added bearing support in extremely soft sediments). However, this sampler is relatively heavy and requires a power winch to operate safely (GLNPO, 1994 (11)).

10.2.3.3 As shown in Annex A1, grab sampler capacities range from about 0.5 to 75 L. If a sampler does not have sufficient capacity to meet the study plan requirements, additional samples can be collected and composited to obtain the necessary sample volume. Grab samplers penetrate to different depths depending on their size, weight, and the bottom substrate. Heavy, large volume samplers such as the Smith-McIntyre, large Birge-Ekman, Van Veen, and Petersen devices can effectively sample to a depth of 30 cm. These samplers might actually sample sediments that are too deep for certain study objectives (that is, not reflective of recently deposited sediments). Smaller samplers such as the small Birge-Ekman, standard and petite Ponar, and standard Shipek devices can effectively collect sediments to a maximum depth of 10 cm. The mini-Shipek can sample to a depth of 3 cm.

10.2.3.4 Another consideration in choosing a grab sampler is how well it protects the sample from disturbance and washout. Grab samples are prone to washout which results in the loss of surficial, fine grained sediments that are often important from a biological and contaminant standpoint. The Ponar, Ted-Young modified grab, and Van Veen samplers are equipped with mesh screens and rubber flaps to cover the jaws. This design allows water to pass through the samplers during descent, reducing disturbance from bow waves at the sediment-water interface. The rubber flaps also serve to protect the sediment sample from washout during ascent. However, meshed screens on samplers may result in wash out of sample after collection, and rubber flaps may be difficult to decontaminate between samples.

10.2.3.5 The use of small or lightweight samplers, such as the small Birge-Ekman, petite Ponar, and mini-Shipek, can be advantageous because of easy handling, particularly from a small vessel or using only a hand line. However, these samplers should not be used in strong currents or high waves. This is particularly true for the Birge-Ekman sampler, which requires relatively calm conditions for proper performance. Lightweight samplers generally have the disadvantage of being less stable during sediment penetration. They tend to fall to one side due to inadequate or incomplete penetration, resulting in unacceptable samples.

10.2.3.6 In certain very shallow water applications, such as a stream assessment, it might be difficult to use even a lightweight sampler to collect a sample. In these cases,

sediment can be collected from depositional areas using a shovel or other hand implement. However, such sampling procedures are discouraged as a general rule and the use of a hand corer or similar device is preferred (see 10.2.4).

10.2.3.7 Fig. 4 summarizes appropriate grab samplers based on two important site factors, depth and sediment particle size. This figure also indicates appropriate grab samplers depending on certain common study constraints such as sample depth and volume desired, and the ability to subsample directly from the sampler (see 11.4 and Guide D 4387). Based on all of these factors, the Ponar or Van Veen samplers are perhaps the most versatile of the grab samplers, hence their common usage in sediment studies.

10.2.3.8 Careful use of grab samplers is required to minimize problems such as loss of fine-grained surface sediments from the bow wave during descent, mixing of sediment layers upon impact, lack of sediment penetration, and loss of sediment from tilting or washout upon ascent (USEPA 2001(1); Environment Canada, 1994 (2); Baudo, 1990 (145); Golterman et al., 1983 (286); Plumb, 1981 (213)). When deploying a grab sampler, the speed of descent should be controlled, with no "free fall" allowed. In deep waters, a winching system should be used to control both the rate of descent and ascent. A ball-bearing swivel should be used to attach the grab sampler to the cable to minimize twisting during descent. After the sample is collected, the sampling device should be lifted slowly off the bottom, then steadily raised to the surface at a speed of about 30 cm/sec (Environment Canada, 1994 (2)).

10.2.4 Core Samplers:

10.2.4.1 Core samplers (corers) are used: (1) to obtain sediment samples for geological characterizations and dating, (2) to investigate the historical input of contaminants to aquatic systems and, (3) to characterize the depth of contamination at a site. Corers are an essential tool in sediments in which 3-dimensional maps of sediment contamination are necessary. Table A1.2 discusses some of the advantages and disadvantages of common corers.

10.2.4.2 Core devices should be used for projects in which it is important to maintain the integrity of the sediment profile, because these devices are considered to be less disruptive than dredge or grab samplers. Core samplers should also be used where it is important to maintain an oxygen-free environment because they limit oxygen exchange with the air more effectively than grab samplers. Cores should also be used where thick sediment deposits are to be representatively sampled (for example, for dredging projects).

10.2.4.3 One limitation of core samplers is that the volume of any given depth horizon within the profile sample is relatively small. Thus, depending on the number and type of analyses needed, repetitive sampling at a site might be required to obtain the desired quantity of material from a given depth. Some core samplers are prone to "plugging" or "rodding" where the friction of the sediment within the core tube prevents it from passing freely and the core sample is compressed or does not sample to the depth required. This limitation is more likely with smaller diameter core tubes and heavy clay sediments. Except for piston corers and vibracorers, there are few

core devices that function efficiently in substrates with significant proportions of sand, gravel, clay, or till.

10.2.4.4 Coring devices are available in various designs, lengths, and diameters (Annex A1). With the obvious exception of hand corers, there are only a few corers that can be operated without a mechanical winch. The more common of these include the standard Kajak-Brinkhurst corer, suitable for sampling soft, fine-grained sediments, and the Phleger corer, suitable for a wider variety of sediment types ranging from soft to sandy, semi-compacted material, as well as peat and plant roots in shallow lakes or marshes (Mudroch and Azcue, 1995 (46)). The Kajak-Brinkhurst corer uses a larger core tube, and therefore recovers a greater quantity of sediment, than the Phleger corer. Both corers can be used with different liner materials including stainless steel and PVC. Stainless steel liners should not be used if trace metal contamination is an issue.

10.2.4.5 Gravity corers are appropriate for recovering up to 3 m long cores from soft, fine-grained sediments. Recent models include stabilizing fins on the upper part of the corer to promote vertical penetration into the sediment, and weights that can be mounted externally to enhance penetration (Mudroch and Azcue, 1995 (46)). A variety of liner materials are available including stainless steel; Lexan®, and PVC. For studies in which metals are a concern, stainless steel liners should not be used.

10.2.4.6 Vibracorers are perhaps the most commonly used coring device in the United States because they collect deep cores in most types of sediments, yielding excellent sample integrity. Vibracorers are one of the only sampling devices that can reliably collect thick sediment samples (up to 10 m or more). Some programs that rely on vibracorers include the Puget Sound Estuary Program, the USEPA Great Lakes National Program ARCS Program (GLNPO 1994 (11)), and the Dredged Materials Management Program. Note that the vibratory action of a vibracore can lead to vertical transport of fines along the wall of the core tube resulting in smearing of the sample. Additionally, unconsolidated materials can be mixed (for example, recently placed or capped materials). Consequently, vibracoring may not be appropriate in cases where higher resolution sampling is required in “loose” materials.

10.2.4.7 Vibracorers have an electric-powered, mechanical vibrator located at the head end of the corer which applies thousands of vertical and horizontal vibrations per minute to help penetrate the sediment. A core tube and rigid liner (preferably of relatively inert material such as cellulose acetate butyrate) of varying diameter depending on the specific vibrator head used, is inserted into the head and the entire assembly is lowered in the water. Depending on the horsepower of the vibrating head and its weight, a vibracorer can penetrate very compact sediments and collect cores up to 6 m long. For example, the ARCS program in the Great Lakes uses a Rossfelder® Model P-4 Vibracorer (Rossfelder Corporation, La Jolla, CA) to collect cores up to 6 m in length; however, this particular model is relatively heavy. Therefore, use of a heavy vibracorer requires a large vessel to maintain balance and provide adequate lift to break the corer out of the sediment and retrieve it (GLNPO, 1994 (11); PSEP, 1997a (33)).

10.2.4.8 When deployed properly, box corers can obtain undisturbed sediment samples of excellent quality. The basic box corer consists of a stainless steel box equipped with a frame to add stability and facilitate vertical penetration on low slopes. Box corers should be used in studies of the sediment-water interface or when there is a need to collect larger volumes of sediment from the depth profile. Because of the heavy weight and large size of almost all box corers, they can be operated only from a vessel with a large lifting capacity and sufficient deck space. Sediment inside a box corer can be subsampled by inserting narrow core tubes into the sediment. The tubes should be machine cut so that the opening is square with the tube shaft, and the ends of the tube should be carefully milled to reduce smearing of the sample on the inside surface of the tube and to improve the ease of penetration of the tube. Core tubes are an ideal sampler for obtaining acceptable subsamples for different analyses at a given station. Carlton and Wetzel (1985 (47)) describe a box corer that permits the sediment and overlying water to be held intact as a laboratory microcosm under either the original in situ conditions or other laboratory controlled conditions. A box corer was developed that enables horizontal subsampling of the entire sediment volume recovered by the device (Mudroch and Azcue, 1995 (46)).

10.2.4.9 Fig. 5 summarizes the core samplers that are appropriate given site factors such as depth and particle size and other study constraints such as sample depth and volume required, and lifting capacity needed to use the sampling device. Given the factors examined for general monitoring studies, the Phleger, Alpine, and Kajak-Brinkhurst corers might be most versatile. For dredged materials evaluations, and projects requiring sediment profile characterizations greater than 3 m in sediment depth, the vibracorer or piston corer are the samplers of choice.

10.2.4.10 Collection of core samples with hand-coring devices should be performed with care to minimize disturbance or compression of sediment during collection. To minimize disruption of the sediment, core samples should be kept as stationary and vibration-free as possible during transport. These cautions are particularly applicable to cores collected by divers.

10.2.4.11 The speed of descent of coring devices should be controlled, especially during the initial penetration of the sediment, to minimize disturbance of the surface and to minimize compression due to frictional drag from the sides of the core liner (Guide D 4823). In deep waters, winches should be used where necessary to minimize twisting and tilting and to control the rate of both descent and ascent. With the exception of piston corers or vibracorers, which are equipped with their own mechanical impact features, for other corers, only the weight or piston mechanism of the sampler should be used to force it into the sediment. The sampler should be raised to the surface at a steady rate, similar to that described for grab samplers. Where core caps are required, it is essential to quickly and securely cap the core samples when the samples are retrieved. The liner from the core sampler should be carefully removed and kept in a stable position until the samples are processed (see Section 11). If there is little to no

overlying water in the tube and the sediments are relatively consolidated, it is not necessary to keep the core sample tubes vertical. If sediment oxidation is a concern (for example, due to potential changes in metal bioavailability or volatile substances in anoxic sediments), then the head space of the core tube should be purged with an inert gas such as nitrogen or argon.

10.3 Sample Acceptability:

10.3.1 Only sediments that are correctly collected with grab or core sampling devices should be used for subsequent physicochemical, toxicity, or bioaccumulation testing. Acceptability of grabs can be determined by noting that the samplers were closed when retrieved, are relatively full of sediment (but not over-filled), and do not appear to have lost surficial fines. At shallow stations when multiple composite samples are being taken to retrieve larger sediment volumes, it is not uncommon to drop the dredge into a previous hole. A visual inspection of the sample surface should be done to determine if only surface sediment has been collected. Slight adjustments in location

may be necessary if operating with a crane or if using a hand line, moving elsewhere in the boat to operate the sampler. Core samples are acceptable if the core was inserted vertically in the sediment and an adequate depth was sampled.

10.3.2 A sediment sample should be inspected as soon as it is secured. If a collected sample fails to meet any of the conditions listed in the previous paragraph, then the sample might need to be rejected and another sample collected at the station. The location of consecutive attempts should be as close to the original attempt as possible and located in the “up-stream” direction of any existing current. Rejected sediment samples should be discarded in a manner that will not affect subsequent samples at that station or other possible sampling stations. Illustrations of acceptable and unacceptable grab samples are provided in Fig. 6.

10.4 Equipment Decontamination:

10.4.1 For most sampling applications, site water rinse of equipment in between stations is normally sufficient (PSEP,

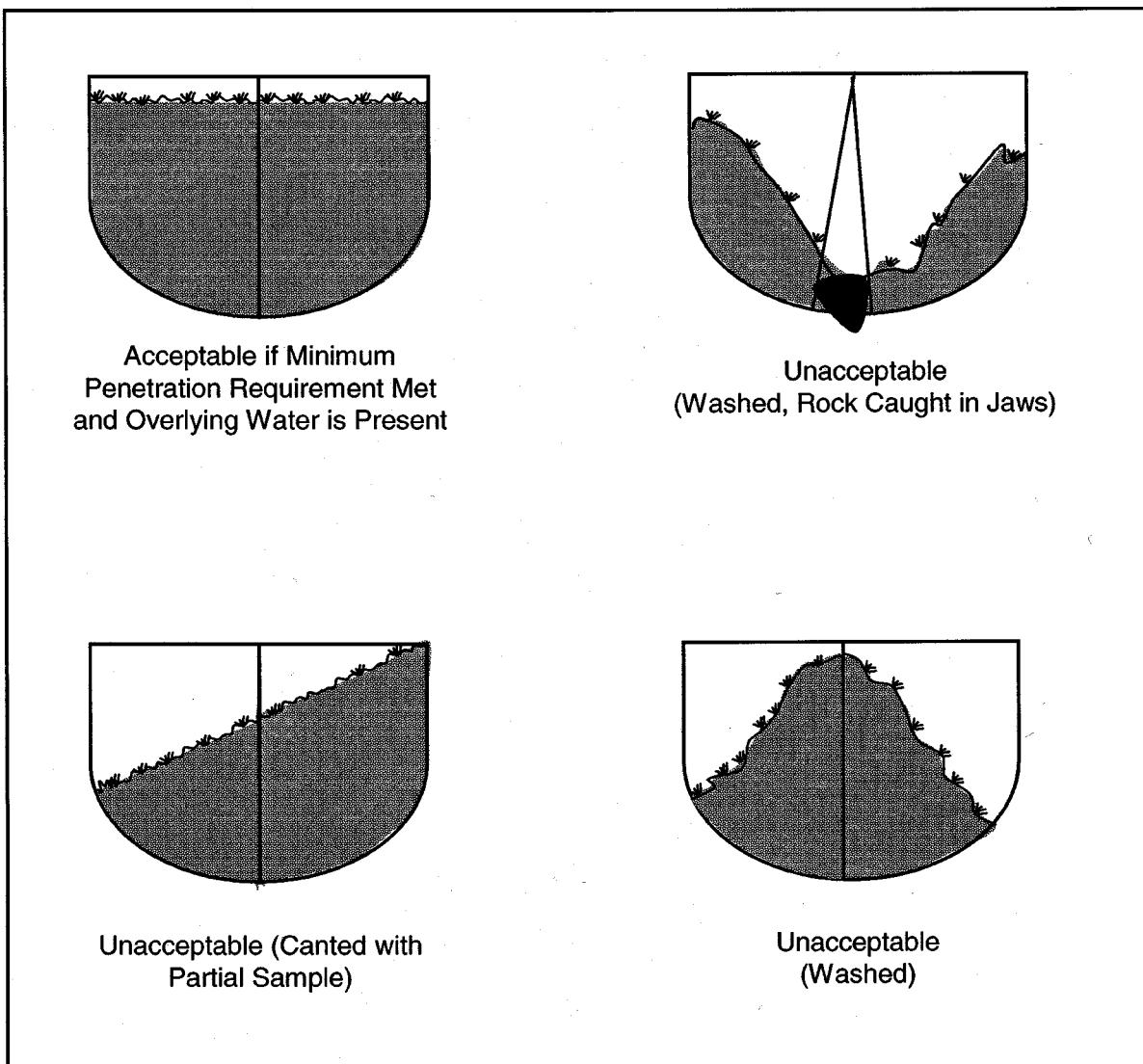


FIG. 6 Illustrations of Acceptable and Unacceptable Grab Samples (USEPA 2001 (1))

1997a (33)). However, if one is sampling many stations, including some that could be heavily contaminated, a site water rinse might not be sufficient to minimize cross-contamination of samples among stations. In these cases, it might be necessary to decontaminate all sampling materials in between stations. This would include the sampling device, scoop, spatula, mixing bowls, and any other utensils that come in contact with sediment samples. See 7.2 for additional detail on cleaning equipment. Alternatively, separate sampling equipment could be used at each station.

10.4.2 If sediment can be collected from the interior of the sampling device, and away from potentially contaminated surfaces of the sampler, it might be adequate to rinse with site water between stations. The interior of the sampler needs to be free of any sediment between sampling stations, and should be either rinsed or physically scrubbed. Particular attention should be paid to corners and seams in the sampling device.

10.4.3 If metals or other inorganic compounds are specifically of concern, sampling and handling equipment should be suspended over a tub and rinsed from the top down with 10 % nitric acid using a pump or squirt bottle (USEPA 1993 (48), 2001(1)). If organic compounds are a specific concern, sampling equipment can be decontaminated using acetone followed by a site water rinse. Wash water from decontamination should be collected and disposed of properly.

10.5 Field Measurements and Observations:

10.5.1 Field measurements and observations are important to any sediment collection study, and specific details concerning sample documentation should be included in the study plan.

10.5.2 Measurements and observations should be documented clearly in a bound field logbook (or on pre-printed sample forms). Preferably, a logbook should be dedicated to an individual project. The investigator's name, project name, project number, and book number (if more than one is required) should be entered on the inside of the front cover of the logbook. All entries should be written in indelible ink, and the date and time of entry recorded. Additionally, each page should be initialed and dated by the investigator. At the end of each day's activity, or entry of a particular event if appropriate, the investigator should enter their initials. All aspects of sample collection and handling as well as visual observations and field conditions should be documented in the field logbooks at the time of sample collection. Logbook entries should also include any circumstances that potentially affected sampling procedures or any field preparation of samples. Data entries should be thorough enough to allow station relocation and sample tracking. Because field records are the basis for later written reports, language should be objective, factual, and free of personal opinions or other terminology that might appear inappropriate.

10.5.3 In describing characteristics of samples collected, some cautions should be noted. First, polarized glasses are often worn in the field to reduce glare, however, they can also alter color vision. Therefore, visual examination or characterization of samples should be performed without sunglasses (GLNPO, 1994 (11)). Second, descriptions of sediment texture and composition should rely on a texture-by-feel or "ribbon"

test in addition to visual determinations (GLNPO, 1994 (11)). In this test, a small piece of suspected clay is rolled between the fingers while wearing protective gloves. If the piece easily rolls into a ribbon it is clay; if it breaks apart, it is silt (GLNPO, 1994 (11)).

10.5.4 Documentation of Sample Collection— Documentation of collection and analysis of sediment and pore-water samples requires all the information necessary to: (1) trace a sample from the field to the final result of analysis; (2) describe the sampling and analytical methodology; and (3) describe the QA/QC program (Mudroch and Accue 1995 (46); Keith, 1993 (49); Table 8). Poor or incomplete documentation of sample collection can compromise the integrity of the sample(s) and thus, the study. In addition, stations that could not, or were not, sampled should be documented with an explanation. Samples should be accompanied by chain-of-custody forms that identify each sample collected and the analyses to be conducted on that sample. Specific guidance on quality assurance procedures regarding sample chain-of-custody is summarized in Section 15.

11. Field Sample Processing, Transport, and Storage of Sediments

11.1 The way in which sediment samples are processed, transported, and stored might alter contaminant bioavailability and concentration by introducing contaminants to the sample or by changing the physical, chemical, or biological characteristics of the sample. Manipulation processes often change availability of organic compounds because of disruption of the equilibrium with organic carbon in the pore water and sediment

TABLE 8 Recommendations on Information to be Documented for Each Sample Collected (PSEP 1997a (33), USEPA 2001 (1))

NOTE—Some geological characterization methods might include an odor evaluation of the sediment as this can provide useful information on physicochemical conditions. However, sediment odor evaluation is potentially dangerous depending on the chemicals present in the sediment (Test Method E 1706) and should therefore be done cautiously, if at all.

Project title, time and date of collection, sample number, replicate number, site identification (for example, name); station number and location (for example, positioning information);

Water depth and the sampling penetration depth;

Details pertaining to unusual events which might have occurred during the operation of the sampler (for example, possible sample contamination, equipment failure, unusual appearance of sediment integrity, control of vertical descent of the sampler, etc.), preservation and storage method, analysis or test to be performed;

Estimate of quantity of sediment recovered by a grab sampler, or length and appearance of recovered cores;

Description of the sediment including texture and consistency, color, presence of biota or debris, presence of oily sheen, changes in sediment characteristics with depth, and presence/location/thickness of the redox potential discontinuity (RPD) layer (a visual indication of black is often adequate for documenting anoxia);

Photograph of the sample is desirable, especially longitudinally-sectioned cores, to document stratification;

Deviations from approved work plans or SOPs.

system. Similarly, oxidation of anaerobic sediments increases the availability of certain metals (Di Toro et al. 1990 (50); Ankley et al. 1996 (51)). Materials and techniques should be selected to minimize sources of contamination and variation, and sample treatment before testing should be as consistent as possible. A flowchart is presented in Fig. 7 that summarizes common sediment processing procedures discussed in this section as well as issues and objectives relevant to each processing step.

11.2 Sample Containers:

11.2.1 Any material that is in contact with a field sample has the potential to contaminate the sample or adsorb components from the sample. For example, samples can be contaminated by zinc from glassware, metals from metallic containers, and organic compounds from rubber or plastic materials. The use of appropriate materials, along with appropriate cleaning procedures, can minimize or mitigate interferences from sample containers.

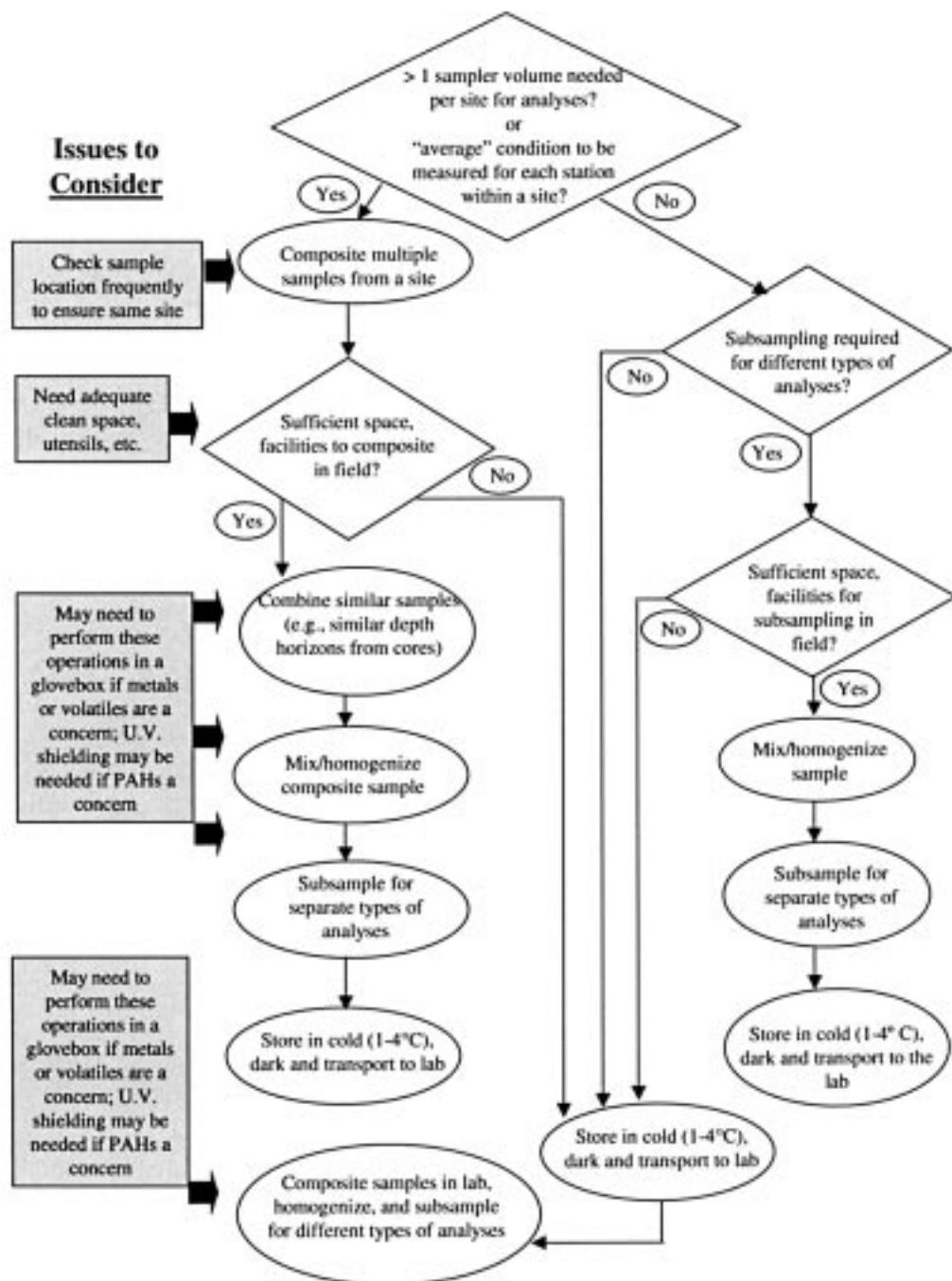


FIG. 7 Flowchart of Suggested Sediment Processing Procedures (USEPA 2001 (1))

11.2.2 Container Material:

11.2.2.1 Equipment and supplies that contact sediments or overlying water should not contain substances that can be leached or dissolved in amounts that adversely affect the test organisms or interfere with chemical or physical analyses. In addition, equipment and supplies that contact sediment or water should be chosen to minimize sorption of test materials from water. Glass, Type 316 stainless steel, nylon, high-density polyethylene, polypropylene, polycarbonate, and fluorocarbon plastics should be used whenever possible to minimize leaching, dissolution, and sorption (Test Method E 1706). 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 9 summarizes the appropriate types of sampling containers and allowable holding times for various types of contaminants associated with sediments.

11.2.2.2 In general, sediments and pore waters with multiple or unknown chemical types should be stored in containers made from high density polyethylene plastic or polytetrafluoroethylene (PTFE 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

TABLE 9 Recommended Sampling Containers, Holding Times, and Storage Conditions for Common Types of Sediment Analyses (USEPA, 1983 (52);1993(48); 2001 (1))

NOTE—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 ₂ SO ₄ , pH<2; R
Metals (except Cr or Hg)	P,G	6 months	HNO ₃ , pH<2; F
Extractable organics (including phthalates, ariosomes, organochlorine pesticides, PCBs, aromatics, isophorone, PAHs, halogenated hydrocarbons, and TCDD)	G, PTFE-lined cap	7 days (until extraction) 30 days (after extraction)	R; F
Purgables (halocarbons and aromatics)	G, PTFE-lined septum	14 days	R; F
Pesticides	G, PTFE-lined cap	7 days (until extraction) 30 days (after extraction)	R; F
Sediment Toxicity (acute and chronic)	P, PTFE	2 weeks ^A	R, dark
Bioaccumulation testing	P, PTFE	2 weeks ^A	R, dark

^A Holding time might be longer depending on the magnitude and type of contaminants present. Test Methods E 1706, E 1367 and Guide E 1688.

provided for these analyses in the event that re-analysis of the sample is required. If samples are contaminated with photoreactive compounds such as PAHs, exposure to light should be minimized by using brown glass containers or clear containers wrapped tightly with an opaque material (for example, clean aluminum foil). Plastic or acid-rinsed glass containers should be used when the chemicals of concern are heavy metals.

11.2.2.3 In general, anything coming in contact with the sediment during sample collection, processing and subsequent testing should be made of non-contaminating materials. However, in certain cases (for example, in situ testing) it may be necessary to use materials (PVC, fiberglass, etc.) that have a potential to leach contaminants. In such instances it is advisable that such materials be soaked or aged for an extended period of time (for example, 7 days) before use to reduce the amount of contaminants potentially leached from these materials (see 11.2.3.2).

11.2.3 Container Preparation:

11.2.3.1 Many vendors have commercially available pre-cleaned containers for a variety of applications. For chemical and toxicological analyses, certified pre-cleaned containers are often a cost-effective way to limit the potential for container contamination of samples. Thus, manufacturer-supplied pre-cleaned containers are often a prerequisite in QAPPs.

11.2.3.2 If new containers are used, materials should be soaked or aged before use (see 7.2, 12.2.2.3, and Test Method E 1706).

11.2.3.3 If a sample is to be refrigerated, the container should be filled to the brim to reduce oxygen exposure. This is particularly important for volatile compounds (for example, AVS). If a sample is to be frozen, the container should be filled to no more than about 90 % of its volume (about 10 % headspace) to allow for expansion of the sample during freezing. See 11.5 for preservation and storage conditions for various types of analyses. For studies in which it is important to maintain the collected sediment under anoxic conditions (for example, where metal contamination is of concern), the container should be purged with an inert gas (for example, nitrogen) before filling and then again before capping tightly. Sediment samples should never be frozen for toxicity or bioaccumulation testing (Test Method E 1706 and Guide E 1688).

11.2.3.4 All sediment containers should be properly labeled with a waterproof marker before sampling. Containers should be labeled on their sides in addition to or instead of labeling the lids. Each label should include, at a minimum, the study title, station location or sample identification, date and time of collection, sample type, and name of collector. Blind sample labeling (that is, a sample code) should be used, along with a sample log that identifies information about each sample (see 9.9) to minimize potential analytical bias. Additional information such as required analyses and any preservative used might also be included on the label although this information is typically recorded on the chain-of-custody form (see 9.9 and 15.6). Labeled containers should be stabilized in an upright position in the transport or storage container (see 11.5, Transport and Storage for further information). Extra containers should be carried on each sampling trip.

11.3 Subsampling and Compositing Samples:

11.3.1 The decision to subsample or composite sediment samples within or among stations depends on the purpose and objectives of the study, the nature and heterogeneity of the sediments, the volume of sediment required for analytical or toxicity assessment, and the degree of statistical resolution that is acceptable. Subsampling and compositing might be accomplished in the field, if facilities, space, and equipment are available, or alternatively, in a laboratory setting following sample transport Table 10.

11.3.2 General Procedures:

11.3.2.1 Subsampling is useful for collecting sediment from a specific depth of a core sample, for splitting samples among multiple laboratories, for obtaining replicates within a sample, or for forming a composite sample.

11.3.2.2 Compositing refers to combining aliquots from two or more samples and analyzing the resulting pooled sample (Keith, 1993 (49)). Compositing is often necessary when a relatively large amount of sediment is needed from each sampling site (for instance, to conduct several different physical, chemical or biological analyses). Compositing might be a practical, cost-effective way to obtain average sediment characteristics for a particular site Table 10, but not to dilute a contaminated sample. Also, if an objective of the study is to define or model physicochemical characteristics of the sediment, it might be important not to composite samples because of model input requirements (EPRI, 1999 (53)).

11.3.3 Grab Samples:

11.3.3.1 If a sediment grab sample is to be subsampled in the laboratory, the sample should be released carefully and directly into a labeled container that is the same shape as the sampler and made of a chemically-inert material (see 11.2 for recommendations on containers). The container needs to be large enough to accommodate the sediment sample and should be tightly sealed with the air excluded.

11.3.3.2 If the grab sample is to be subsampled in the field, it is desirable to subsample from the sampler directly to

TABLE 10 Recommendations for Subsampling or Compositing Sediment Samples (USEPA 2001 (1))

Overlying water should be siphoned off, not decanted, from grab samplers prior to subsampling.

All utensils that are used to process samples should be made of inert materials such as 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 h (and preferably within 24 h) of collection.

Sufficient sample homogenization, prior to placing in containers, is critical for accurate measurements and correct sediment quality determinations.

If rigorous evaluation of metal contamination is a focus of the study, or if anaerobic conditions need to be maintained for other reasons, it might be necessary to homogenize, subsample, and composite samples in an oxygen-free glovebox or other suitable apparatus.

Similar depth horizons or geologic strata should be subsampled when compositing core samples.

minimize sediment handling and associated artifacts. Therefore, the sampler should allow access to the surface of the sample without loss of water or fine-grained sediment (see 10.1 for sampler descriptions). This typically dictates the use of a grab sampler with bucket covers that are either removable or hinged to allow access to the surface of the sediment sample (for example, Ponar, VanVeen).

11.3.3.3 Before subsampling from the grab sampler, the overlying water should be removed by slow siphoning using a clean tube near one side of the sampler (WDE, 1995 (29); PSEP, 1997a (33)). If the overlying water in a sediment sampler is turbid, it should be allowed to settle if possible.

11.3.3.4 The general subsampling and compositing process for grab samples is illustrated in Fig. 8. Subsampling can be performed using a spoon or scoop made of inert, non-contaminating material. Sediment that is in direct contact with the sides of the grab sampler should be excluded as a general precaution against potential contamination from the device. Subsamples may be combined or placed into separate clean, pre-labeled containers. If the sample is to be frozen, it is advisable to leave at least about 10 % head space in the container to accommodate expansion and avoid breakage. Sediment samples should never be frozen for toxicity or bioaccumulation testing (Test Method E 1706 and Guide E 1688).

11.3.3.5 There are two alternatives for compositing sediment samples from grab samplers Fig. 8: (1) compositing and homogenizing (mixing) in the field and (2) compositing in the field and homogenizing in the laboratory.

11.3.3.6 In some studies (for example, where metals are the contaminants of concern), it might be necessary to subsample a grab sample under oxygen-free conditions to minimize oxidative changes. In these cases, a hand-coring device should be used for subsampling. The core should be inserted immediately upon retrieval of the sampler, then removed and placed into a glove box or bag which is flushed with a constant, controlled volume of inert gas. The sediment within the core can then be extruded under oxygen-free conditions into deaerated containers. The presence of oxygen during handling and storage might be relatively unimportant (Brumbaugh et al. 1994 (27)) or very important (Besser et al. 1995 (54)), depending on the sediment characteristics, the contaminants of concern, and the study objectives.

11.3.4 Core Samples:

11.3.4.1 Subsampling sediment core samples is usually done to focus the assessment on a particular sediment horizon or horizons, or to evaluate historical changes or vertical extent in contamination or sedimentation rates. Whenever subsampling of retrieved sediment cores is required, particularly for analysis of contaminants, the sediment should be extruded from the core liners and subsampled as soon as possible after collection. This can be accomplished in the field if appropriate facilities and equipment are available, or in the laboratory after transport.

11.3.4.2 Systematic subsampling Fig. 9 involves removing the sediment from the core in sections of uniform thickness. Each incremental core section corresponds to a particular sediment depth interval. In remedial dredging and geological

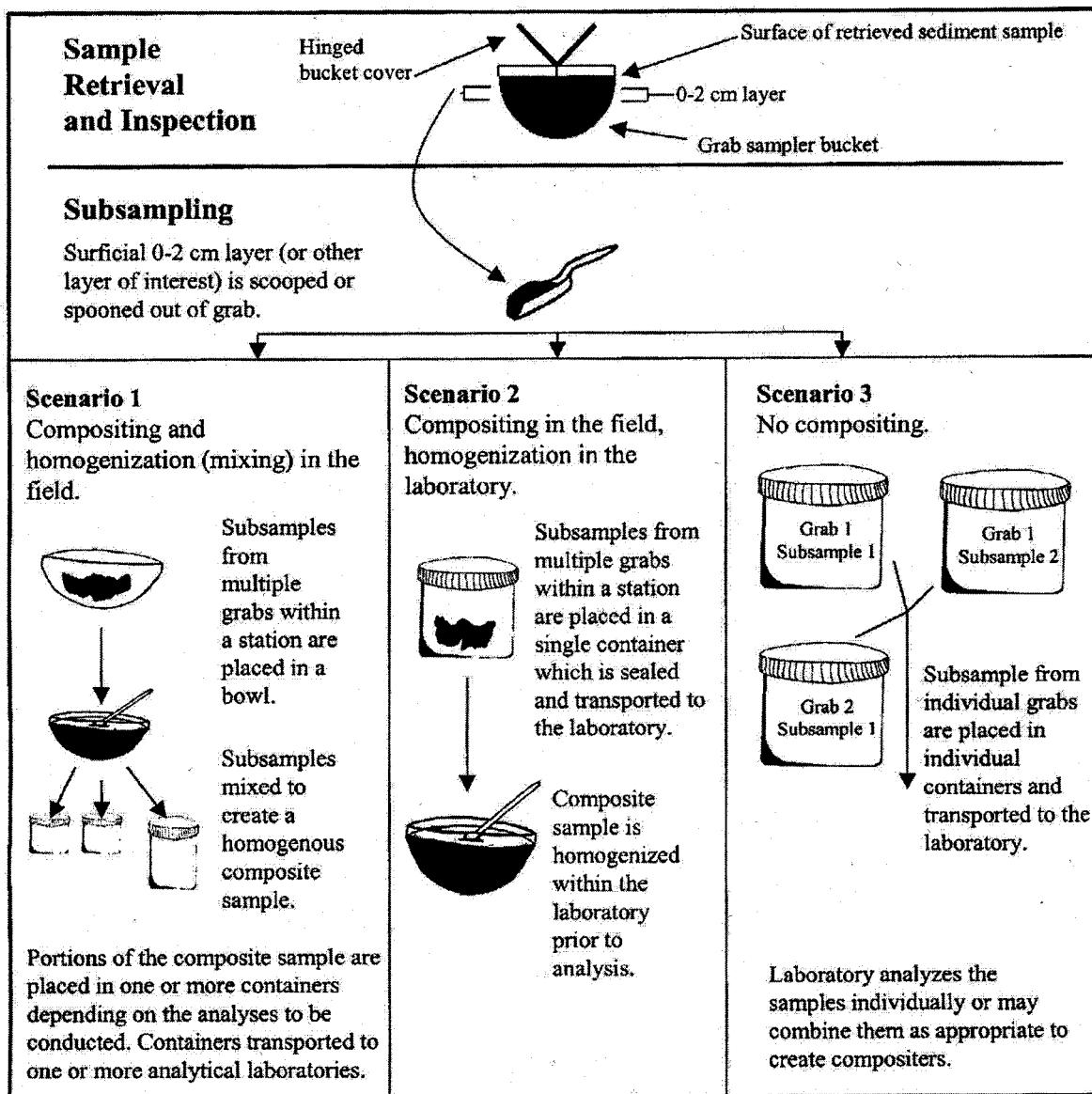


FIG. 8 Alternatives for Subsampling and Compositing Sediment Grab Samples (USEPA 2001(1))

applications, longer sections (for example, 25 to 50 cm) are typically used to characterize a site.

11.3.4.3 The depth horizon(s) sampled will depend on the study objectives as well as the nature of the substrate. For toxicological studies, the biologically active layer and sedimentation rates at the site are important factors determining which core sections are sampled. In these studies, subsampling depth intervals may include the 0 to 2 cm layer for recent deposition or greater than the 2-cm layer if the deposition rate is known to be higher, and the 0 to 5 cm or 0 to 15 cm layers for biological activity, depending on resident organisms. Many investigations have project-specific depths corresponding to study requirements, such as dredging depths for navigation or remediation dredging. In many regional or national environmental monitoring programs (for example, USEPA EMAP, NOAA Status and Trends), the uppermost surficial layer is

sampled because information on the horizontal distribution of sediment contaminants is desired (USEPA, 2000d (35), Wolfe et al. 1993 (22)).

11.3.4.4 There are various methods for subsampling sediment cores including gradual extrusion, dissection of a core using a jig saw, reciprocating saws, use of a segmented gravity corer, a hand corer, or scoops and spoons. Cutting devices range from stainless steel knives to teflon or nylon string. Note that metal saws frequently generate debris that can contaminate a sample. An electric sheet metal cutter has been used on plastic core liners or aluminum core tubes creating a ribbon of material as opposed to chips left behind with a metal saw (David Moore, MEC Analytical, Carlsbad, CA, personal communication).

11.3.4.5 A piston-type extruder that applies upward pressure on the sediment is an instrument commonly used to gradually

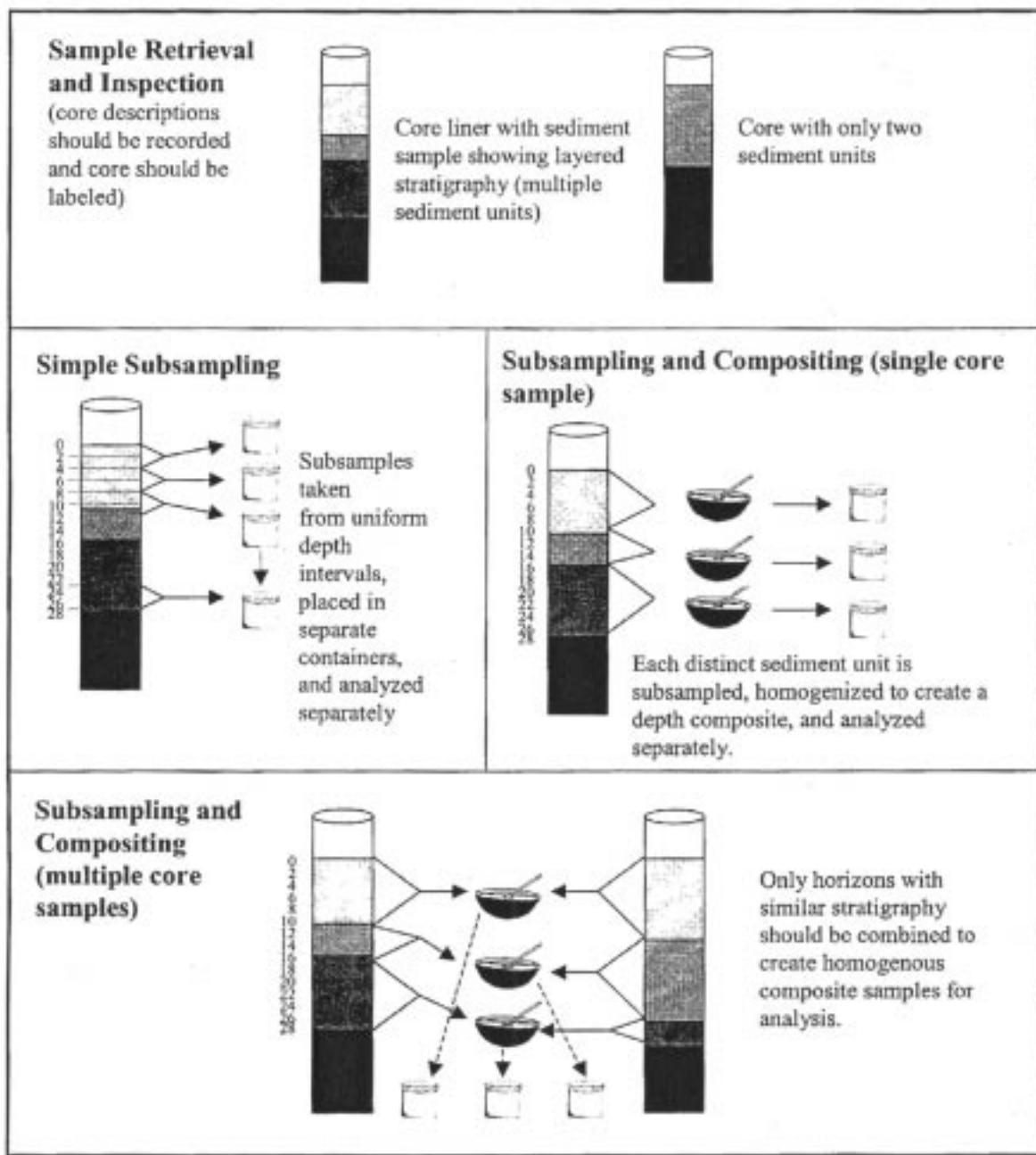


FIG. 9 Alternatives for Subsampling and Compositing Sediment Core Samples (USEPA 2001 (1))

expose a core for sectioning in some monitoring programs where specific sediment depths have been defined a priori (Kemp et al. 1971 (55)). The capped core liner containing the sediment and overlying water is uncapped at the lower end and placed vertically on top of the piston. The top cap is removed and the water is siphoned off to minimize disturbance of the sediment-water interface. The core liner is then pushed slowly down until the surface of the sediment is at the upper end of the liner. Sediment sections are collected by pushing the liner down and cutting the exposed sediment into sections of the desired thickness using a stainless steel or Teflon® cutter

(Environment Canada, 1994 (2); Mudroch and Azcue, 1995 (46)). A 1- to 2-mm outer layer of sediment that has been in contact with the plastic or metal liner should be removed and discarded, if possible, to avoid contamination. Each sediment subsample should be placed into a labeled, clean, and chemically-inert container, or, if subsamples are being composited, into an appropriately sized mixing bowl. The size of the container should be as close to the volume of the sediment as possible to minimize the head space in the container. If it is desirable to maintain an oxygen-free environment during subsampling, then all handling or manipulations should take

place in a glove box or bag filled with an inert gas and modified to accommodate the core liner through an opening (Environment Canada, 1994 (2); Mudroch and MacKnight, 1994 (36)).

11.3.4.6 Cores of more consolidated material can be mounted onto a horizontal U-shaped rail and the liner cut using a saw mounted on a depth-controlling jig. The final cut can then be made with a sharp knife to minimize contamination of the sediment by liner material, and the core itself can be sliced with Teflon® or nylon string. The core then becomes two D-shaped halves that can be easily inspected and subsampled (46). Sediment in contact with the saw blade should not be used for toxicity tests or metals analyses due to potential contamination from the saw blade. Another alternative for sectioning and subsampling is a segmented gravity corer described by Aanderaa Instruments of Victoria, BC, Canada. The core tube of the sampler consists of a series of rings placed on top of one another. Subsampling is carried out by rotating the rings around its other axis so that it cuts sediment layers of similar thickness. This segmented core tube is suitable for sampling fine-grained sediments and allows one person in the field to subsample the core into 1-cm sections (Mudroch and Azcue 1995 (46)).

11.3.4.7 Sediment from box-core samples can be effectively subsampled with a small hand corer after the overlying water has been carefully siphoned off and discarded. Hand corers with small inner diameters less than 3 cm tend to compact sediments, so this equipment needs to be used with care. Spoons or scoops have also been used to subsample surface sediments from a box corer (Environment Canada, 1994 (2)).

11.3.4.8 Like grab samples, core samples may be composited or subsampled in the field or laboratory after evaluating them for acceptability. Although there might be occasions when it is desirable to composite incremental core depths, only horizons of similar stratigraphy should be composited. Depending on the study objectives and desired sampling resolution, individual horizons within a single core can be homogenized to create one or more “depth composites” for that core, or corresponding horizons from two or more cores might be composited Fig. 9. Composite samples should be homogenized before analysis or testing.

11.4 Homogenization:

11.4.1 Homogenization refers to the complete mixing of sediment to obtain consistency of physicochemical properties throughout the sample before using in analyses. Homogenization is typically performed on individual samples, as well as on composited samples and can be done either in the field or the laboratory Table 11.

11.4.2 Depending on the objective of the study, unrepresentative materials (for example, twigs, shells, leaves, stones, wood chips and sea grass) might be removed and documented before homogenization (see 12.3 for techniques to remove unrepresentative material). The need for removal of larger matter depends on the analyses to be conducted.

11.4.3 Mixing should be performed as quickly and efficiently as possible, because prolonged mixing can alter the particle-size distribution in a sample and cause oxidation of the sediments (Ditsworth et al. 1990 (56); Stemmer et al. 1990a,b (57), (58)). This can alter the bioavailability of contaminants,

TABLE 11 Recommendations for Homogenizing Sediment Samples (USEPA 2001 (1))

Use a sufficiently large, precleaned glass or stainless steel mixing bowl to homogenize the sample.
Use clean glass polyethylene, or stainless steel implements (for example, spoon) to mix sediment.
Mixing should be performed as quickly and efficiently as possible while attempting to reduce oxidation of the sample.
Intensive manual mixing of wet sediment, in a suitably large container, is usually sufficient to homogenize the sample Ingersoll and Nelson, 1990 (103).
Regardless of the mixing method selected, the effectiveness of the method should be demonstrated using a homogenate replicate

particularly metals, by increasing or decreasing their availability Ankley et al. 1996 (51)). If metal contaminants or volatile chemicals are a concern, samples should be mixed in a glove box under an inert atmosphere and quickly partitioned into sample containers for analysis.

11.4.4 Homogenate replicates consist of two or more subsamples, taken from different locations within a mixed sample, and then comparing analytical results of the replicate samples (sometimes called a split sample). After the sediment has been homogenized, it is generally partitioned among sample containers. Partitioning sediments for chemical or biological testing may be accomplished using various methods. In one method, a number of small portions are removed from random locations in the mixing container and distributed randomly in all sample jars until the appropriate volume of sediment is contained in each sample jar for each analysis. During distribution, the sediment can be periodically mixed using a glass rod or porcelain spatula to minimize stratification effects due to differential settling, especially if the sediment is prone to rapid settling. An alternative is to use a splitter box designed to contain and then divide the homogenized sediment.

11.5 Sample Transport and Storage:

11.5.1 Transport and storage methods should be designed to maintain structural and chemical qualities of sediment samples. Sediments collected using grab samplers are usually transferred from the sampler to containers that may or may not serve as the storage container. The containers might be stored temporarily in the field or they might be transported immediately to a laboratory for storage. If sediment core samples are not sectioned or subsampled in the field, they may be stored upright, in the core liner, for intact transportation to the laboratory. If sectioning or subsampling takes place in the field, then the subsamples may also be transferred to sample containers and stored temporarily. The sample containers with the field-collected sediments are then placed into a transport container and shipped to the laboratory. Proper storage conditions Table 9 should be achieved as quickly as possible after sampling. For those parameters that are preserved via refrigeration (for example, toxicity or bioaccumulation tests), samples should be stored in the field in refrigerated units on board the sampling vessel or in insulated containers containing ice or frozen ice packs. Sediment samples should never be frozen for toxicity or bioaccumulation testing (Test Method E 1706 and Guide E 1688).

11.5.2 For samples that can be preserved via freezing (for example, some metal and organic chemical analyses), dry ice can be used to freeze samples for temporary storage and transport (USEPA, 1983 (52), 1993 (48)). Pelletized dry ice has been used effectively to store core samples. It is important to know chilling capacities and efficiencies to determine that temperature regulation is adequate. Care should be taken to prevent refrigerated samples from freezing and to keep frozen samples from thawing. Freezing changes the sediment volume depending on the water content, and it permanently changes the structure of the sediment and potentially alters the bioavailability of sediment associated contaminants (Test Method E 1706).

11.5.3 Logistics for sample transport will be specifically tailored to each study. In some cases it is most efficient to transfer samples to a local storage facility where they can be either frozen or refrigerated. Depending on the logistics of the operation, field personnel can transport samples to the laboratory themselves or can use an overnight courier service. If a freight carrier is employed, the user needs to be aware of any potentially limiting regulations (for example, regarding the use of ice or dry ice). Samples should be cooled to that temperature before placement in the transport container. Light should be excluded from the transport container.

11.5.4 Core samples should be transported as intact core liners (tubes). Before sample transport, the entire space over the sediment in the core liner should be filled with site water, and both ends of the core liner should be completely sealed to prevent mixing of the sediment inside. The cores should be maintained in an upright position particularly if the sample is not highly consolidated material, and secured in either a transport container (for example, cooler or insulated box) with ice or ice packs, or in a refrigerated unit that can maintain a temperature near 4°C (Environment Canada, 1994 (2)). If the transport container cannot accommodate long core samples such as from vibracores or piston corers (core liners > 1 m), then the core samples can be cut into 1-m lengths, and the ends securely capped such that no air is trapped inside the liners (see 11.4).

11.5.5 Impregnating unconsolidated sediment cores with epoxy or polyester resins will preserve sediment structure and texture (Ginsburg et al. 1966 (59); Crevello et al. 1981 (60)), but not the chemical characteristics of the sediment. Therefore, this procedure should not be used for transporting or storing sediment samples for chemical characterization or biological testing (Environment Canada, 1994 (2)).

11.6 Sample Holding Times:

11.6.1 Because the chemicals of concern influencing sediment characteristics are not always known, it is desirable to hold the sediments after collection in the dark at 4°C (Test Method E 1706). Traditional convention has held that toxicity or bioaccumulation tests should be started as soon as possible following collection from the field, although actual recommended storage times range from two weeks (USEPA 2001 (1)) to less than eight weeks (USEPA-USACE 1998 (61)). Discrepancies in recommended storage times reflected a lack of data concerning the effects of long-term storage on the physical, chemical, and toxicological characteristics of the

sediment. However, numerous studies have recently been conducted to address issues related to sediment storage (Dillon et al., 1994 (62); Becker et al., 1995 (63), Carr and Chapman, 1995 (64), Moore et al., 1996 (65), Sarda and Burton, 1995 (66), Sijm et al., 1997 (67), DeFoe and Ankley, 1998 (68)). The conclusions and recommendations offered by these studies vary substantially and appear to depend primarily upon the type or class of chemical(s) present. Considered collectively, these studies suggest that the recommended guidance that sediments be tested sometime between the time of collection and 8 weeks storage is appropriate. Additional guidance is provided below.

11.6.2 Extended storage of sediments that contain high concentrations of labile chemicals (for example, ammonia, volatile organics) may lead to a loss of these chemicals and a corresponding reduction in toxicity. Under these circumstances, the sediment should be tested as soon as possible after collection, but not later than within two weeks (Sarda and Burton, 1995 (66)). Sediments that exhibit low-level to moderate toxicity can exhibit considerable temporal variability in toxicity, although the direction of change is often unpredictable (Carr and Chapman, 1995 (64); Moore et al., 1996 (65); DeFoe and Ankley, 1998 (68)). For these types of sediments, the recommended storage time of <8 weeks may be most appropriate. In some situations, a minimum storage period for low-to-moderately contaminated sediments may help reduce variability. For example, DeFoe and Ankley, 1998 (68) observed high variability in survival during early testing periods (for example, <2 weeks) in sediments with low toxicity. De Foe and Ankley, 1998 (68) hypothesized that this variability partially reflected the presence of indigenous predators that remained alive during this relatively short storage period. Thus, if predatory species are known to exist, and the sediment does not contain labile contaminants, it may be desirable to store the sediment for a short period before testing (for example, 2 weeks) to reduce potential for interferences from indigenous organisms. Sediments that contain comparatively stable compounds (for example, high molecular weight compounds such as PCBs) or which exhibit a moderate-to-high level of toxicity, typically do not vary appreciably in toxicity in relation to storage duration (Moore et al., 1996 (65), DeFoe and Ankley, 1998 (68)). For these sediments, long-term storage (for example, >8 weeks) can be undertaken.

11.6.3 Researchers may wish to conduct additional characterizations of sediment to evaluate possible effects of storage. Concentrations of chemicals of concern could be measured periodically in pore water during the storage period and at the start of the sediment test Kemble et al., 1994 (69). Ingersoll et al., 1993 (70) recommend conducting a toxicity test with pore water within two weeks from sediment collection and at the start of the sediment test. Freezing might further change sediment properties such as grain size or chemical partitioning and should be avoided (Schuytema et al., 1989 (71)). Sediment should be stored with no air over the sealed samples (no head space) at 4°C before the start of a test (Shuba et al., 1978 (72)). Sediment should be stored in containers constructed of suitable materials as outlined in 11.2.

11.6.4 Sediment cores collected for stratigraphical or geological studies can be stored at 4°C in a humidity-controlled room for several months without any substantial changes in sediment properties (Mudroch and Azcue, 1995 (46)).

12. Sample Manipulations

12.1 Manipulation of sediments in the laboratory is often required to achieve certain desired characteristics or forms of material for toxicity or bioaccumulation testing and chemical analysis. As all manipulation procedures alter some qualities of field samples, it is important to evaluate the effect that these changes might have on the study objective and on each measurement. Therefore, all procedures used to prepare sediment samples should be described in the study plan and documented. Generally, manipulation procedures should be designed to maintain sample representativeness in terms of toxicity and chemistry by minimizing procedural artifacts.

12.1.1 This section discusses methods for several common manipulations performed in the laboratory including sieving, spiking, organic carbon modification and formulated sediments, sediment dilution, and elutriate preparation. Other sediment manipulations, such as salinity adjustments or pre-treatment of sediment ammonia (done in conjunction with toxicity testing in certain regulatory programs) are not discussed in this standard as these are described elsewhere (for example, PSEP, 1995 (73), USEPA 1994 (74)).

12.2 Sieving:

12.2.1 In general, sieving should not be done on sediment samples because this process can change the physicochemical characteristics of the sediment sample. For example, wet sieving of sediment through fine mesh (=500 µm openings) has been shown to result in decreased percent total organic carbon and decreased concentrations of total PCBs, which might have been associated with fine suspended organic matter lost during the sieving process (Day et al. 1995 (75)). Sieving can also disrupt the natural chemical equilibrium by homogenizing or otherwise changing the biological activity within the sediment (Environment Canada, 1994 (2); Test Method E 1706).

12.2.2 In some cases, however, sieving might be necessary to remove indigenous organisms, which can interfere with subsequent toxicity testing and confound interpretations of analytical results (USEPA, 1994 (74); 2000d (35); Practice D 3976). Indigenous organisms can be problematic in toxicity testing because they may be the same species as the test organism, they may be a species similar in appearance to test organisms, or they might prey on the test organisms. Similarly, in bioaccumulation tests, indigenous organisms might be similar in appearance to the test organisms (Test Method E 1706 and Guide E 1688).

12.2.3 If sieving is performed, it should be done for all samples to be tested, including control and reference sediments, if the objective of the study is to compare results among stations (Test Method E 1706). It might be desirable to obtain certain measurements (for example, dissolved and total organic carbon, acid volatile sulfide [AVS], and simultaneously extracted metals [SEM]) both before and after manipulation, to document changes associated with sieving (USEPA, 2000d (35)). In addition, it might be desirable to document the effect of sieving on the sediment sample by conducting comparative

toxicity tests using sieved and unsieved sediment (Environment Canada, 1994 (2)).

12.2.4 Sieving Methods:

12.2.4.1 *Press Sieving*—If sieving is necessary, press sieving is the preferred method. In this method, sediment particles are hand-pressed through a sieve using chemically inert paddles (Giesy et al. 1990 (76); Johns et al. 1991 (77)). Matter retained by the screen, such as organisms, shell fragments, gravel, and debris, should be recorded in a log book and discarded (USEPA/USACE, 1991 (32)). Samples with high debris, vegetation, or clay content might be difficult to press through a single sieve with a mesh size less than 1 mm; such samples might need to be pressed through a series of sieves with progressively smaller openings. Water should not be added to sediment when press sieving, as this could result in changes in contaminant concentration and bioavailability. Samples that are going to be used for both chemical analysis and toxicity or bioaccumulation tests should be sieved together, homogenized, and then split for their respective analyses.

12.2.4.2 *Wet Sieving*—If sediments cannot be hand-pressed sieved, wet sieving might be required, however, this type of sieving increases the likelihood of contaminant loss. Wet sieving involves swirling sediment particles within a sieve using water to facilitate the mechanical separation of smaller from larger particles. A slurry made with water that has separated from the sediment during storage or transport might be sufficient to wash particles through the sieve. Wet samples that might have settled during transit should be stirred to incorporate as much field water as possible. In some cases, addition of a small volume of site water, deionized water, or reconstituted water to the wet sample might be required. Mechanical shakers or stirring with a nylon brush can also facilitate wet sieving (Mudroch and MacKnight, 1994 (36)).

12.2.4.3 In general, smaller mesh sieves are preferred to reduce loss of fines. Sieves made of stainless steel, or plastic woven polymers (for example, polyethylene, polypropylene, nylon, and Teflon) with mesh sizes that vary from 0.24 to 2.0 mm have been used to sieve sediment for toxicity tests (Keilty et al. 1988a;b; (78),(79); Giesy et al. 1990 (76); Lydy et al. 1990(81); Stemmer et al. 1990a;b (57), (58); Johns et al. 1991 (77); Landrum and Faust, 1991 (80)). Non-metallic sieves are preferred if metals are of interest. Stainless steel sieves are acceptable if organic compounds are of interest. Stainless steel (provided the mesh is not soldered or welded to the frame), nylon, or Nitex-type plastic sieves should be used when other inorganic constituents are of concern or are to be analyzed (PSEP 1995) (73).

12.2.4.4 Generally, sieving through a 10-mesh (2-mm openings) sieve is acceptable as a basis to discriminate between sediment and other materials. For toxicity testing, a mesh size of 1.0 mm has been used (Environment Canada, 1994 (2)) which will remove most adult amphipods. However, a mesh of 0.25 mm might be needed to remove immature amphipods and most macrofauna (Landrum et al. 1992 (82); Robinson et al. 1988 (83); Day et al. 1995 (75)). In marine sediments, sieves with a mesh size of 0.5 mm are effective in removing most of the immature amphipods (Swartz et al. 1990 (84); PSEP, 1995 (73)).

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

12.3 Formulated Sediment and Organic Carbon Modification:

12.3.1 Formulated Sediments—Formulated sediments (also called reconstituted, artificial, or synthetic sediments) are mixtures of materials that mimic the physical components of natural sediments (Test Method E 1706). While they have not been used routinely, formulated sediments potentially offer advantages over natural sediments for use in chemical fate and biological effects testing. However, formulated sediments also have limitations. They do not possess the natural microbial, meiofaunal, and macrofaunal communities or the complex organic and inorganic gradients prevalent in natural sediments. The lack of biological activity, diagenesis, and oxidation-reduction (redox) potential gradients undoubtedly alters some sorption and desorption properties, which might in turn alter contaminant fate and effects. The current lack of understanding of physicochemical controls on bioavailability in different sediment environments precludes broad-scale use of formulated sediments (Test Method E 1706).

12.3.2 A formulated sediment should: (1) support the survival, growth, or reproduction of a variety of benthic invertebrates, (2) provide consistent acceptable biological endpoints for a variety of species, and (3) be composed of materials that have consistent characteristics (USEPA, 2000d (35), Test Method E 1706). Characteristics should include: (1) consistency of materials from batch to batch, (2) contaminant concentrations below concentrations of concern, and (3) availability to all individuals and facilities (Kemble et al. 1999 (85)). Physicochemical characteristics that might be considered when evaluating the appropriateness of a sediment formulation include percent sand/clay/silt, organic carbon content, cation exchange capacity (CEC), redox potential, pH, and carbon:nitrogen:phosphorous ratios (USEPA, 2000d (35); Test Method E 1706).

12.3.3 The specific material source should be carefully selected, as characteristics can vary significantly among product types. For example, USEPA (2000d (35)) found that for three different sources of kaolinite clay, the percentage of clay ranged from 57 to 89 %, depending on individual product

specifications. There are a number of suppliers of various sediment components (USEPA, 2000d (35)). A critical component of formulated sediments is the source of organic carbon. It is not clear that any one source of organic carbon is routinely superior to another source (Test Method E 1706).

12.3.4 Organic Carbon Modification—Organic carbon content of natural as well as formulated sediments can be modified to assess the effect on contaminant fate and bioavailability. Many studies have modified sediment carbon because total organic carbon (TOC) content has been shown to be a major determinant of non-ionic organic chemical bioavailability (Di Toro et al. 1991 (86); DeWitt et al. 1992 (87); and Kosian et al. 1999 (88)). While TOC modifications might be necessary to achieve study objectives, it should be recognized that organic carbon manipulations can change the particle composition and size distribution, thereby potentially affecting contaminant equilibrium. Thus, results from such experiments should be interpreted with care. Also, the sample needs to be equilibrated (see 12.4.1) following addition of the new source of organic carbon, before conducting analyses.

12.3.5 Some recipes have used peat as the source of organic carbon, however, the quality and characteristics of peat moss can vary from bag to bag (Test Method E 1706). Other sources of organic carbon include humus, potting soil, maple leaves, composted cow manure, rabbit chow, cereal leaves, chlorella, trout chow, Tetramin®, Tetrafin®, and alpha cellulose. Of these, only peat, humus, potting soil, composted cow manure, and alpha cellulose have been used successfully in sediment testing without fouling the overlying water; other sources have caused dissolved oxygen concentrations to fall to unacceptable levels (Kemble et al. 1999 (85)).

12.3.6 Five studies compared organic carbon sources in formulated sediments. A study of 31 different organic carbon recipes by Environment Canada (1995) (89) compared effects on sediment homogeneity, density, and turbidity. Cerophyll and trout chow were selected as the optimal organic carbon sources with high clay (kaolin at 50 or 75 % total concentration) and fine sand.

12.3.7 Ribeiro et al. (1994) (90) suggested the use of synthetic alpha-cellulose as a carbon source amended with humic acid. The use of alpha-cellulose in formulated sediment has since been evaluated by Kemble et al. (1999 (85), Sawyer and Burton (1994 (91), and Fleming and Nixon (1996 (92)). Ribeiro et al. (1994 (90)) found that sorption was dependent on the amount of organic carbon present. Kemble et al. (1999 (85)) found that growth of *Hyalella azteca* was better in 10 % than in 2 % alpha-cellulose. Both alpha-cellulose and conditioned red maple leaves were found to be suitable as organic carbon amendments for reference toxicant testing with *Hyalella azteca* (96 h exposures) when spiked with cadmium, zinc, or anthracene (Sawyer and Burton, 1994 (91)).

12.3.8 Use of alpha cellulose as a carbon source for sediment-spiking studies has not been adequately evaluated, but it appears to be promising. Alpha cellulose is a consistent source of organic carbon that is relatively biologically inactive and low in concentrations of chemicals of concern. Furthermore, Kemble et al. (1999 (85)) reported that conditioning of formulated sediment was not necessary when alpha cellulose

was used as a carbon source for a negative control sediment. Compared with other sources of organic carbon, alpha cellulose is highly polymerized and would not serve as a food source, but rather would serve to add texture or provide a partitioning compartment for chemicals. Reductions in organic carbon content have been achieved by diluting sediment with clean sand (see 12.5; Clark et al. 1986 (93); Clark et al. 1987 (94); Tatem, 1986(95); Knezovich and Harrison, 1988) (96)). However, this can change sediment characteristics resulting in non-linear responses in toxicity (Nelson et al. 1993 (97)). Combustion has also been used to remove fractions of organic carbon (Adams et al. 1985 (98); IJC, 1988 (99)). However, this method results in substantial modification of the sediment characteristics, including oxidization of some inorganic components.

12.3.9 The ratio of carbon to nitrogen to phosphorous might be an important parameter to consider when selecting an organic carbon source. This ratio can vary widely among carbon sources (Test Method E 1706, USEPA 2000d (35)). For example, carbon can range from 30 to 47 %, nitrogen from 0.7 to 45 mg/g, and phosphorous from below detection limits to 11 µg/g for several different carbon sources (USEPA, 2000d (35)).

12.3.10 A variety of formulations have been used successfully in sediment toxicity testing (Test Method E 1706 and USEPA 2000d (35)). At this time, no one formulation appears to be universally better than others.

12.4 Sediment Spiking:

12.4.1 Test sediment can be prepared by manipulating the properties of a control or reference sediment (Test Method E 1706). Mixing time (Stemmer 1990a, 1990b (57) (58)) and aging (Landrum 1989, Word et al. 1987, Landrum and Faust 1992(100),(101),(102)) of spiked sediment can affect bioavailability of chemicals in sediment. Many studies with spiked sediment are often started only a few days after the chemical has been added to the sediment. This short time period may not be long enough for sediments to equilibrate with the spiked chemicals. Consistent spiking procedures should be followed in order to make interlaboratory comparisons. Limited studies have been conducted comparing appropriate methods for spiking chemicals in sediment. Additional research is needed before more definitive recommendations for spiking of sediment can be outlined in this standard. The guidance provided in the following sections has been developed from a variety of sources. Spiking procedures that have been developed using one sediment or test organism may not be applicable to other sediments or test organisms.

12.4.2 Spiking involves adding one or more chemicals to sediment for either experimental or quality control purposes. Spiking environmental samples is used to document recoveries of an analyte and thereby analytical bias. Spiked sediments are used in toxicity tests to determine effects of material(s) on test species. The cause of sediment toxicity and the interactive effects of chemicals can be determined by spiking a sediment with chemicals or complex waste mixtures (97). Sediments spiked with a range of concentrations can be used to generate either point estimates (for example, LC₅₀) or a minimum concentration at which effects are observed (lowest-observable-effect concentration; LOEC). Results of tests may

be reported in terms of a Biota-sediment accumulation factor (BSAF) Ankley et al., 1992b (104). The influence of sediment physico-chemical characteristics on chemical toxicity can also be determined with sediment-spiking studies Swartz et al., 1994(105). Spiking tests can also provide information concerning chemical interactions and transformation rates. The design of spiking experiments, and interpretation of results, should always consider the ability of the sediment to sequester contaminants, recognizing that this governs many chemical and biological processes (O'Donnell et al. 1985 (106); Stemmer et al. 1990a,b (57),(58); Northcott and Jones, 2000 (107), Test Method E 1706). In preparation for toxicity and bioaccumulation tests, references regarding the choice of test concentrations should be consulted (USEPA 2000d (35), Environment Canada 1995 (89), Test Method E 1706). Table 12 summarizes general recommendations for spiking sediments with a chemical or other test materials.

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

12.4.4 The test material(s) should be at least reagent grade, unless a test using a formulated commercial product, technical-grade, or use-grade material is specifically needed. Before a test is started, the following should be known about the test

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

Regardless of the spiking technique used, care should be taken to ensure complete and homogenous mixing.

Replicate subsamples should be analyzed to confirm homogeneous mixing.

Moisture content should be determined on triplicates for each sample so that the spike concentration can be normalized on a dry weight basis.

Wet spiking is recommended over dry spiking methods.

Generally speaking, the jar rolling method is more suitable than hand mixing for spiking larger batches of sediment.

To ensure chemical equilibrium between the sediment and pore water in toxicity testing, spike sediments should be stored for at least one month, unless other information is available for the spiking material and sediment type.

Direct addition of organic solvent carriers should be avoided because they might alter sediment chemistry and affect contaminant bioavailability. Shell coating methods should be used instead as this eliminates many of the disadvantages of solvent carriers.

material: (1) the identity and concentration of major ingredients and impurities, (2) water solubility in test water, (3) log Kow, BCF (from other test species), persistence, hydrolysis, and photolysis rates of the test substrate, (4) estimated toxicity to the test organism and to humans, (5) if the test concentration(s) are to be measured, the precision and bias of the analytical method at the planned concentration(s) of the test material, and (6) recommended handling and disposal procedures. Addition of test material(s) to sediment may be accomplished using various methods, such as a: (1) rolling mill, (2) feed mixer, or (3) hand mixing. Modifications of the mixing techniques might be necessary to allow time for a test material to equilibrate with the sediment. Mixing time of spiked sediment should be limited from minutes to a few hours, and temperature should be kept low to minimize potential changes in the physico-chemical and microbial characteristics of the sediment USEPA, 2000 (108). Duration of contact between the chemical and sediment can affect partitioning and bioavailability Word et al., 1987 (101). Care should be taken to evenly distributed the spiked material in the sediment. Analyses of sediment subsamples is advisable to determine the degree of mixing homogeneity Ditworth et al., 1990(109). Moreover, results from sediment-spiking studies should be compared with the response of test organisms to chemical concentrations in natural sediments (144).

12.4.5 Organic chemicals have been added: (1) directly in a dry (crystalline) form; (2) coated on the inside walls of the container (Ditworth et al. (109)); or (3) coated onto silica sand (for example, 5 % w/w of sediment) which is added to the sediment (D.R. Mount, USEPA, Duluth, MN, personal communication). In techniques 2 and 3, the chemical is dissolved in solvent, placed in a glass spiking container (with or without sand), then the solvent is slowly evaporated. The advantage of these three approaches is that no solvent is introduced to the sediment, only the chemical being spiked. When testing spiked sediments, procedural blanks (sediments that have been handled in the same way, including solvent addition and evaporation, but contain no added chemical) should be tested in addition to regular negative controls. Metals are generally added in an aqueous solution (Di Toro et al. (111)). Ammonia has also been successfully spiked using aqueous solutions (Besser et al. (112)). Spiking blanks should also be included in these analyses.

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

12.4.7 If the test contains both a negative control and a solvent control, the survival, growth, or reproduction of the organisms tested should be compared in the two controls. If a

statistically significant difference is detected between the two controls, only the solvent control may be used for meeting the acceptability of the test and as the basis for calculation of results. The negative control might provide additional information on the general health of the organisms tested. If no statistically significant difference is detected, the data from both controls should be used for meeting the acceptability of the test and as the basis for calculation of results (Guide E 1241 and Test Method E 1706). If performance in the solvent control is markedly different from that in the negative control, it is possible that the data are compromised by experimental artifacts and may not accurately reflect the toxicity of the chemical in natural sediments.

12.4.8 Preparation for Spiking:

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

12.4.8.2 Regardless of the spiking technique used, care should be taken to homogenize the sediment. Chemical analyses should be conducted to verify that concentrations of the spiked contaminants are uniform throughout the mixed material. Three or more subsamples of the spiked sediment should be randomly collected to determine the concentration of the substance being tested. In general, the coefficient of variation (CV) should be = 20 % for homogeneity of mixing to be considered sufficient (Northcott and Jones, 2000 (107)).

12.4.8.3 Temperatures should be kept cool during spiking preparation (for example, 4°C) due to rapid physicochemical and microbiological alterations which might occur in the sediment that, in turn, might alter bioavailability and toxicity (Test Method E 1706, Environment Canada 1995 (89)). If spiking PAH compounds, it might be important to conduct spiking in the dark, or at least under low light as PAH toxicity has been shown to increase under ultraviolet light (Ankley et al. 1994 (113)).

12.4.8.4 A subsample of the spiked sediment should be analyzed for at least the following parameters: moisture content, pH, ammonia, total organic carbon (TOC), acid volatile sulfide (AVS), particle size distribution, and background levels of the chemical(s) to be spiked. Further characterization may include analyses of total volatile residue, pore water salinity (before and after any sieving), chemical oxygen demand, sediment oxygen demand, oxidation-reduction potential (Eh), metals, total chlorinated organic content, chlorinated organic compounds, and polycyclic aromatic hydrocarbons (see Section 15 for more information on physicochemical parameters often measured on sediments). It is particularly important to determine the TOC concentration if the sediment is to be spiked with a non-ionic organic compound, as organic carbon is the primary binding phase for such compounds (Di Toro et al. 1990 (50)). Similarly, the concentration of AVS (the primary binding phase for cationic metals in anoxic sediments) and TOC should be measured after spiking with a cationic metal (Ankley et al. 1996 (51); Leonard et al. 1999 (114)). The

organic carbon composition may also be an important characteristic to determine in the sediment (for example, the C:N ratio; Landrum et al. 1997 (115)). Further, bioavailability may be more controlled by the desorption characteristics of the compound from sediment (for example, this can be measured by a Tenax® desorption method that appears to correlate well with bioaccumulation; Ten Hulscher et al. 2003 (116)).

12.4.8.5 The sediment moisture content measurement is used to calculate the amount of chemical spiked on a dry weight basis. Generally, the moisture content should be determined on triplicates for each sample by measuring the weight lost following 24 h of oven-drying at 105°C. After drying, the samples should be cooled to room temperature in a desiccator before taking dry weight measurements (Yee et al. 1992 (117)). The mean wet density, expressed as mg water/cm³, is measured by using the same drying method on known sediment volumes. This allows spiking to be normalized from a volume basis to an equivalent dry weight basis.

12.4.9 Methods for Spiking:

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

12.4.9.2 In addition to the above techniques, sediments may be spiked by hand stirring using a scoop or spatula, as long as the homogeneity of the mixture is verified. Eberbach and gyro-rotary shakers have also been used effectively to mix spiked sediments (Stemmer et al. 1990a (57)). Less commonly, chemical(s) are added to the water overlying the sediment and allowed to sorb with no mixing (Stephenson and Kane, 1984; (118) O'Neill et al. 1985 (119); Crossland and Wolff, 1985 (120); Pritchard et al. 1986 (121)).

12.4.9.3 *Sediment Rolling*—This sediment rolling technique requires a specific jar-rolling apparatus (for example, Ditsworth et al. 1990 (56)). Many other jar-rolling apparatuses are available, ranging in size and options available. This “rolling mill” method has been used to homogenize large volumes of sediments spiked with metals and non-ionic organic compounds. The primary disadvantage of this method is that the mixing apparatus needs to be constructed or purchased. The jar-rolling apparatus used by Ditsworth et al. (1990 (56)) consists of eight parallel, horizontal rollers powered by an electric motor through a reduction gear, belts, and pulleys, which rotate cylindrical vessels containing the substrate mixtures. Mixing is accomplished gravimetrically by slowly rolling the jars (gallon-sized jars can be rolled at about 15 revolutions per minute). Optimally wetted, individual substrate particles adhere to each other and to the wall of the revolving jar until they cascade or tumble down the surface of the substrate mass. Water may be added to the substrate before rolling to adjust the sediment-to-water ratio for optimal mixing. If oxidation is a concern (for example, if the sample will be analyzed for metals), jar contents might need to be main-

tained in an inert atmosphere. If PAHs are of concern then jars should be shielded from light (Ankley et al. 1994 (113)).

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

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

12.4.9.6 After rolling, the jars should be gently shaken to settle sediment that adhered to the walls. They may be set upright and stored overnight in the dark at room temperature or at an alternate temperature (for example, 4°C) depending on the study objectives. After equilibration (see 12.4.10) and before distributing the sample to test chambers, additional rolling for two hours will help integrate interstitial water into the sediment.

12.4.9.7 *Sediment Suspension Spiking*—The sediment suspension technique (Cairns et al. 1984 (122); Schuytema et al. 1984 (123); Stemmer et al. 1990a; b (57), (58); Landrum and Faust, 1991(80); Landrum et al. 1992 (82)) is the simplest of the three spiking techniques and requires the least equipment. The method involves placing water and sediment together in a 1-L beaker. The desired amount of toxicant, dissolved in water, is added to the beaker. The mixture should be stirred at a moderate speed with a stir bar, or mechanical stirrer, for a minimum of four hours. The sediment in the beakers should then be allowed to settle and equilibrated at the appropriate test temperature as specified in the method. The excess water overlying the sediment is decanted and discarded, and the sediment is distributed to the test containers (Environment Canada, 1995 (89)).

12.4.9.8 *Slurry Spiking*—The slurry technique (Birge et al., 1987 (124); Francis et al., 1984 (125); Landrum and Faust, 1991 (80); Landrum et al., 1992 (82)) requires a minimum of

equipment and involves less water than the sediment suspension technique. A 250-g dry weight sample of sediment is placed in a 500-mL Erlenmeyer flask. Via a 25-mL aliquot of distilled, deionized water, a sufficient concentration of the materials of interest is added to obtain the desired sediment concentration (mg/kg, dry weight basis). Control (unspiked) sediment receives a 25-mL aliquot of distilled, deionized water having no added materials. The sealed flask may be mixed using various methods such as continuous agitation in a shaker for five days (Birge et al. 1987 (124)) or vigorous shaking for 60 s, twice daily for seven days (Francis et al. 1984 (125)). Following mixing, the sediment suspensions should be centrifuged to remove water. The moisture content of the sediment should be about 15 to 20 % after centrifugation. After removal of excess water, the prepared sediment can be placed in the exposure chambers and covered with water according to the specific methods. This procedure often yields sediment having its original moisture content.

12.4.10 Equilibration Time:

12.4.10.1 Before distributing the spiked sediment to containers for toxicity or bioaccumulation testing, or chemical analyses, the spiked sediments should be stored for a sufficient time to approach chemical equilibrium in the test material between the sediment and interstitial water (see 12.4.6). Equilibration times for spiked sediments vary widely among studies (Burton, 1991 (126)), depending on the spiking material and sediment type. For metals, equilibration time can be as short as 24 h (Jenne and Zachara, 1984 (127); Nebeker et al. 1986 (128)), but one to two weeks is more typical (Test Method E 1706). For organic compounds with low octanol-water partition coefficients (*K_{ow}*), equilibration times as short as 24 h have been used (DeWitt et al. 1989 (293)). Some organic contaminants might undergo rapid microbiological degradation depending on the microbial population present in the sample. In these cases, knowledge of microbial effects might be important in defining an appropriate equilibration period. Organic compounds with a high partition coefficient might require two months or more to establish equilibrium (Landrum et al. 1992 (82)). Boundaries for the sorption time can be estimated from the partition coefficient, using calculations described by Karickhoff and Morris (1985a,b (129),(130)). It is important to recognize that the quantity of spiked chemical might exceed the capacity of the test sediment system, prohibiting equilibrium.

12.4.10.2 Unless definitive information is available regarding equilibration time for a given contaminant and sediment concentration, a one-month equilibration period is recommended, with consideration that two months might be needed in some instances (see 12.4.10, USEPA 2000d (35)). Periodic monitoring during the equilibration time is highly recommended to empirically establish stability of interstitial water concentrations (USEPA, 2000d (35)). Sediment and interstitial water chemical concentrations should also be monitored during long-term toxicity tests to determine the actual chemical concentrations to which test organisms are exposed, and to verify that the concentrations remain stable over the duration of the test.

12.4.11 Use of Organic Solvents:

12.4.11.1 Direct addition of organic solvents should be avoided if possible, because organic solvents can alter geochemistry and bioavailability (USEPA, 2000d (35)). However, many organic materials require use of a solvent to adequately mix with the sediment. If an organic solvent is to be used, the solvent should be at a concentration that does not affect test organisms and should be uniform across treatments. Further, both solvent control and negative control sediments should be included in tests with solvents. The solvent concentration in the control should equal the treatment concentration, and should be from the same batch used to make the stock solution (Test Method E 1706).

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

12.4.11.3 To reduce the possibility of solvent-related artifacts, the spiking process should include a step which allows the solvent to evaporate before addition of sediment and water followed by rolling (McLeese et al. 1980 (131); Muir et al. 1982 (132); Adams et al. 1985 (98)). Highly volatile organic compounds have been spiked into sediments using co-solvents followed by shaking in an aqueous slurry. When highly volatile compounds are used, immediate testing in covered flow-through systems is recommended (Knezovich and Harrison, 1988 (96)).

12.4.11.4 There is some uncertainty concerning artifacts introduced by the use of solvents. The use of a polar, water soluble carrier such as methanol was found to have little effect on the partitioning of non-ionic compounds to dissolved organic matter at concentrations up to 15 % carrier by volume (Webster et al. 1990 (133)). However, another study showed that changes in partitioning by a factor of about two might occur with 10 % methanol as a co-solvent for anthracene sorption (Nkedi-Kizza et al. 1985 (134)). The effect of carrier volume on partitioning of organic chemicals in sediments is equivocal. However, because solvents might be either directly or indirectly toxic to the test organisms, caution should be taken to minimize the amount of carrier used. In addition, the use of a carrier such as acetone might result in faster equilibration of spiked organic compounds (Schults et al. 1992 (135)).

12.4.11.5 Shell coating techniques which introduce dry chemical(s) to wet sediment have also been developed, principally to eliminate the potential disadvantages of solvent carriers. The chemical may be either coated on the inside walls of the container (Ditsworth et al. 1990 (56); Burgess et al. 2000 (136)) or coated onto silica sand (Kane-Driscoll and Landrum, 1997 (137); Cole et al. 2000 (138); see 12.4.5). In each shell coating method, the chemical is dissolved in solvent, placed in a glass spiking container (with or without sand), and the solvent is slowly evaporated before addition of the wet sediment. Wet sediment then sorbs the chemical from the dry

surfaces. It is important that the solvent be allowed to evaporate before adding sediment or water.

12.5 Preparation of Sediment Dilutions:

12.5.1 Spiked or field-contaminated sediments can be diluted with whole sediment to obtain different contaminant concentrations for concentration-effects testing. The diluent sediment should have physicochemical characteristics similar to the test sediment, including organic carbon content and particle size, but should not contain concentrations of contaminants above background levels (Test Method E 1706, Burton 1991 (126)). Diluent sediment has included formulated sediment as well as reference or control sediment. Diluted sediment samples should be homogenized and equilibrated in accordance with procedures described in 11.5 and 12.4.10.

12.5.2 The diluent sediment should be combined with the test sediment in ratios determined on a dry weight basis to achieve the desired nominal dilution series. Volume to volume dilutions have also been performed (for example, Schlekat et al. 1995 (139); Johns et al. 1985 (140)), but weight to weight dilutions are preferred because they provide more accurate control and enable a more straightforward calculation of dose-response curves.

12.5.3 Results from dilution experiments should be interpreted with care. There can be non-linear responses due to non-equilibrium, non-linear sorption-desorption processes that cannot always be adequately controlled (Nelson et al. 1993 (97)). Nelson et al. (1993) (97) found that analyses of diluted sediments did not match nominal concentrations as estimated by physical characteristics and suggested that chemical characterization is needed to determine effects of manipulations (that is, mixing) and resulting changes (that is, oxygenation of complexing agents such as acid volatile sulfides). Hayward (2003 (141)) successfully conducted sediment dilution studies with field-collected sediments by matching the physical characteristics of the sediments, and by including a prolonged (3 month) equilibration period of the diluted sediment before conducting toxicity testing in the laboratory or field-colonization studies.

12.6 Preparation of Sediment Elutriates:

12.6.1 Sediment toxicity studies have evaluated aqueous extractions of suspended sediment called elutriates. The elutriate method was initially developed to assess the effects of dredging operations on water quality (USACE, 1976 (142)). Elutriate manipulations are also applicable to any situation where the resuspension of sediment-bound toxicants is of concern, such as bioturbation and storms, and that might disturb sediments and affect water quality (USEPA/USACE, 1991(32), 1998 (34); Ankley et al. 1991 (110)). USEPA/USACE (1998) (34) lists eighteen freshwater, estuarine, or marine aquatic organisms as candidates for elutriate toxicity testing. Standard effluent toxicity test procedures are also appropriate for elutriates, including tests with various vascular and non-vascular plant species (Ingersoll, 1995 (143)).

12.6.2 Elutriate tests are not intended to reflect the toxicity of interstitial waters or whole sediments, as there are differences in contaminant bioavailability in the two types of media (Harkey et al. 1994 (144)). In general, elutriates have been found to be less toxic than bulk sediments or interstitial water

fractions (Burgess et al. 1993 (145); Ankley et al. 1991 (110)), although in some studies elutriates have been found to be more toxic (Hoke et al. 1990 (146)) or equally as toxic (Flegel et al. 1994 (147)) relative to interstitial water.

12.6.3 While there are several procedural variations, the basic method for elutriate preparation involves combining various mixtures of water and sediment (usually in the ratio of 4 parts water to 1 part sediment, by volume) and shaking, bubbling or stirring the mixture for 1 h (Ross and Henebry, 1989; Daniels et al. 1989 (148); Ankley et al. 1991 (110); Burgess et al. 1993 (145); USEPA/USACE, 1991(32), 1998 (34)). It is likely that chemical concentrations will vary depending on the elutriate procedure used. The water phase is then separated from the sediment by settling or centrifugation. Once an elutriate has been prepared, it should be analyzed or used in biological tests immediately, or as soon as possible thereafter. It should be stored at 4°C for not longer than 24 h, unless the method dictates otherwise (Environment Canada, 1994 (2); USEPA/USACE, 1991 (32), 1998 (34)). For toxicity test exposures exceeding 24 h, fresh elutriate should be prepared daily.

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

13. Collection of Interstitial Water

13.1 Sediment interstitial water, or pore water, is defined as the water occupying the spaces between sediment or soil particles (Terminology E 943). Interstitial water might occupy about 50 % (or more) of the volume of a depositional (silt-clay) sediment. The interstitial water is in contact with sediment surfaces for relatively long periods of time and therefore, might become contaminated due to partitioning of the contaminants from the surrounding sediments. In addition, interstitial waters might reflect ground water - surface water transition zones in upwelling or downwelling areas. In these areas, their chemistry might be more reflective of ground or surface waters at the site. Therefore, flow, residence time, and other physicochemical factors (for example, pH, temperature, redox potential, organic carbon, sulfides, carbonates, mineralogy) might have varying roles in determining whether interstitial waters are contaminated.

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

13.2 General Procedures:

13.2.1 Interstitial water sampling has become especially important because interstitial water toxicity tests yield additional information not provided by whole-sediment elutriate or sediment extract tests (Carr and Chapman 1992 (149); SETAC 2003 (150)). Furthermore, interstitial water toxicity tests are useful in sediment toxicity identification evaluation (TIE) studies (for example, Burgess 1996 (151); Carr 1998 (152); Burton et al. 2001 (153)) as test procedures and sample manipulation techniques can be faster and easier to conduct than whole-sediment toxicity tests (SETAC, 2003 (150)). Thus, the collection of interstitial water has become increasingly important in sediment quality monitoring programs.

13.2.2 Interstitial water sampling is most suitable for sediment types ranging from sandy to uncompacted silt-clays (Sarda and Burton, 1995 (154); SETAC, 2003 (150)). Such sampling is not typically performed on sediments with coarse particle size (such as gravel) or on hard, compacted clays, as the potential for interstitial water contamination in these sediment types is relatively low.

13.2.3 As with all sampling discussed in this standard, the principle aim is to use procedures that minimize changes to the in situ condition of the water. It should be recognized that most sediment collection and processing methods have been shown to alter interstitial water chemistry (for example, Schults et al. 1992 (135); Bufflap and Allen, 1995 (155); Sarda and Burton, 1995 (154)), thereby potentially altering contaminant bioavailability and toxicity.

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

13.2.5 Both in-situ and laboratory-based or ex-situ methods might be appropriate for many study objectives. It is important that the same procedures are used for all stations sampled in a study so that appropriate comparisons can be made. Furthermore, the sediment depth at which interstitial water is sampled (either using in-situ or ex-situ extraction methods) should match the depth of interest in the study (see 10.1, SETAC 2003 (150)). For example, samples for dredging remediation should be sampled to the depth to be disturbed by dredging activity, whereas samples for a status and trends survey should be collected at the biologically active depth (often <15 cm). Fig. 10 summarizes the major considerations for selecting in-situ or ex-situ procedures in a given study.

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

13.3 In-situ Collection:

13.3.1 In situ methods might be superior to ex-situ methods for collecting interstitial water, as they are less subject to sampling or extraction related artifacts and therefore, might be more likely to maintain the chemical integrity of the sample (Sarda and Burton 1995 (154), SETAC 2003 (150)). However, in situ methods have generally produced relatively small volumes of interstitial water, and are often limited to wadeable or diver-accessible water depths. These logistical constraints have limited their use and applicability in sediment monitoring studies.

13.3.2 The principal methods for in situ collection of interstitial water involve either deployed “peepers” (Bufflap and Allen, 1995 (155); Brumbaugh et al. 1994 (27); Adams, 1991 (156); Carignan and Lean, 1991 (157); Carignan et al. 1985 (292); Bottomley and Bayly, 1984 (158)) or suction techniques (Watson and Frickers, 1990 (159); Knezovich and Harrison, 1988 (96); Howes et al. 1985 (160)). A summary of these methods is provided in Table 13. Both methods have a high likelihood of maintaining in situ conditions. In cases where in situ deployment is impractical, peepers or suction devices can be placed in relatively undisturbed sediments collected by core or grab samplers (see Section 10).

13.3.3 Peeper Methods:

13.3.3.1 Peepers are small chambers with membrane or mesh walls containing either distilled water or clean water of the appropriate salinity or hardness. Samples are collected by burying the devices in sediments and allowing surrounding interstitial waters to infiltrate. In principle, dissolved solutes will diffuse through the porous wall into the peeper and the contained water will reach equilibrium with the ambient interstitial water. The design concept for sediment peepers originated as modifications of the dialysis bag technique used by Mayer (1976 (161)) and Hesslein (1976 (162)), and has been modified for use in laboratory sediment toxicity tests (Doig and Liber, 2000 (163)). The initial designs consisted of either a flat base plate or a cylindrical dialysis probe (Bottomley and Bayly, 1984 (158)) with compartments covered by dialysis membranes and a manifold for collection of multiple samples at various depths in the sediment profile Fig. 11. Further modifications to these designs have incorporated sampling ports, large sample compartments, and various types of membranes with different pore sizes. These modifications are usually required based on specific project objectives regarding sample volumes and contaminants of interest.

13.3.3.2 Various peeper devices have been recently used effectively to collect interstitial water. For example, a simplified design using a 1 µm polycarbonate membrane over the opening of a polyethylene vial was successful in capturing elevated levels of copper and zinc (Brumbaugh et al. 1994 (27)). Other designs have been used to collect non-ionic organic compounds in a variety of aquatic systems (Bennett et al. 1996 (164); Axelman et al. 1999 (165)) and in overlying water (Huckins et al. 1990 (166)).

13.3.3.3 Peepers have also been used to expose organisms to sediments in situ (Burton et al. 2001 (153)). Burton et al. (1999 (167)) successfully introduced organisms to aerobic sediments using peepers. However, anoxic sediments are not amenable to in situ organism exposure.

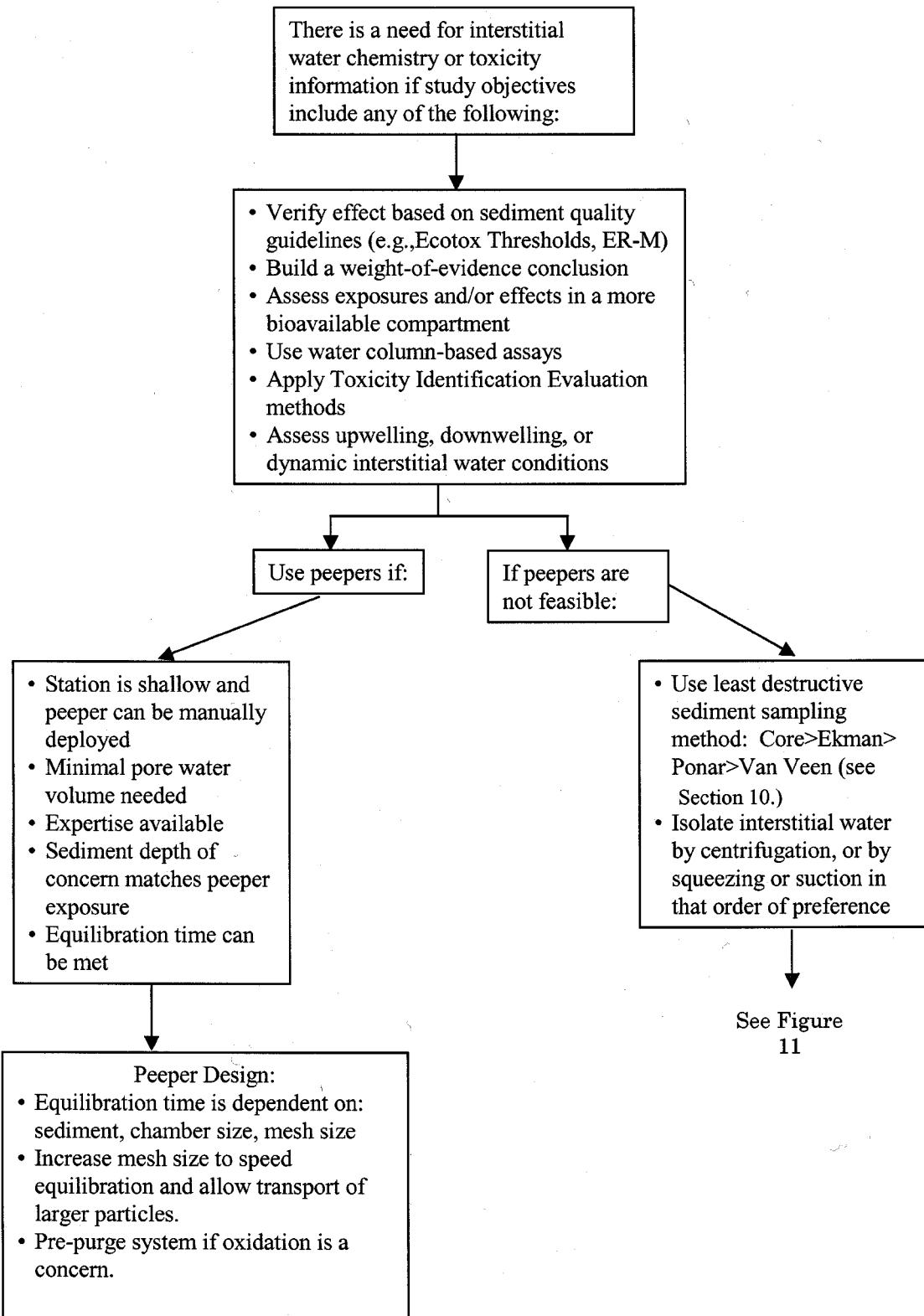


FIG. 10 Considerations for Selecting the Appropriate Type of Interstitial Water Sampling Method (USEPA 2001 (1))

13.3.3.4 Different materials might be advisable in constructing peepers depending on the contaminants of concern. For example, for many contaminants, peepers constructed from

acrylic material appear to yield interstitial water samples with minimal chemical artifacts (Burton et al. 2001 (153)). Some polymer materials might be inappropriate for studies of certain

TABLE 13 In-situ Interstitial Water Collection Methods (Sarda and Burton 1995(154), SETAC 2003 (150))

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

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

non-ionic organic compounds. Cellulose membranes are also unsuitable, as they decompose too quickly. Plastic samplers can contaminate anoxic sediments with diffusible oxygen (Carignan et al. 1994 (168)).

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

13.3.3.6 Following initial placement, the equilibration time for peelers may range from hours to a month, but a deployment period of one to two weeks is most often used (Adams, 1991 (156); Call et al. 1999 (169); Steward and Malley, 1999 (170)). Equilibration time is a function of sediment type, study objectives, contaminants of concern, and temperature (for example, Skalski and Burton, 1991(171); Carr et al. 1989(172); Howes et al. 1985(160); Simon et al. 1985 (174); Mayer, 1976 (161)). Membrane pore size also affects equilibration time, with larger pore sizes being used to achieve reduced equilibration times (Sarda and Burton, 1995 (154)). For example, using a peeper with a 149-µm pore size, Adams (1991 (156)) reported equilibration of conductivity within hours of peeper insertion into the sediment. Thus, it appears that equilibration time is a function of the type of contaminant, sediment type, peeper volume, and mesh pore size.

13.3.3.7 Peelers with large-pored membranes, while shortening equilibration time, also allow particulates to enter the chamber. The larger solids tend to settle to the bottom of the peeper chamber, and caution should be used to avoid collecting the solids when retrieving the water sample from the chamber. Colloidal particles will remain suspended in the sample and thereby present an artifact, but the concentration of such particles is typically lower than that found in laboratory-centrifuged samples (Chin and Gschwend, 1991 (173)).

13.3.3.8 In several studies, analysis of interstitial water from replicate peelers has demonstrated variable heterogeneity in water quality characteristics (Frazier et al. 1996 (175); Sarda and Burton, 1995 (154)). The potential for high variability in interstitial water chemical characteristics should be taken into account when developing the sampling design.

13.3.4 *Suction Methods*—There are a variety of suction devices for collecting interstitial water. A typical suction device consists of a syringe or tube of varying length, with one or more ports located at the desired sampling positions. The device is inserted into the sediment to the desired depth and a manual, spring-operated, or vacuum gas suction is applied to directly retrieve the water sample. A variation on this approach employs a peeper-like porous cup or perforated tube with filters. The unit is inserted into the sediment for a period of time, allowing interstitial water to infiltrate the chamber before suction is applied. The samples are then retrieved by suction. Another variation that has been used successfully employs an air stone embedded into the sediment that forces interstitial water upward where it can be collected via syringe or tube. All of these suction methods generally yield smaller quantities of interstitial water than peelers, and chemical (toxicological) artifacts are more likely due to greater potential exposure of interstitial water to oxygen.

13.3.5 Processing of Field-Collected Interstitial Water Samples:

13.3.5.1 Following sample retrieval, interstitial water might need to be recovered and stabilized quickly to prevent oxidative changes or volatilization (Carignan, 1984 (176)). Containers should be filled with no headspace to minimize changes in dissolved oxygen and contaminant bioavailability. Procedures for stabilization are dependent on the analyses to be performed. When non-volatile compounds are the target analytes, acidification is often stipulated, while organic carbon and methane may be stabilized with saturated mercury chloride (Mudroch and MacKnight, 1994 (36)). Samples for chemical analyses should be preserved immediately, if appropriate, or cooled to 4°C as soon as possible.

13.3.5.2 Samples to be analyzed for toxicity are normally cooled to 4°C as soon as possible for transport to the laboratory. USEPA methods for toxicity testing of surface waters and effluents (USEPA 1991 (177)) recommend that

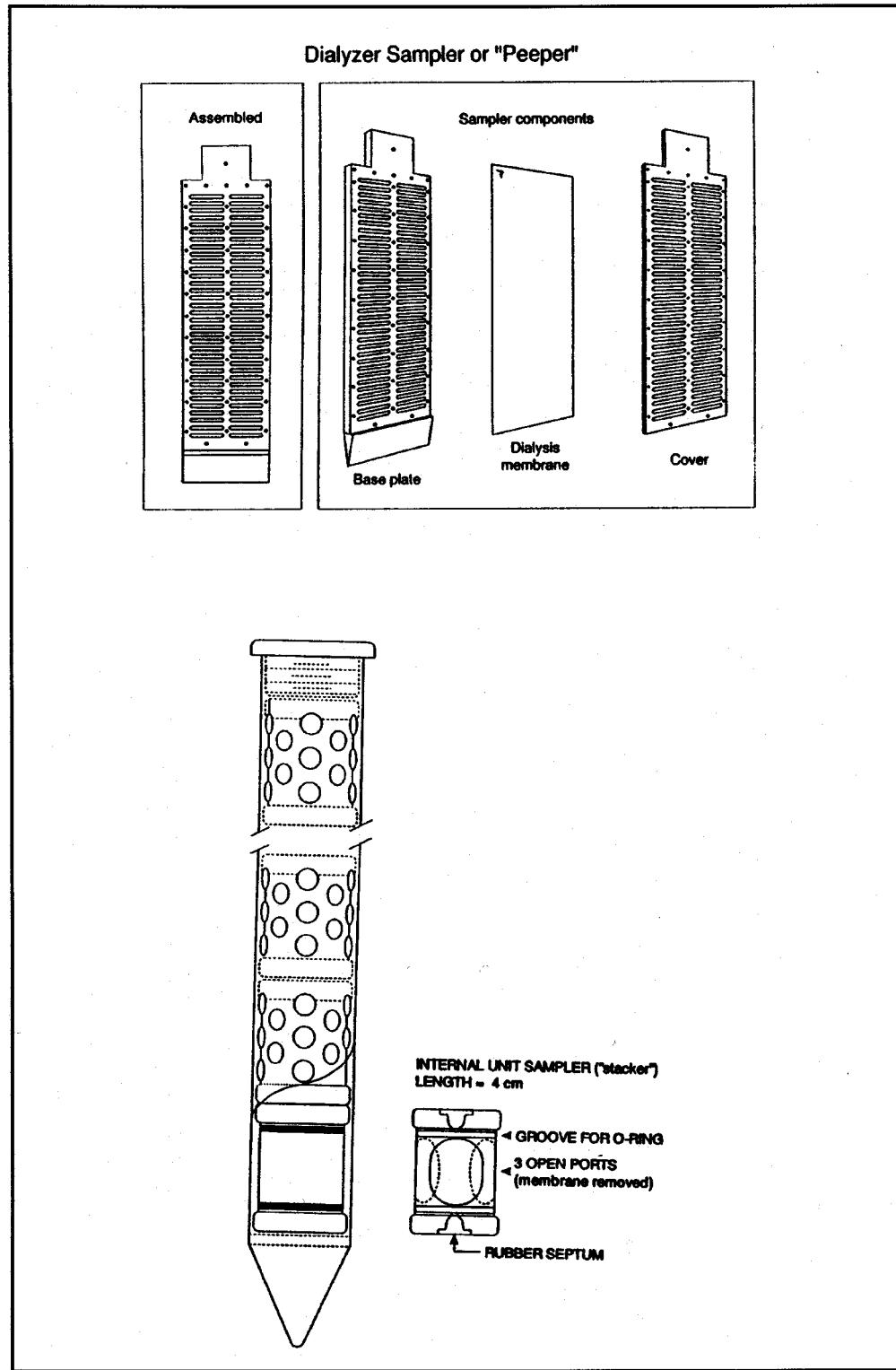


FIG. 11 Front View and Components of Peeper Sampling Devices (Top: Plate Device; Bottom: Cylindrical Probe; USEPA 2001 (1))

samples not be frozen in storage or transport. However, recent information suggests that freezing of interstitial water may not affect toxicity in some cases (Ho et al. 1997 (178), Carr and Chapman, 1995 (179), SETAC 2003 (150)). Unless a demon-

stration of acceptability is made for the sites of interest, interstitial water samples should not be frozen before biological testing.

13.4 Ex-situ Extraction of Interstitial Water:

13.4.1 Ex-situ interstitial water collection methods are often necessary when relatively large volumes of interstitial water are required (such as for toxicity testing), when in-situ collection is not viable, or when a brief sampling time is important. While these extraction methods can be done in the field or in the laboratory, extraction in the laboratory, just before analysis or testing, is preferable to maintain as close to its original state as possible during transport and storage (SETAC 2003 (150), Table 14). Guidance in this section reflects recommendations presented in several recent publications, including proceedings from two workshops dealing with interstitial water extraction and handling methods, and use in toxicity applications: (1) a dredged materials management program workshop on interstitial water extraction methods and sample storage in relation to tributyltin analysis (Hoffman, 1998 (180)) and (2) a workshop on interstitial water toxicity testing including interstitial water extraction methods and applications (SETAC 2003 (150)).

13.4.2 General Procedures:

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

13.4.2.2 Regardless of the method used, interstitial water should be preserved immediately for chemical analyses, if appropriate, or analyzed as soon as possible after sample collection if unpreserved (such as for toxicity testing; Hoffman, 1998 (180); SETAC 2003 (150)). Significant chemical changes can occur even when interstitial water is stored for periods as short as 24 h (Hulbert and Brindle, 1975 (182); Watson et al. 1985 (181); Kemble et al. 1999 (85); Sarda and Burton, 1995 (154); SETAC 2003 (150)).

13.4.2.3 If sediments are anoxic, as most depositional sediments are, sample processing, including mixing of interstitial water that has separated from the sediment, should be con-

ducted in an inert atmosphere or with minimal atmospheric contact. Exposure to air can result in oxidation of contaminants, thereby altering bioavailability (Bray et al. 1973 (183); Lyons et al. 1979 (184); Howes et al. 1985 (160)). Air exposure can also result in loss of volatile sulfides, which might increase the availability of sulfide-bound metals (Allen et al. 1993 (185); Bufflap and Allen, 1995 (155)). In addition, iron and manganese oxyhydroxides are quickly formed upon exposure to air. These compounds readily complex with trace metals, thus altering metals-related toxicity (Bray et al. 1973 (183); Troup et al. 1974 (186); Burton, 1991 (126); Bufflap and Allen, 1995 (155)). Maintaining anoxic processing conditions is not necessary when study objectives are concerned with exposures to aerobic sediments, or if target contaminants are unaffected by oxidation in short-term toxicity testing.

13.4.3 Centrifugation:

13.4.3.1 Centrifugation is the generally preferred laboratory method for collection of interstitial water (SETAC 2003 (150)). It is a relatively simple procedure that allows rapid collection of large volumes of interstitial water. It also facilitates the maintenance of anoxic conditions (if required). However, centrifugation, like other ex-situ procedures, might yield chemical or toxicological artifacts due to the extraction procedures themselves, which might alter the natural equilibrium between interstitial water and sediment.

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

13.4.3.3 Interstitial water has been isolated over a range of centrifugal forces and durations (Landrum et al. 1987 (187); Giesy et al. 1988 (188); Schults et al. 1992 (135); Burgess et al. 1993 (145); Ankley et al. 1990 (189); Schubauer-Berigan and Ankley, 1991 (190); Ankley and Schubauer-Berigan, 1994 (191); Kemble et al. 1994 (69)). For toxicity testing of interstitial waters, some sources recommend that sediments be centrifuged at $10\,000 \times g$ for a 30 min period (Environment Canada, 1994 (2)). Such high speed centrifugation is often necessary to remove most colloids and dispersible clays (Adams, 1991 (156); Chin and Gschwend, 1991 (173); Brownawell and Farrington, 1986 (192); Ankley and Schubauer-Berigan, 1994 (191)), which can introduce interferences to chemical or toxicological analysis. However, such high speed centrifuges are not commonly available. Furthermore, many materials (glass, plastic) are not able to withstand high centrifugation speeds. Finally, it should be noted that toxicity is typically reduced with high speed centrifugation due to the removal of particle-associated contaminants (Sasson-Brickson and Burton 1991(193); Schults et al. 1992 (135); Ankley and Schubauer-Berigan, 1994 (191); Bufflap and Allen, 1995 (155)).

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

Centrifugation is the generally preferred laboratory method for the extraction of interstitial water.

Extraction of interstitial water should be completed as soon as possible.

Interstitial water that has accumulated on the surface of the homogenized sediment sample should be mixed into the sediment before the sample is partitioned among centrifuge bottles.

Unless other program-specific guidance is available, sediments should be centrifuged at high speed (for example, 8000 to $10\,000 \times g$) for 30 min.

Unless site-specific information suggests otherwise, centrifuging should be at 4°C to minimize temperature-mediated biological and chemical processes.

Interstitial water should be preserved immediately for chemical analyses or analyzed as soon as possible after extraction, unpreserved. For toxicity testing, interstitial water should be stored at 4°C for not longer than 24 h, unless the test method dictates otherwise.

Filtration should be avoided unless required by a test method because it might reduce interstitial water toxicity. Double (serial) centrifugation (low speed followed by high speed) should be used instead.

13.4.3.4 Based on research to date, both slower and faster centrifugation speeds (and associated differences in colloid or suspended solids removal) may be appropriate depending on the study objectives. High speed centrifugation may not be appropriate because one is interested in toxicity potential of the interstitial water in its entirety (that is, including colloidal material). However, if one is interested in comparing interstitial water contaminant concentrations to model exposure compartments for example (EPRI, 2000 (194)), then high speed centrifugation might be necessary. As our knowledge is still limited in this area, it is perhaps most important to note that centrifugation speed can have an effect on the observed toxicity and chemical characteristics. Therefore, a consistent centrifugation procedure (including speed and time) should be identified and used throughout a study for all samples.

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

13.4.3.6 When centrifuging coarse sand, it might be desirable to use a modified centrifuge bottle to aid interstitial water recovery (USEPA/USACE, 1998 (34)). The modified bottle is equipped with an internal filter that can recover 75 % of the interstitial water, as compared to 25 to 30 % recovery from squeezing (Saager et al. 1990 (195)).

13.4.3.7 As discussed in 11.2, all containers have limitations with regards to adsorption or leaching of chemicals, ease of use, and reliability. For example, polytetrafluoroethylene (PTF) bottles have been used successfully up to $2500 \times g$ when filled to 80 % of capacity, but collapse at 3000 g (Burgess et al. 1993 (145)). Polycarbonate bottles have been used successfully for tributyltin analyses in interstitial water (Hoffman, 1998 (180)). If small volumes of water are required for testing, higher speed centrifugation can be performed with glass tubes (up to 10 000 g, Word et al. 1987 (101)). Larger glass tubes, however, can not be centrifuged at such high speeds. If metal toxicity is not a concern, then high speed centrifugation in larger stainless steel centrifuge tubes is suitable. If test samples are contaminated with photoreactive compounds such as PAHs, exposure of the sample to light should be minimized to limit degradation or alteration of potentially toxic compounds.

13.4.4 Sediment Squeezing:

13.4.4.1 Isolation of interstitial water by squeezing has been performed using a variety of procedures and devices (Reeburgh, 1967 (196); Kalil and Goldhaker, 1973 (197); Jahnke, 1988 (198); Carr et al. 1989 (172); Long et al. 1990 (28); Watson and Frickers, 1990 (159); Adams, 1991 (156); Carr and Chapman, 1995 (179); Carr, 1998 (152)). Low-pressure mechanical squeezers can be constructed, and may provide specialized capacities such as collection of interstitial water profiles from core samples (Bender, et al. 1987(199)). In all cases, the interstitial water is passed through a filter that is a part of the squeezing apparatus.

13.4.4.2 Squeezing has been shown to produce a number of artifacts due to shifts in equilibrium from pressure, tempera-

ture, and gradient changes (for example, Froelich et al. 1979 (201); Kriukov and Manheim, 1982 (200); Bollinger et al. 1992 (206); Schults, 1992 (135)). Squeezing can affect the electrolyte concentration in the interstitial water particularly with a decrease in chemical concentrations near the end of the squeezing process. However, others reported that squeezing did not produce artifacts in interstitial water toxicity studies (Carr and Chapman 1995 (179); Carr 1998 (152); SETAC 2003 (150)). It is therefore recommended that if squeezing is performed, moderate pressures be applied along with electrolyte (conductivity) monitoring during extraction (Kriukov and Manheim, 1982 (200)). Squeezing should also be performed at *in situ* ambient temperatures, as significant alterations to interstitial water composition can occur when squeezing is conducted at temperatures different from ambient conditions (for example, Mangelsdorf et al. 1969 (202); Bischoff et al. 1970; Sayles et al. 1973 (203)).

13.4.4.3 Other sources of interstitial water alteration during squeezing are: contamination from overlying water; internal mixing of interstitial water during extrusion; and solid-solution reactions as interstitial water is expressed through the overlying sediment. As interstitial waters are displaced into upper sediment zones, they come in contact with solids with which they are not in equilibrium. This inter-mixing causes solid-solution reactions to occur. Most interstitial water chemical species are rapidly transformed, as observed with ammonia and trace metals (Rosenfield, 1979 (204); Santschi et al. 1997 (205)). Bollinger et al. (1992) (206) found elevated levels of several ions and dissolved organic carbon in squeezed samples as compared to samples collected by *in situ* peepers. The magnitude of the artifact will depend on the characteristics of the contaminant and redox potential.

13.4.5 Pressurized and Vacuum Devices:

13.4.5.1 Other methods for extraction of interstitial water from sediment samples can include vacuum filtration (Jenne and Zachara, 1987 (127); Knezovich and Harrison, 1987 (207); Winger and Lasier, 1991 (208)), gas pressurization (Reeburgh, 1967 (196)), and displacement (Adams, 1991 (156)). These methods typically recover only small volumes of interstitial water and are not commonly used.

13.4.5.2 Use of a hand vacuum with an aquarium stone is an effective vacuum filtration method (Winger and Lasier, 1991(208); Sarda and Burton, 1995 (154)). The procedure typically involves attaching the air stone to a 50 mL syringe via plastic tubing, inserting it into the sediment to the desired depth, and then applying suction. This method can recover relatively large volumes of interstitial water; Santschi et al. (1997 (205)) used this procedure to extract up to 1,500 mL from 4 L of sediment. Sarda and Burton (1995 (154)) found that ammonia concentrations in water obtained by this procedure were similar to those collected by *in situ* peepers. Drawbacks to this method include loss of equilibrium between the interstitial water and the solids, filter clogging, and oxidation (Brinkman et al. 1982 (209)).

14. Physicochemical Characterization of Sediment Samples

14.1 *General Information*—It is often necessary or desirable to determine certain physicochemical characteristics of

sediments in the laboratory, in conjunction with toxicity testing or chemical analysis for inorganic or organic contaminants. This characterization should include measurement of certain parameters known to mediate the availability of contaminants in sediment (Test Method E 1706). Bulk chemical concentrations alone should not be used to evaluate bioavailability (USEPA 1998 (210)). The following parameters are generally measured: pH (pore water), ammonia (pore water), total organic carbon, particle size distribution (for example, percent sand, silt and clay), percent water content, salinity or hardness of pore water, and conductivity of pore water. Depending on the experimental design or study objectives, more extensive characterization may be necessary. Several additional characteristics that may assist in study implementation, data interpretation, or QA/QC (that is, assessing sediment integrity, artifact production, optimal extraction and test procedures) include: sediment biochemical oxygen demand (BOD), sediment chemical oxygen demand (COD), sediment oxygen demand (SOD), cation exchange capacity (CEC), Redox (Eh) or oxidation-reduction potential (ORP), total inorganic carbon, total volatile solids, acid volatile sulfides (AVS), simultaneously extracted metals (SEM), metals, petroleum hydrocarbons, other organic compounds (pesticides, PCBs, PAHs, and TCDD-dioxin), oil and grease, and dissolved organic carbon (DOC) in the pore water. Measurements of many sediment physicochemical characteristics use analytical techniques originally developed for soils and waters, and the literature should be consulted for details regarding recommended methodology (Black, 1965 (211); USGS, 1969 (212); Plumb, 1981(213); Page et al. 1982 (214)). The following sections provide rationale for making each type of sediment physicochemical measurement, along with brief descriptions of measurement techniques, and references for further information and specific procedures.

14.2 pH in Pore Water:

14.2.1 Sediment pH is often one of the single most important factors controlling speciation and equilibria for many chemicals including sulfides, ammonia, cyanide, and metals, all of which ionize under the influence of pH. The USEPA ammonia water-quality criterion, for example, is dependent in part on pH because ammonia toxicity is largely governed by the unionized ammonia fraction which is pH-dependent (USEPA, 1999 (215)). Metal (Cd, Cu, Ni, Pb, and Zn) speciation and bioavailability are also known to be affected by pH (Schubauer-Berigan and Ankley, et al. 1991 (190); Ho et al. 1999 (216)).

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

tial is calculated as the difference between millivolts measured at the known pH of two buffers.

14.2.3 Plumb (1981 (213)) and Gonzalez (1995 (217)) described a method for measuring pH in sediment using a pH probe and meter. The probe was inserted into the sediment and pH directly measured after at least a 5 min equilibration time. Electrodes have also been used for direct measurements of pH in sediment pore water, or in a 1 to 1 mixture of sediment to water (Jackson, 1958 (218)). Direct measurement of sediment pH is also possible using electrodes with “spear tip” designs allowing for greater penetration into the sample. Detailed methods for measuring pH in water and sediment are also described by USEPA (1979 (219);1983 (52);1986 (220);1987 (43)).

14.3 Ammonia in Pore Water:

14.3.1 Nitrogen, a nutrient associated with over-enrichment of aquatic environments, exists in several forms, including ammonia. Ammonia is highly soluble in water where it is found in an un-ionized form (NH_3) and in an ionized form as NH_4^+ . The extent of ionization is dependent on pH, temperature, and salinity (in seawater). Ammonia in sediments and pore water is generally the result of microbial degradation of nitrogenous organic material such as amino acids (Ankley et al. 1990 (189)). Pore water concentrations of ammonia as high as 50 mg/L have been measured in otherwise uncontaminated sediments (Murray et al. 1978 (221); Kristensen and Blackburn, 1987 (222)), while ammonia in pore waters from contaminated sediments can range from 50 to more than 200 mg/L (Ankley et al. 1990 (189); Schubauer-Berigan and Ankley, 1991 (190)). Elevated concentrations of ammonia (Sims et al. 1995a (223); Moore et al. 1997 (224)) and hydrogen sulfide (Sims et al. 1995b (225)) have frequently been found in deeper dredged sediment samples compared to surficial sediment samples.

14.3.2 The toxic effects of ammonia are generally considered to be associated with the un-ionized fraction (NH_3) rather than the ionic components (NH_4^+ and NH_4SO_4^-), which co-exist in equilibria. This equilibrium is highly dependent on pH, temperature, pressure, salinity, and ionic concentrations of ammonia. The more toxic un-ionized ammonia fraction can be calculated using known total ammonia values and measurements of pH, pressure, salinity, and temperature as described by Whitfield (1978 (226)) and Thurston et al (1981 (227)).

14.3.3 USEPA (1983 (52)), and APHA (1995 (228)) describe five methods available to measure ammonia in the pore water: the titrimetric method; the ammonia-selective electrode method; the ammonia-selective electrode method using known addition; the phenate method; and, the automated phenate method.

14.3.4 A preliminary distillation step may be required if interferences are present (APHA, 1995 (228)). Interferences (for example, sample constituents that interact with procedural reagents) are described in detail in the APHA 1995 (228) and Guide D 1426. Once distilled, the sample can be analyzed using any of the methods listed above.

14.3.5 The distillation and titration methods are frequently used when ammonia concentrations are greater than 5.0 mg/L. The ammonia-selective electrode method is appropriate when

concentrations range between 0.03 and 1400 mg NH₃-N/L. Ammonia readings are calibrated against ammonia standards. To verify meter readings, confirmatory subsamples can be preserved and analyzed for ammonia using the standard Nessler technique described in APHA (1995) (228). For the phenate method, APHA (1995) (228) recommends distillation with sulfuric acid when interferences are present (Bower and Holm-Hansen, 1980 (229)). The automated phenate method is suitable for pore waters with ammonia concentrations in the range of 0.02 and 2.0 mg NH₃-N/L.

14.4 Total Organic Carbon Content (TOC):

14.4.1 The total organic carbon (TOC) content of sediment is a measure of the total amount of oxidizable organic material. TOC is the sum of dissolved organic carbon (DOC), particulate organic carbon (POC) or suspended organic carbon (SOC), and colloids. TOC is an important parameter to measure in sediments because it is a major determinant of non-ionic organic chemical bioavailability (Di Toro et al. 1991 (86)). Metal bioavailability is also affected by the amount of TOC present in sediments. TOC is usually expressed as a percentage of the bulk sediment, and is used to normalize the dry-weight sediment concentration of a chemical to the organic carbon content of the sediment. USEPA Equilibrium Partitioning Guidelines estimate bioavailability as a function of contaminant concentration sorbed to sediment organic carbon and contaminant concentration in the pore water under equilibrium conditions (USEPA, 1998 (210)). Recently, the presence of soot carbon from the combustion of organic carbon (for example, fossil fuels) has been recognized as a fraction of the TOC in sediment. Soot carbon may alter the geochemistry and bioavailability of some organic contaminants (Gustaffson et al. 1997 (230)). Methods for determining organic carbon in sediment have been reviewed (Schumacher 2002 (231)).

14.4.2 The organic carbon content of sediments has been measured using several methods including: wet oxidation titration, modified titration, and combustion after removal of carbonate by the addition of HCl and subsequent drying. USEPA methods (1986 (220); 1987 (43)), including SW-846 and 430/9-86-004, are often used to measure TOC. Plumb (1981) (213) recommends one of two methods to separate organic from inorganic carbon before analyzing for TOC: (a) ignition and using HCl as the acid for pre-treating sediment, or (b) differential combustion, which uses thermal combustion to separate the two forms of carbon.

14.4.3 USEPA/USACE guidance (1998) (34) recommends that TOC analyses be based on high-temperature combustion rather than on chemical oxidation because some classes of organic compounds are not fully degraded by combined chemical and ultraviolet oxidation techniques. Inorganic carbon (for example, carbonates and bicarbonates) can be a significant proportion of the total carbon in some sediments. Therefore, samples should be treated with acid to remove the inorganic carbon before TOC analysis. The procedure described by the Puget Sound Estuary Program (PSEP, 1997a (33)) is recommended for TOC analysis because this method uses high-temperature combustion using an induction furnace. USEPA recommends a similar method using catalytic combustion and non-dispersive infrared detection (Leonard, 1991

(232)) for quantifying TOC. Because of interferences associated with TOC measurement in high carbonate sand areas in Florida and in Hawaii, some investigators have not been able to use acid addition to remove inorganic carbon and have instead used the Lloyd Kahn method (Kahn 1988 (233); David Moore MEC Analytical, Carlsbad, CA; personal communication).

14.4.4 Several methods for measuring the total organic carbon (TOC) content of sediments exist (See Nelson and Sommers 1996 (234) for a review). However, acceptable methods should at a minimum include the following steps:

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

14.4.4.2 *Sample Preparation*—Each sediment sample should have macroscopic pieces of shells (for example, >1 mm) removed and then be pulverized and homogenized. Each sediment sample should be treated by direct addition with a strong non-oxidizing acid (for example, HCl) for about 18 h to remove inorganic carbon; sample pH should be about 2 after acidification (Yamamoto and Kayanne, 1995 (235)). Each sediment sample is oven dried following acid treatment (60 to 70°C; Weliky et al. 1983 (236); Yamamoto and Kayanne, 1995 (235)). Each sediment sample is stored in a desiccator until analysis. As noted, desiccation is highly recommended, however if not possible, a pre- and post-acidification sample weight should be performed to correct for water uptake (Hedges and Stern, 1984 (237)).

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

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

14.5.1 Particle size is used to characterize the physical characteristics of sediments. Because particle size influences both chemical and biological characteristics, it can be used to normalize chemical concentrations and account for some of the variability found in biological assemblages (USEPA 1998 (210)) or in laboratory toxicity testing (USEPA, 2000d (35); Hoss et al. 1999 (238)). Particle size can be characterized in varying detail. The broadest divisions that generally are considered useful for characterizing particle size distributions are percentages of gravel, sand, silt, and clay. However, each of these size fractions can be subdivided further so that additional characteristics of the size distribution are determined (PSEP, 1996 (239)).

14.5.2 Particle size determinations can either include or exclude organic material. If organic material is removed before analysis, the "true" (that is, primarily inorganic) particle size distribution is determined. If organic material is included in the analysis, the "apparent" (that is, organic plus inorganic) particle size distribution is determined. Because true and apparent distributions may differ, detailed comparisons between samples analyzed by these different methods are questionable. Therefore, if comparisons among samples between studies is desired, sediment particle size should be measured using consistent methods (PSEP, 1996 (239)). For interpretation of biological effects or chemical partitioning in sediment, the "apparent"

particle size distribution may be more desirable to determine compared to the measurement of the “true” particle size distribution (Word et al. 2004 (239)).

14.5.3 Sediment particle size can be measured by a number of different methods (Allen, 1975 (241); Plumb, 1981 (213); PSEP, 1996 (239)). The best method will depend on the particle properties of the sample (Singer et al. 1988 (242)). Particle size distribution is often determined by either wet sieving the sample (USEPA, 1979 (219); Plumb, 1981 (213); PSEP, 1996 (239); Singer et al. 1988 (242)), the hydrometer method (Day, 1965 (243); Patrick, 1958 (244)), the pipette method (USGS, 1969 (212); Rukavina and Duncan, 1970 (245)), settling techniques (Sanford and Swift, 1971 (246)), by use of laser diffraction, or X-ray absorption (Duncan and Lattaie, 1979 (247); Rukavina and Duncan, 1970 (245)). The pipet method may be superior to the hydrometer method (Sternberg and Creager, 1961 (248)). Combinations of multiple methods may provide refined measurements of particle size distribution. Gee and Bauder (1986 (249)) used sieving and pipeting after soluble salts were removed. Gonzalez (1995 (217)) used a combination of sieve and hydrometer methods. Folk (1968 (250)) and Buchanan (1984 (251)) discuss additional methods to measure particle size.

14.5.4 Recommended methods for measuring sediment particle size distribution are those of PSEP (1996 (239)) and USEPA (1995 (252)). Percent gravel, sand, silt, and clay are determined as apparent distribution using a minimum sediment sample size of 100 g taken from a homogenized sediment sample. Organic matter should be removed before analysis by oxidation using hydrogen peroxide. Wet-sieving followed by dry sieving (mechanical shaking) separates the two coarse particle size groups. The silt-clay fraction is subdivided using a pipet technique that depends upon the differential settling rates of the two different particle size fractions. All fractions are dried to a constant weight. Cooled samples are stored in a desiccator and weighed.

14.5.5 Particle analyzers may be preferable over pipette methods for the evaluation of fine fractions due to the introduction of human error in the sampling process (for example, Beckman Coulter LS100Q laser diffraction particle size analyzer or Micromeritics Sedigraph; Syvitski et al. 1991(253)). To obtain an accurate determination of particle sizes for the fine fraction, the Coulter (particle size) counter method may be employed (McCave and Jarvis, 1973 (254); Vanderpleeg, 1981 (255)). This method gives the fraction of particles with an apparent spherical diameter. In a review of the available methods, Swift et al. (1972) (256) found the Coulter counter method to be the most versatile method overall; however, it does not provide settling information. Another potential method for determining the particle size distribution of a very fine fraction is through the use of electron microscopy (Leppard et al. 1988 (257)). Collection techniques for very fine material can result in aggregation of larger colloidal structures (Leppard, 1986 (258); Leppard et al. 1988 (257)). In general, particle settling methods are preferred to sediment sizing methods. Unless there is a large amount of organic matter, particle size should be determined with the organic matter present.

14.6 Percent Water or Moisture Content:

14.6.1 Water content is a measurement of sediment moisture usually expressed as a percentage of the whole sediment weight. Sediment moisture content is measured as the difference between wet weight of the sediment and dry weight following oven drying at 50 to 105°C to a constant weight. Percent water is used to convert sediment concentrations of substances from wet-weight to a dry-weight. Methods for determining moisture content are described by Plumb (1981 (213)) and Vecchi (1999 (259)). Additional methods are provided in USEPA (1987 (43)).

14.7 Salinity of the Pore Water (Marine Sediments):

14.7.1 Salinity is a measure of the mass of dissolved salt in a given mass of solution. The most reliable method to determine the true or absolute salinity is by complete chemical analysis. However, this is time consuming and costly. Therefore, indirect methods are more suitable. Indirect methods include conductivity, density, sound speed, or refractive index (APHA, 1995) (228). Salinity is then calculated from the empirical relationship between salinity and the indirect measurement. Conductivity measurements have the greatest precision, but respond only to ionic solutes (APHA, 1995 (228)). Density measurements respond to all solutes. APHA (1995 (228)) recommends the electrical conductivity method, because it is sensitive and easily performed. APHA (1995 (228)) also recommends the density method, using a vibrating flow densitometer.

14.7.2 A salinity refractometer can be used for quick readings of salt density in solutions such as sea water. These refractometers are easy to read, non-corrosive, and lightweight. They have dual scales and an adjustable focus. Temperature and non-temperature compensating refractometers are available. Most refractometers are accurate to 1 ppt and read specific gravity (1.000 to 1.070 in 0.001 divisions) and parts per thousand (0 to 100 in 1 part per thousand divisions).

14.8 Conductivity of the Pore Water (Freshwater Sediments):

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

14.9 Acid Volatile Sulfide (AVS):

14.9.1 Measurement of acid volatile sulfides (AVS) and simultaneously extracted divalent metal (SEM) concentrations associated with AVS extraction can provide insight into the bioavailability of metals in anaerobic (anoxic) sediments (Di

Toro et al. 1990 (50); Ankley et al. 1996 (51)). AVS is the reactive solid-phase sulfide fraction that is extracted by cold hydrochloric acid. AVS appears to affect the bioavailability of most divalent metal ions as the sulfide ions have a high affinity for divalent metals. This affinity results in the formation of insoluble metal sulfides with greatly reduced bioavailability. AVS concentrations in freshwater and marine sediments can range from less than 0.1 to greater than 50 µmol AVS/g of sediment (Di Toro et al. 1990 (50)).

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

14.9.3 The easily extractable sulfide fraction can be measured using the acid purge and trap technique. The sample sulfide is solubilized in cold hydrochloric acid. The analytical method involves conversion of sulfides to aqueous H₂S. This may be measured with a sulfide probe or by following a wet chemistry method. In the latter method, silver sulfide is precipitated in a gas-tight assembly and flushed with nitrogen to eliminate oxidation. The precipitate is filtered, dried, and weighed. The weight is compared with the weight obtained from a non-acidified sample, and the difference is attributed to the AVS fraction (Di Toro et al. 1990 (50)).

14.10 Simultaneously Extracted Metals:

14.10.1 A model for predicting toxicity from divalent trace metals (Di Toro et al. 1990 (50)) is based on the binding of these metals to AVS. Where the sum of the moles of the SEM, including Ag, Cd, Cu, Ni, Pb, and Zn is exceeded by the molar concentration of AVS, the metals are insoluble and largely unavailable to biota. The extraction of AVS and SEM metals should be achieved using a single methodology so that recoveries associated with each measure are consistent. Simultaneous extraction improves the efficiency of the methodology.

14.10.2 SEM can be measured in filtered aliquots by atomic absorption methods (Di Toro et al. 1990 (50)). Recent SEM analysis methods use inductively coupled plasma atomic emission spectrometry (ICP-AES; Berry et al. 1999 (260)). Other methods for analysis of metals are described in 14.11.

14.11 Metals:

14.11.1 Low levels of trace metals occur naturally in the environment but highly elevated levels in sediment are generally associated with anthropogenic contaminant loads. Metals are partitioned in sediments as soluble free ions, soluble organic and inorganic complexes, easily exchangeable ions, precipitates of metal hydroxides, precipitates with colloidal ferric and manganic oxyhydroxides, insoluble organic complexes, insoluble sulfides, and residual forms (Gambrell et al. 1976 (261)).

14.11.2 Current instrument methods available for the analysis of trace metals include electrochemistry (for example, differential pulse polarography), spectrophotometry (for example, silver diethyldithiocarbamate), atomic absorption spectrophotometry, atomic emission spectrophotometry, x-ray fluorescence (XRF), and neutron activation (PSEP 1997b (262)).

The most commonly used instrumental method to analyze sediments for metals is atomic absorption spectrophotometry (PSEP, 1997b (262)). Inductively coupled plasma mass spectrometry (ICP-MS) or ICP-AES allow for simultaneous determination of many metals at concentrations below a part per billion with little pretreatment (Crecelius et al. 1987 (263); Berry et al. 1999 (260)).

14.11.3 The concentration of salt in marine or estuarine samples may interfere with metals analyses (USEPA/USACE, 1998(34)). Therefore, acid digestion and atomic absorption spectroscopy should be coupled with an appropriate technique to control for this interference. Methods in USEPA (1986 (220)) are recommended for the analysis of mercury in sediments and EPRI (1986 (264)) methods are recommended for the analysis of selenium and arsenic. USEPA methods for cadmium, hexavalent chromium, copper, lead, mercury, nickel, selenium, silver, and zinc are described by USEPA (1986 (220)). PSEP (1997b (262)) suggests that mercury can be extracted using vacuum distillation and analyzed by gas chromatography/mass spectrometry.

14.12 Petroleum Hydrocarbons and Polycyclic Aromatic Hydrocarbons:

14.12.1 Petroleum hydrocarbons are oil and grease constituents which remain in solution after contact with silica gel. Petroleum distillates, also called hydrocarbons or petrochemicals, refer to a broad range of compounds that are extracted by distillation during the refining of crude oil. During the fractional distillation of petroleum, crude oil is heated to allow various compounds to turn from liquid into gas, and then are captured as they rise, cool, and condense. Lighter, more volatile compounds rise higher before they condense and are collected on distillation trays. Heavier, less volatile compounds such as diesel fuel and oil are collected on lower distillation trays. Waxes and asphalts are collected from the bottom after the other products have volatilized.

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

14.12.3 Aromatic and aliphatic hydrocarbons were analyzed in sediments by Page et al. (1995a, b (44),(45)). Sediment samples were spiked with the appropriate surrogates, mixed with equal amounts of sodium sulfate to dry the samples, and extracted with a methylene chloride acetone mixture (Method 3550, USEPA, 1986 (220)). The concentrated extracts were partitioned on an alumina column into saturated and unsaturated hydrocarbon fractions (Method 3611, USEPA, 1986 (220)). The fractions were concentrated using the appropriate pre-injection volume, spiked with the appropriate internal

standards, and analyzed by gas chromatography with flame ionization detection (GC/FID) and gas chromatography with mass spectrometry detection (GC/MS) operating in the selected ion monitoring (SIM) mode. The method of internal standards (Method 8000, USEPA, 1986 (220)) using the average relative response factors generated from the linear initial calibration was used to quantify the target compounds. All data were corrected for the recovery of the appropriate surrogate compound. Their relative abundances could then be used for identification and quantification purposes.

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

14.12.5 Kaplan et al. (1996 (265)) extracted hydrocarbons using anhydrous Na₂SO₄ with methylene chloride and sonication. The total solvent extract was then concentrated with Kuderna-Danish equipment. The concentrate was further concentrated using a gentle stream of dry nitrogen. An aliquot was then injected directly into the gas chromatography.

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

14.13.1 Analytical techniques for measuring organic compounds require five general steps: drying the sample, extraction, drying the extract, clean up of the extract, and analysis of the extract. PSEP (1997c (266)) recommends centrifugation or sodium sulfate to dry the sample and a solvent extraction, with application of shaker/roller, or sonication. Sample drying with sodium sulfate is recommended for samples weighing about 10 g (after overlying water is decanted). The sediment and sulfate mixture is extracted and the extract is processed (MacLeod et al. 1985 (267)).

14.13.2 Soxhlet® extraction (USEPA, 1986 (220)) involves distillation with a solvent such as acetone, dichloromethane/methanol (2:1), dichloromethane/methanol (9:1), and benzene/methanol (3:2). USEPA (1983 (52)) recommends sonication with solvent mixtures and a 30-g subsample of sediment.

14.13.3 Drying the extract can be accomplished through separatory funnel partitioning as needed to remove water and sodium sulfate or by using a Kuderna-Danish apparatus and rotary evaporation with purified nitrogen gas for concentration to smaller volumes (PSEP, 1997c (266)). Using the separatory funnel partitioning method, the wet sample is mixed with methanol and centrifuged. The supernatant is decanted and extracted later. Extraction of the sample is continued using less polar solvents and the water/methanol and solvent extracts are combined and dried.

14.13.4 According to PSEP (1997c (266)) elemental sulfur can be removed from the sediment sample with vigorous mechanical agitation using a Vortex or Genie⁴ or using activated copper. Organic interferences can be removed with gel permeation chromatography (GPC) described in USEPA

(1983(52)), bonded octadecyl columns (PSEP, 1997c (266)), high performance liquid chromatography (HPLC) described by Metro (1981 (268)), silica gel (PSEP, 1997c (266)), or alumina (USEPA, 1983) (52). Instrumental analyses for volatiles and semivolatiles and pesticides/PCBs are performed using gas chromatography/mass spectrophotometry (GC/MS; PSEP, 1997c (266)) and gas chromatography/electron capture detection (GC/ECD; Burgess and McKinney, 1997 (269)).

14.14 Oil and Grease:

14.14.1 Oil and grease tests for sediments measure material recovered that is soluble in a non-ionic solvent under acidic conditions. Oil and grease compounds are substances such as hydrocarbons, vegetable oils, animal fats, waxes, soaps, and greases. Many solvents can dissolve other substances (for example, sulfur compounds, organic dyes, and chlorophyll). Therefore, oil and grease is operationally defined by the solvent used and the analytical method used to perform the analysis. There are two basic methods used to analyze oil and grease: the gravimetric technique and the IR (infrared spectrophotometer) technique. Both are described by PSEP (1996 (239)).

14.15 Total Sulfides:

14.15.1 Total sulfides represent the combined amount of acid-soluble H₂S, HS-, and S²⁻ in a sample. Sulfides are often measured because they are common in some sediments, particularly those that are anoxic, and they can be toxic to aquatic organisms. PSEP (1996 (239)) describes a method to measure total sulfides in sediments. Oxygen is removed from the sample using nitrogen gas, methyl orange and hydrochloric acid is added, and the mixture is heated. Amine solution and iron chloride are added to develop a colorimetric reaction product and sample absorbance is measured spectrophotometrically. Elevated concentrations of ammonia (Sims et al. 1995a (223), Moore et al. 1997 (224)) and hydrogen sulfide (Sims et al. 1995b (225)) have frequently been found in deeper dredged sediment samples compared to surficial sediment samples.

14.15.2 Methods for measuring sulfides in aqueous samples include: potentiometric methods described by Practice D 3976 and APHA (Method 4500, 1995 (228)). Sulfide ions are measured using a sulfide ion-selective electrode in conjunction with a double-junction, sleeve type reference electrode (Phillips et al. 1997 (270)). Potentials are read using a pH meter or a specific ion meter having a direct concentration scale for the sulfide ion. Samples are treated with sulfide anti-oxidant buffer that fixes the solution pH at a high alkaline level and retards air oxidation of sulfide ion in solution. This ensures that the sulfide measured represents total sulfides as S = ion rather than the HS- or H₂S found at lower pH values.

14.15.3 APHA (Method 4500, 1995 (228)) provides qualitative as well as quantitative methods to determine aqueous sulfide concentrations. Qualitative methods include the antimony test, the silver-silver sulfide electrode test, the lead acetate paper test, and the silver foil test. Quantitative methods include the photometric method, the automated photometric methylene blue colorimetric methods, and the iodometric titration method for standardizing stock solutions.

14.16 Sediment Oxygen Demand (SOD):

⁴ Genie is a trademark of Scientific Industries Inc. 70 Orville Drive, Bohemia, New York 11716.

14.16.1 Sediment can exhibit significant rates of oxygen uptake attributable to either: (1) a benthic ecosystem supported by soluble organic substances in the water column, (2) naturally occurring sediments derived from aquatic plants and animals, and (3) detritus discharged into the water body by natural runoff. When numerical modeling is required to predict dissolved oxygen concentrations, the rate of dissolved oxygen consumed by the benthic ecosystem is defined as the sediment (benthic) oxygen demand (SOD) in g O₂/m²-day.

14.16.2 Two approaches for measuring SOD were reviewed by Truax et al. (1995 (271)) including in-situ respirometry and laboratory respirometry methods. Numerous techniques have been developed for each approach. Generally, in-situ methods are considered more credible than laboratory measurements although both apply the same technique. A given amount of sediment is enclosed in a chamber with a known water volume and oxygen uptake is measured over time. The SOD rate is then calculated based on the area of the enclosed sediment, the volume of water in the chamber, and the rate of uptake.

14.16.3 In situ sediment oxygen demand measurement methods were described by Uchrin and Ahlert (1985 (272)). A cylindrical respirometer, a dissolved oxygen probe with stirring mechanism, and a dissolved oxygen meter were used. Ambient dissolved oxygen was measured using the probe/meter as well as by using the Winkler method (APHA, 1995 (228)) in the laboratory to determine the effect of respiration on total dissolved oxygen uptake. The respirometer was deployed in a level area at the bottom of the water body. Dissolved oxygen were recorded initially and at 15-min intervals thereafter to determine the SOD rate.

14.17 *Sediment Biochemical Oxygen Demand (BOD):*

14.17.1 Biochemical oxygen demand (BOD) is a measure of the dissolved oxygen consumed by microbial organisms while assimilating and oxidizing the organic matter in a sample (PSEP, 1996 (239)). The test is an empirical methodology in which consistent laboratory procedures are used to determine the relative oxygen uptake of environmental samples. The test measures the amount of molecular oxygen used during a specified incubation period to biochemically degrade organic material and to oxidize reduced forms of nitrogen (APHA, 1995 (228)).

14.17.2 Plumb (1981 (213)) described a method to analyze BOD in sediments using freshwater bacteria as a “seed” and buffered distilled water. PSEP (1996 (239)) described an alternative procedure to analyze BOD in marine sediments using marine bacteria as the “seed” and filtered, oxygenated seawater. USEPA (1987 (43)) methods should also be consulted.

14.18 *Sediment Chemical Oxygen Demand (COD):*

14.18.1 Chemical oxygen demand (COD) is a measure of the oxygen equivalent of organic matter content in a sample that is susceptible to oxidation by a strong chemical oxidant at elevated temperature and reduced pH. The test was devised to augment the biochemical oxygen demand test. Chemical oxygen demand can be related empirically to biochemical oxygen demand, organic carbon, or total volatile solids (PSEP, 1996 (239)).

14.18.2 PSEP (1996 (239)) described a method for analyzing sediment COD using a closed reflux/colorimetric method. Dichromate (Cr₂O₇) ions are used to oxidize organic matter to carbon dioxide and water and to provide oxygen. The dichromate ions remaining after the reaction are measured by titration and the amount of oxygen consumed is then calculated.

14.18.3 Four standards procedures for measuring COD in water are available in APHA (1995 (228)): the open reflux method, the closed reflux method, the titrimetric method, and the closed reflux/colorimetric method. USEPA (1983 (52)) methods for the colorimetric and titrimetric method are described in USEPA (1979 (219)). Semi-automated methods are described in USEPA (1993 (48)).

14.19 *Cation Exchange Capacity of Sediments:*

14.19.1 Cation exchange capacity (CEC) is a parameter that provides information relevant to metal bioavailability studies (Black, 1965 (211)). Cations or positively charged elements (such as calcium, magnesium, hydrogen, and potassium), are attracted to negatively charged surfaces of clay and organic matter. There is a continuous exchange of cations between sediment and water. CEC is a measure of the sediment’s ability to retain cationic elements. It is also a measure of clay activity and mineralogy, which is used to calculate mineralization rates, leaching rates, and to predict interactions with contaminants. The degree of CEC is dependent on the kind and amount of suitable surfaces such as organic matter and clay. High cation exchange capacities are associated with high clay contents and high organic matter and changes in CEC are typically associated with changes in organic carbon content and pH of the sediment. Organic matter generally supplies a greater number of exchange sites than clay particles.

14.19.2 Various methods have been recommended to determine bioavailable fractions of metals in sediments (Chao and Zhou, 1983 (273); Crecelius et al. 1987 (263); Kersten and Forstner, 1987 (274); Di Toro et al. 1990 (50)). CEC can be measured by treating samples with ammonium acetate so that all exchangeable sites are occupied by NH₄⁺ ion, digesting the samples with sodium hydroxide during distillation, and titrating to determine the ammonium ion concentration. The amount of exchangeable cations are expressed as milliequivalents of ammonium ion exchanged (meq) per 100 g of dried sample. More detailed methods are provided in Bascomb (1964 (275)), Black (1965 (211)), Klute (1986 (276)), and USEPA (1986 (220)).

14.20 *Redox Potential (Eh) of Sediments:*

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

14.20.2 Potentiometric measurements of Eh using a millivolt reader can be obtained with a platinum electrode relative

to a standard hydrogen electrode (Plumb, 1981 (213)). APHA (1995 (228)) does not recommend the standard hydrogen electrode as it is fragile and impractical. Instead, their method uses a silver-silver-chloride or calomel reference electrode. APHA (1995 (228)) recommends a graphite rather than platinum electrode for sediments. Once the Eh equilibrium is reached, the difference between the platinum or graphite electrode and the reference electrode is equal to the redox potential of the system. For a more detailed explanation on how to calculate the Eh potential see APHA (1995 (228)). Gonzalez (1995 (217)) also describes a detailed method that can be used to measure sediment Eh.

14.20.3 There are a number of problems associated with the accurate measurement and interpretation of Eh in sediments, particularly in marine sediments. Therefore, considerable attention should be paid to the use of proper equipment and techniques. Some of the problems identified by Whitfield (1969 (277)) and Murdoch and Azcue (1995 (46)) include measurement inaccuracy due to disturbance of the sediment sample during insertion of the electrode, instability and poor reproducibility of the measurements, and differential responses of platinum electrodes under different environmental conditions. A comprehensive description of the limitations of sediment Eh measurement is beyond the scope of this standard. Rather, it is recommended that published studies on the problems associated with measuring and interpreting sediment Eh be consulted before any attempt is made to measure these parameters in sediment samples (Berner, 1963 (278); Morris

and Stumm, 1967 (279); Whitfield, 1969 (277); Tinsley, 1979 (280); Bates, 1981(281)). The recommended procedure for measuring pH and Eh in the field are described in detail in Table 15.

14.21 Total Inorganic Carbon:

14.21.1 Inorganic carbon has been measured as a complement to microbial activity (Bregnard et al. 1996 (282)), to determine the fate of an organic contaminant in biodegradation studies (West and Gonsior, 1996 (283)), and to determine the percent carbon unaccounted for in fate transport predictions of hydrophobic contaminants (Tye, et al. 1996 (284)). Often the total inorganic carbon (TIC) fraction in samples is many times greater than the TOC fraction and presents an interference in the measurement of TOC. There are several options to eliminate TIC interferences when trying to measure TOC. One option is to compensate for the IC interference by measuring total carbon (TC) and total inorganic carbon (see 14.4). The difference between the two is the TOC.

14.21.2 TIC is determined by acidifying the sample to convert the inorganic carbon (that is, carbonates, bicarbonates, and dissolved CO₂) to carbon dioxide. Carbon dioxide is purged from the sample and then detected by a non-dispersive infrared detector (NDIR) calibrated to directly display the mass of carbon dioxide measured. This mass is proportional to the mass of TIC. Other instrumentation for the analysis of TIC is described in West and Gonsior (1996 (283)) and Tye et al. (1996 (284)).

14.22 Total Volatile Solids (TVS):

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

Equipment and solutions used in the measurements:

A portable, battery-operated pH/Eh meter, batteries, and a power cord for recharging the meter.
 Combination glass and platinum electrodes or other electrodes suitable for the measurements.
 Plastic test-tube-shaped containers or other containers for storing the electrodes in solutions during transport in the field.
 Commercially-available or laboratory-prepared pH buffer solutions (pH 4 and 7) in plastic bottles with lids.
 Freshly-prepared solution for calibration of Eh electrode in a plastic bottle with a tight lid.
 Freshly-prepared solution of saturated potassium chloride for storage of the electrodes.
 Other solutions necessary for proper functioning of electrodes as outlined by manufacturers.
 Deionized water and wash bottle for storing and rinsing the electrodes between measurements.
 Several small and larger plastic beakers for holding solutions, rinsing electrodes, etc..
 Support stands, rods, clamps to secure electrodes in solutions and during measurements.
 Large plastic containers for storage and transport of used buffers and Eh-calibration solutions.
 Notebook and pens, soft paper tissue.

Preparation of equipment before the field trip:

Check batteries of the portable pH/Eh meter and replace/recharge them, if necessary.
 Prepare calibration solutions.
 Check and test the pH and Eh electrodes.
 Mark the electrodes vertically at desired intervals for insertion into the sediment samples.
 Store the electrodes according the manufacturers instructions.
 Pack all equipment for transport to the field and take spare electrodes if available.

Measurements in the field:

Allocate a space where measurements will be carried out. Within this space, all equipment should be assembled, checked for proper functioning, and prepared for measurement of the first sample.
 Place grab sampler and sediment cores with recovered sediment in such a way that they will remain steady without disturbing the sediment samples during the measurements.
 Insert electrodes carefully into the undisturbed sediment samples to avoid any air. contamination, particularly around the Eh electrode. Care must be taken not to generate any open space between the electrode and the sediment. Proper insertion of the electrode without disturbing the sediment is the most important step in measuring the Eh.
 Insert electrodes into the sediment to the depth marked. Switch the pH/Eh meter to the pH scale and the value recorded within 1 minute after inserting the electrode into the sample. Switch the meter to the mV scale for recording the Eh value. The potential usually drifts considerably over the first 10 to 15 min, and then stabilizes. After stabilization, record the mV value. In measuring Eh of sediments from waters with low ionic strength, such as most freshwater bodies, it is recommended to "acclimatize" the electrodes in the water prior to measurement, particularly the electrodes that were stored in saturated potassium chloride solution. This will reduce the drifting of the potential after inserting the electrode into the sediment.
 Remove both electrodes, wash them with distilled water to remove all adhering sediment particles, and dry them gently with a soft paper tissue.
 Calibrate the electrodes after each five measurements. The electrodes may need less frequent calibration if pH and Eh are being measured in a sediment core.

14.22.1 Total volatile solids represent the fraction of total solids that are lost on ignition at a higher temperature than that used to determine total solids. Total volatile solids are used as a crude estimate of the amount of organic matter in total solids (PSEP, 1996 (239)). In this regard, total volatile solids are often measured instead of, or in addition to, organic carbon content.

14.22.2 Total volatile solids are operationally defined by ignition temperature. Total volatile solids content does not always represent the organic content of a sample because some organic material may be lost at the drying temperature and some inorganic material (for example, carbonates, chlorides) may be lost at the ignition temperature. Because of the temperature dependence of total volatile solids, valid inter-study comparisons require the use of consistent drying and ignition temperatures (PSEP, 1996 (239)).

14.22.3 Total volatile solids measurements are generally made by igniting the sediments at $550 \pm 10^{\circ}\text{C}$ until a constant weight is achieved and reporting the percent ash-free dry weight (McLeese et al. 1980 (131); APHA, 1995 (228); Keilty et al. 1988a (78)). Plumb (1981) (213) and PSEP (1996) (239) describe standard methods for determining the total volatile solid content of sediments. Additional methods are provided in USEPA (1987) (43).

14.23 Dissolved Organic Carbon in Pore Water:

14.23.1 Dissolved organic carbon (DOC) often consists of humic substances, and is the fraction of the organic carbon pool that is dissolved in water and passed through a $0.45\text{ }\mu\text{m}$ glass fiber filter. DOC is an indicator of the chemically reactive organic fraction and accurately measures the dissolved organic load. Sediment pore waters can be rich in humic acids. Fifty to 90 % of the pore water DOC can be colloidal which is a significant factor because organic chemicals will preferentially partition to pore water DOC (Resendes et al. 1992 (285); Burgess 1996 (151)). Dissolved organic carbon (DOC) often consists of humic substances, and is the fraction of the organic carbon pool that is dissolved in water and passed through a $0.45\text{ }\mu\text{m}$ glass fiber filter. DOC is an indicator of the chemically reactive organic fraction and accurately measures the dissolved organic load. Sediment pore waters can be rich in humic acids. Fifty to 90 % of the pore water DOC can be colloidal which is a significant factor because organic chemicals will preferentially partition to pore water DOC (Resendes et al. 1992 (285); Burgess 1996 (151)).

14.23.2 Hermann (1996 (286)) and Gilek et al. (1996 (287)) measured DOC using a TOC apparatus and infrared detection of CO_2 . Borga et al. (1996 (288)) measured DOC using atomic emission spectrometry (ECP-AES). The APHA (Method 5310, 1995 (228)) methods for total organic carbon that can be applied to the measurement of DOC are (*a*) the combustion-infrared method; (*b*) the persulfate-ultraviolet oxidation method; and (*c*) the wet-oxidation method. Adjustments for inorganic carbon interference may be required.

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

14.24.1 Alkalinity is defined as the acid-neutralizing (that is, proton-accepting) capacity of water. It is the sum of all the titratable bases, and a measure of the quality and quantity of constituents in the pore water that result in a shift in the pH

toward the alkaline side of neutrality. The measured value may vary significantly with the pH end-point used. Studies have shown that effects of certain contaminants such as metals are influenced by alkalinity as it alters speciation and bioavailability.

14.24.2 APHA (1995 (228)) recommends a color-change titration method to measure alkalinity which is also described by Test Method D 1067. The sample is titrated with standard alkali or acid to a designated pH and the endpoint is determined electrometrically or by the color change of an internal standard. In addition, Test Method D 1067 describes two additional methods: (*1*) a titration curve is developed to identify inflection points, a standard acid or alkali is added to the sample by small increments and pH is recorded after each addition, and the total volume of acid or alkali is plotted against the observed pH values; and (*2*) pH is determined, standard acid is added to lower the pH to 4.0 or less, the solution is boiled with hydrogen peroxide, and titrated, while hot, to the phenolphthalein endpoint or, when cooled, electrometrically with standard alkali to pH 8.2, the desired endpoint. The color-change titration method is most commonly used.

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

14.24.4 APHA (Method 2340, 1995 (228)) describes two methods to measure hardness: (*1*) the calculation method and (*2*) the EDTA titrimetric method. Test Method D 1126 describes the APHA (1995 (228)) EDTA titrimetric method. Calcium and magnesium ions in water are sequestered by the addition of EDTA. The endpoint of the reaction is measured by means of Chrome Black T3, which is red in the presence of calcium and magnesium and blue when both are sequestered. APHA recommends the calculation method because it is more accurate. The method uses direct determinations of calcium and magnesium to determine hardness. The APHA EDTA titration method is most often used.

15. Quality Assurance

15.1 General Procedures:

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

15.1.2 Developing and maintaining a quality assurance (QA) program requires an ongoing commitment by project management and also includes the following: (*1*) appointment of a quality assurance officer with the responsibility and authority to develop and maintain a QA program, (*2*) preparation of a Quality Assurance Project Plan with Data Quality Objectives, (*3*) preparation of written descriptions of Standard

Operating Procedures (SOPs) for sediment sampling and manipulations, instrument calibration, sample chain-of-custody, laboratory sample tracking system, and (4) provision of adequate, qualified technical staff and suitable space and equipment to provide reliable data. Program specific guidance for developing and maintaining a QA program should be followed as appropriate. Examples of program guidance for developing a quality assurance program can be found in USEPA (1994 (74); 1995 (252); 2000d (35)), PSEP (1997a) (33), WDE (1995) (29), and USEPA/USACE (1991 (32), 1998 (34)).

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

15.2 QA/QC Procedures for Sediment Collection and Manipulation:

15.2.1 To establish the appropriateness of the sample collection procedure for sample integrity and to establish that data of suitable quality, a program of scheduled field QC samples, such as field replicates (duplicates, splits, field spikes), field blanks (rinsate equipment), bottle, trip, and background (up-gradient) samples. All field QC samples should be handled exactly as the sediment samples and should be treated as blind samples so as to minimize bias in the analysis. A random portion of the samples should also be analyzed by a third party to evaluate the primary laboratory's performance. QC replicates (duplicates, splits) should be collected for analysis by the primary laboratory to determine analytical variability (USEPA 1995 (252)).

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

cal spiking), maintain sediment physical properties, and include replication and blank samples.

15.3 The Quality Assurance Project Plan (QAPP):

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

15.3.2 The QAPP uses input from the sampling design derived from the Data Quality Objectives Process (see Section 9, 9.6, and USEPA, 2000a (12)) to specify the above elements. This Plan should be reviewed by an independent person (for example, quality assurance officer or staff member not involved in the project directly) for accuracy and completeness. A key element of a QAPP is Standard Operating Procedures (see 15.4). Further information on preparing a QAPP and resources necessary can be found in USEPA (2000e (289)).

15.4 *Standard Operating Procedures*—Standard operating procedures are written descriptions of routine methods and should be provided for all methods used. A large number of field and laboratory operations can be described in standard operating procedures. General types of procedures that benefit from standard operating procedures include field measurements ancillary to sample collection (for example, water quality measurements or mixing model input measurements); chain-of-custody, sample handling, and shipment; and routine analytical methods for chemical analyses and toxicological analyses. Standard operating procedures are used to establish that all persons conducting work are following the same procedures and that the procedures do not change over time. All personnel should be familiar with the standard operating procedures before work is initiated. Deviations from standard operating procedures might affect data quality and integrity. If it is necessary to deviate from approved standard operating procedures, these deviations need to be documented and approved through an appropriate chain-of-command.

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

15.6 Sample Tracking Documentation:

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

are put into a secure location and the transfer is conducted when the appropriate personnel are present.

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

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

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

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

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

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

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

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

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

15.9.5 Corrective actions relative to sample collection and manipulation may include, but are not limited to, review of the data and calculations, flagging or qualification of suspect data, or possible re-sampling. A review that provides a preliminary check of all "out of limit" events is performed as soon as the data for a given parameter or test is tabulated and verified for accuracy. "Out of limit" events are flagged to determine whether new samples are required.

16. Report

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

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

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

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

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

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

17. Keywords

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

ANNEX

(Mandatory Information)

A1. DESCRIPTION OF SAMPLERS USED TO COLLECT SEDIMENT OR BENTHIC INVERTEBRATES**A1.1 Significance**

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

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

D 4387-84 (2002) Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates

D 4556-85 (2002) Guide for Selecting Stream-Net Sampling Devices for Collecting Benthic Macroinvertebrates

D 4342-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Ponar Grab Sampler

D 4343-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Ekman Grab Sampler

D 4344-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Smith-McIntyre Grab Sampler

D 4345-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Van Veen Grab Sampler

D 4346-84 (1997) Practice for Collecting Benthic Macroinvertebrates with Okean 50 Grab Sampler

D 4347-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Shipek (Scoop) Grab Sampler

D 4348-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Holme (Scoop) Grab Sampler

D 4401-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Petersen Grab Sampler

D 4407-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Orange Peel Grab Sampler

D 4557-85 (2002) Practice for Collecting Benthic Macroinvertebrates with Surber And Related Type Samplers

D 4558-85 (2002) Practice for Collecting Benthic Macroinvertebrates with Drift Net

E 1468-92 (2002) Practice for Collecting Benthic Macroinvertebrates with Basket Sampler

E 1469-92 (2002) Practice for Collecting Benthic Macroinvertebrates with Multiplate Sampler

A1.2 Terminology Specific to this Annex

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

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

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

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

A1.3 Significance and Use

A1.3.1 Grab samplers for collecting sediments or benthic macroinvertebrates: Qualitative and quantitative samples of macroinvertebrates inhabiting sediments or substrates are often collected using a grab sampler. In view of the advantages and limitations regarding the penetration of the sediment by many grab samplers and their closing mechanisms, it is not possible to recommend any single instrument as suitable for general use. However, the Petersen grab is considered the least effective bottom grab sampler and, therefore, has limited application. The type and size of the grab sampler or device selected for use will depend on such factors as the size of boat, hoisting gear available, the type of substrate or sediment to be sampled, depth of water, current velocity, and whether sampling is in sheltered areas or in open waters of large rivers, reservoirs, lakes, and oceans. A great variety of instruments have been described and choice of a grab sampler will depend largely on what is available, what is suitable for the sampling area, and what can be obtained without difficulty. This annex describes the following grab samplers for collecting sediment or benthic macroinvertebrates: (1) Ponar, (2) Ekman, (3) Petersen, (4) Smith-McIntyre, (5) Van Veen, (6) Orange-Peel, (7) Okean 50, (8) Shipek, and (9) Holme. Tables A1.3 and A1.4 describe advantages and disadvantages of commonly used grab or core samplers.

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

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

Grab Sampling Device	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Ponar Grab	Freshwater lakes, rivers, and estuaries, reservoirs	Hard sediments, except hard clay; some-what less efficient in softer sediments	Sample area 523 cm ² ; efficient and versatile; not entirely adequate for deep burrowing organisms in soft sediments; quantitative and qualitative sampling obtained; sediment inhabiting macro-invertebrates	Better penetration than other grabs; side plates and screens pre-vent washout and shock wave that accompany other grabs	Requires boat, winch, and cable; jaws can be blocked and part of sample lost	Better for quantitative sampling than Petersen grab	Brinkhurst (297, 298) Elliot and Drake (299) Elliott and Tullet (300) Flannagan (301) Howmiller (302) Hudson (303) Lewis, Mason, Weber (304) Powers and Robertson (305) Weber (306) Klemm et al. (353)
Petite	Freshwater lakes, rivers, and estuaries, reservoirs	Hard sediments, except hard clay; some-what less efficient in softer sediments	Sample area 232 cm ² ; efficient and versatile; not entirely adequate for deep burrowing organisms in soft sediments; sediment inhabiting macro-invertebrates	Better penetration than other grabs; side plates and screens prevent washout and shock wave that accompany other grabs; can be operated by hand	Jaws can be blocked and part of sample lost; insufficient in swiftly moving water to 1 m/s velocity		Klemm et al.(353) Merrit et al. (354) Gerritsen et al. (355)
Ekman Grab	Freshwater lakes, reservoirs, where there is little current; usually small bodies of water	Soft sediments only	Sample area 232 cm ² ; efficient in soft sediments; extra weights can be used for deeper penetration; quantitative and qualitative obtainable; sediment inhabiting macroinvertebrates	Can be operated by hand; can be operated in shallow, sand or mud bottom streams; comes in a range of sizes	Jaws can fail to penetrate; only partial cylinder cut from substrate, small surface area coverage jaws can be blocked and part of sample lost; inefficient in deep water or moderate to strong currents		Beatties (307) Burton and Flannagan (308) Ekman (309,310) Elliot and Drake (299) Elliott and Tullett (300) Flannagan (301) Howmiller (302) Hudson (303) Lanz, (311) Lewis, Mason, Weber (304) Lind (312) Milbrink and Wiederholm (313) Rowe and Clifford (314) Lewis et al.(356) Klemm et al. (353) Merritt et al. (354) Gerritsen et al.(355)
Tall	Same as above	Same as above	Sample area 232 cm ² Same as above	Same as above	Same as above		Paterson and Fernando (315) Schwoerbel (316)
Large	Same as above	Same as above	Sample area 523 cm ² Same as above	Same as above	Same as above		Rawson (317) Welch (318) Weber (306)
Extra Large	Same as above	Same as above	Sample area 929 cm ²	Same as above	Same as above		
Petersen Grab	Freshwater lakes, reservoirs; adaptable to rivers, estuaries, and oceans	Sand, gravel, mud, clay	Sample penetration limited sample area from 0.06 to 0.099 m ² ; sediment inhabiting macroinvertebrates	Gives reasonable quantitative samples when used carefully; comes in a range of sizes	Fairly heavy; need boat and power winch; jaws maybe blocked by sand, etc.; inadequate for deep burrowing organisms; questionable value for strictly quantitative samples; hard to use in adverse weather conditions	Least preferred grab sampler	Barnes (319) Birkett (320) Brinkhurst, (297,298) Davis (321) Edmondson and Winberg (322) Davis (321) Elliott and Tullett (300) Holme and McIntyre (323) Hudson (303) Howmiller (302) Lewis, Mason, Weber (304) Lind (312) Petersen (324) Thorson (325) Welch (318) Weber, (306) Petersen and Boysen Jensen (326)Klemm et al.(353)

TABLE A1.1 *Continued*

Grab Sampling Device	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Smith-McIntyre Grab	Marine and estuaries; adaptable to large rivers, lakes	Sand, gravel, mud, clay, and similar substrates	Sample area limited to 0.1 m ² with approximately 4 cm deep in hard sand; reasonably quantitative; sediment inhabiting macro-invertebrates	Reasonable quantitative samples; the trigger plates provide added leverage essential to its penetration of substrate	Heavy; need boat and power winch; spring-loaded jaws, hazardous; jaws can be blocked; inadequate for deep burrowing organisms	Widely acceptable sampling device for use in marine and estuary habitats	Carey and Heyamoto (327) Carey and Paul (328) Elliott and Tullett (300) Holme (329,330) Hopkins (331) Hunter and Simpson (332) McIntyre (333) Smith and McIntyre (334) Tyler and Shackley (335) Wigley (336) Word (337) Klemm et al.(353)
Van Veen Grab	Marine and estuaries, adaptable to freshwater areas	Sand, gravel, mud, clay, and similar substrates	Sample area 0.1 m ² and 0.2 m ² ; reasonable penetration; to depth of approximately 5 to 7 cm; sediment inhabiting macroinvertebrates	Jaws close tighter than Petersen grab; samples most sediment types; comes in a range of sizes	Need large boat, power winch and cable line; blockage of jaws may cause sample loss; not useful for deep burrowing organisms	Limited application	Barnes (319) Beukema (338) Birkett (320) Elliott and Drake (299) Elliott and Tullet (300) Holme (329,330) Lassig (339) Longhurst (340) McIntyre (333) (341) Nichols and Ellison (342) Schwoerbel (316) Ursin (343) Wigley (336), Word (344,345) Word (337) Klemm et al (353)
Orange-Peel Grab	Marine waters, deep lakes	Sandy substrates, cobble, rubble stone	Sample area 0.025 m ² ; penetration depth about 18 cm; qualitative sampler, not a satisfactory quantitative sampler; should not be used in critical quantitative work that is to be compared with results from other sampling areas; sediment inhabiting macro-invertebrates	Comes in a range of sizes	Need large boat, powered which and cable line; blocking of jaws may cause sample loss	Limited application; reconnaissance sampling only	Briba and Reys (346) Elliott and Tullett (300) Hartman (347) Hopkins (331) Merna (348) Packard (349) Reish (350) Thorson (325) Word (344)Klemm et al (353)
Okean 50 Grab	Marine, estuarine, also large rivers	Sand, gravel, mud, clay, similar substrates	Sample area 0.25 m ² ; should be lowered slowly for quantitative work; moderately deep penetration in hard sand; better for quantitative sampling than Petersen grab; sediment inhabiting macroinvertebrates	Moderately deep penetration in hard sand; gauze covered window at top of each bucket to allow water to escape while grab is closing; offer some resistance to swift currents; lowering of grab desirable for deep sea sampling; may also have hinged doors instead of screened windows; rapid rates of lowering are possible; comes in a range of sizes	Heavy; requires large boat, powered winch and cable line; jaws may be blocked and sample lost; not entirely adequate for deep burrowing organisms; should be lowered slowly for quantitative sampling		Elliott and Tullett (300) Holme (329,330) Holme and McIntyre (323) Lisitsin and Udintsen (351) Zhadin (352)

TABLE A1.1 *Continued*

Grab Sampling Device	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Shipek Grab	Estuarine areas, also large freshwater lakes	Sand, gravel, mud, clay, and similar substrates	Sample area 20 cm ² , approximately 10 cm deep at left; sediment inhabiting macroinvertebrates	Scoop type sampler	Heavy; requires boat, powered winch and line; should be lowered on a near vertical line; inadequate for deep burrowing organisms; sampled area may be rather small for quantitative work	Limited application	Barnes (319) Elliott and Tullett (300) Flannagan (301) Holme (329,330) Holme and McIntyre (323)
Holme Grab	Marine, estuarine areas, deep lakes	Sand, gravel, mud, clay, and similar substrates	Sample area 0.05 m ² , approximately 15 cm. in hard sand, etc., sediment inhabiting macroinvertebrates	Scoop type sampler; comes with a single scoop or double scoop	Heavy; requires boat, powered winch and line; springs of scoop may be difficult to reset; inadequate for deep burrowing organisms	Limited application	Barnes (319) Elliott and Tullett (300) Holme (329,349) (39) Holme and McIntyre (323) Thorson (325)

water column from all types of substrates in flowing waters. These devices are used to determine the drift of benthic organisms from a variety of substrate types at one time.

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

A1.4 General Hazards

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

A1.4.2 Exercise caution when handling the samplers.

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

A1.5 Descriptions of Samplers

A1.5.1 Ponar Grab Sampler:

A1.5.1.1 A Ponar Grab Sampler (Fig. A1.1) is designed to obtain quantitative samples of macroinvertebrates from sediments in lakes, rivers, estuaries, oceans, and similar habitats. This device is most useful for collecting benthic macroinvertebrates from a wide range of bottom substrate types, for example, coarse sand, fine gravel, clay, mud, marl, and similar

substrates. The sampler can be used in swift currents and deeper waters. The sampler is available in a range of sizes from 23 to 15 cm.

A1.5.1.2 The Ponar grab sampler has paired jaws that should penetrate beneath the surface of the substrate without disturbing the water surface boundary layer of the substrate, close when positioned properly on the bottom, and retain discrete samples of sediment while it is brought to the surface for processing. The Ponar collects a sample from an area of about 523 cm². A small version, the petite Ponar grab, takes a sample area of 232 cm² and can be used in habitats where there may be an unusual abundance of macroinvertebrates, thus eliminating the need to subsample. The Ponar grab sampler is used to collect qualitative and quantitative samples from different aquatic habitats containing benthic macroinvertebrates living on or in various types of substrates.

A1.5.1.3 Hazards:

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

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

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

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

A1.5.1.4 Procedure:

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

(2) The device should have a controlled lowering speed and should be lowered slowly because free-fall may airplane the

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

Stream-Net Samplers	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Surber sampler	Shallow, flowing waters, depth recommended	Mud, sand, gravel, or rubble substrates	Depends on experience and ability of user; area sampled 0.1 m ² ; performance depends on current and substrate; size of macroinvertebrates collected depends on mesh size; variety of mesh sizes may be used.	Easily transported or constructed; samples a unit area; partial screen enclosure	Does not produce quantitative samples consistently; clogging with sand or algae; difficult to set in some substrate types, that is, large rubble; cannot be used efficiently in still or deep water.	Can be modified to fit difficult situations.	Elliot and Tullett (357) Ellis and Rutter (358) Lane (359) Merritt, Cummins, and Resh (360) Needham and Usinger (361) Pollard and Kinney (362) Rutter and Ettinger (363) Resh (364) Rutter and Poe (365) Surber (366) (367) Welch (368) Kroger (369) Klemm et al (353)
Portable invertebrate box sampler	Same as above	Same as above	Same as above	Same as above; completely enclosed; limits escape of organisms; stable platform; can be used in weed beds.	Same as above	Same as above	Resh, et al (370)
Hess sampler	Same as above	Same as above	Same as above	Same as above; completely enclosed; limits escape of organisms; can be used in weed beds.	Same as above	Same as above	Canton and Chadwick (371) Elliott and Tullett (357) Hess (372) Merritt, Cummins, and Resh (360) Pollard and Kinney (362) Resh (364) Usinger (373) Welch (368) Resh, et al (370) Klemm et al.(353)
Hess stream bottom sampler	Shallow, flowing waters, depth recommended	Mud, sand, gravel, or rubble substrates	Depends on experience and ability of user; area sampled 0.1 m ² ; performance depends on current and substrate; size of macroinvertebrates collected depends on mesh size; variety of mesh sizes may be used.	Easily transported, or constructed; samples a unit area completely enclosed; limits escape of organisms; can be used in weed beds.	Does not produce quantitative samples consistently; clogging with sand or algae; difficult to set in some substrate types that is, large rubble; cannot be used efficiently in still or deep water.	Can be modified to fit difficult situations	
Stream-bed fauna sampler	Same as above	Same as above	Same as above	Same as above	Same as above	Same as above	
Drift nets	Flowing rivers and stream	Drifting benthic macroinvertebrates from all substrate types.	Effective in collecting all taxa which drift in the water column; performance depends on current velocity and sampling period; size of macroinvertebrates collected depends on mesh size used.	Low sampling error; less time, money, effort; collects macroinvertebrates from all substrates; usually collects more taxa.	Unknown where organisms come from; terrestrial species may make up a large part of sample in summer and periods of wind and rain.	Limited application	Allen (374) Allan and Russek (375) Bailey (376) Berner (377) Chaston (378) Clifford (379) (380) Cushing (381,382) Dimond (383) Edington (384) Elliott (385,386,387,388,389,390) Ferrington (391) Hales and Gaufin (392) Hildebrand (393) Holt and Waters (394) Hynes (395) Klemm

TABLE A1.2 *Continued*

Stream-Net Samplers	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Drift nets	Flowing rivers and stream	Drifting benthic macroinvertebrates from all substrate types.	Effective in collecting all taxa which drift in the water column; performance depends on current velocity and sampling period; size of macroinvertebrates collected depends on mesh size used.	Low sampling error; less time, money, effort; collects macroinvertebrates from all substrates; usually collects more taxa.	Unknown where organisms come from; terrestrial species may make up a large part of sample in summer and periods of wind and rain.	Limited application	Keifer and Maughan (396) Larimore (397) Larkin and McKone (398) Lehmkuhl and Anderson (399) McClay (400) Merritt, Cummins, and Resh (360) Minshall and Winger (401) Modde and Schulmbach (402) Muller (403,404) Mullican, Sansing, and Sharber (405) Mundie (406,407) Pearson and Franklin (408) Pearson and Kramer (409,410) Pearson, Kramer, and Franklin (411) Pfitzer (412) Radford and Hartland-Rowe (413) Reisen and Prins (414) Resh (364) Resh, et. al (370) Spence and Hynes (415) Tanaka (416) Tranter and Smith (417) Waters (418,419, 420,421,422, 423,424,425, 426,427) Weber (428) Wilson and Bright (429) Winner, Boesel, and Farrell (430) Wojtalik and Waters (431)

device, causing the device to land improperly or causing a pressure wave and blowout of the surface layer of sediment when the grab reaches the bottom.

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

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

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

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

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

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

A1.5.2 *Ekman Grab Sampler:*

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

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

A1.5.2.3 *Hazards:*

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

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

NOTE—Modified from Klemm et al., 1990 (30); Environment Canada, 1994(2); PSEP, 1997a (33); WDE, 1995(29); USEPA 2001 (1).

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

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

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

A1.5.2.4 Procedure:

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

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

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

NOTE—Modified from Klemm et al., 1990 (30); Environment Canada, 1994(2); PSEP, 1997a (33); WDE, 1995(29); USEPA/ACE, 1998 (34)); USEPA (2001) (1).

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



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

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

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

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

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

A1.5.3 Petersen Grab Sampler:

A1.5.3.1 The Petersen Grab Sampler is designed to obtain quantitative samples of macroinvertebrates from sediments in

lakes, reservoirs, and similar habitats and is adaptable to rivers, estuaries, and oceans. This device (Fig. A1.3) is useful for sampling sand, gravel, marl, and clay in swift currents and deep waters. This sampler is available in a range of sizes that will sample an area from 0.06 to 0.099 m².

A1.5.3.2 The Petersen grab sampler has paired jaws that should penetrate the intended substrate without disturbing the water surface boundary layer of the substrate, close when positioned properly on the bottom, and retain the sample of sediment while it is brought to the surface for processing. The Petersen grab has been modified to improve its efficiency and reliability. Modified versions of the Petersen grab sampler may have a screened window at the top of each jaw to allow water to escape while the grab is descending and closing. While some modifications may close or function better, the sampling

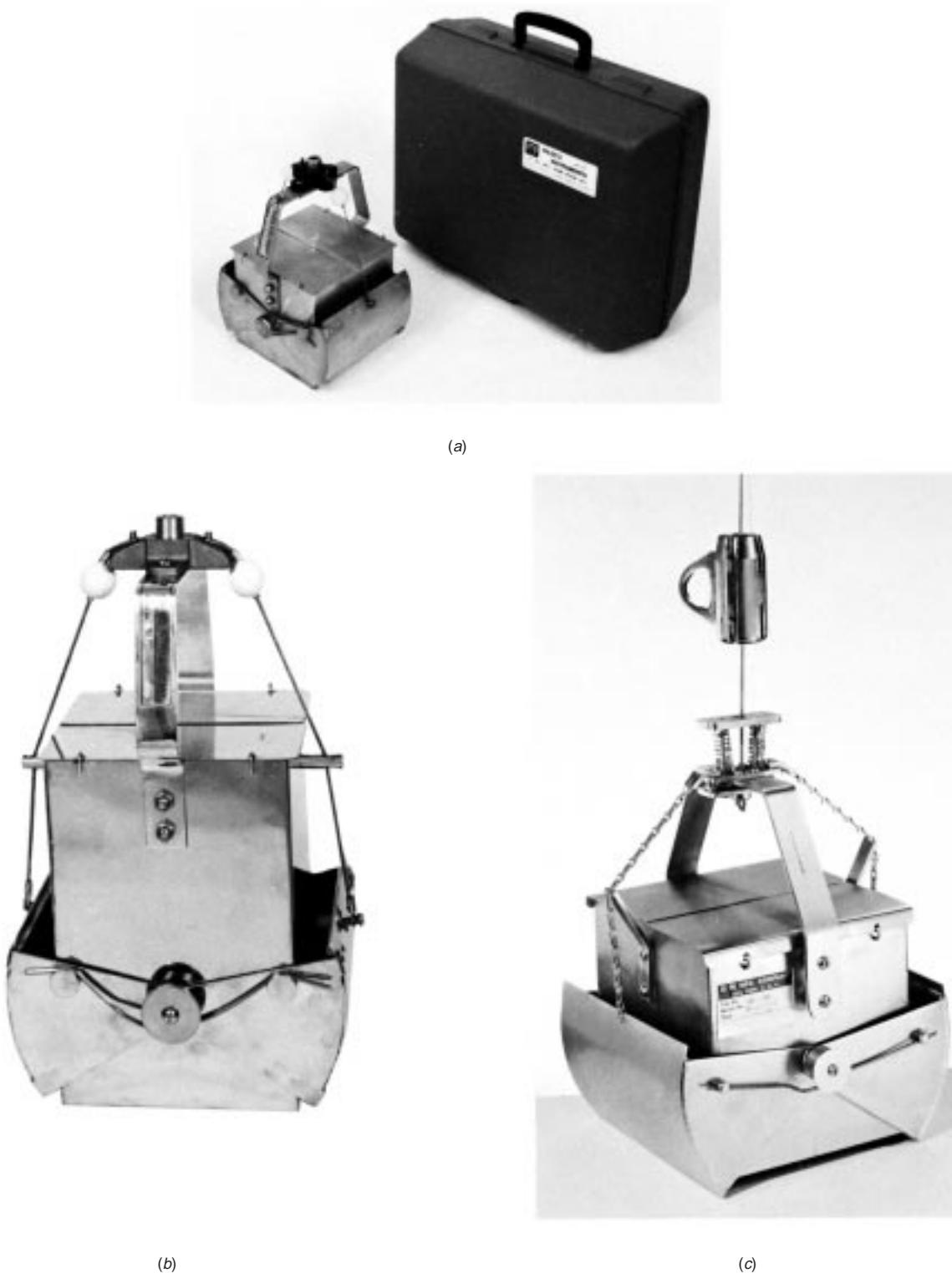


FIG. A1.2 Ekman Grabs. (a) Wildco Ekman Grab, Standard Design with Case; (b) Wildco Ekman Grab, tall design, (Photographs courtesy of Wildlife Supply Co.; (c) Ekman Box Sediment Grab (Birge-Ekman Design), (Photograph courtesy of Kahl Scientific Instrument Corp.)

characteristics remain the same. Most of the modified versions are intended for use in estuarine and marine waters. A small version can be hauled aboard by hand and held with one hand for washing procedures.

A1.5.3.3 This grab sampler has limited application, and is not recommended for quantitative benthic work. A consensus of aquatic biologists consider the use of this device the least preferable grab sampler and would use it only in limited

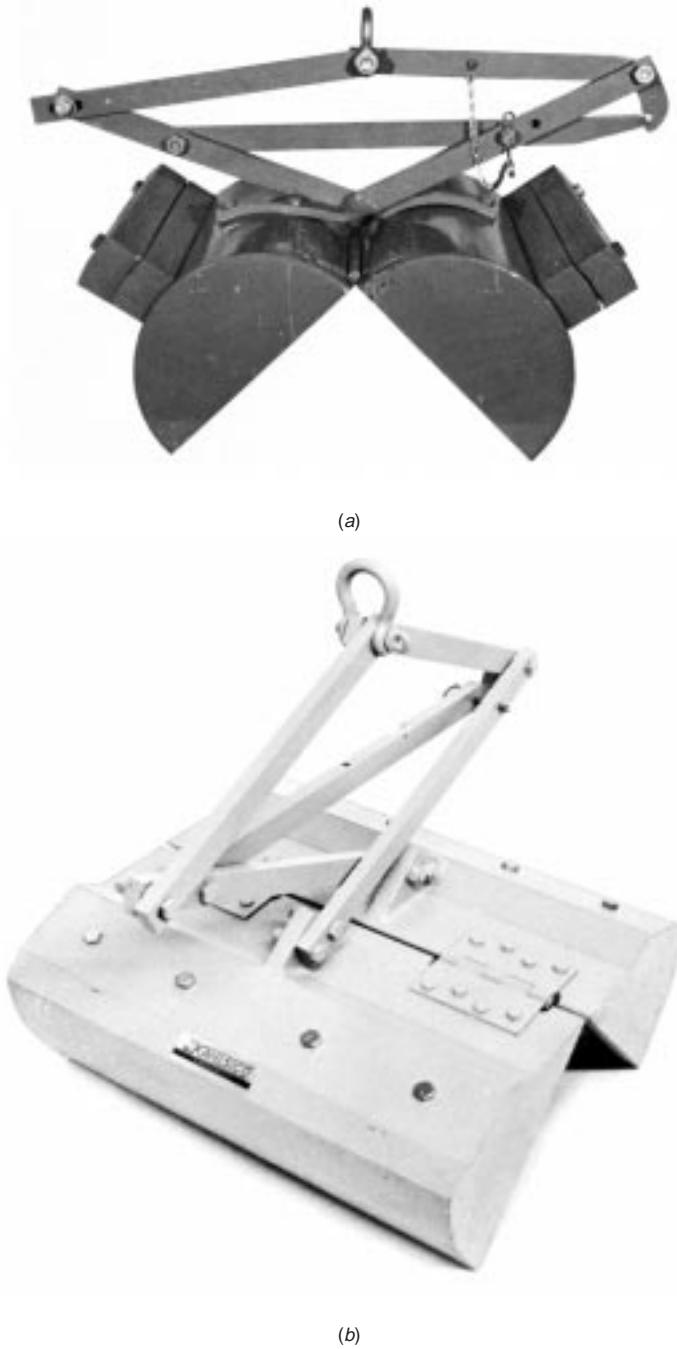


FIG. A1.3 Petersen Grabs (a) Wildco Petersen Grab (Photograph courtesy of Wildlife Supply Co.); (b) Kahl Petersen Grab (Photograph courtesy of Kahl Scientific Instrument Corp.)

applications. The grab should only be used with consideration of its defects when quantitative estimates are attempted.

A1.5.3.4 Hazards:

- (1) This grab sampler cannot be used under adverse weather conditions.
- (2) It is advisable to use a winch and cable to lower and raise the sampler.
- (3) Ideally a stationary boat or platform should be used when taking samples.

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

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

A1.5.3.5 Procedure:

- (1) The Petersen grab sampler should be inspected for mechanical defects prior to use.
- (2) The sampler is slowly lowered to the bottom when open to avoid disturbing lighter materials of the substrate.

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

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

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

A1.5.4 Smith-McIntyre Grab Sampler:

A1.5.4.1 Smith-McIntyre Grab Sampler (Fig. A1.4) is designed to obtain quantitative samples of macroinvertebrates from sediments in rough weather in hard sand bottoms in lakes, streams, estuaries, and oceans. This device is useful for sampling macroinvertebrates from sand, gravel, mud, clay, and similar substrates and is useful under adverse weather conditions. This device samples a surface area of 0.1 m².

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

A1.5.4.3 Hazards:

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

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

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

A1.5.4.4 Procedure:

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

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

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

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

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

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

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

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

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

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

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



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

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

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

A1.5.5 *Van Veen Grab Sampler:*

A1.5.5.1 Van Veen Grab Sampler (Fig. A1.5) is designed to give quantitative samples of macroinvertebrates from sediments in estuaries, oceans, and similar habitats, and is adaptable to freshwater areas including large rivers. This device is useful for sampling sand, gravel, mud, clay and similar substrates. This sampler is available in two sizes, 0.1 m² and 0.2 m².

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

A1.5.5.3 *Hazards:*

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

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

A1.5.5.4 *Procedure:*

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

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

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

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

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

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

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

A1.5.6 *Orange-Peel Grab Sampler:*

A1.5.6.1 Orange-Peel Grab Sampler (Fig. A1.6) is designed to obtain quantitative samples of macroinvertebrates from sediments in marine waters and deep lakes. This device is useful for sampling sand, cobble, rubble stone, and similar substrates. The sampler is available in a range of sizes but the 1600 cm³ is generally used although larger sizes are available. The sampler should not be used in critical quantitative work that is to be compared with results of other areas and is recommended as a reconnaissance sampler only.



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



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

A1.5.6.2 The Orange-Peel grab sampler has four curved jaws that close to encircle a hemisphere of sediment and should penetrate the intended substrate without disturbing the water surface boundary of the substrate, close when positioned properly on the bottom, and retain discrete samples of sediment while it is brought to the surface for processing. A modification of the Orange Peel, described by Reish (1959 (**350**)) has a trigger mechanism and more efficient closing jaws, and the volume of sample to surface-area sampled relationship has been worked out. The surface area of this device also varies with penetration depth or volume sampled. The device penetrates to a maximum depth of 18 cm, but this depth will vary.

A1.5.6.3 Hazards:

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

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

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

A1.5.6.4 Procedure:

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

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

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

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

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

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

A1.5.7 Okean 50 Grab Sampler:

A1.5.7.1 Okean 50 Grab Sampler (See Holme, 1971 for illustration (**330**)) is designed to obtain quantitative samples of sediment and macroinvertebrates primarily in marine, estuarine, and large river habitats. This device is useful for collecting macroinvertebrates from sand, gravel, mud, clay, and similar substrates. The sampler is available in various sizes, generally a sampling area of 0.25 m^2 . The Okean 50 grab sampler is used to collect qualitative and quantitative samples from different aquatic habitats containing benthic macroinvertebrates living on or in various types of substrates.

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

device is being lowered. The Okean 50 grab sampler retains many of the disadvantages of the Petersen grab but is better for sampling in deep water.

A1.5.7.3 Hazards:

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

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

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

A1.5.7.4 Procedure:

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

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

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

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

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

A1.5.8 Shipek (Scoop) Grab Sampler:

A1.5.8.1 The Shipek (Scoop) Grab Sampler (Fig. A1.7) is designed to obtain quantitative samples of macroinvertebrates from sediments in marine waters and large inland bodies of water. This device is useful for sampling macroinvertebrates from sand, gravel, mud, clay, and similar substrates. It is designed to take a sediment sample with a surface area of 20 cm² to about 10 cm deep at the center.

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

A1.5.8.3 Hazards:

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

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

A1.5.8.4 Procedure:

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

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

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

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

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

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

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

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

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

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

A1.5.9 Holme (Scoop) Grab Sampler:

A1.5.9.1 The Holme (Scoop) Grab Sampler (Fig. A1.8) is designed to obtain quantitative samples of sediment and macroinvertebrates primarily in marine and estuarine waters and large deep freshwater lakes. This device is useful for sampling macroinvertebrates from sand, gravel, mud, clay, and similar substrates. This sampler is designed to take a sediment sample with a surface area of 0.05 m² and approximately 15 cm deep at the center. The device comes with a single scoop or double scoops.

A1.5.9.2 The Holme (scoop) grab sampler has a semicylindrical scoop mounted on the bottom of a heavy frame and needs to be positioned properly on the bottom to take a scoop to retain discrete samples of sediment through 180°. The device penetrates to a depth of about 150 mm. The sampler may be modified to include double scoops each of 0.05 m² or larger.

A1.5.9.3 Hazards:

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

A1.5.9.4 Procedure:



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

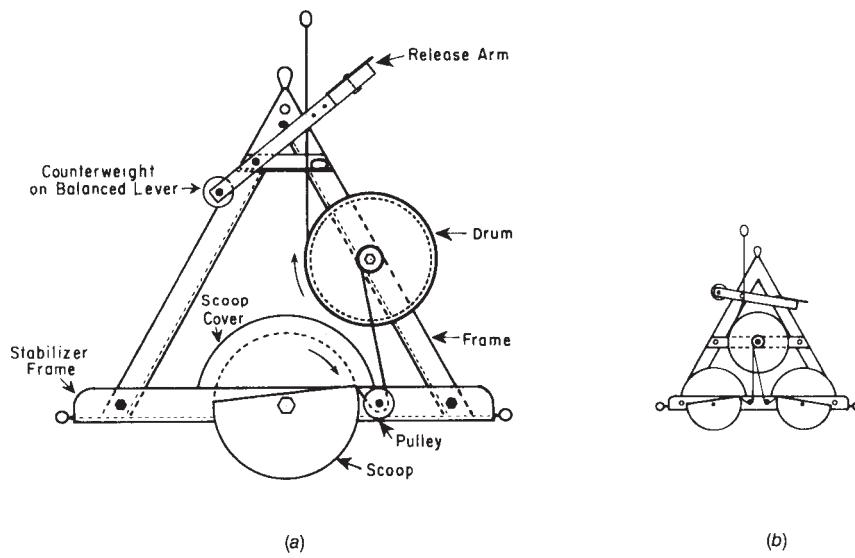


FIG. A1.8 Holme Grabs. (a) Single Holme; (b) Double Holme (See Holme and McIntyre (1971), pages 103–105)

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

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

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

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

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

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

A1.5.10 Surber Sampler:

A1.5.10.1 The Surber sampler (Figs. A1.9 and A1.10) is

designed to obtain a qualitative or quantitative sample of macroinvertebrates from a unit area. The device is used in shallow flowing streams and shallow areas of rivers with mud, sand, gravel, or rubble substrates. Modification of its basic design has resulted in other sampling devices, such as the portable invertebrate box sampler (Fig. A1.11). The latter closed-box-type sampler is preferred, if available. A variety of mesh sizes is available and mesh size should be selected based on the objectives of the study; the finer the mesh, the more organisms (instars) will be collected. These devices sample an area of 0.1 m². The device is restricted to use in shallow streams or shallow areas of rivers, and it depends on a water velocity of not less than 0.05 m/s to wash the sample into a net.

A1.5.10.2 The Surber sampler consists of two 30.5-cm frames, hinged together; one frame rests on the substrate, the

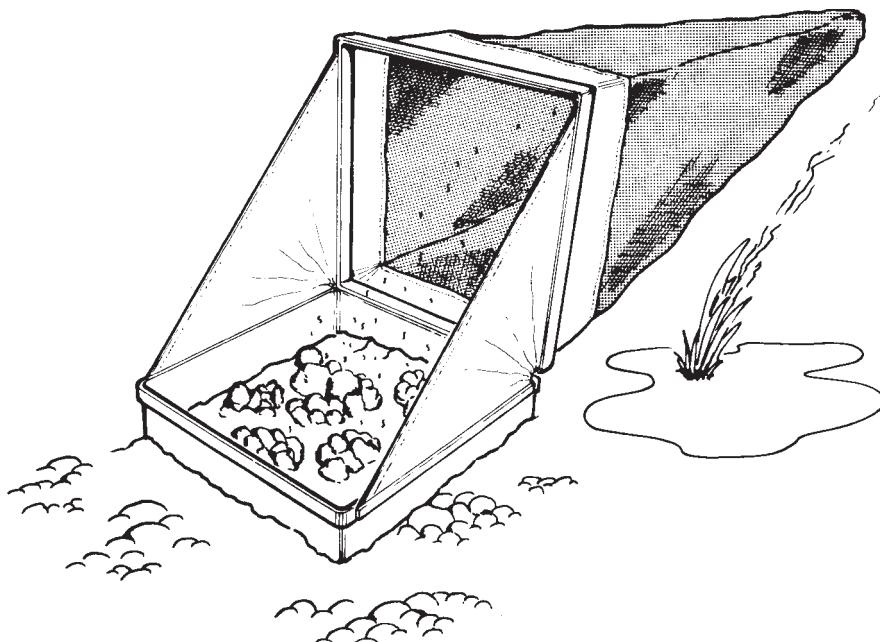


FIG. A1.9 Surber Sampler (Illustration courtesy of Kahl Scientific Instrument Corp., P.O. Box 1166, El Cajon, CA 92022-1166)

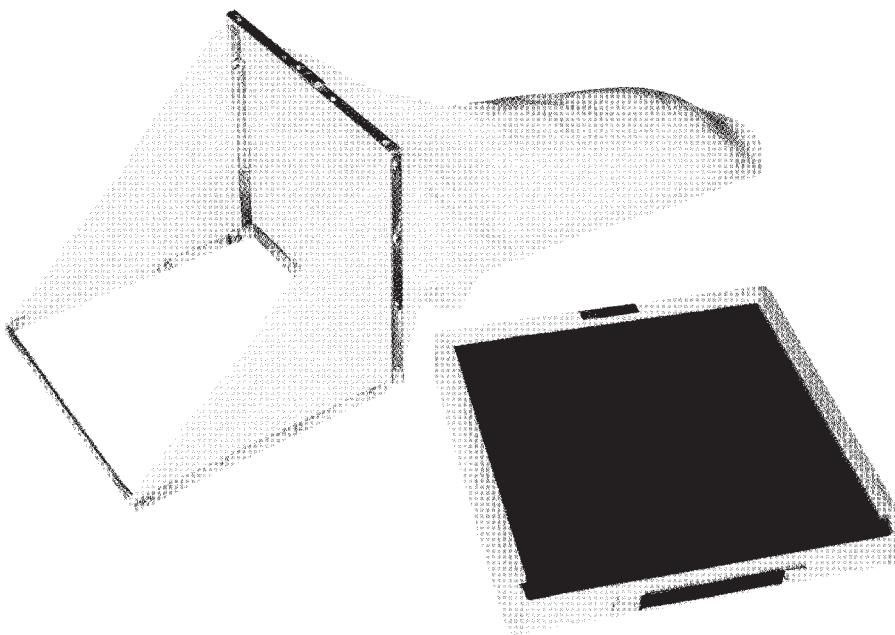


FIG. A1.10 Surber Sampler (Photograph courtesy of Wildlife Supply Co., 301 Cass St., Saginaw, MI 48602)

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

A1.5.10.3 The Hess (cylindrical) sampler (Fig. A1.12) is designed to obtain a qualitative or quantitative sample of macroinvertebrates from a unit area. The device is used in shallow flowing streams and shallow areas of rivers with mud, sand, gravel, or rubble substrates. Modification of its basic design has resulted in other sampling devices, such as the Hess stream bottom sampler (Fig. A1.13) and stream-bed fauna sampler (Fig. A1.14). A variety of mesh sizes is available, and mesh size should be selected based on the objectives of the study; the finer the mesh, the more organisms (instars) will be collected. The area sampled by these devices is dependent on their diameter and is comparable to the Surber sampler. These devices sample an area of 0.1 m².

A1.5.10.4 The net used to collect macroinvertebrates can vary in mesh size, length, taper, and material, for example, canvas, taffeta, or nylon monofilament. The net is usually made of nylon, and a variety of mesh sizes is available. The mesh size used will depend on the objectives of the study. A mesh size of 0.35 mm, for example, will retain most instars of

aquatic insects. While a smaller mesh size might increase the number of smaller invertebrates and young instars collected, it will clog more easily and exert more resistance to the current than a larger mesh, possibly resulting in a loss of organisms due to backwashing from the sample net.

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

A1.5.10.6 The Surber, portable invertebrate box, Hess, Hess stream bottom, and stream-bed fauna samplers sample an area of 0.1 m².

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

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

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

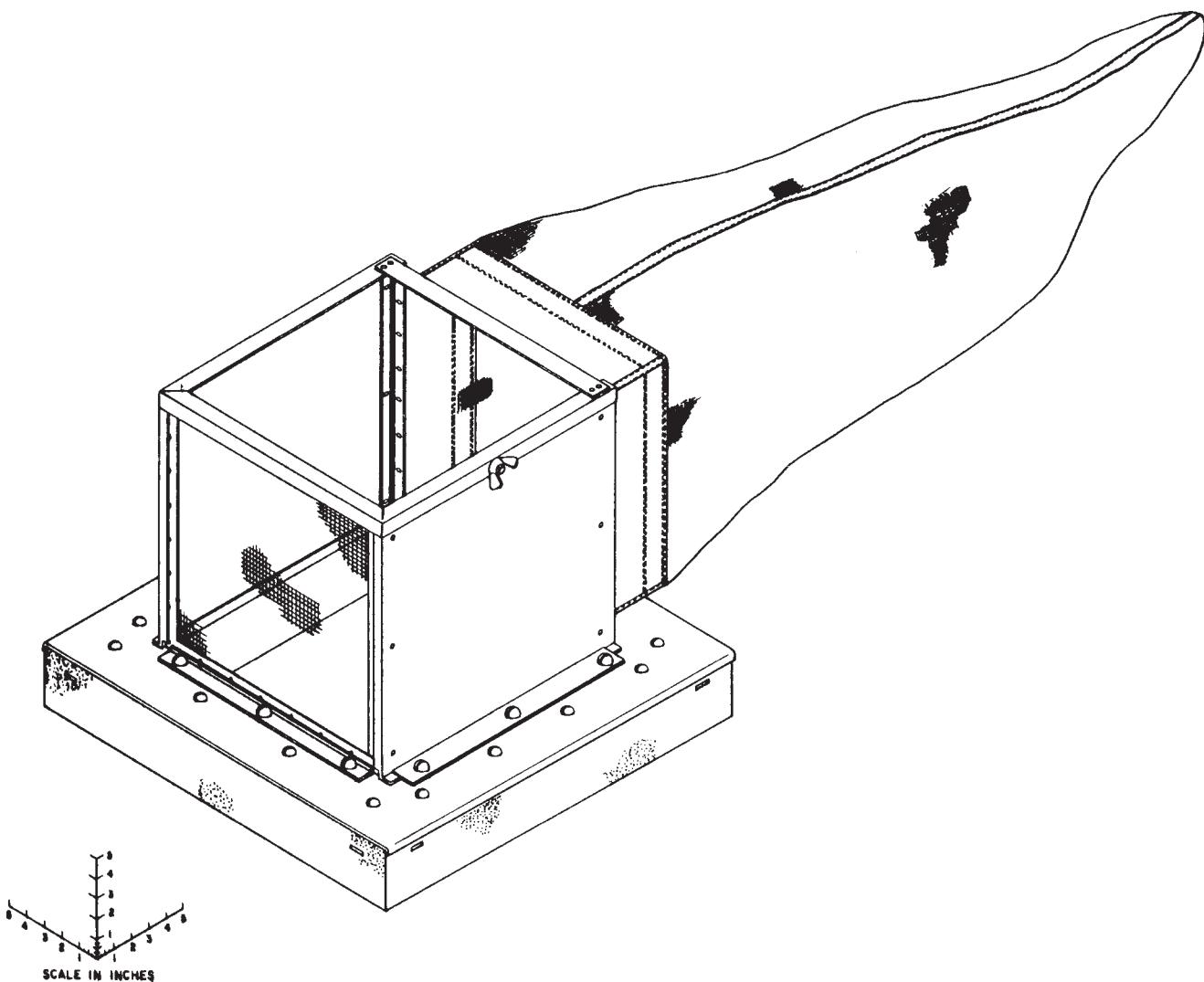


FIG. A1.11 Portable Invertebrate Box Sampler (Illustration courtesy of Ellis-Rutter Associates, P.O. Box 401, Punta Gorda, FL 33950)

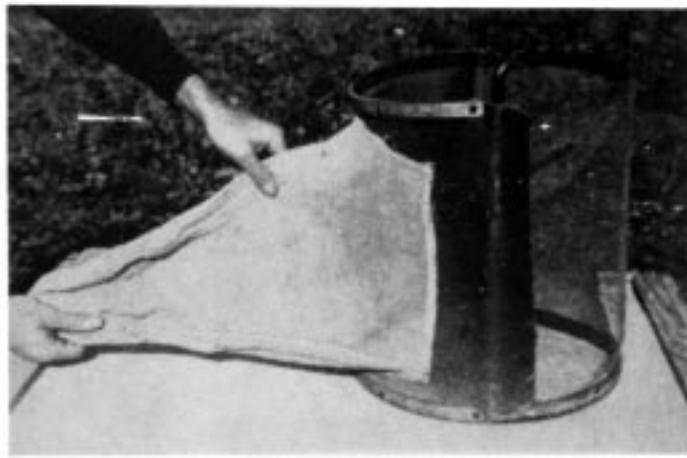


FIG. A1.12 Hess Sampler (Photograph courtesy of Billy G. Isom)

used consistently by one experienced person in similar habitats. If the water velocity is very great, resistance provided by the small mesh of the net or debris washed into it, or both, may

result in a backwashing effect that washes benthic organisms out of the sample area of the Surber sampler or top of the other samplers.



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

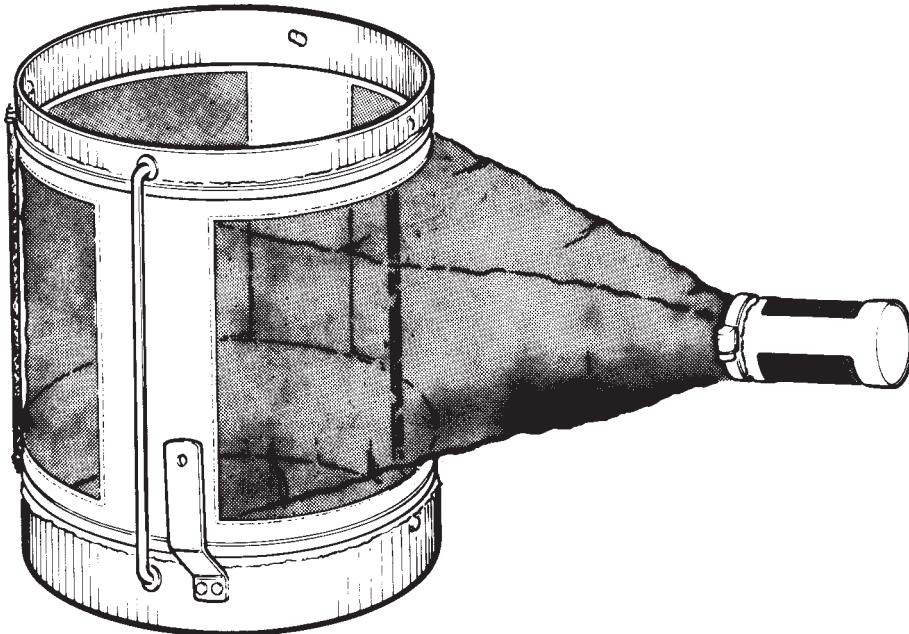


FIG. A1.14 Stream-Bed Fauna Sampler (Photograph courtesy of Kahl Scientific Instrument Corp., P.O. Box 1166, El Cajon, CA 92022-1166)

A1.5.10.10 *Hazards:*

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

A1.5.10.11 *Procedure:*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

A1.5.11 Drift Net Samplers:

A1.5.11.1 Drift net samplers (Figs. A1.15 and A1.16) are designed to obtain qualitative and quantitative samples of macroinvertebrates which drift in flowing streams and rivers with a velocity of not less than 0.05 m/s. Drift nets vary in size,

but the type commonly used has an upstream opening of 15 by 30 cm, and the collection bag is 1.3 m long. A variety of mesh sizes is available, and mesh size should be selected based on the objectives of the study; the finer the mesh, the more organisms (instars) will be collected.

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

A1.5.11.3 Drift nets are useful for collecting macroinvertebrates that actively or passively enter the water column or that are dislodged from the substrate; naturally or by stress. They are particularly well-suited for synoptic surveys because they are light weight and easily transported. Thousands of organisms, including larvae of stoneflies, mayflies, caddisflies, and midges and other Diptera, may be collected in a sampling period of only a few hours. The drift net efficiently collects organisms originating from all types of substrates and a wide spectrum of microhabitats in lotic (flowing) waters. The device is restricted to flowing rivers or streams with a current velocity of more than 0.05 m/s.

A1.5.11.4 The typical drift net consists of a bag of nylon or nylon monofilament; a variety of mesh sizes can be used depending on the objectives of the study. The U.S. Standard No. 30 (0.595-mm mesh openings) net is often used for



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

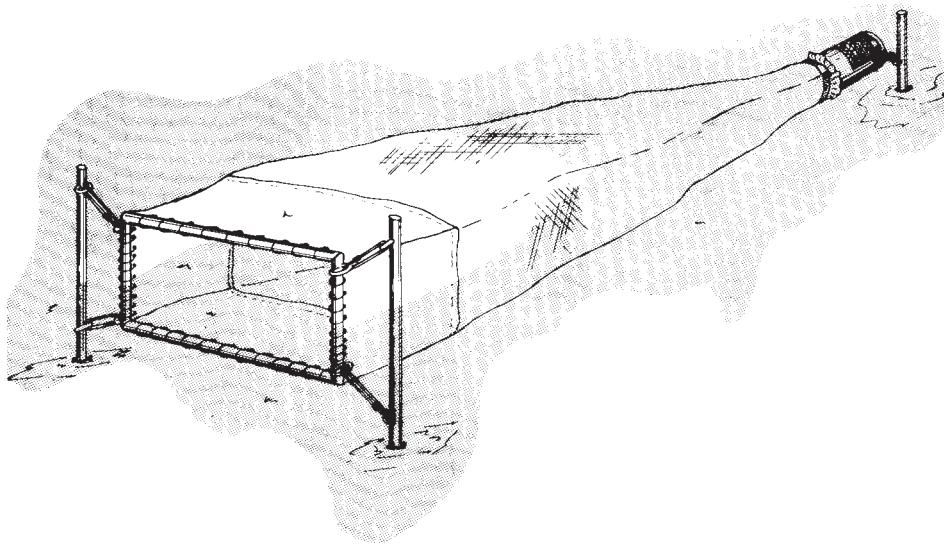


FIG. A1.16 Drift Net (Photograph courtesy of Kahl Instrument Corp., P.O. Box 1166, El Cajon, CA 92022-1166)

collecting macroinvertebrates. The frame typically consists of a 0.045-m²(15 by 30-cm) rod structure anchored into the stream bed by a pair of steel rods. Cable clamps are used to secure the nets to the rods.

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

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

A1.5.11.7 The drift net frame can be fitted anteriorly with a mouth reducing rectangular plexiglass enclosure (Rutter and Ettinger, 1977 (433)) to increase filtration efficiency. The type of drift net and mesh size utilized will depend on the objectives of the study and the physical characteristics of the flowing water.

A1.5.11.8 Alternatives to the typical drift net include the waterwheel drift sampler (Pearson and Kramer, 1969 (434)) which might be useful in large rivers or streams which can be reached by automobile. An automatic drift sampler (Muller, 1965 (435)) can be constructed that eliminates the need for an attendant at the sampling site during collection of as many as eight consecutive samples. A modified emergence-trap drift sampler (Mundie, 1964 (436); Cushing, 1964 (437)) is useful in streams with extremely high drift, where water is very turbid, or where a long sampling period is desired without clogging. The drift collection usually represents a wide spectrum of the habitats found in a stream.

A1.5.11.9 A benthic sample shows only what taxa were existing in the particular area (usually some fraction of a square meter, etc.) that was sampled. The great variation among benthic samples, even in a limited area, illustrates the necessity of several samples and the influence of selecting the collecting sites. One drift sample might be adequate for collecting the majority of invertebrate taxa in a stream reach, whereas a large number of benthic samples would be needed to cover the

variety of bottom habitats even in a uniform reach of the stream. Quantitative benthic sampling is seldom extended to include stream banks, organic substrates (logs, etc.), and areas of dense vegetation. The drift net collects organisms from all these areas. Drift net collections often require much less sorting work than a series of benthic samples.

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

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

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

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

A1.5.11.14 Certain aquatic organisms enter the drift only sporadically and might be missed even though common in the benthos. The relative abundance of macroinvertebrates in a drift sample often differs significantly from their relative abundance on the stream bottom. A slight current is necessary if a drift collection is to be taken (greater than 0.05 m/s).

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

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

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

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

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

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

A1.5.11.21 Hazards:

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

A1.5.11.22 Procedure:

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

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

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

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

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

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

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

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

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

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

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

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

(13) Drift nets have also been used from small boats in large rivers (Rutter and Ettinger, 1977 (433)).

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

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

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

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

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

(19) Subdividing the sample substantially reduces analysis time with large samples (Waters, 1969b (440) and Weber (306)).

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

(21) Conversion equations and other statistical aspects of drift sampling are given by Elliott, 1981 (299).

(22) An equation for converting the data to number per 100 m³ of water flow is:

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

where:

X = number of organisms per 100 m³,
a = number of organisms in the net (density),
b = number of minutes of the sampling interval,
c = current velocity, m/min, and
d = area of the net opening (m²).

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

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

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

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

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

A1.5.12 *Basket Samplers:*

A1.5.12.1 Basket samplers are a highly effective device for evaluating the biological integrity of surface waters and for studying macroinvertebrate communities (441-466). The materials used in the basket sampler are natural or artificial materials of various compositions and configurations. The device is placed in water for a predetermined exposure period and depth for the colonization of macroinvertebrate communities. Basket samplers are used to collect qualitative and quantitative samples from lentic and lotic waters containing benthic macroinvertebrates living on various types of substrates. Physical factors such as stream velocity and depth may variably affect the degree of colonization. The sampling method is selective for drifting organisms (biased for insects) and for those that preferentially attach to or live on hard surfaces. Basket samplers are excellent for water quality monitoring; contain uniform substrate types at each station for better comparison; provide quantitatively comparable data; contain negligible amounts of debris, permitting quick laboratory processing; and usually do not require additional weight for stability. Basket samplers sample a known area at a known depth for a known exposure period. Basket samples provide no measure of the biota and condition of the natural substrate at a station. They record only biota accumulated during the exposure period.

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

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

A1.5.12.4 The type of basket sampler normally used (Fig. A1.17) is a cylindrical "barbecue" basket 11 in. (28 cm) long and 7 in. (17.8 cm) in diameter that is filled with approximately 17 lb (7.7 kg) of natural rocks varying from 1 to 3 in. (2.5 to 7.6 cm) in diameter (Mason 1967, 1971 (456,457)). A hinged door on the side provides access to the contents. An estimated 3.2 ft² (0.3 m²) of surface area is provided for colonization by macroinvertebrates. A 1/8-in. (3.2-mm) wire cable is passed through the long axis of the basket; one end is fastened with a cable clamp, and the other end is fixed to the float. A5-gal (19-L) metal container filled with polyurethane foam can be used as a float. A 3/8-in. (9.5-mm) steel rod threaded at each end is passed through the long axis of the float and fastened at each end by nuts. Three inch by 1 1/8-in. by 1/8-in. (76.2 by 25.6 by 3.2-mm) strap iron serves as a swivel at each end, secured on the rods by nuts. The wire cable used to suspend the basket is attached to the swivels by holes drilled for that purpose. The

float can be attached to a stationary structure, or the basket can be anchored to the bottom in shallow water.

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

A1.5.12.6 A collapsible type of basket sampler has been used for comparing populations surrounding rocky substrates (Bull, 1968 (447)). The sampler consists of a collapsible basket surrounded by a nylon netting bag that can be loaded with materials simulating the natural substrate on which it lies. A rim around the top helps retain the substrate material. When lowered to the bottom, the basket sampler collapses to form a substrate area that is eventually colonized. When the basket is raised off the bottom, the basket extends to its original hemispherical shape, and the surrounding net bag limits the loss of invertebrates during retrieval.

A1.5.12.7 *Hazards:*

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

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

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

A1.5.12.8 *Procedure:*

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

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

(3) For uniformity of depth, suspend the basket samplers from floats on 1/8-in. (3.2-mm) steel cable. If vandalism is a problem, use subsurface floats or put the samplers on supports placed on the bottom. Regardless of the installation technique, use uniform procedures (for example, the same depth and exposure period, sunlight, current velocity, and habitat type).

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

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

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

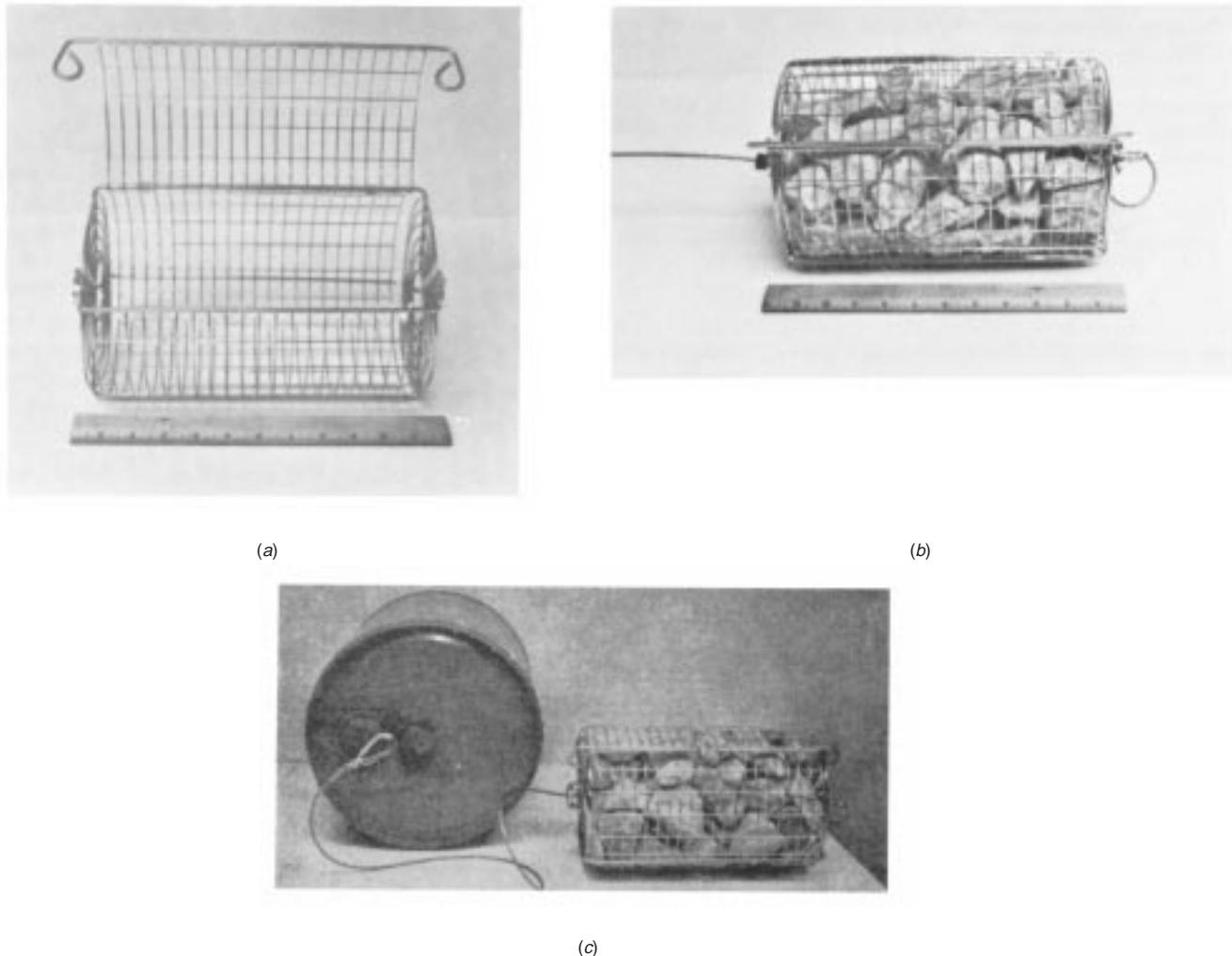


FIG. A1.17 Cylindrical “Barbecue” Basket Sampler: (A) Basket Sampler Empty; (B) Basket Sampler Containing Limestone Rocks and Ready for Installation; and (C) Basket Sampler Containing Limestone Rocks and Attached to 5-gal (19-L) Metal Container Filled with Polyurethane Foam. (Barbecue Baskets Available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

(7) In shallow streams with sheet rock bottoms, basket samplers can be secured to $\frac{3}{8}$ -in. (0.95-cm) steel rods that are driven into the substrate or secured to rods that are mounted on low, flat, rectangular blocks half way between the water surface and the stream bed. However, these should be anchored securely to the rock bottom to avoid loss during floods.

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

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

(10) In shallow water, approach the basket samplers from downstream, lift the sampler quickly, and place the entire

sampler in a polyethylene bag or jug containing the selected fixative. The fixative should be used only if the specimens collected require special processing for identification.

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

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

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

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

A1.5.13 *Multiplate Samplers:*

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

A1.5.13.2 The sampler can be purchased or constructed from readily available materials. Multiple-plate samples have been constructed of 8 or more tempered hardboard or ceramic material cut in 76 mm (3 in.) square or circular plates and separated by a specific arrangement of spacers. The plates and spacers are placed on a 1/4-in. eyebolt. Total surface area of the 8-plate sampler is approximately 939 cm²(0.09 m²), and the 14 plate sampler is 1160 cm² (0.116 m²). The 14 plate, tempered hardboard, multiple-plate sampler weighs about 1 lb (0.45 kg).

A1.5.13.3 *Description of the Modified Hester-Dendy Multiple-Plate Sampler*—The modified multiple-plate (Fig. A1.18) is constructed of 0.25 in. (0.3 cm) tempered hardboard or ceramic material with 3 in. (7.6 cm) round or square plates and 1 in. (2.5 cm) round spacers that have 5/8-in. holes drilled in the center (Fuller, 1971 (472) and Cairns, 1982 (479)). The plates are separated by spacers on a 0.25-in. (0.63 cm) diameter eyebolt, held in place by a nut at the top and bottom. A total of 14 large plates and 24 spacers are used in each sampler. The top nine plates are each separated by a single spacer, plates 9 and 10 are separated by two spacers, plates 11 and 12 are separated by three spacers, and plates 13 and 14 are separated by four spacers. The hardboard sampler is about 5.5-in. (14 cm) long, 3-in. (7.6 cm) diameter, exposes about 1160 cm²(0.116 m²) of surface area for the attachment of organisms, and weighs about 1 lb (0.45 kg). The ceramic sampler is 6.5-in. long and weighs 2.2 lb (1 kg). The ceramic plates can be chemically cleaned, oven dried and reused indefinitely as they are stable and unaffected by long-term immersion in water. The sampler will not warp with time; therefore, the spacings between plates do not change, assuring replicate and efficient sampling. Each

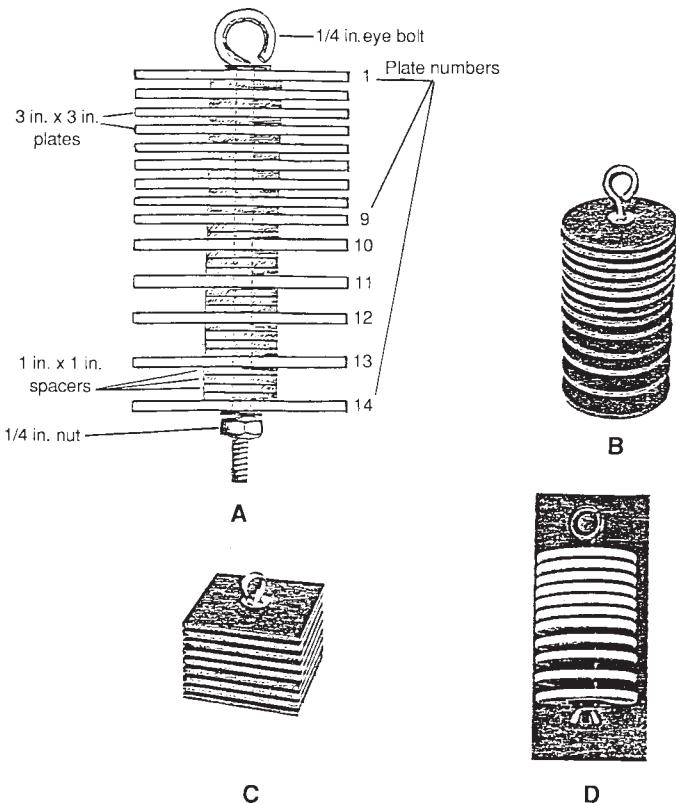


FIG. A1.18 Artificial multiple-plate samplers: (a) schematic drawing of multiple-plate sampler; (b) modified round; (c) original square, tempered hardboard, Hester-Dendy samplers; and (d) round ceramic multiple-plate macroinvertebrate sampler

sampler is supplied with a 20-ft (6 m) long nylon suspension rope. The total weight is 2.2 lb (1 kg). Sturdy wire stakes for holding the sampler above the riverbed are recommended accessories.

A1.5.13.4 Another type of modified Hester-Dendy multiple-plate artificial substrate sampler (Ohio EPA, 1987 (483)) is constructed of 1/8-in. tempered hardboard cut into 3-in. (7.6 cm) square plates and 1-in. (2.5 cm) square spacers. A total of eight plates and twelve spacers are used for each sampler. The plates and spacers are placed on a 1/4-in. eyebolt so that there are three single spaces, three double spaces, and one triple space between the plates. The total surface area of the sampler, excluding the eyebolt, is 145.6 in.²(939 cm² or 0.09 m²). Five samplers are placed in streams tied to a concrete construction block which anchors them in place and prevents the multiple-plates from coming into contact with the natural substrates.

A1.5.13.5 The recommended exposure period for multiple-plate sampler is six weeks, and the time of exposure may be critical to development of a relatively abundant and diverse community of organisms. Three replicate samples at each station are an absolute minimum. Collecting five replicate samples at each station will increase statistical precision and accuracy. Multiple-plate samplers are a highly effective device for evaluating the biological integrity of surface waters and for studying macroinvertebrate communities (467-487). Multiple-plate samplers are used to collect qualitative and quantitative samples from lentic and lotic waters containing benthic macroinvertebrates living on various types of substrates. Physical

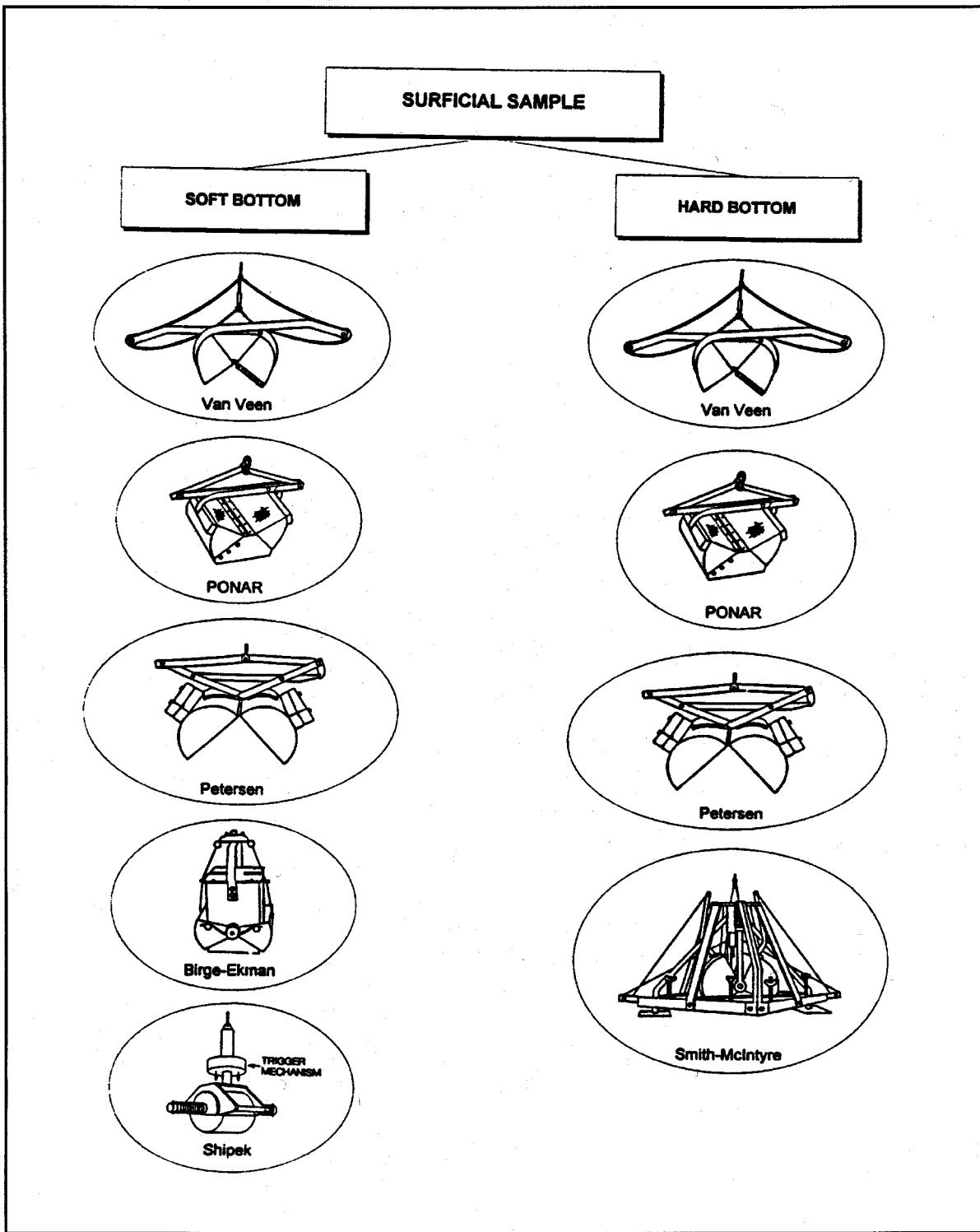


FIG. A1.19 Some Recommended Devices for Collecting Surficial Sediments (drawings from Murdoch and Azcue 1995 (46); USEPA 2001 (1))

factors such as stream velocity and depth may variably affect degree of colonization. The sampling method is selective for drifting organisms (biased for insects) and for those which preferentially attach to or live on hard surfaces.

A1.5.13.6 Multiple-plate samplers are excellent for water quality monitoring, contain uniform substrate type at each station for better comparison, give quantitatively comparable data, contain negligible amounts of debris permitting quick

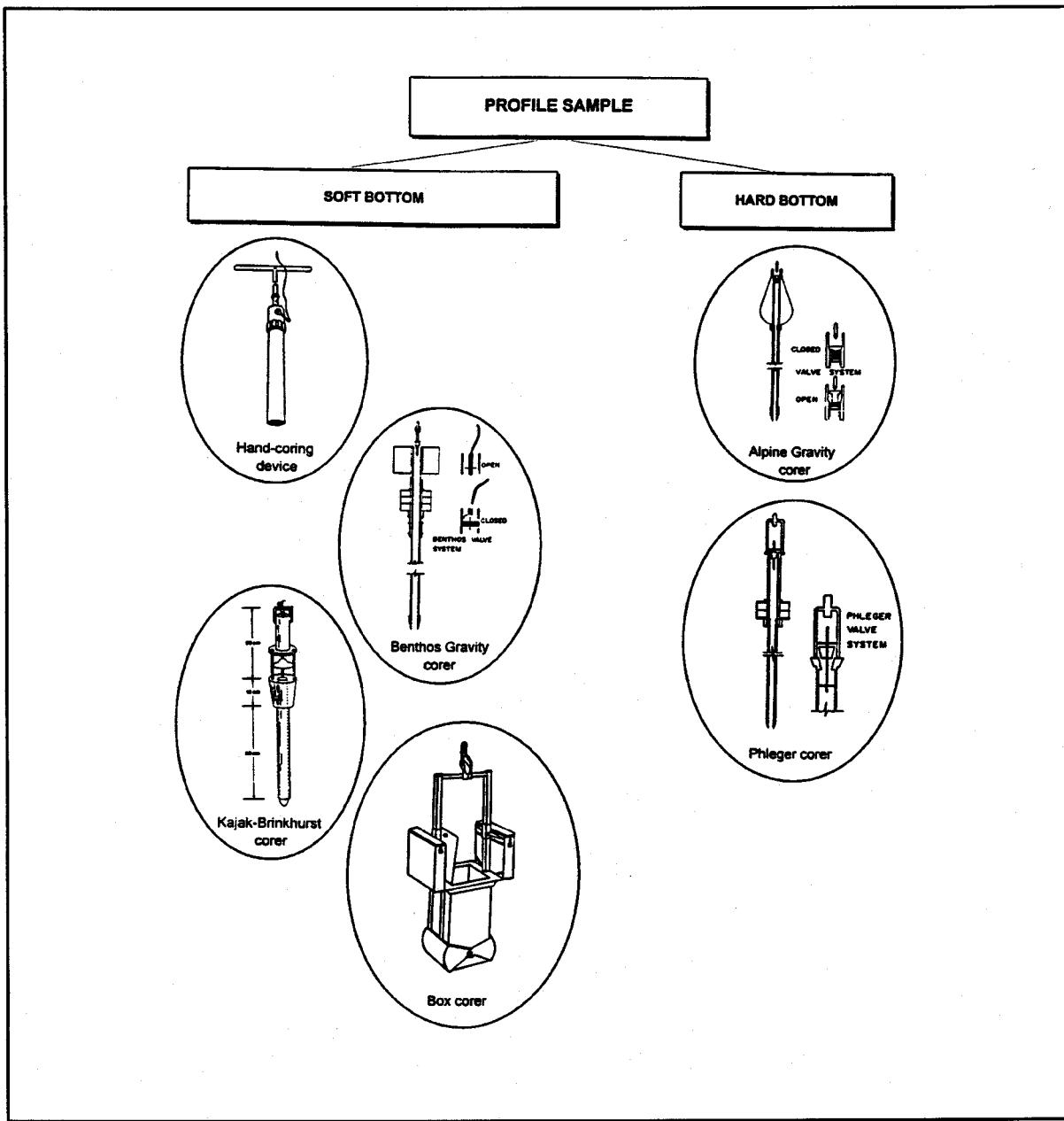


FIG. A1.20 Some Recommended Devices for Obtaining Sediment Profiles (drawings from Murdoch and Azcue 1995(46); USEPA 2001 (1))

laboratory processing, but may require additional weight for stability. Multiple-plate samplers sample a known area at a known depth for a known exposure period. Multiple-plate samples provide no measure of the biota and condition of the natural substrate at a station. They record only biota accumulated during exposure period. The distinct advantages of the multiple-plate sampler are its small size and light weight. It is the most adaptable of the recommended benthic invertebrate artificial substrate devices.

A1.5.13.7 Hazards:

- (1) Samplers and floats may be difficult to anchor; they may be a navigation hazard.
- (2) Samplers are susceptible to vandalism and often lost.

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

A1.5.13.8 Procedure:

(1) In deep water three multiple-plate samplers are suspended from floats, cement structures, or rods driven into the stream-bed or lake-bed and positioned well up in the euphotic zone of good light penetration (1 to 3 ft, or 0.3 to 0.9 m) for maximum abundance and diversity of macroinvertebrates. A 4-ft (1.2 m) depth is acceptable unless the water is exceptionally turbid.

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

(3) For uniformity of depth, suspend the multiple-plate samplers from floats on $\frac{1}{8}$ -in. (3.2 mm) steel cable. If vandalism is a problem, use subsurface floats or put the sampler on supports placed on the bottom. Regardless of the installation technique, use uniform procedures (for example, the same depth and exposure period, sunlight, current velocity, and habitat type).

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

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

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

(7) In shallow streams with sheet rock bottoms, multiple-plate samplers can be secured to $\frac{3}{8}$ -in. (0.95 cm) steel rods that are driven into the substrate or secured to rods that are mounted on low, flat, rectangular blocks half-way between the water surface and the stream bed. However, these should be anchored securely to the rock bottom to avoid loss during floods.

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

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

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

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

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

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

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SUMMARY OF CHANGES

The primary technical changes from the previous version of this standard (E 1367-99) are summarized in this section.

- (1) Information from USEPA (2001) (1) and Environment Canada (1994) were used to update the sections dealing with collection, storage, and manipulation of sediments.
- (2) Information from the following standards were consolidated in Annex A1 (once this Annex has been approved, there will be a ballot started to withdraw these 15 standards:
- D 4387-84 (2002) Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates
 - D 4556-85 (2002) Guide for Selecting Stream-Net Sampling Devices for Collecting Benthic Macroinvertebrates
 - D 4342-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Ponar Grab Sampler
 - D 4343-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Ekman Grab Sampler
 - D 4344-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Smith-McIntyre Grab Sampler
 - D 4345-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Van Veen Grab Sampler

- D 4346-84 (1997) Practice for Collecting Benthic Macroinvertebrates with Okean 50 Grab Sampler
- D 4347-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Shipek (Scoop) Grab Sampler
- D 4348-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Holme (Scoop) Grab Sampler
- D 4401-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Petersen Grab Sampler
- D 4407-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Orange Peel Grab Sampler
- D 4557-85 (2002) Practice for Collecting Benthic Macroinvertebrates with Surber And Related Type Samplers
- D 4558-85 (2002) Practice for Collecting Benthic Macroinvertebrates with Drift Net
- E 1468-92 (2002) Practice for Collecting Benthic Macroinvertebrates with Basket Sampler
- E 1469-92 (2002) Practice for Collecting Benthic Macroinvertebrates with Multiplate Sampler

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Designation: D4700 – 91 (Reapproved 2006)

Standard Guide for Soil Sampling from the Vadose Zone¹

This standard is issued under the fixed designation D4700; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers procedures that may be used for obtaining soil samples from the vadose zone (unsaturated zone). Samples can be collected for a variety of reasons including the following:

- 1.1.1 Stratigraphic description,
- 1.1.2 Hydraulic conductivity testing,
- 1.1.3 Moisture content measurement,
- 1.1.4 Moisture release curve construction,
- 1.1.5 Geotechnical testing,
- 1.1.6 Soil gas analyses,
- 1.1.7 Microorganism extraction, or
- 1.1.8 Pore liquid and soils chemical analyses.

1.2 This guide focuses on methods that provide soil samples for chemical analyses of the soil or contained liquids or contaminants. However, comments on how methods may be modified for other objectives are included.

1.3 This guide does not describe sampling methods for lithified deposits and rocks (for example, sandstone, shale, tuff, granite).

1.4 In general, it is prudent to perform all field work with at least two people present. This increases safety and facilitates efficient data collection.

1.5 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.6 *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 prior to use.*

1.7 *This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional*

judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.

2. Referenced Documents

2.1 ASTM Standards:²

- D653 Terminology Relating to Soil, Rock, and Contained Fluids
- D1452 Practice for Soil Exploration and Sampling by Auger Borings
- D1586 Test Method for Penetration Test (SPT) and Split-Barrel Sampling of Soils
- D1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes
- D2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)
- D3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils
- D4220 Practices for Preserving and Transporting Soil Samples

3. Terminology

3.1 Definitions:

3.1.1 Except where noted, all terms and symbols in this guide are in accordance with the following publications. In order of consideration they are:

3.1.1.1 Terminology D653.

3.1.1.2 *Compilation of ASTM Standard Terminology*,³ and
3.1.1.3 *Webster's New Collegiate Dictionary*.⁴

3.1.2 For definitions and classifications of soil related terms used, refer to Practice D2488 and Terminology D653. Additional terms that require clarification are defined in 3.2.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

³ *Compilation of ASTM Standard Terminology*, Sixth edition, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA, 1986.

⁴ *Webster's New Collegiate Dictionary*, Fifth edition, 1977.

¹ This guide is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.21 on Groundwater and Vadose Zone Investigations.

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Type of Sampler	Obtains Core Sample Yes No	Most Suitable Core Types Csh. Cohless	Operation in Stoney Soils Fav. Unfav.	Most Suitable Soil Moisture Conditions Wet Dry Inter.	Access to Sample Sites During Poor Soil Conditions Yes No	Relative Sample Size Sm. Lg.	Labor Requirements Sngl. 2/More
A. Drill Rig Samplers							
1. Multipurpose Drill Rig	◆	◆	◆	◆	◆	◆	◆
2. Split-barrel Drive Sampler	◆	◆	◆	◆	◆	◆	◆
3. Thin-Walled Tube Sampler	◆	◆	◆	◆	◆	◆	◆
4. Piston Sampler	◆	◆	◆	◆	◆	◆	◆
5. Continuous Sample Tube System	◆	◆	◆	◆	◆	◆	◆
6. Hand Held Power Auger	◆	◆	◆	◆	◆	◆	◆
B. Hand Operated Samplers							
1. Screw-Type Auger	◆	◆	◆	◆	◆	◆	◆
2. Barrel Auger	◆	◆	◆	◆	◆	◆	◆
a. Post-Hole Auger	◆	◆	◆	◆	◆	◆	◆
b. Dutch Auger	◆	◆	◆	◆	◆	◆	◆
c. Regular Barrel Auger	◆	◆	◆	◆	◆	◆	◆
d. Sand Auger	◆	◆	◆	◆	◆	◆	◆
e. Mud Auger	◆	◆	◆	◆	◆	◆	◆
3. Tube-Type Sampler	◆	◆	◆	◆	◆	◆	◆
a. Soil Sampling Tube	◆	◆	◆	◆	◆	◆	◆
(1) Wet Tip	◆	◆	◆	◆	◆	◆	◆
(2) Dry Tip	◆	◆	◆	◆	◆	◆	◆
b. Vehmeyer Tube	◆	◆	◆	◆	◆	◆	◆

FIG. 1 Criteria for Selecting Soil Sampling Equipment

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *cascading water*—perched ground water that enters a well casing via cracks or uncovered perforations, trickling, or pouring down the inside of the casing.

3.2.2 *sludge*—a water charged sedimentary deposit.

3.2.2.1 *Discussion*—The water-formed sedimentary deposit may include all suspended solids carried by the water and trace elements that were in solution in the water. Sludge usually does not cohere sufficiently to retain its physical shape when mechanical means are used to remove it from the surface on which it deposits, but it may be baked in place and be adherent.

4. Summary of Guide

4.1 Sampling vadose zone soil involves inserting into the ground a device that retains and recovers a sample. Devices and systems for vadose zone sampling are divided into two general groups, namely the following: samplers used in conjunction with hand operated devices; and samplers used in conjunction with multipurpose or auger drill rigs. This guide discusses these groups and their associated practices.

4.2 The discussion of each device is organized into three sections, describing the device, describing sampling methods, and limitations and advantages of its use.

4.3 This guide identifies and describes a number of sampling methods and samplers. It is advisable to consult available site-specific geological and hydrological data to assist in determining the sampling method and sampler best suited for a specific project. It is also advisable to contact a local firm providing the services required as not all sampling and drilling methods described in this guide are available nationwide.

5. Significance and Use

5.1 Chemical analyses of liquids, solids, and gases from the vadose zone can provide information on the presence, possible source, migration route, and physical-chemical behavior of contaminants. Remedial or mitigating measures can be formulated based on this information. This guide describes devices and procedures that can be used to obtain vadose zone soil samples.

5.2 Soil sampling is useful for the reasons presented in Section 1. However, it should be recognized that the general method is destructive, and that resampling at an exact location is not possible. Therefore, if a long term monitoring program is being designed, other methods for obtaining samples should be considered.

6. Criteria for Selecting Soil Samplers

6.1 Important criteria to consider when selecting devices for vadose zone soil sampling include the following:

6.1.1 Type of sample: An encased core sample, an uncased core sample, a depth-specific representative sample, or a sample according to requirements of the analyses,

6.1.2 Sample size requirements,

6.1.3 Suitability for sampling various soil types,

6.1.4 Maximum sampling depth,

6.1.5 Suitability for sampling soils under various moisture conditions,

6.1.6 Ability to minimize cross contamination,

6.1.7 Accessibility to the sampling site, and

6.1.8 Personnel requirements.

6.2 The sampling devices described in this guide have been evaluated for these criteria. The results are summarized in Fig. 1.

7. Sampling with Hand Operated Devices

7.1 These devices, that have mostly been developed for agricultural purposes, include:

7.1.1 Screw-type augers,

7.1.2 Barrel augers,

7.1.3 Tube-type samplers,

7.1.4 Hand held power augers, and

7.1.5 Trench sampling with shovels in conjunction with machine excavations.

7.2 The advantages of using hand operated devices over drill rigs are the ease of equipment transport to locations with poor vehicle access, and the lower costs of setup and decontamination. However, a major disadvantage is that these devices are limited to shallower depths than drill rigs.

7.3 Screw-Type Augers:

7.3.1 *Description*—The screw or ship auger is essentially a small diameter (for example, 1.5 in. (3.81 cm)) wood auger from which the cutting side flanges and tip have been removed (1)⁵ (see Fig. 2(a)). According to the Soil Survey Staff (1), the spiral part of the auger should be about 7 in. (18 cm) long, with the distances between flights about the same as the diameter (for example, 1.5 in.) of the auger. This facilitates measuring the depth of penetration of the tool. Variations on this design include the closed spiral auger and the Jamaica open spiral auger (2) (see Fig. 2(b) and (c)). The auger is welded onto a length of solid or tubular rod. The upper end of this rod is threaded, to accept a handle or extension rods. As many extensions are used as are required to reach the target sampling

⁵ The boldface numbers in parentheses refer to the list of references at the end of the text.

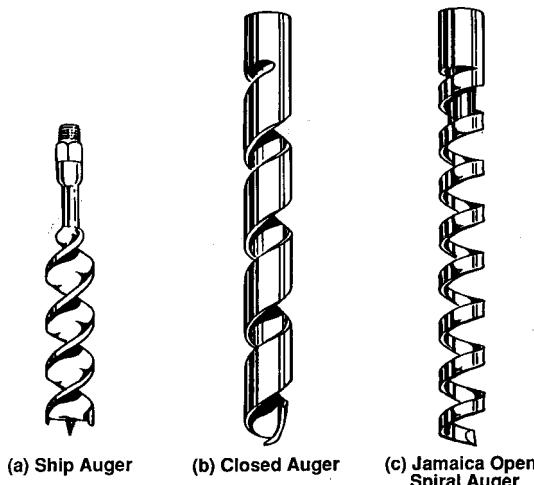


FIG. 2 Screw Type Augers

depth. The rod and the extensions are marked in even increments (for example, in 6-in. (15.24-cm) increments) above the base of the auger to aid in determining drilling depth. A wooden or metal handle fits into a tee-type coupling, screwed into the uppermost extension rod.

7.3.2 Sampling Method—For drilling, the auger is rotated manually. The operator may have to apply downward pressure to start and embed the auger; afterwards, the auger screws itself into the soil. The auger is advanced to its full length, and then pulled up and removed. Soil from the deepest interval penetrated by the auger is retained on the auger flights. A sample can be collected from the flights using a spatula. A foot pump operated hydraulic system has been developed to advance augers up to 4.5 in. (11.43 cm) in diameter. This larger diameter allows insertion of other sampling devices into the drill hole, once the auger is removed, if desired (3).⁶

7.3.3 Comments—Samples obtained with screw-type samplers are disturbed and are not truly core samples. Therefore, the samples are not suitable for tests requiring undisturbed samples, such as hydraulic conductivity tests. In addition, soil structures are disrupted and small scale lithologic features cannot be examined. Nevertheless, screw-type samplers are still suitable for use in collecting samples for the purpose of detecting contaminants. However, it is difficult to avoid transporting shallow soils downward when reentering a drill hole. When representative samples are desired from a discrete interval, the borehole must be made large enough to insert a sampler and extend it to the bottom of the borehole without touching the sides of the borehole. It is suggested that a larger diameter auger be used to advance and clear the borehole, then a smaller diameter auger sampler be used to obtain the sample. Screw-type augers work better in wet, cohesive soils than in dry, loose soils. Sampling in very dry (for example, powdery) soils may not be possible with these augers as soils will not be retained on the auger flights. Also, if the soil contains gravel or rock fragments larger than about one tenth of the hole diameter, drilling may not be possible (4).

7.4 Barrel Augers:

7.4.1 Description—The barrel auger consists of a bit with cutting edges welded to a short tube or barrel within which the soil sample is retained, welded in turn to shanks. The shanks are welded to a threaded rod at the other end. Extension rods are attached as required to reach the target sampling depth. Extensions are marked in increments above the base of the tool. The uppermost extension rod contains a tee-type coupling for a handle. The auger is available in carbon steel and stainless steel with hardened steel cutting edges (5, 6).

7.4.2 Sampling Method—The auger is rotated to advance the barrel into the ground. The operator may have to apply downward pressure to keep the auger advancing. When the barrel is filled, the unit is withdrawn from the soil cavity and a sample may be collected from the barrel.

7.4.3 Comments—Barrel augers generally provide larger samples than screw-type augers. The augers can penetrate shallow clays, silts, and fine grained sands (7).⁶ The augers do not work well in gravelly soils, caliche, or semi-lithified deposits. Samples obtained with barrel augers are disturbed and are not core samples. Therefore, the samples are not suitable for tests requiring undisturbed samples, such as hydraulic conductivity tests. Nevertheless, the samplers are still suitable for use in collecting samples for the purpose of detecting contaminants. Because the sample is retained inside the barrel, there is less of a chance of mixing it with soil from a shallower interval during insertion or withdrawal of the sampler. The following are five common barrel augers:

- 7.4.3.1 Post-hole augers (also called Iwan-type augers),
- 7.4.3.2 Dutch-type augers,
- 7.4.3.3 Regular or general purpose barrel augers,
- 7.4.3.4 Sand augers, and
- 7.4.3.5 Mud augers.

7.4.4 Post-Hole Augers—The most readily available barrel auger is the post-hole auger (also called the Iwan-type auger) (8). As shown in Fig. 3, the barrel consists of two-part cylindrical leaves rather than a complete cylinder and is slightly tapered toward the cutting bit. The taper and the cupped bit help to retain soils within the barrel. The barrel is available with a 3 to 12-in. (7.62 to 30.48-cm) diameter. There

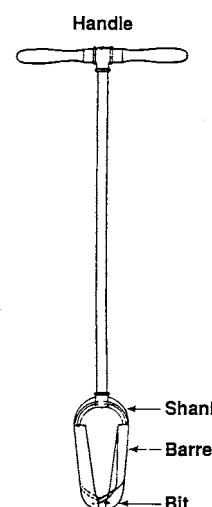


FIG. 3 Post-Hole Type Barrel Auger

⁶ This reference is manufacturer's literature, and it has not been subjected to technical review.

are two types of drilling systems, one has a single rod and handle, and the other has two handles. In stable, cohesive soils, the auger can be advanced up to 25 ft (7.62 m) (8).

7.4.5 Dutch-Type Augers—The Dutch-type auger (commercially developed by Eijkenkamp) is a smaller variation of the post-hole auger design. As shown in Fig. 4, the pointed bit is continuous with two, narrow part-cylindrical barrel segments, welded onto the shanks. The barrel generally has a 3 in. (7.62 cm) outside diameter. This tool is best suited for sampling wet, clayey soils.

7.4.6 Regular or General Purpose Barrel Augers—A version of the barrel auger commonly used by soil scientists and county agricultural agents is depicted in Fig. 5(a) and (b). As shown, the barrel is a complete cylinder. As with the post-hole auger, the cutting blades are cupped so that soil is loosened and forced into the barrel as the unit is rotated and pushed into the ground. Each filling of the barrel corresponds to a depth of penetration of 3 to 5 in. (7.62 to 12.70 cm) (1). The most popular barrel diameter is 3.5 in. (8.89 cm), but sizes ranging from 1.5 to 7 in. (3.81 to 17.78 cm) are available (6).⁶ Plastic, stainless steel, PTFE (polytetrafluoroethylene), or aluminum liners can also be used (6).⁶ Extension rods are available in 4 ft (1.22 m) lengths. The rods can be made from standard black pipe, from lightweight conduit or from seamless steel tubing. The extensions have evenly spaced marks to facilitate determining sample depth. The regular barrel auger is suitable for use in loam type soils.

7.4.7 Sand Augers—For dry, sandy soils it may be necessary to use a variation of the regular barrel auger that includes a specially-formed bit to retain the sample in the barrel (see Fig. 5(c)). Sand augers with 2, 3, or 4-in. (5.08, 7.62, or 10.16-cm) diameters are available (5).⁶

7.4.8 Mud Augers—Another variation on the regular barrel auger design is available for sampling wet, clayey soils. As shown in Fig. 5(d), the barrel is designed with open sides to facilitate extraction of samples. The bits are the same as those used on the regular barrel auger (6).⁶ Mud augers with 2, 3, or 4-in. (5.08, 7.62, or 10.16-cm) diameters are available (5).⁶

7.5 Tube-Type Samplers:

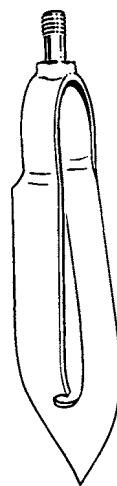
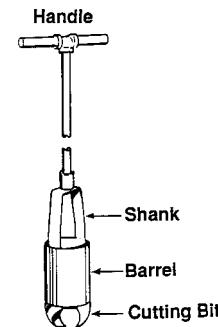


FIG. 4 Dutch Type Auger



(a) Regular Barrel Auger



(b) Regular Barrel Auger



(c) Sand Auger



(d) Mud Auger

FIG. 5 Barrel Auger Variations and Soil Moisture

7.5.1 Tube-type samplers generally have proportionally smaller diameters and greater body lengths than those of barrel augers.

7.5.2 For sampling, these units are perched into the soil causing the tube to fill with material from the interval penetrated. The assembly is then pulled to the surface and a sample can be collected from the tube. Since the device is not rotated, a nearly undisturbed sample can be obtained. Commercial units are available with foot lever attachments, a hydraulic apparatus, or drop-hammers to aid in driving the sampler into the ground (5).⁶ Vibratory heads have also been developed to advance tube-type samplers (9).⁶

7.5.3 These units are not as suitable for sampling in compacted, gravelly soils as are the barrel augers. They are preferred if an undisturbed sample is required. Commonly used varieties of the tube type samplers include:

7.5.3.1 Soil sampling tubes (also called Lord samplers),

7.5.3.2 Veihmeyer tubes (also called King tubes),

7.5.3.3 Thin-walled tube samplers (also called Shelby tubes),

7.5.3.4 Ring-lined barrel samplers, and

7.5.3.5 Piston samplers.

7.5.4 Soil Sampling Tubes:

7.5.4.1 Description—As depicted in Fig. 6, the soil sampling tube consists of a hardened cutting tip, a cut-away barrel, and an uppermost threaded segment. The cut-away barrel allows textural examination and easy removal of soil samples. Generally, the tube is constructed from high strength alloy steel (10).⁶ The samplers are available with 6, 12, 15, 18, and 24-in. (15.24, 30.48, 38.10, 45.72, and 60.96-cm) lengths (5, 6). The tubes are available with 1.13 or 0.88-in. (2.87 or 2.22-cm)

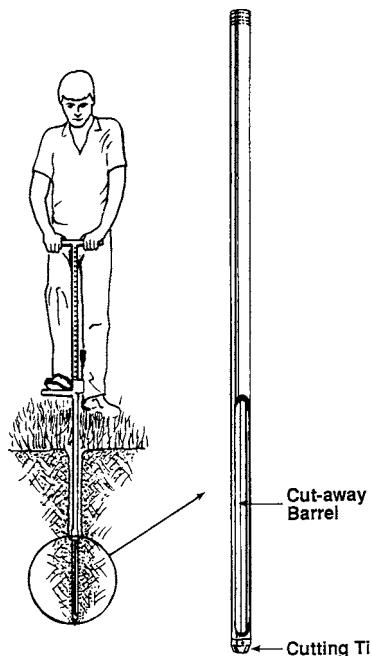


FIG. 6 Soil Sampling Tube

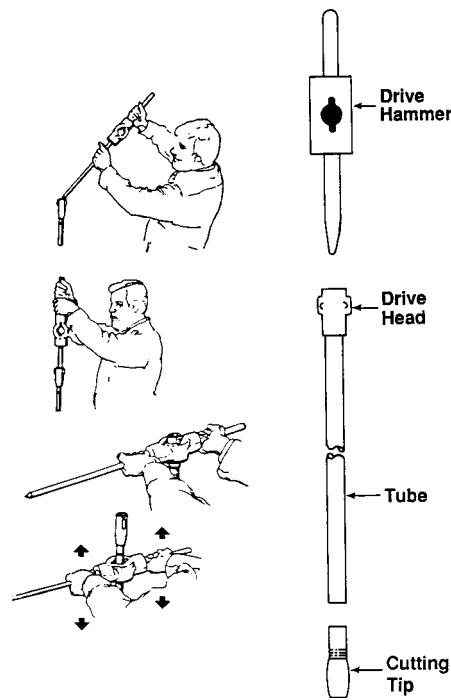


FIG. 7 Veihmeyer Tube

outside diameter. Two modified versions of the tip are available, for sampling in wet or dry soils. The sampling tube is attached to extension rods to attain the target sampling depth. A cross-handle is attached to the uppermost rod. Extension rods are made of lightweight, durable metal. They are available in a variety of lengths depending on the manufacturer. Markings on the extensions and the sampler facilitate determining sample depths.

7.5.4.2 Sampling Method—The sampler is pushed into the ground by leaning on the unit's handle. Once the sampler has reached the bottom of the sampling interval, it is twisted to break soil continuity at the tip. Depending on the type of cutting edge, the tube sampler may obtain samples varying in diameter from 0.69 to 0.75 in. (1.75 to 1.91 cm).

7.5.4.3 Comments—The soil sampling tube works best in soft, clayey, cohesive soils. If the soil contains cobbles or rock fragments larger than about one-half the cutting tip diameter, satisfactory sampling may not be possible. If the soil is cohesionless, it will not be retained in the tube. With time, the cutting tip will be damaged and worn dull. Most units are designed so that this part can be replaced.

7.5.5 Veihmeyer Tubes:

7.5.5.1 Description—The Veihmeyer tube is a long, complete cylinder. As shown in Fig. 6, this unit consists of a bevelled tip, that is threaded into the lower end of the tube, and a drive head threaded onto the upper end of the tube. The sampler is constructed of hardened steel. The tube is generally marked in even increments (for example, 1 ft or 0.30 m). These samplers are available in 4 to 16-ft (1.22 to 4.88-m) lengths with a 0.75-in. (1.91-cm) inside diameter.

7.5.5.2 Sampling Method—The lower guide rod of the drop hammer is slipped into the upper tube, through the drive head (see Fig. 7). The hammer is used to pound the sampler into the ground. The sampler is then retrieved by pulling or jerking up

on the hammer to force the sampler out of the soil cavity. Samples are extruded by forcing a rod through the tube.

7.5.5.3 Comments—Prior to sampling, the inside of the tube is sometimes coated with a lubricant to facilitate extrusion. However, the types of analyses to be performed on the samples should be considered to determine if the presence of lubricant will cause interference. Because the Veihmeyer sampler is a solid-walled tube and is fitted with a drop hammer, it can generally be used in more resistant soils than the soil sampling tube.

7.5.6 Thin-Walled Tube Samplers:

7.5.6.1 Description—Thin-walled tube (Shelby Tube) samplers are readily available with 2, 3, and 5-in. (5.08, 7.62, and 12.70-cm) outside diameters and are commonly 30 in. (76.20 cm) long. The 3 by 30-in. (7.62 by 76.20-cm) outside diameter long sampler is most common. The advancing end of the sampler is rolled inwardly and has a cutting edge with a smaller diameter than the tube inside diameter. The cutting edge inside diameter reduction, defined as a "clearance ratio," is usually in the range of 0.0050 to 0.0150 or 0.50 to 1.50 % (Refer to Practice D1587). The sampler tube is usually connected with set screws to a sampler head that in turn is threaded to connect with extension rods. Plastic and PTFE sealing caps for use after sampling are readily available for the 2, 3, and 5-in. (5.08, 7.62, and 12.70-cm) diameter tubes (refer to Practices D4220). Shelby tubes are commonly available in carbon steel but can be manufactured from other metal (see Fig. 8).

7.5.6.2 Sampling Method—The Shelby tube is pushed into soil by hand, with a jack-like system or with a hydraulic piston. The sample recovered is often less than the distance pushed, that is, the recovery ratio is less than 1.0. The recovery ratio is less than 1.0 because of soil compaction during sampling, and

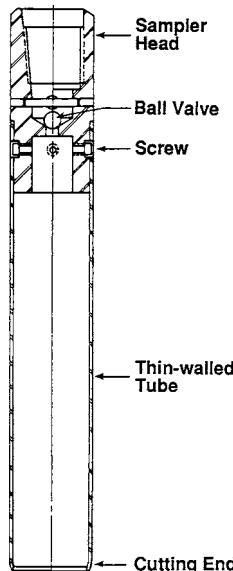


FIG. 8 Thin-Walled Tube Sampler

because friction between soil and the inner tube walls becomes greater than the shear strength of the soil in front of the tube. Consequently, soil in front of the advancing end of the tube is displaced laterally rather than entering the tube (11). In general, shorter tubes provide less-disturbed samples than longer tubes. Samples are extruded from the Shelby tube with a hydraulic ram. As with all sampling devices, the most disturbed portion of the sample in contact with the tube is considered unrepresentative. Wilson et al. (12) developed a paring device to remove this outer layer of the core during extrusion.

7.5.6.3 Comments—Shelby tubes are best used in clays, silts, and fine-grained sands. If the soils are cohesionless, they may not be retained in the tube. If firm to very hard soils are encountered, driving (hammering) the sampler may be required. However, this should be avoided as the tube may buckle under the drive stress.

7.5.7 Ring-Lined Barrel Samplers :

7.5.7.1 Description—As described in Practice D3550, the ring-lined barrel sampler consists of a one piece barrel or two split barrel halves, a drive shoe, rings, and a sampler head (see Fig. 9). The rings, that are usually brass, fit snugly inside the barrel and are designed to be directly inserted into geotechnical testing apparatuses when removed from the barrel. Most samplers are designed to hold at least two rings. The barrel is commonly 3.5 in. (8.89 cm) inside diameter and 3.94 to 5.91 in. (10 to 15 cm) long (5).⁶ With these lengths, the barrel can be fitted with a variety of liners ranging in length from 1 to 2.36 in. (2.54 to 6 cm).

7.5.7.2 Sampling Method—The ring-lined barrel sampler can be driven or pushed into soil. Once retrieved, the sampler is disassembled, and the sample-filled rings are removed. The rings are usually removed as one unit and placed into a capped container. Alternately, the individual soil-filled rings can be capped with plastic or PTFE and then sealed with wax or adhesive tape (refer to Practices D4220).

7.5.7.3 Comments—Because barrel samplers are more rigid than thin-walled tubes, they can be driven into hard soils and

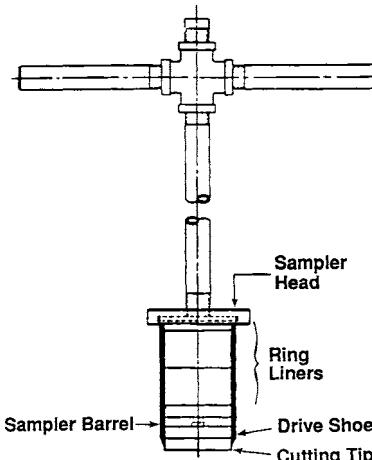


FIG. 9 Hand Operated Ring-Lined Barrel Sampler

soils containing sands and gravels that might damage thin-walled tubes. The sampler provides samples in rings which can be handled without further disturbance of the soil. Because of this, these devices are most often used when geotechnical or chemical analyses are to be performed.

7.5.8 Piston Samplers:

7.5.8.1 Description—Locally saturated (for example, by perched ground water), or cohesionless soils, and very soft soils or sludges may not be retained in most samplers, even when fitted with retainer baskets or flap valves. Piston samplers can be used in these situations. The sampler consists of a sampling tube, extension pipe attached to the tube, an internal piston, and rods connected to the piston and running through the extension pipe (see Fig. 10). These samplers are often built, as needed, out of common PVC (for use in sludge) or steel pipe

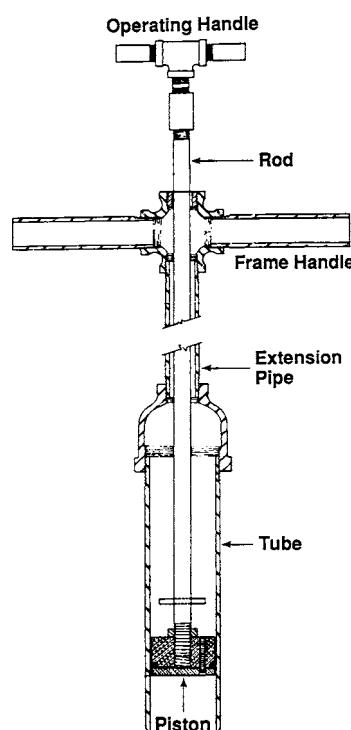


FIG. 10 Hand Operated Piston Sampler

fittings. The sampling tube commonly has a 0.75 to 3-in. (1.91 to 7.62-cm) inside diameter and is 8 in. to 9 ft (20.32 cm to 2.74 m) long (13). A variation designed for sampling peat has a cone shaped piston (8).

7.5.8.2 Sampling Method—The sampler can be pushed into the ground with the handle or driven into the ground with a drop hammer (13). As the tube is advanced, the piston is held stationary or pulled upward with the attached rods. Once the tube has been advanced through the sampling interval, it is rotated to break suction that might have developed between the soil and the outside wall of the tube. The sampler is then pulled to the surface keeping the piston rod fixed with respect to the extension pipe. The sample is retained because of suction that develops between the piston and the sample. Upon retrieval, the sample is extruded by using the piston to force the sample out of the tube. Sharma and De Dalta (14) described a cylindrical sampler for use in puddled soils that would flow back out of most samplers. The design includes a basal shutter that retains the sample while the sampler is withdrawn from the soil.

7.5.8.3 Comments—Because the sampler depends on development of suction between the sample and the piston, it may not work in unsaturated, coarse-grained sands and gravels. This is due to the high air permeability of such material that prevents the creation of high suction.

7.6 Hand Held Power Augers:

7.6.1 Description—A very simple, commercially available auger consists of a solid flight auger attached to and driven by a small air-cooled engine (see Fig. 11). Two handles on the head assembly allow two operators to guide the auger into the soil. Throttle and clutch controls are integrated into grips on the handles. Augers are available with diameters ranging from 2 to 16 in. (5.08 to 40.64 cm). The auger sections are commonly 3 ft (0.91 m) long.

7.6.2 Sampling Method—As the auger rotates into soil, cuttings advance up the flights and are discharged at the surface. Soil samples can be collected from the surface discharge, or from the auger flights after pulling the auger out of the ground. Alternatively, samples can be collected with other samplers (for example, a thin-walled tube) after auger removal.

7.6.3 Comments—As discussed in 7.3, if samples are collected from surface discharge or from the flights, they are

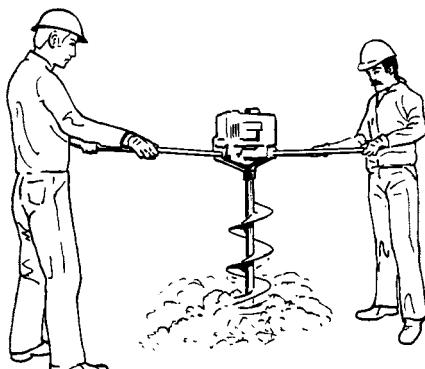


FIG. 11 Hand Held Power Auger

disturbed and are not suitable for some uses. In addition, if samples are collected from surface discharge, it is difficult to determine the depth from which the soil came and uncontrolled mixing of soil from different depth intervals can occur. The auger operates well in most soils. However, if the soil is cohesionless, it may not be retained on the flights and sampling in that fashion may not be possible. If the soil contains cobbles or boulders, drilling may not be possible. If the auger “hangs up” on an obstruction, the machine will start to rotate at the surface. Otherwise, the operator should not attempt to stop rotation of the machine by grabbing the handles. An alternate design that transfers the torque to a separate engine prevents this problem (15).⁶ As previously stated, it is prudent to perform the field work with at least two people present.

7.7 Trench Sampling:

7.7.1 Description—Soils may be sampled from a trench or pit excavated for that purpose. Excavation is usually performed by a backhoe, and samples are collected with knives, trowels, or shovels. Occasionally, samples are collected from the sides or the bottom of the trench or pit with hand augers or tube-type samplers.

7.7.2 Sampling Method—Excavation is performed under the guidance of the sampling technician. Sampling is performed only after the backhoe has moved away from the trench or pit. When the trench or pit is in unstable material or is more than a few feet deep, the sampling technician should only enter the trench or pit after it has been shored up or the sidewalls have been cut back to within the angle of repose (see Occupational Safety and Health Administration regulations). Otherwise, samples are more commonly collected at the surface from the bucket of the backhoe as excavation occurs.

7.7.3 Comments—The maximum sampling depth for the trench or pit method is dictated by the reach of the backhoe, the soil type and the moisture content of the soil. Maximum depths of up to 20 ft (6.10 m) can be obtained in moist clays. Maximum depths of less than 10 ft (3.05 m) are common in dry sands. Samples collected from the backhoe bucket should be taken from the center of the material to prevent collecting soil contaminated by the bucket surface, and to prevent inclusion of materials that may have fallen from above the desired sampling interval. However, when this is done, it is difficult to accurately estimate the depth from which the sample was obtained. Trenches are useful for obtaining lithologic information since cross sections of the vadose zone can be studied and photographed. Trench or pit sampling is often used in areas with difficult access since backhoes are designed to travel on rough terrain. However, because the process involves excavating a much larger hole than drilling methods, chances of encountering underground utilities are increased, and proper backfilling and compaction of the trench is often very difficult.

8. Multipurpose and Auger Drill Rigs

8.1 Vadose zone samplers used in conjunction with drill rigs are identical to those used to sample below the water table. However, commonly used drill rigs such as cable tool and rotary units are not recommended as they generally require the introduction of drilling fluids to the soils to be sampled. Air rotary drilling is also undesirable for obtaining samples for

pore liquid or gas extraction. In most cases, hollow-stem augers with some type of cylindrical sampler provide the greatest level of assurance that soil sampled within the vadose zone was not carried downward by the drilling or sampling process. For some situations, such as sampling firm to very hard ground, using multipurpose auger-core-rotary drill rigs will be necessary. For some geologic circumstances the use of solid stem augers will provide an adequate drilling method.

8.2 Multipurpose Auger-Core-Rotary Drill Rigs:

8.2.1 Multipurpose auger-core-rotary drill rigs are generally equipped with rotary power and vertical feed control to advance both hollow-stem augers and continuous flight (solid stem) augers to depths greater than 100 ft (30.48 m). These same drills have secondary capability for rotary and core drilling. The larger of these drills are typically mounted on 20 000 to 30 000-lb (9070 to 13605-kg) GVW trucks. The same multipurpose drill rigs are available on both rubber-tired and track-driven all-terrain carriers. The smaller of the multipurpose drills are typically mounted on trailers or one-ton, 4 by 4 trucks.

8.2.2 When equipped with augers, the sampling process is identical to that for auger drill rigs. When multipurpose auger-core-rotary drill rigs or auger drill rigs are used, the speed of drilling and sampling is much greater than with hand operated equipment. Therefore it is useful to have a larger crew to efficiently handle, log, identify, and preserve the samples.

8.3 *Auger Drill Rigs*—Auger drill rigs are similar to multipurpose auger-core-rotary drill rigs. They are manufactured specifically for efficient auger drilling but do not have the pumps and hoists that are required for efficient core or rotary drilling. The rigs can be equipped with either solid stem or hollow stem augers. There are relatively few auger drills available in comparison to multipurpose auger-core-rotary drills.

9. Auger Drilling and Sampling

9.1 Solid Stem Auger Drilling and Sampling (see Practice D1452):

9.1.1 *Description*—The tools used for solid-stem auger drilling include: auger sections, the drive cap, and the cutter head (see Fig. 12). Auger sections are typically 5 ft (1.52 m) long and are interchangeable for assembly in an articulated but continuously flighted column. Augers are available in diameters up to 24 in. (60.96 cm). The cutter head is attached to the lowermost or leading flight of the auger column. It is about 0.5 in. (1.25 cm) larger in diameter than the flights. Head types include fish tail or drag bits for use in cohesionless materials, and clay or stinger bits for use in more consolidated material (16).

9.1.2 *Sampling Method*—As the auger column is rotated into soil, cuttings are retained on the flights. The augers are then removed from the hole and samples are taken from the retained soil. Samples obtained with solid stem augers are disturbed and are not core samples. Therefore, the samples are not suitable for analyses requiring undisturbed samples, such as hydraulic conductivity tests. This sampling method can provide an adequately clean borehole in some clayey and silty soils. However, when using the method in caving or squeezing

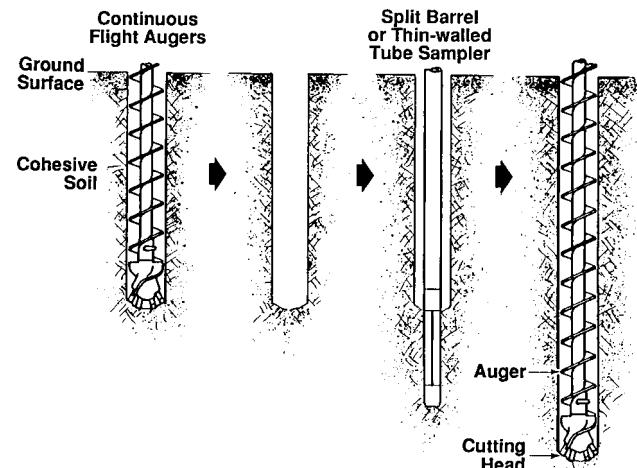


FIG. 12 Solid Stem Auger Sampling

ground, the quality and the origin of the recovered samples are questionable because soils from different intervals may have mixed. Therefore, when representative samples from discrete depths are desired, the borehole should be made large enough to insert a smaller diameter auger or another sampler (for example, a thin-walled tube) to the bottom of the borehole, without touching the sides of the borehole (see Fig. 11), to collect a discrete sample from the interval ahead.

9.1.3 *Comments*—Typical drilling depths with solid stem augers range from 50 to 120 ft (15.24 to 36.58 m). The greater drilling depths are attained in firm, silty and clayey soils. However, the depth to which the hole will remain open for sampling once the auger column has been removed is usually less than the maximum drilling depth. If cascading water or cohesionless soils are encountered, it can be expected that the hole will cave at that depth. The sample depth measurement, as taken from its location on an auger, is not precise. This is because soil may move up the flights in an uneven fashion as the auger column is advanced. As with hollow-stem augers, solid stem augers are often painted by the driller or manufacturer. It is prudent to remove this paint before drilling. The majority of the paint can be removed by drilling through sandy soils or by sand blasting. As with all sampling devices, decontamination (for example, steam cleaning) should be performed between holes when chemical analyses are to be performed on the samples. This is especially important with the solid stem auger as it doubles as the drilling and sampling tool.

9.2 Bucket Auger Drilling and Sampling:

9.2.1 *Description*—The bucket auger is a large diameter cylindrical bucket with auger-type cutting blades on the bottom. The bucket can have a diameter ranging from 12 in. (30.48 cm) up to 6 ft (1.83 m) with lengths varying from 24 to 48 in. (60.96 to 121.92 cm) (17). The bottom is hinged to allow cuttings to be emptied out (see Fig. 13).

9.2.2 *Sampling Method*—The bucket is rotated to depth in the vadose zone until the bucket is full. Therefore, depending on the bucket length, sampling intervals can range from 24 to 48 in. (60.96 to 121.92 cm). Sampling consists of extracting small diameter core samples from the interior of the bucket after lowering the full bucket to the ground (see Section 7).

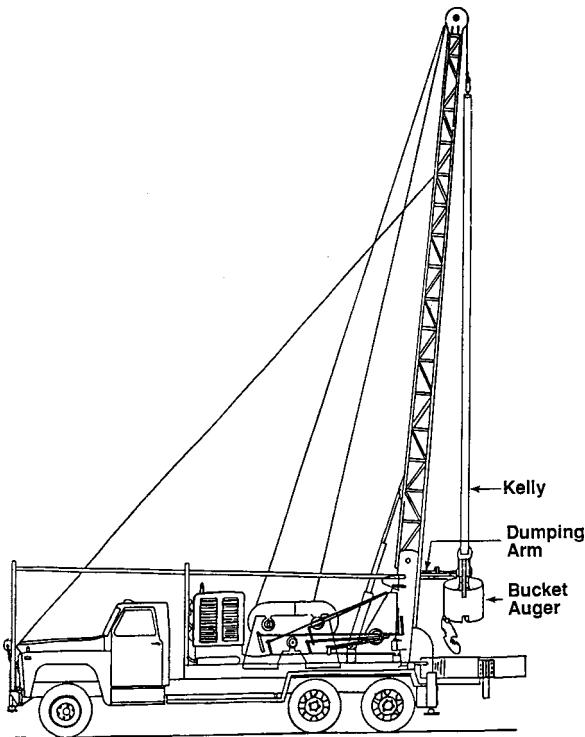


FIG. 13 Bucket Auger and Drilling Rig

This approach minimizes problems with undiscernible mixing of discrete portions to be sampled.

9.2.3 Comments—The bucket auger is best suited for sampling from relatively stable clays as the caving problems discussed in 9.1.3 are amplified by the larger hole diameter. Boulders can impede drilling and may have to be individually removed from the hole before sampling can continue (15)⁶. Generally, boulders up to one-third or one-fourth the bucket diameter can be picked up by the bucket. Common sampling depths are less than 50 ft (15.24 m) but holes up to 250 ft (76.20 m) deep have been drilled (16, 17).

9.3 Hollow Stem Auger Drilling and Sampling:

9.3.1 Description—Outer components of the hollow stem auger system include: hollow auger sections, the hollow auger head, and the drive cap. Inner components include: the pilot assembly, the center rod column, and the rod-to-cap adaptor (see Fig. 14). The auger head contains replaceable carbide teeth that pulverize the formation during flight column rotation. The cutting diameter is somewhat greater than the flighting diameter because of the protruding teeth. Auger sections are typically 5 ft (1.52 m) long and are interchangeable for assembly in an articulated but continuously flighted column. Drilling progresses in 5 ft (1.52 m) or shorter increments and sampling can be accomplished at any depth within that increment. Upon advancement of a 5 ft (1.52 m) increment, another 5 ft (1.52 m) section of hollow-stem auger and center rod is added. Hollow-stem augers are readily available with 2.25, 2.75, 3.25, 3.75, 4.25, 6.25, and 8.25-in. (5.72, 6.99, 8.26, 9.53, 10.80, 15.88, and 20.96-cm) inside diameters.

9.3.2 Sampling Method—The auger column and pilot assembly are advanced to the top of the desired sampling interval. Sampling is accomplished by removing the pilot

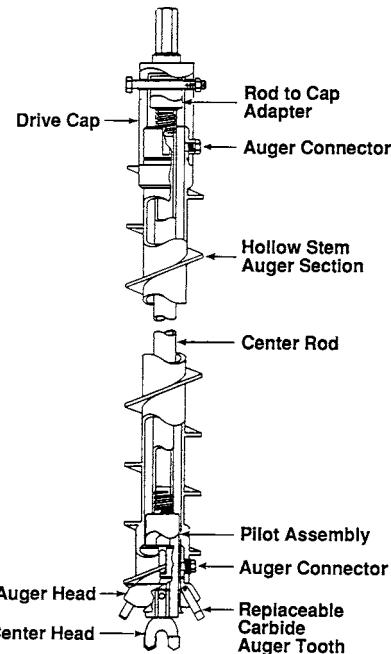


FIG. 14 Hollow-Stem Auger Components

assembly and center rod, if they are used, and inserting the sampler through the hollow stem of the auger column (see Fig. 15). The sampler may be lowered to the sampling depth by attaching it to center rods or by using a wireline assembly (12). When the sampler is attached to center rods, a sample is collected by pushing or driving the sampler into undisturbed soil with the rig hydraulic system or with a drop hammer. When a wireline is used, the sampler is locked into place ahead of the lower-most auger and advanced into the sampling interval by rotating the auger column (18).⁶ Hollow stem augers with a 6.25-in. (15.88-cm) inside diameter allow the use of 5-in. (12.70-cm) outside diameter Shelby tubes and 4.5-in. (11.43-cm) outside diameter split barrel samplers (see 9.4).

9.3.3 Comments—The purpose of the center head (pilot) assembly is to prevent soils from entering the auger column as it is advanced (19). Driscoll (17) suggests that the assembly may be omitted when drilling through hard, silty and clayey soils as these materials will usually form a 2 to 4 in. (5.08 to 10.16 cm) long plug at the auger opening. However, Hackett (19) recommends that the pilot assembly be used when detailed

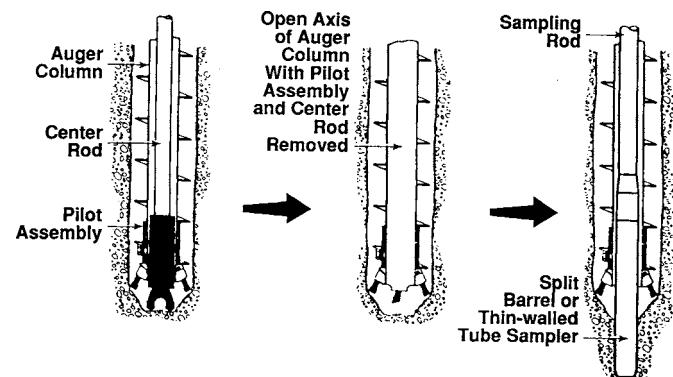


FIG. 15 Hollow-Stem Auger Sampling

samples are required. When perched water is encountered, "heaving sands" that move up into the auger column upon pilot assembly removal during sampling, may be a concern. Various one-way plugs that allow sampling, but that prevent sand from moving into the auger column, are described in Hackett (19). The important capability of being able to obtain samples that do not contain mixed material from shallow sources in the hole is enhanced by using the hollow-stem auger method. However, because the sections are hollow, decontamination of the auger interiors between holes to prevent cross contamination is difficult. High pressure steam cleaners are usually necessary to remove caked-on soils and contaminants. Hollow stem augers may advance rapidly through unconsolidated materials.

9.4 Sampling Devices:

9.4.1 Sampling devices used in conjunction with hollow stem augers and occasionally in holes advanced by solid stem augers include:

9.4.1.1 Thin-walled tube samplers (also called Shelby tubes),

9.4.1.2 Split-barrel drive samplers (also called Split spoons),

9.4.1.3 Ring-lined barrel samplers,

9.4.1.4 Continuous sample tube systems, and

9.4.1.5 Piston samplers.

9.4.2 These samplers are either pushed or driven in sequence with an increment of drilling or advanced simultaneously with the advance of a hollow stem auger column.

9.4.3 Thin-Walled Tube Samplers:

9.4.3.1 *Description*—The thin-walled tube sampler consists of a tube connected to a head with screws. The head is threaded to connect with standard drill rods. The head contains a ball check valve. Thin-walled tube (Shelby tube) samplers are readily available with 2, 3, and 5-in. (5.08, 7.62, and 12.70-cm) outside diameter and are commonly 30 in. (76.20 cm) long. The 3 by 30 in. (7.62 by 76.20 cm) outside diameter long sampler is most common. The advancing end of the sampler is constructed with an inward lip, machined to a cutting edge, that has a smaller diameter than the tube inside diameter. The cutting edge inside diameter reduction, defined as a "clearance ratio," is usually in the range of 0.0050 to 0.0150 or 0.50 to 1.50 % (refer to Practice D1587). PTFE or plastic sealing caps and other sealing devices for use after sampling are readily available for the 2, 3, and 5-in. (5.08, 7.62 and 12.70-cm) diameter tubes (refer to Practices D4220). Shelby tubes are commonly available in carbon steel but can be manufactured from other metal (see Fig. 8).

9.4.3.2 *Sampling Methods*—When a Shelby tube is pushed into soil, the length of the sample recovered is often less than the distance pushed, that is, the recovery ratio is less than 1.0 (see 7.5.6.2). In addition, a portion of the sample frequently remains in the borehole after retrieval of the sampler. This is due to suction that develops at the sampler-soil interface. This suction may be broken by twisting the sampler prior to retrieval or by advancing the auger column below the base of the sampler before retrieval (20). Samples are extruded from the Shelby tube with a hydraulic ram. As with all sampling devices, the portion of the sample in contact with the tube is

considered disturbed and unrepresentative. Wilson et al. (12) developed a paring device to remove this outer layer of the core during extrusion.

9.4.3.3 *Comments*—The ball check valve was originally intended to provide a vent for drilling fluids when pushing the tube into soil, and also to prevent the column of fluid within the drill stem from forcing the sample out of the tube during retrieval. Since drilling fluids are not used when sampling in the vadose zone, these considerations are not important. However, the valve does provide a vent for air displaced as the sampler is pushed into soil. Shelby tubes are best used in clays, silts, and fine grained sands. They can be pushed with the hydraulic system of most drill rigs in fine grained sands that are loose to moderately consolidated or in clays and silts that are soft to firm. If the soils are cohesionless, they may not be retained in the tube. If consolidated or hard soils are encountered, driving the sampler may be required. However, some tubes may buckle under the drive stress. A spring-loaded barrel has been developed to protect the Shelby tube from buckling when sampling these soils (21).⁶

9.4.4 Split-Barrel Drive Samplers:

9.4.4.1 *Description*—The split-barrel drive sampler consists of two split-barrel halves, a drive shoe, and a sampler head containing a ball check valve, all of which are threaded together (see Fig. 16). The most common size has a 2-in. (5.08-cm) outside diameter and a 1.5-in. (3.81-cm) inside diameter split barrel with a 1.375-in. (3.49-cm) inside diameter drive shoe. This sampler is used extensively in geotechnical exploration (Refer to Test Method D1586). When fitted with a 16 gauge liner for encased cores, the sampler has a 1.375-in. (3.49-cm) inside diameter throughout. A 3-in. (7.62-cm) outside diameter by 2.5-in. (6.35-cm) inside diameter split-barrel sampler with a 2.375-in. (6.03-cm) inside diameter drive shoe is also available (22).⁶ Other split-barrel samplers in the size

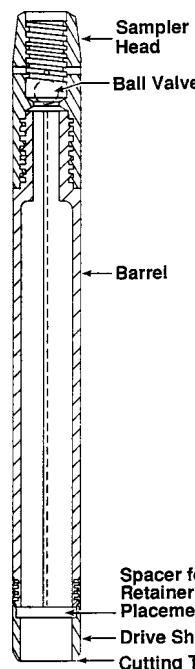


FIG. 16 Split-Barrel Drive Sampler

range of 2.5-in. (6.35-cm) to 4.5-in. (11.43-cm) outside diameter are manufactured but are less common. A plastic or metal retainer basket, or a flap valve is often fitted into the drive shoe to prevent samples from falling out during retrieval.

9.4.4.2 Sampling Method—As described in Test Method D1586 the sampler is threaded onto drilling rods and is lowered to the bottom of the boring. The sampler is then driven into the soil with blows from a drop hammer attached to the drill rig. The hammer usually weighs 140 lb and is operated by the driller. The sampler is extracted from the soil in a manner that will ensure maximum sample recovery. A sample is obtained by disassembling the drive shoe and head, and splitting the barrel to expose the core of soil. Material disturbed by contact with the barrel can be scraped away, or a less disturbed interior portion collected with a spatula.

9.4.4.3 Comments—Split barrel drive samplers can be used in all soil types if the larger grain sizes can enter through the opening of the drive shoe. Because the sampler can be fitted with a retainer basket, it is typically used in place of thin-walled tubes when cohesionless soils are to be sampled.

9.4.5 Ring-Lined Barrel Samplers :

9.4.5.1 Description—As described in Practice D3550, the ring-lined barrel sampler consists of a one piece barrel or two split-barrel halves, a drive shoe, rings, a waste barrel and a sampler head containing a ball check valve (see Fig. 17). The rings fit snugly inside the barrel and are designed to be directly inserted into geotechnical testing apparatus when removed from the barrel. Most samplers are designed to hold at least six rings. The waste barrel provides a space above the rings into which disturbed soil, originally at the bottom of the hole, can move. The samplers are commonly available with 2, 3, and 4-in. (5.08, 7.62, and 10.16-cm) outside diameter.

9.4.5.2 Sampling Method—The ring-lined barrel sampler can be driven or pushed into soil. It is important to insert the sampler deep enough to allow all disturbed soil to move through the rings and into the waste barrel. Once retrieved, the sampler is disassembled, and the sample filled rings are carefully removed. The rings are usually removed as one unit and placed into a capped container. Alternately, the individual soil filled rings can be capped with plastic or PTFE and even sealed with wax or adhesive tape (refer to Practices D4220).

9.4.5.3 Comments—Because ring-lined barrel samplers are more rigid than thin-walled tubes, they can be driven into soils containing sands and gravels that might damage thin-walled tubes. The sampler provides samples in rings that can be handled without further disturbance of the soil. Because of this, these devices are most often used when geotechnical or chemical analyses are to be performed.

9.4.6 Continuous Sample Tube System:

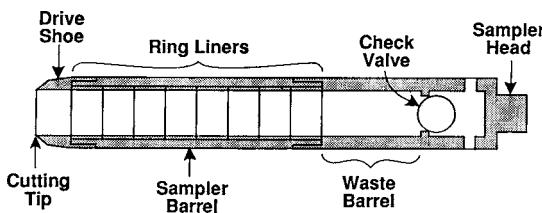


FIG. 17 Ring-Lined Barrel Sampler

9.4.6.1 Description—Continuous sample tube systems that fit within a hollow-stem auger column are readily available in North America. The barrel is typically 5 ft (1.52 m) long, and fits within the lead auger of the hollow auger column. The sampler is prevented from rotating as the auger column is turned (20). For many conditions the sampler provides continuous, 5-ft (1.52-m) samples (see Fig. 18). The assembly can be split- or solid-barrel and can be used with or without liners of various metallic and nonmetallic materials (20). Two clear, plastic, 30 in. (76.20 cm) long liners are often used. The sampler may also be fitted with a plastic or metal retainer basket, or a flap valve to prevent cohesionless soils from falling out of the sampler during retrieval (20).

9.4.6.2 Sampling Method—The sampler is locked in place inside the auger column with its open end protruding a short distance beyond the end of the column. While advancing the column, soil enters the non-rotating sampling barrel. After a 5-ft (1.52-m) advance, the sampler is withdrawn, and the liner (if used) is removed and capped.

9.4.6.3 Comments—The continuous sample tube system replaces the pilot head assembly in the hollow-stem auger column. Because of this, sampling speed is greatly increased since the pilot assembly does not have to be removed before taking a sample. The continuous sample tube system is best used in clays, silts, and in fine grained sands. It can be used to sample soils that are much more consolidated or harder than can be sampled with Shelby tubes.

9.4.7 Piston Samplers:

9.4.7.1 Description—Locally saturated (for example, perched ground water), or cohesionless soils, and very soft soils or sludges may not be retained in most samplers, even when they have been fitted with retainer baskets or flap valves. Piston samplers are often used under these conditions. The sampler consists of a sampling tube, an internal piston, and a drive head. The piston fits snugly inside the tube. The piston is attached to a rod assembly or a cable that leads to the surface.

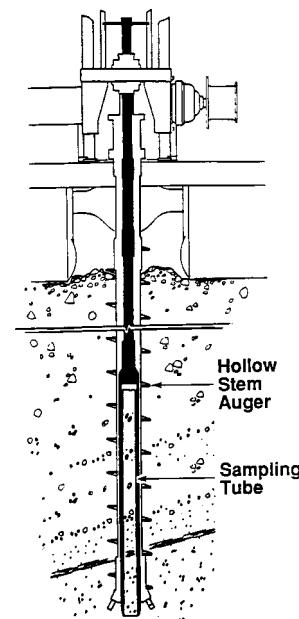


FIG. 18 Continuous Sample Tube System

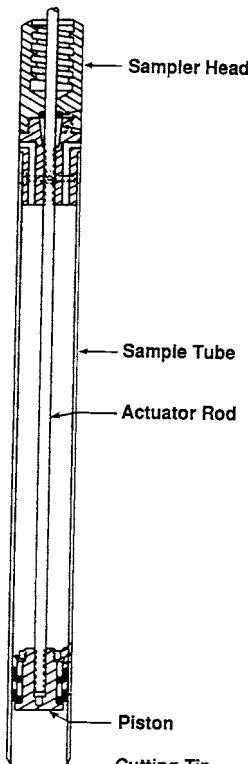


FIG. 19 Piston Sampler

Tubes made of steel are available in 5.5 and 30-in. (13.97 and 76.20-cm) and 5-ft (1.5-m) lengths with 0.75, 2, 3, 4, and 5-in. (1.91, 5.08, 7.62, 10.16, and 12.70-cm) inside diameter (22).

23). When equipped with a hardened steel drive shoe, the tube can be fitted with a liner made of aluminum clear PVC, or another material (see Fig. 19) (24). A version of the sampler designed for peat sampling has a cone shaped piston (8).

9.4.7.2 *Sampling Method*—Prior to sampling, the piston is placed at the base (advancing end) of the tube. The sampler is then attached to drill rods and lowered down the borehole or hollow-stem auger column to the bottom of the hole (top of the sampling interval). The sampler is then pushed or driven into the sampling interval. As the tube moves downward, the piston remains stationary and in contact with the top of the soil sample. When the sampler is withdrawn, soil is retained because of suction that develops between the piston and the soil core within the sampler. This suction is stronger than the suction at the bottom of the sampler that would tend to extract soil from the sampler. Even so, it is often useful to twist the sampler with the drill rods prior to retrieval, to break suction at the bottom end and ensure that the sample will not be pulled out of the sampler.

9.4.7.3 *Comments*—Average recovery ratios greater than 0.9 can be attained with this sampling tool (24, 25). However, because the sampler depends on development of suction between the sample and the piston, it may not work in unsaturated, coarse grained sands and gravels. This is due to the high air permeability of such material that prevents the creation of suction with the sampler. Samples collected with piston samplers are relatively undisturbed. Zapico et al. (24) described techniques for extracting fluid samples directly from liners, and for converting liners into permeameters.

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Addendum 6. Sample Inventory Form

Project Name:							Contact:								
Sample ID / Station ID	Date	Time	Sample Type			No. Containers	Sample Designation (Primary / Secondary)	Analyses Requested							Notes
			Sediment	Soil	Grab			PCBs	Aroclors	Cong / Hom	High Res	PCDD / PCDF	Mercury	TOC	
Relinquished By	Signature			Date/Time		Received By			Signature			Date/Time			

Page ____ of ____

Addendum 7. Field Chain-of-Custody Form

CHAIN OF CUSTODY RECORD

PROJ. NO.		PROJECT NAME				NO. OF CON- TAINERS	REMARKS						
SAMPLERS: <i>(Signature)</i>													
STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION								
Relinquished by: <i>(Signature)</i>			Date / Time		Received by: <i>(Signature)</i>		Relinquished by: <i>(Signature)</i>			Date / Time		Received by: <i>(Signature)</i>	
Relinquished by: <i>(Signature)</i>			Date / Time		Received by: <i>(Signature)</i>		Relinquished by: <i>(Signature)</i>			Date / Time		Received by: <i>(Signature)</i>	
Relinquished by: <i>(Signature)</i>			Date / Time		Received for Laboratory by: <i>(Signature)</i>		Date / Time		Remarks				
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