Work Plan for Confirmation Sediment Sampling in Springstead and Hogtown Creeks

Cabot Carbon/Koppers Superfund Site

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October 16, 2015



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1 Introduction

Springstead and Hogtown Creeks (creeks) are located downstream of the Cabot Carbon/Koppers Superfund Site (Site) in Gainesville, Florida (Figure 1). In 2011, Cabot conducted a removal action to address sediments in isolated areas within the creeks, which were affected with pine tar residuals from Cabot Carbon's historic operations (Gradient, 2011). The purpose of this proposed work is to collect sediment quality data to determine if there are any remaining potential risks to human health or the environment associated with creek conditions.

1.1 Background and Setting

The creeks have been the subject of numerous sediment and surface water quality investigations over the last three decades (US EPA, 1980; IT, 1987; Hunter/ESE, 1990; ACEPD, 1994; ACEPD, 2006, 2007, 2009, 2010). These include the early remedial investigations in the 1980s (US EPA, 1980; IT, 1987), a CERCLA remedial investigation and risk assessment upon which the US EPA Record of Decision (ROD) (US EPA, 1990) was developed (Hunter/ESE, 1990), and the more recent sediment studies conducted by the Alachua County Environmental Protection Department (ACEPD, 2006, 2007, 2009, 2010). While the objective of some of these studies was general characterization of conditions, others were undertaken in response to citizen observations of tar within the creek(s), *i.e.*, were biased towards areas believed to be affected by tar. The investigations generally found the presence of sporadic tar deposits and low levels of volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) in sediments.

The recent studies undertaken by ACEPD (ACEPD, 2009, 2010) were the most comprehensive in scope. These studies included a thorough field reconnaissance survey (*i.e.*, the advancement of probes at hundreds of locations in the creeks), which identified and delineated the presence of tarry material in about 10 areas in the creeks, and a follow-up focused sediment sampling that detected polycyclic aromatic hydrocarbons (PAHs) in sediment samples. While PAHs are minor constituents of pine tar, they are also associated with several other natural and anthropogenic sources, including wood treating chemicals (*e.g.*, creosote). Given the urbanized setting of the Hogtown and Springstead Creek watersheds, PAHs in the creeks likely have numerous such point and non-point sources:

- Point Sources: Although Cabot Carbon operations ceased in 1966, historical inputs, including storm and waste water discharges and a breach of the former pine products lagoon by a developer, likely released pine tar to the creeks. The Koppers wood treating facility operated from 1916 to 2010 and discharged untreated storm water via a drainage ditch that traversed the Koppers property from south to north and discharged to Springstead Creek just downstream of its confluence with the North Main Street Terrace Ditch. The concentration of PAHs in surficial sediments (0 to 6 inches) in the Koppers drainage ditch (average: 49 mg/kg, max: 124 mg/kg; AMEC, 2009) are consistent with Koppers' historical operations being a significant source of PAHs to the creeks. Discharges from several long-standing and small commercial businesses (e.g., gas stations, body shops, welders, etc.), particularly along North Main Street Terrace Ditch, also flow into the creek.
- Non-point sources: Given the urban setting of the creeks, runoff from roads, parking lots, and driveways are also expected to contribute PAH-containing oils and petroleum residues to the

creeks. Furthermore, the high density residential areas along the creeks also contribute runoff that can contain various chemicals (ACEPD, 2007).

The urban setting of the creeks and industrial and commercial uses of the area are critical elements to consider in evaluating sediment quality in the creeks, particularly the presence of PAHs.

1.2 Summary of Previous Tar Removal Projects

In 2011, Cabot designed and implemented a tar removal plan that relied on the findings of the ACEPD studies and supplemental verification surveys. Cabot's remedial actions involved the removal of visible and accessible pine tar deposits that had been delineated by ACEPD, with post-remediation sampling results generally showing insignificant risks to human and ecological receptors, as described in further detail below (Gradient, 2011). Figures 2 to 5 present a comparison of the post-remediation sampling results to relevant ecological and human health benchmarks.

- The ACEPD study found tar-affected sediments at depths greater than 8 inches in depositional areas of the creeks, with clean sediments overlying the tar. Total concentrations of PAHs in the tar-affected sediments were generally on the order of 20 to 30 mg/kg, with two samples on the order of 100 mg/kg compared to the PAH Probable Effects Concentration (PEC) of 23 mg/kg, which is the ecological risk-based sediment cleanup goal specified in the 2011 ROD (US EPA, 2011). Furthermore, low levels of PAHs were detected in background sediment samples, thus confirming that PAHs have multiple natural and anthropogenic sources in this setting.
- Based on pre-remediation PAH concentrations in sediments, and the depth and inaccessibility of the tar, risks to human health and ecological risks were insignificant. Thus, Cabot's remedial efforts in the creeks were targeted towards addressing pine tar related impacts with the goal of mitigating potential future risks that could result from buried tar deposits being mobilized to the surface.
- In 2011, Cabot removed tar deposits that were identified by ACEPD in 2009/2010 to a "visibly clean" endpoint and backfilled excavated areas with clean material. This action was consistent with the subsequently issued 2011 ROD for the Site (US EPA, 2011). Tar could not be removed from a few locations due to property owners denying access, despite Cabot's best efforts, and/or other access constraints (e.g., concerns regarding bank slope stability). However, tar at these locations was present at depth and was inaccessible.
- Post-excavation monitoring showed that PAH concentrations were, on average (95% UCLM), less than or comparable to the total PAH PEC (23 mg/kg) (Figures 2 and 3), the sediment cleanup goal specified in the 2011 ROD (US EPA, 2011). At the few locations with exceedances, the PAH concentrations over time are expected to attenuate and attain background concentrations or comply with the PAH Threshold Effects Concentration (TEC) of 1.6 mg/kg.
- Furthermore, while not specified in the 2011 ROD, the post-tar removal BAP-TEQ levels on average (95% UCLM) were either below or comparable to the ACTL (0.5 to 1.0 mg/kg), the human health benchmark developed by the University of Florida (Figures 4 and 5).

The following sections of the work plan present the objectives, rationale, and approach for Cabot's proposed confirmation sediment sampling program.

2 Sediment Sampling Plan

2.1 Sampling Objectives

Cabot will conduct sediment sampling in the creeks to address the following objectives:

- Confirm that any residual tar deposits and related impacts in the creek sediments that could not be
 accessed in the prior removal action remain buried, inaccessible, and pose insignificant human
 and environmental risks; and
- Characterize PAH concentrations in creek sediments in a more comprehensive manner, *i.e.*, including upstream locations representative of background conditions and areas where tar was previously not found by ACEPD.

The following sub-sections discuss the proposed sampling approach.

2.2 Sediment Sampling

The proposed sampling will include a comprehensive visual survey and sediment chemical characterization program in North Main Street Terrace Ditch, Springstead Creek, and Hogtown Creek. The visual survey and sediment sampling will include: background locations, locations that were not previously remediated due to property access or bank stability constraints, locations that showed exceedances of relevant PAH benchmarks, and areas that were previously not sampled due to the absence of tar.

The proposed scope of work for the visual survey and sediment sampling event is described below and depicted in Figure 1.

- A visual survey with sediment probes will be conducted in transects perpendicular to the flow of the creeks with the goal of evaluating if any residual tar is present. The survey will be conducted specifically at the 6 creek locations where tar deposits could not be removed during Cabot's prior tar removal efforts due to property access and bank stability issues, as well as the 3 creek locations that showed post-remedy exceedances of PAH benchmarks. In addition, sediment probes will be advanced in transects every 500 feet along North Main Street Terrace Ditch, and Springstead and Hogtown Creek at a total of approximately 16 locations, as shown in Figure 1. If tar deposits are encountered, key characteristics of the deposits (e.g., dimensions, depth below creek bed, stability, and location) will be documented and photographs of the tar deposit(s) will be taken.
- Grab sediment samples will be generally collected every 500 feet at the same transects in the creeks that are visually surveyed (Figure 1), but sample locations may be adjusted based on field conditions. In particular, the field considerations that will affect the selection of sediment sample locations are discussed below:
 - Given that there are sections of the creek with exposed Hawthorn Clay deposits, an effort will be made to collect samples only from locations with sediment present.

- At each transect, a grab sediment sample will be collected from the inundated portion of the creek. However, if the visual survey indicates the presence of tar deposits in non-inundated areas or exposed sandbanks, an additional sample will be collected from this area.
- Sediment samples will be collected mainly from the top 6 inches of the sediment horizon, since potential human and ecological receptors would be exposed to shallow sediments. Sediment samples will only be collected in the 6 to 24 inch depth horizon at locations that show evidence of tar deposits at these depths during the visual survey. The deeper sediment samples will be collected with the objective of assessing the risk associated with tar deposits, if mobilized to the surface in the future. However, if tar deposits are not found at depth, sediment sampling is not needed since prior sampling results showed that PAH impacts were mainly associated with the presence of tar. Further, as discussed above, human and ecological exposure occurs primarily in the shallow sediment horizon, i.e., in the top 6 inches.
- Finally, in order to characterize background sediment quality, approximately 6 to 8 samples will be collected from sections of North Main Street Ditch and Springstead Creek that are upstream of the Site, and from the portion of Hogtown Creek that is upstream of the confluence of Springstead and Hogtown Creeks (see Figure 1). The sample locations in these areas will be selected based on field conditions and will generally target depositional areas.

The results of this proposed sampling program will provide a comprehensive understanding of sediment quality in Springstead and Hogtown creeks. It is envisioned that samples will be collected from approximately 30 locations, with multiple samples (shallow and deep) to be collected if tar is encountered. The sediment quality data will be used to evaluate the potential risks to human health and the environment, if any, associated with creek sediments.

2.3 Sediment Sampling Methodology

In general, the visual survey and sediment sampling event will be conducted in accordance with Section B.2.1.1 of the Site-specific QAPP. The specifics of the sampling methodology are described as follows:

- Sampling personnel will walk/wade through the creek to access the visual survey and sampling locations. The survey and sampling will start at the downstream end of the study area and proceed upstream.
- At each location, a metal-tipped fiberglass probe will be inserted into the creek bed at 3-4 evenly spaced locations perpendicular to the flow. Upon removal from the creek bed, the probe tip and rod will be inspected for the presence or absence of tar. If tar is observed on the probe, then additional probing of the creek bottom will continue until the lateral extent is determined.
- The survey locations will be surveyed with a portable GPS device and the coordinates will be recorded in the field notebook. A photograph will be taken of each probe location and, if applicable, pin flags will be used to designate the lateral extent of the deposit in the photograph. The results of each probing attempt into the creek bottom will be documented in the field notebook.
- The probing rod will be wiped clean with a paper towel soaked with isopropanol to remove any tar before use at the next location.
- The sediment sampling will commence following the completion of the visual survey. The sampling personnel will be positioned downstream of the selected sample location during sample collection and care will be taken to avoid disturbing the sediment in the sample location prior to sample collection.

- Sediment samples will be collected using a stainless steel or Teflon coated scoop or spoon from the targeted sediment depth horizon. Each sample will be screened with a flame ionizing detector (FID) and the resulting measurements will be recorded in the field logbook or on a sediment sampling form.
- A description of the sediment at the sample location, including color, texture, make-up, and any other field observations (*e.g.*, staining or odor) will also be recorded in the field notebook or on a sediment sampling form. A photograph will be taken of each sample location and sediment sample.
- The sample locations will be surveyed with a portable GPS device and the sample coordinates will be recorded in the field notebook or designated sampling form.
- The sample number, laboratory analytical method, and date of collection will be recorded on the laboratory chain of custody record. Samples will be labeled with the prefix SD- and a number indicating the sample location in order of collection. The sample label will use a suffix to indicate whether the sample was collected from the inundated (I) or exposed (E) portion of the creek, and another suffix to indicate if the sample was collected from the shallow (S) or deep (D) sampling regime, *e.g.*, SD-3IS or SD-2ED. Samples will be placed in a cooler with wet ice and shipped via overnight carrier to Test America's Savannah laboratory. All samples will be handled consistent with the procedures in Section B.3 of the Site-specific QAPP.

2.3.1 Sediment Sampling and Analysis Plan

All of the sediment samples will be submitted for laboratory analysis of PAHs using Method 8270D. In addition, 10 to 12 sediment samples will be analyzed for total organic carbon (TOC) using the Walkley-Black Method.

Quality control samples will be collected along with the proposed sediment samples. These samples will include duplicates, equipment rinsate blanks, field blanks, and travel blanks. One matrix spike per sample delivery group will be specified on the chain of custody. All field and analytical work will comply with the quality control guidelines in the Site-specific QAPP approved March 6, 2015 (Gradient, 2015). The supplemental quality assurance documents added in this work plan include the following:

- A list of materials and supplies for the Site updated to include requirements for sediment sampling, presented in Table 1;
- US EPA Region 4 and FDEP sediment sampling Standard Operating Procedures (SOPs) listed in Table 2 and provided in Appendix A; and
- An example sediment sampling form provided in Appendix B.

Sediment samples will be analyzed for PAHs and total organic carbon (in selected samples); samples will not be analyzed for dioxins. The reason is that the Cabot Carbon process was a pyrolytic process – not comparable to, for instance, a wood burning stove or fireplace. Therefore, operating conditions would not have been suitable for dioxin formation.

Specifically, the pine processing operations consisted of pyrolyzing pine tree stumps (*i.e.*, destructive distillation of wood in the <u>absence of oxygen</u>) at high temperatures (up to 400°C) in retorts built of reinforced concrete that were purged with steam to create an oxygen-starved environment. The primary products of this process were light and heavy pine oils and pine tar, and the residue from the combustion process was charcoal (Cabot Corporation; Hunter/ESE, 1989).

According to U.S. EPA, dioxins can be formed during combustion of organic materials, only if the following conditions are met: a) oxygen and chlorine are present; b) a transition metal catalyst, such as aluminum or copper chloride, is present¹; and c) temperatures are within the range of approximately 200 °C to 450 °C (US EPA, 2006).

None of these required conditions were present in the Cabot Carbon pine processing operation. The process was conducted in an oxygen-starved environment, the chlorine² content of the pine wood used was low, and a metal catalyst was not present because the retorts were concrete structures. Accordingly, the conditions in the former Cabot Carbon retorts were very unfavorable for dioxin production. This conclusion is supported by the results from the 2009 ACEPD sediment investigation, which examined the dioxin content of pine tar affected sediments. The study demonstrated a poor correlation between PAHs and dioxins, leading ACEPD to conclude that the dioxins detected "could be due to other sources" (ACEPD, 2009).

2.3.2 Sediment Quality Data Evaluation

The PAH benchmark that is selected to evaluate the sediment sampling results will depend on whether the sample was collected from the inundated portion of the creek or an exposed sandbank. The PEC will be applied to analytical results for samples collected in inundated and exposed areas in the creeks to assess potential ecological risks. The ACTL for BAP-TEQ (0.5 to 1.0 mg/kg) will be applied to any analytical results for samples collected in the non-inundated areas (*i.e.*, exposed creek bottom and sandbank) to assess potential human health risks, since the risk associated with human exposure to impacted sediments is likely to be more significant in the non-inundated portions of the creek, where impacted sediments may not be washed off during contact. Further, the background sampling results will be considered in the evaluation of the sediment quality data, particularly if background PAH concentrations are found to be higher than the relevant ecological and human health benchmarks.

¹ Reactive particulate matter, such as fly ash, also potentially could serve as a catalyst in the formation of dioxin; however, fly ash is not formed in a sealed and anaerobic environment, and would not have been present in the Cabot Carbon retorts.

² A literature review indicates that the chlorine content of pine wood and related pyrolysis products is 0.1% (wt/wt) or lower (Oasmaa *et al.*, 2010; He *et al.*, 2012), which is at least an order of magnitude lower than the chlorine-in-fuel threshold associated with increased levels of dioxin production (US EPA, 2005).

3 Schedule

This field program will be initiated within 8 weeks of receiving US EPA approval of the work plan and is anticipated to take 1 to 2 weeks to complete. Analytical data packages received from the laboratory for this program will be validated in accordance with the Site-specific QAPP. The results of this investigation will be presented in a report, which will be submitted to US EPA approximately 8 weeks after the complete and validated data package becomes available.

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Tables

Table 1 Instruments and Equipment Needed to Conduct Ongoing Work at the Site

Instrument/Equipment Description	Function	Purpose for This Site	Models/Types Typically Used at the Site
Soil probes		Determining lateral and vertical extent of tar deposits that may remain in sediment.	Fiberglass shaft with metal tip (Corona,
·	Probing the top 0-40 inches of sediment	·	NUPULA or equivalent)
Digital camera	Taking and saving digital photographs.	Photographing collected sediment samples.	Nikon COOLPIX L120, I-Phone, Samsung
Digital carriera	raming and saving digital priotographs.	Thotographing concerca scanners sumples.	Galaxy or equivalent with minimum 5
			Megapixel resolution
Global Positioning System (GPS)	Determining point locations on a global reference	Documenting sediment sampling locations.	Garmin Handheld (Oregon 450 T or
8 2 7 2 2 7	system.	the standard production of the standard producti	Equivalent)
Waders	·	Keeping personnel dry when standing in the stream bed to collect sediment samples.	Rubber of Neoprene Chest Waders (Frogg
	Keeping personnel dry when operating in aquatic	neeping personner ary microstrating in the stream searce contest seament samples.	Togg Cascade, Amphib Max or equivalent)
	systems.		1088 Gassauc, 1 ampinio marcon equitations,
Mixing bowls	Mixing materials	Mixing composite sediment samples.	Glass/stainless steel (Pyrex, Crestware or
	•		equivalent) 1-2 quart mixing bowl
Water Quality Meter	Measures water quality parameters (e.g., pH,	Measuring water quality parameters during water sampling and well development activities. See EPA Region 4	YSI 6920
	temperature, specific conductivity, ORP, turbidity,	SOPs SESDPROC-100-R3, SESDPROC-102-R4, SESDPROC-101-R5, SESDPROC-113-R1, SESDPROC-103-R3, and	YSI 556, with the YSI 5083 flow-through cel
	dissolved oxygen).	SESDPROC-106-R3.	
Turbidity Meter	Measures turbidity in liquids.	Measure turbidity in water samples. See EPA Region 4 SOP SESDPROC-103-R3.	LaMott 2020
Photo Ionizing Detector (PID)	Detects presence of VOCs.	Detects presence of VOCs in soil samples, well headspace, and air monitoring for health and safety.	Mini-RAE 3000
Flame Ionizing Detector (FID)	Detects presence of VOCs.	Detects presence of VOCs in soil samples, well headspace, and air monitoring for health and safety.	PhotoVac Microfid
Water Level Indicator	Measures depth to water.	Determine depth to water in wells, sumps, and piezometers. See EPA Region 4 SOP SESDPROC-105-R2.	Solonist Model 101 P2
Peristaltic Pump	Pumps liquids.	Well development. Purging and water sampling. See EPA Region 4 SOP SESDPROC-203-R3.	Geotech Geopump 2
Submersible Pumps and Control	Pumps liquids.	Well development. Purging and water sampling. Liquid transfer from tanks. See EPA Region 4 SOP SESDPROC-	Geotech SS GeoSub
Boxes		203-R3.	Grundfos Redi Flow II
			Proactive SS Monsoon Pump
			Little Giant
Sonic Drill Rig	Drills borings/wells. Advances casings.	Install wells and soil borings. Over-drill casings to abandon wells.	Geoprobe 8140 Mini Sonic
Rotary Drill Rig	Drills borings/wells.	Install wells/borings/casings/piezometers.	BK-81
			Barber Rotary Rig
Direct Push Drill Rig	Drills borings/wells.	Install borings/wells/piezometers.	Geoprobe 6620
In Situ Water Samplers	Collects water inside wells.	Collect downhole water samples.	Pro Hydro SNAP Sampler
Bailers	Collect liquids from wells.	Develop low producing wells and collect product from wells.	Teflon
Steam Cleaner		Equipment decontamination (e.g., drill rig).	Various
Skid Steer	Lifting/grading/hauling.	Haul materials and supplies. Create access paths. Restore site conditions.	Various
Excavator	Digging.	Site Clearing, test pits.	Various
Loader		Loading bulk soils, site preparation, restoration. Site grading.	Various
Vac Truck	Collects liquids and sludges.	Collect IDW for off-site disposal or transportation to lift station.	Various As Supplied By Contractor
Hand Tools	2	Repair, maintenance, assembly.	Various
Water Storage Tanks	Contain liquids.	Store purge/development water/drilling fluids/aquifer testing water.	Portable Poly-Tanks
T 1 (C) D (Steel Frac Tanks
Trucks/SUVs	Callest resourced arraying of a list floored.	Transport personnel.	Various
Syringes	Collects solids	Soil and sediment sampling for specific VOC methods. See EPA Region 4 SOP SESDPROC-300-R3.	Plastic
Scoops Packet Panatrameter	Collects solids.	Soil/sediment sampling. See EPA Region 4 SOP SESDPROC-300-R3.	ANAC
Pocket Penetrometer	Measures soil compaction rates. Provides soil colors for comparison with accompanying	Determine soil consolidation in soil cores. Helps determine soil colors in a consistent repeatable method.	AMS
Soil Color Chart		neips determine son colors in a consistent repeatable method.	Munsell
Generator	numbers. Generate portable electricity.	Electricity for pumps, office trailer, power tools, portable lighting.	Various
Welder	Metal fabrication.	Weld casing segments together for steel isolation casings.	Various
VVCIGCI			
Cutting Torch	Cutting and working metals	Cutting steel casings nines	\/arious
Cutting Torch Office Trailer	Cutting and working metals. Mobile office.	Cutting steel casings pipes. On-site office. Store equipment and supplies during field operations.	Various William Scotsman

Notes:

IDW = Investigation-Derived Waste; ORP = Oxidation Reduction Potential; VOC = Volatile Organic Compound.

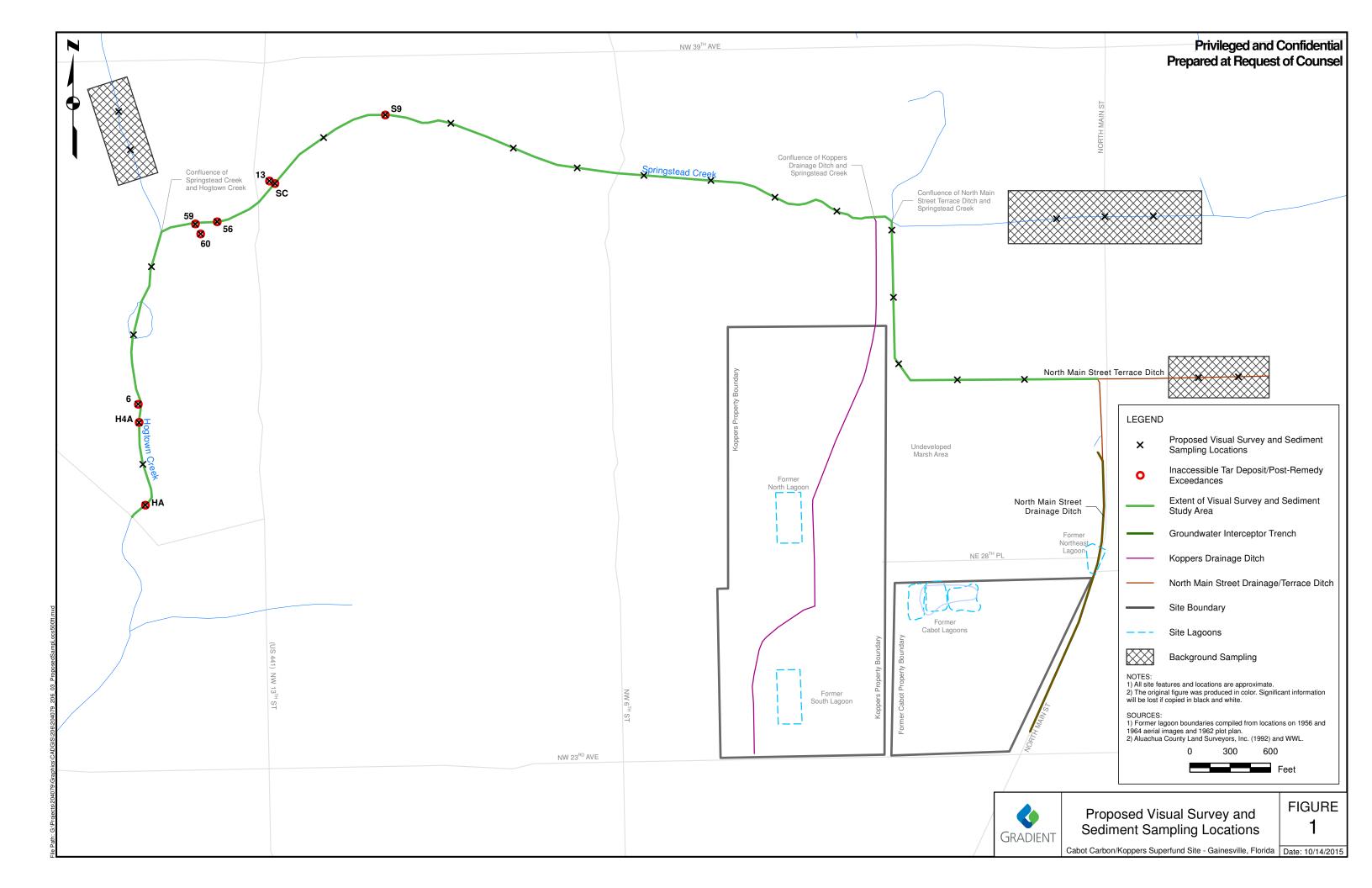
Table 2 Sediment Sampling SOP References

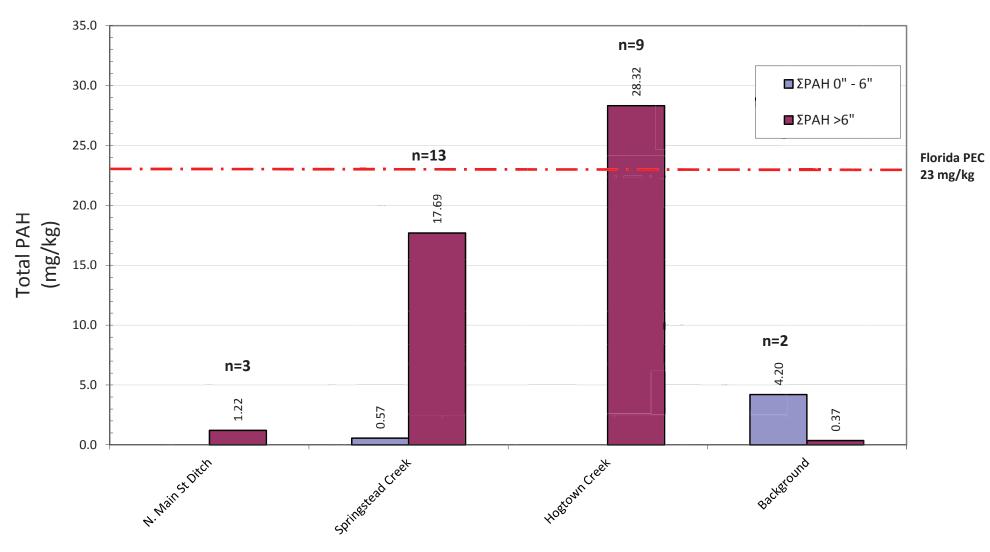
	Sampling Type	SOP Title and Reference Number	Source of SOP	Equipment Type	Modified for Project (Yes/No)
Sodi	Sediment Sampling	Sediment Sampling - FS4000	FDEP (2014)	Scoops, Spoons	No
	Sediment Sampling	Sediment Sampling - SESDPROC-200-R3	EPA Region IV SESD	Scoops, Spoons	

Notes:

SOP = Standard Operating Procedures; FDEP = Florida Department of Environmental Protection; SESD = Science and Ecosystem Support Division.

Figures





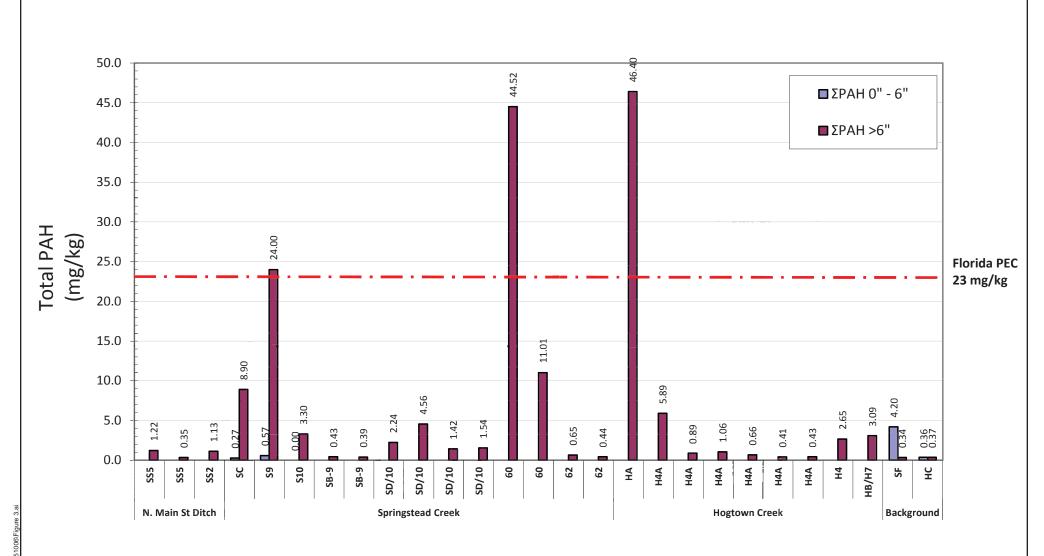
- 1) PECs are not applicable to deep sediments.
- 2) 95% UCLM calculated using ProUCL 4.1 for Springstead Creek and Hogtown Creek deep samples (> 6").
- 3) For shallow samples (0"-6"), N Main St. Ditch and Background samples, maximum values assumed due to limited data set.



Post-Excavation Sediment Data: Total PAH v. Florida PEC 95% UCLM FIGURE 2

Cabot Carbon/Koppers Superfund Site - Gainesville, Florida

File Path: \\camfs\G_Drive\Projects\204079\Graphics\N

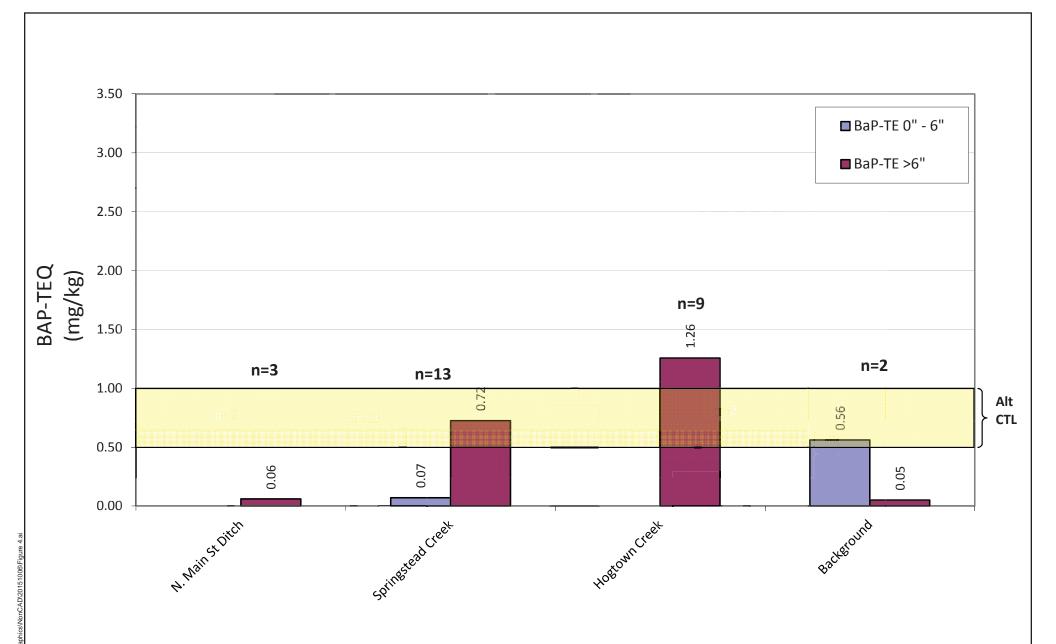


1) PECs are not applicable to deep sediments.



Post-Excavation Sediment Data Total PAH vs. Florida PEC All Data FIGURE 3

Cabot Carbon/Koppers Superfund Site - Gainesville, Florida

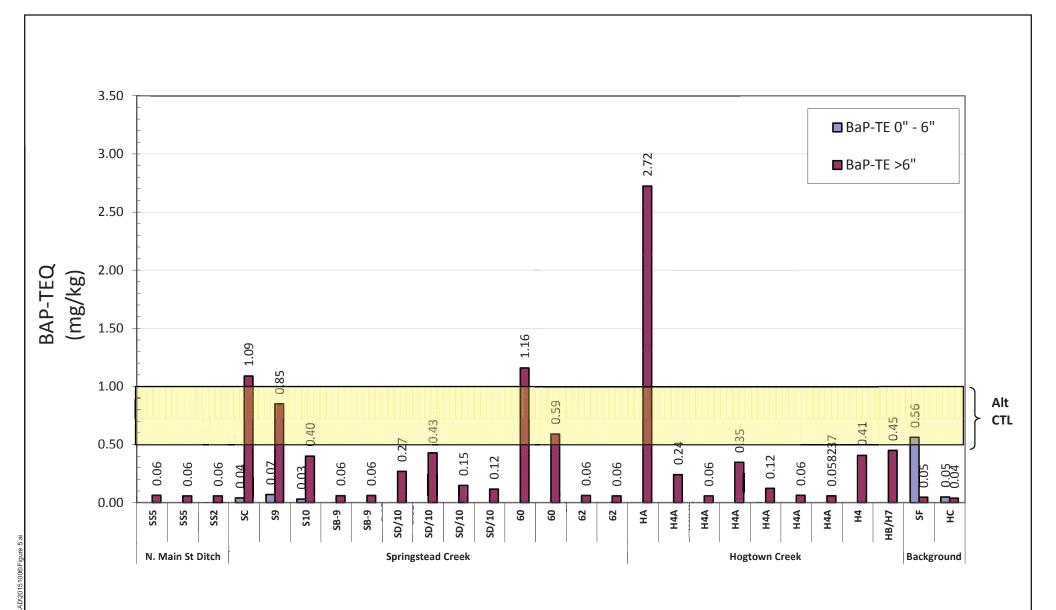


- 1) "Alt CTL": Alternate Cleanup Target Levels for BAP-TEQ were developed by the University of Florida (Roberts and Stuchal, 2010a,b).
- 2) 95% UCLM calculated using ProUCL 4.1 for Springstead Creek and Hogtown Creek deep samples (> 6").
- 3) For shallow samples (0"-6"), N Main St. Ditch and Background samples, maximum values assumed due to limited data set.



Post-Excavation Sediment Data: BAP-TEQ vs. ACTLs 95% UCLM FIGURE 4

Cabot Carbon/Koppers Superfund Site - Gainesville, Florida



"Alt CTL": Alternate Cleanup Target Levels for BAP-TEQ were developed by the University of Florida (Roberts and Stuchal, 2010a,b).



Post-Excavation Sediment Data: BAP-TEQ vs. ACTLs All Data FIGURE 5

Cabot Carbon/Koppers Superfund Site - Gainesville, Florida

Appendix A – Sediment Sampling Standard Operating Procedures

FS 4000. SEDIMENT SAMPLING

See also the following Standard Operating Procedures:

- FA 1000 Administrative
- FC 1000 Field Cleaning
- FD 1000 Documentation
- FM 1000 Field Mobilization
- FQ 1000 Quality Control
- FS 1000 General Sampling
- FS 7400 Benthic Macroinvertebate Sampling
- FT 1000 2000 Field Testing
- 1. Introduction and Scope

Sediments occur in freshwater and marine environments such as streams/rivers, ponds/lakes, canals, ditches, wetlands, lagoons, and estuaries. Recently exposed sediment (due to low water levels) may also be sampled, but exposure to air could affect the characteristics of the sediment (for example, redox) and hence, the interpretation of the results of chemical or other analyses. The following methods are for physical, chemical, and toxicological sampling. See FS 7400 for benthic invertebrate sampling.

- 1.1. Select sampling locations for sediments depending upon the project objectives. Collect:
 - 1.1.1. Sediment samples as an adjunct to surface water samples;
 - 1.1.2. A series of sediment samples for compositing to determine water or sediment quality in a system;
 - 1.1.3. Sediment samples above and below an outfall to document degradation due to a point source discharge; or
 - 1.1.4. Sediment samples if stressed shore vegetation or visible surface water contamination is evident.
- 1.2. Decisions related to the selection of sampling locations will not be discussed in this document.
- 1.3. Collect, preserve and containerize surface water samples prior to collecting sediment samples (see FS 2100).
- 2. EQUIPMENT AND SUPPLIES
 - 2.1. Refer to Table FS 4000-1 and FS 1000, Tables FS 1000-1, 1000-2 and 1000-3, for selection of sampling equipment and construction.
 - 2.2. For information on the selection of appropriate sample containers, see FS 1000 Table FS 1000-6.
 - 2.3. For information on cleaning requirements for sample containers, equipment and utensils, see FC 1000.
 - 2.4. For information on documentation requirements, see FD 1000.
 - 2.5. For information on preservation and holding time requirements, see FS 1000 Table FS 1000-6.
- 3. SAMPLE COLLECTION PROTOCOLS

Take sediment samples using one of three different types of equipment: scoops, corers and dredges or grab samplers.

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- 3.1. Soil sampling equipment is generally not applicable to sediments because of the low cohesion of the medium.
- 3.2. When selecting the appropriate sampling equipment, consider sampling location (edge or middle of lagoon), depth of water and sediment, sediment grain size (fineness), water velocity and analytes of interest.
- 3.3. Direct collection with the appropriate sample container may be appropriate in very low water or where sediment is exposed.
- 3.4. Use dredges for hard or rocky substrates. They are heavy enough to use in high velocity streams.
- 3.5. Use coring devices in quiescent waters, unless water depth precludes effective sample collection.

3.6. Scoops or Similar Equipment

- 3.6.1. Scooping is generally most useful around the margin or shore of the water body or by wading in shallow waters.
 - 3.6.1.1. Stand facing the direction of flow and approach the location from the downstream direction.
 - 3.6.1.2. Take precautions not to disturb the bottom prior to scooping.
 - 3.6.1.3. Scoop the sample in the upstream direction of flow.
- 3.6.2. For obtaining samples several feet from shore or from a boat, DEP recommends attaching the scoop to an extendible pole.
- 3.6.3. Transfer sample to the appropriate sample container(s), using a clean non-reactive utensil.
- 3.6.4. Label, preserve to 4°C with wet ice and complete field notes.

3.7. Corers

- 3.7.1. Coring devices can be easily fabricated from many materials. Although stainless steel, glass or Teflon must be used for sampling extractable organics, volatile organics and inorganics, aggregate organics, petroleum hydrocarbons and oil & grease, other inexpensive material (e.g., PVC, carbon steel, etc.) may be used for inorganic non-metallics and metals.
- 3.7.2. Some corers are simple "push tubes," whereas other more sophisticated models may be finned, gravity driven devices.
- 3.7.3. A core may be useful for preserving the historical layering of sediments.
- 3.7.4. Upon descent, water displacement is minimal with core samplers, which minimizes the shock wave produced by other equipment such as dredges.
- 3.7.5. The corer is an acceptable choice for sampling fine sediments in static waters, especially those containing trace organics and metals.
- 3.7.6. Corer diameter, grain size and sample consistency will determine if the sample will remain in the corer upon withdrawal.
- 3.7.7. Sample washout can be a problem and there are several ways to reduce or prevent it.
 - 3.7.7.1. Fit the leading edge of the corer with a nosepiece or core catcher that physically keeps the sample from slipping back out of the corer. The core catcher material must also be compatible with the analytes of interest.
 - 3.7.7.2. A second option is fit the top or back end with a check valve which creates negative pressure on the back of the sample as it is being pulled from the substrate and prevents surface water from washing out the top portion of the sample.

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- 3.7.8. Rotate the corer, if needed, as it is pushed into the sediment.
 - 3.7.8.1. Rotate be around its axis (do not rock the coring device back and forth).
 - 3.7.8.2. Rotation improves penetration and prevents compaction of the sample as it is pushed to the full length of the corer.
- 3.7.9. Upon withdrawal from the water surface, place a cap on the bottom to prevent the sample from sliding out.
- 3.7.10. Corers can also be fitted with liners. This is advantageous if a complete core is desired that has not been in contact with the atmosphere. It is also advantageous if the coring device is not constructed of the proper material (e.g., PVC) and one of the analytes requires a sampler of inert construction (glass, stainless steel or Teflon).
- 3.7.11. As the core is extruded, carefully remove the sample with a clean, non-reactive utensil and transfer into the appropriate sample container(s).
- 3.7.12. Label, preserve to 4°C with wet ice and complete field notes.

3.8. Dredges or Grab Samplers

- 3.8.1. The three main types of devices used in freshwater are the Ekman, Peterson and Ponar. Heavier oceanographic dredges are used in marine and estuarine waters.
- 3.8.2. Refer to Table FS 4000-1 for additional types of dredges. The Peterson and Ponar dredges are suitable for hard or rocky substrates or deep water bodies.
 - 3.8.2.1. The Peterson and Ponar are virtually the same, except that the Ponar has been adapted with a top screen and side plates to prevent sample loss upon ascent. For this reason, the Ponar is the dredge of choice for rocky substrates. These dredges are heavy enough to use in streams with fast currents.
 - 3.8.2.2. Open the jaws and place the cross bar into the proper notch.
 - 3.8.2.3. Lower the dredge to the bottom, making sure it settles flat.
 - 3.8.2.4. When tension is removed from the line, the cross bar will drop, enabling the dredge to close as the line is pulled upward during retrieval.
 - 3.8.2.5. Pull the sampler to the surface. Check to make sure the jaws are fully closed and that no sample was lost while lifting the dredge.
 - 3.8.2.6. Carefully open the jaws, remove the sample with a clean, non-reactive utensil and transfer the sample into the appropriate sample container(s), label, preserve to 4°C with wet ice and complete field notes.
- 3.8.3. The Ekman is designed for sampling soft substrates (e.g., sand, silt or mud) in areas with little current.
 - 3.8.3.1. Open the spring-loaded jaws and attach the chains to the pegs at the top of the sampler.
 - 3.8.3.2. Lower the dredge to the bottom, making sure it settles flat.
 - 3.8.3.3. Holding the line taut and send down the messenger to close the jaws of the dredge.
 - 3.8.3.4. Pull the sampler to the surface. Check to make sure the jaws are fully closed and that no sample was lost while lifting the dredge.
 - 3.8.3.5. Carefully open the jaws, remove the sample with a clean, non-reactive utensil and transfer the sample into the appropriate sample container(s).
 - 3.8.3.6. Label, preserve to 4°C with wet ice and complete field notes.

4. PROCEDURES FOR COMPOSITING

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- 4.1. The following is not a complete discussion regarding all available sampling protocols nor the appropriateness or inappropriateness of compositing sediment samples. The appropriateness of compositing sediment samples will depend on the data quality objectives of the project. However, it is sometimes advantageous to composite sediment samples to minimize the number of samples to be analyzed when sampling highly contaminated areas. Obtain permission from the DEP program.
 - 4.1.1. Select sampling points from which to collect each aliquot.
 - 4.1.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.
 - 4.1.3. Combine the aliquots of the sample directly in the sample container with no pre-mixing.
 - 4.1.4. Record the amount of each aliquot (volume or weight).
 - 4.1.5. Label container, preserve on wet ice to 4°C and complete field notes.
 - 4.1.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.
- 5. COLLECTION OF INTERSTITIAL OR PORE WATER SAMPLES
 - 5.1. ASTM identifies interstitial water or pore water as the "water occupying the space between sediment...particles." It "is often isolated to provide either a matrix for toxicity testing or an indication of the concentration and partitioning of contaminants with a sediment matrix." See *Standard Guide for Collection, Storage, Characterization and Manipulation of Sediments for Toxicological Testing and for Selection of Samplers Used to Collect Benthic Invertebrates*, ASTM E 1391-03(2008) ASTM International. Collect pore water using available technology that will preserve the integrity of the analytes of interest during collection. Pore water may be extracted in the laboratory from field-collected sediments. Consult the detailed discussion in the ASTM guidance if pore water is to be extracted and analyzed as part of the sampling design. Use of pore water wells (e.g., shallow PVC wells) or pore water equilibrators (e.g., Plexiglas plates with built-in wells or other appropriate construction) is also acceptable.
 - 5.2. Collect adequate amounts of sample in the field to obtain desired quantities of pore water for testing. Sandy sediments retain less water than fine sediments do; thus, the substrate type will dictate the amount of additional sample needed. In all cases, consult the laboratory conducting the analyses to provide estimates of the amount of sediment necessary to obtain the desired quantity of pore water.

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Appendix FS 4000 Tables, Figures and Forms

Table FS 4000-1: Summary of Bottom Sampling Equipment [from ASTM E 1391-94]

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TABLE FS 4000-1 Summary of Bottom Sampling Equipment [from ASTM E 1391-94]

Device	Use	Advantages	Disadvantages
Teflon or glass tube	Shallow wadeable waters or deep waters if SCUBA available. Soft or semi-consolidated deposits.	Preserves layering and permits historical study of sediment deposition. Rapid - samples immediately ready for laboratory shipment. Minimal risk of contamination.	Small sample size requires repetitive sampling.
Hand corer with removable teflon or glass liners	Same as above except more consolidated sediments can be obtained.	Handles provide for greater ease of substrate penetration. Above advantages.	Careful handling necessary to prevent spillage. Requires removal of liners before repetitive sampling. Slight risk of metal contamination from barrel and core cutter.
Box corer	Same as above.	Collection of large sample undisturbed, allowing for subsampling.	Hard to handle.
Gravity corers, such as Phleger Corer	Deep lakes and rivers. Semiconsolidated sediments.	Low risk of sample contamination. Maintains sediment integrity relatively well.	Careful handling necessary to avoid sediment spillage. Small sample, require repetitive operation and removal of liners. Time consuming.
Young grab (Teflon or kynar-lined, modified 0.1- m ² Van Veen)	Lakes and marine areas.	Eliminates metal contamination. Reduced bow wake.	Expensive. Requires winch.
Ekman or box dredge	Soft to semi-soft sediments. Can be used from boat, bridge, or pier in waters of various depths.	Obtains a larger sample than coring tubes. Can be subsampled through box lid.	Possible incomplete jaw closure and sample loss. Possible shock wave, which may disturb the "fines". Metal construction may introduce contaminants. Possible loss of "fines" on retrieval.
PONAR grab sampler Petite PONAR grab sampler	Deep lakes, rivers and estuaries. Useful on sand, silt, or clay.	Most universal grab sampler. Adequate on most substrates. Large sample obtained intact, permitting subsampling.	Shock wave from descent may disturb "fines." Possible incomplete closure of jaws results in sample loss. Possible contamination from metal frame construction. Sample must be further prepared for analysis.
BMH-53 piston corer	Waters of 4 to 6 ft deep when used with extension rod. Soft to semiconsolidated deposits.	Piston provides for greater sample retention.	Cores must be extruded on-site to other containers. Metal barrels introduce risk of metal contamination.
Van Veen dredge	Deep lakes, rivers and estuaries. Useful on sand, silt, or clay.	Adequate on most substrates. Large sample obtained intact, permitting subsampling.	Shock wave from descent may disturb "fines." Possible incomplete closure of jaws results in sample loss. Possible contamination from metal frame construction. Sample must be further prepared for analysis.
BMH-60 grab sampler	Sampling moving waters from a fixed platform.	Streamlined configuration allows sampling where other devices could not achieve proper orientation.	Possible contamination from metal construction. Subsampling difficult. Not effective for sampling fine sediments.
Petersen grab sampler	Deep lakes, rivers and estuaries. Useful on most substrates.	Large sample; can penetrate most substrates.	Heavy. May require winch. No cover lid to permit subsampling. All other disadvantages of Ekman and Ponar.
Shipek grab sampler	Used primarily in marine waters and large inland lakes and reservoirs.	Sample bucket may be opened to permit subsampling. Retains fine- grained sediments effectively.	Possible contamination from metal construction. Heavy. May require winch.
Orange-Peel grab Smith- McIntyre grab	Deep lakes, rivers and estuaries. Useful on most substrates.	Designed for sampling hard substrates.	Loss of fines. Heavy. May require winch. Possible metal contamination.
Scoops Drag Buckets	Various environments, depending on depth and substrate.	Inexpensive, easy to handle.	Loss of fines on retrieval through water column.

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Region 4 U.S. Environmental Protection Agency Science and Ecosystem Support Division Athens, Georgia

OPER ATING PROCEDURE

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Title: Sediment Sampling	
Effective Date: August 21, 2014	Number: SESDPROC-200-R3
A	uthors
Name: Kevin Simmons Title: Environmental Scientist, Regional E	ALL THE STREET STREET STREET
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Signaturally & for Pill 1 "	Date: 8/2.0/14

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

Hi	story		Effective Date
SESDPROC-200-R3, Sed SESDPROC-200-R2.	iment Sampling,	replaces	August 21, 2014
General: Corrected any typograph errors. Throughout the document m quality system was replaced with F FBQS.			
Cover Page: Changed the Enforce from Archie Lee to Acting Chief, Jo Assessment Branch Chief from B Ackerman. Changed the FQM from	ne Ecological Chief, Laura		
Revision History: Changes were nonly including the most recent changes			
Throughout the document: an Moisture" was changed to "Po			
SESDPROC-200-R2, Sed SESDPROC-200-R1.	iment Sampling,	replaces	September 8, 2010
SESDPROC-200-R1, Sed SESDPROC-200-R0.	iment Sampling,	replaces	November 1, 2007
SESDPROC-200-R0, Sedime	nt Sampling, Origina	Issue	February 05, 2007

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Contents

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting sediment samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when collecting and handling sediment samples in the field. On the occasion that SESD field investigators determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a sediment sample, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

SESD Operating Procedure for Control of Records, SESDPROC-004, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

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SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-104, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

SESD Operating Procedure for Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

United States Office of Occupational Health and Safety (US OSHA). 1981. Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples (PM-273), Memo from David Weitzman, Work Group Chairman, US EPA. April 13, 1981.

Effective Date: August 21, 2014

1.5 General Precautions

1.5.1 *Safety*

Proper safety precautions must be observed when collecting sediment samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting sediment samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and stored in a secure place.

2 Special Sampling Considerations

2.1 Sediment Samples for Volatile Organic Compounds Analysis

If samples are to be analyzed for volatile organic compounds (VOCs), they should be collected in a manner that minimizes disturbance of the sample. The sample for VOC analysis should be collected directly from the sample device, if possible, before it is emptied into the pan. It may not be possible to do this with certain types of sediment sampling equipment, such as the Ponar dredge. In cases such as these, the VOC aliquots should be collected from the dredge contents immediately after they have been deposited in the pan and prior to any mixing. The sample shall be placed in the appropriate container (En Core® Sampler or other Method 5035 compatible container) with no headspace. *Samples for VOC analysis are not homogenized*. Preservatives may be required for some samples with certain variations of Method 5035. Consult the method description below in Section 2.2, Sediment Sampling (Method 5035) or the principal analytical chemist to determine if preservatives are necessary.

In some cases, the sediment may be soft and not lend itself to collection by plunging En Core® Samplers or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sample device, i.e., the En Core® Sampler barrel or syringe, prior to sample collection, and to carefully place the sediment in the device, filling it fully with the required volume of sample.

2.2 Sediment Sampling (Method 5035)

The following sampling protocol is recommended for site investigators assessing the extent of VOCs in sediments at a project site. Because of the large number of options available, careful coordination between field and laboratory personnel is needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method best applicable to the investigation can be made.

2.2.1 Equipment

Sediment for VOC analyses may be retrieved using any of the SESD sediment sampling methods described in Sections 3 through 6 of this procedure. Once the sediment has been obtained, the En Core® Sampler, syringes, stainless steel spatula, standard 2-oz. sediment VOC container, or pre-prepared 40 ml vials may be used/required for sub-sampling. The specific sample containers and the sampling tools required will depend upon the data quality objectives established for

the site or sampling investigation. The various sub-sampling methods are described below.

2.2.2 Sampling Methodology - Low Concentrations

When the total VOC concentration in the sediment is expected to be less than 200 μ g/kg, the samples may be collected directly with the En Core® Sampler or syringe. If using the syringes, the sample must be placed in the sample container (40 ml preprepared vial) immediately to reduce volatilization losses. The 40 ml vials should contain 10 ml of organic-free water for an un-preserved sample or approximately 10 ml of organic-free water and a preservative. It is recommended that the 40 ml vials be prepared and weighed by the laboratory (commercial sources are available which supply preserved and tared vials). When sampling directly with the En Core® Sampler, the vial must be immediately capped and locked.

A sediment sample for VOC analysis may also be collected with conventional sampling equipment. A sample collected in this fashion must either be placed in the final sample container (En Core® Sampler or 40 ml pre-prepared vial) immediately or the sample may be immediately placed into an intermediate sample container with no head space. If an intermediate container (usually 2-oz. sediment jar) is used, the sample must be transferred to the final sample container (En Core® Sampler or 40 ml pre-prepared vial) as soon as possible, not to exceed 30 minutes.

NOTE: After collection of the sample into either the En Core® Sampler or other container, the sample must immediately be stored in an ice chest and cooled.

Sediment samples may be prepared for shipping and analysis as follows:

En Core® Sampler - the sample shall be capped, locked, and secured in a plastic bag.

Syringe - Add about 3.7 cc (approximately 5 grams) of sample material to 40-ml pre-prepared containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag. Note: When using the syringes, it is important that no air is allowed to become trapped behind the sample prior to extrusion, as this will adversely affect the sample.

Stainless Steel Laboratory Spatulas - Add between 4.5 and 5.5 grams (approximate) of sample material to 40 ml containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag.

2.2.3 Sampling Methodology - High Concentrations

Based upon the data quality objectives and the detection level requirements, this high level method may also be used. Specifically, the sample may be packed into a single 2-oz. glass container with a screw cap and septum seal. The sample container must be filled quickly and completely to eliminate head space. Sediments containing high total VOC concentrations may also be collected as described in Section 2.2.2, Sampling Methodology - Low Concentrations, and preserved using 10 ml methanol.

2.2.4 Special Techniques and Considerations for Method 5035

Effervescence

If low concentration samples effervesce from contact with the acid preservative, then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or unpreserved as needed, or all samples must be collected unpreserved.

To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence (rapid formation of bubbles) then preservation by acidification is not acceptable, and the sample must be collected un-preserved.

If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with organic-free water. An appropriate amount of organic-free water, equal to the amount of preservative solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured.

Sample Size

While this method is an improvement over earlier ones, field investigators must be aware of an inherent limitation. Because of the extremely small sample size, sample representativeness for VOCs may be reduced compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Holding Times

Sample holding times are specified in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version. Field investigators should note that the holding time for an un-preserved VOC sediment sample is 48 hours. Arrangements should be made to ship the sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may preserve and/or analyze the sample within 48 hours of collection.

Percent Solids

Samplers must ensure that the laboratory has sufficient material to determine percent solids in the VOC sediment sample to correct the analytical results to dry weight. If other analyses requiring percent solids determination are being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 oz.) for percent solids determination will be required.

Safety

Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all required safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Use protective gloves when handling the methanol vials. Store methanol away from sources of ignition such as extreme heat or open flames. The vials of methanol should be stored in a cooler with ice at all times.

Shipping

Methanol and sodium bisulfate are considered dangerous goods, therefore shipment of samples preserved with these materials by common carrier is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulations must be followed when shipping methanol and sodium bisulfate. Consult the above documents or the carrier for additional information. Shipment of the quantities of methanol and sodium bisulfate used for sample preservation falls under the exemption for small quantities. A summary of the requirements for shipping samples follows. Refer to the code for a complete review of the requirements.

1. The maximum volume of methanol or sodium bisulfate in a sample container is

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limited to thirty (30) ml.

- 2. The sample container must not be full of methanol.
- 3. The sample container must be stored upright and have the lid held securely in place. Note that the mechanism used to hold the cap in place must be able to be completely removed so weight is not added to the sample container, as specified in Method 5035.
- 4. Sample containers must be packed in an absorbent material capable of absorbing spills from leaks or breakage of the sample containers.
- 5. The maximum sample shuttle weight must not exceed 64 pounds.
- 6 The maximum volume of methanol or sodium bisulfate per shipping container is 500 ml.
- 7 The shipper must mark the sample shuttle in accordance with shipping dangerous goods in acceptable quantities.
- 8. The package must not be opened or altered until no longer in commerce.

The following summary table lists the options available for compliance with SW846 Method 5035. The advantages and disadvantages are noted for each option. SESD's goal is to minimize the use of hazardous material (methanol and sodium bisulfate) and minimize the generation of hazardous waste during sample collection.

Table 1: Method 5035 Summary

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES
1	Collect 2 – 40 ml vials with ~5 grams of sample and 1 – 2 oz. glass w/septum lid for screening and % solids	Screening conducted by lab	Presently a 48 hour holding time for unpreserved samples
2	Collect 3 EnCore® Samplers and 1 – 2oz. glass w/septum lid for screening and % solids	Lab conducts all preservation/preparation procedures	Presently a 48 hour holding time for preparation of samples
3	Collect 2 – 40 ml vials with 5 grams of sample and preserve w/methanol or sodium bisulfate, and 1 – 2 oz. glass w/septum lid for screening and % solids	High level VOC samples may be composited Longer holding time	Hazardous materials used in field
4	Collect 1 – 2 oz. glass w/septum lid for analysis and % solids	Lab conducts all preservation/preparation procedures	May have significant VOC loss

2.3 **Special Precautions for Trace Contaminant Sediment Sampling**

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers with samples suspected of containing high concentrations of contaminants shall be stored separately. All background samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area if sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.

Page 12 of 23 SESDPROC-200-R3 Samplers must use new, verified and certified-clean disposable or nondisposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, or SESD Operating Procedure for Field Cleaning and Decontamination at the FEC, SESDPROC-206, for collection of samples for trace metals or organic compound analyses.

2.4 Sample Homogenization

- 1. If sub-sampling of the primary sample is to be performed in the laboratory, transfer the entire primary sample directly into an appropriate, labeled sample container(s). Proceed to step 5
- 2. If sub-sampling the primary sample in the field or compositing multiple primary samples in the field, place the sample into a glass or stainless steel homogenization container and mix thoroughly. Each aliquot of a composite sample should be of the same volume.
- 3. All sediment samples must be thoroughly mixed to ensure that the sample is as representative as possible of the sample media. *Samples for VOC analysis are not homogenized.* The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:
 - The material in the sample pan should be divided into quarters and each quarter should be mixed individually.
 - Two quarters should then be mixed to form halves.
 - The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

- 4. Place the sample into an appropriate, labeled container(s) using the alternate shoveling method and secure the cap(s) tightly. Threads on the container and lid should be cleaned to ensure a tight seal when closed.
- 5. Return any unused sample material back to the location from which the sample was collected.

2.5 Quality Control

If possible, a control sample should be collected from an area not affected by the possible contaminants of concern and submitted with the other samples. The control sample should be collected at an upstream location in the same stream or conveyance from which the primary samples area collected. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by sampling tools.

2.6 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-004. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Procedure for Sample and Evidence Management, SESDPROC-005.

3 General Considerations

3.1 General

The sediment sampling techniques and equipment described in the following Sections 4, 5 and 6 of this procedure document are designed to minimize effects on the chemical and physical integrity of the sample. If the procedures in this section are followed, a representative sample of the sediment should be obtained.

3.2 Equipment Selection Considerations

The physical location of the investigator when collecting a sample may dictate the equipment to be used. Wading is the preferred method for reaching the sampling location, particularly if the stream has a noticeable current (is not impounded). However, wading may disrupt bottom sediments causing biased results; therefore, the samples should be collected facing upstream. If the stream is too deep to wade, the sediment sample may be collected from a platform such as a boat or a bridge.

To collect a sediment sample from a water body or other surface water conveyance, a variety of methods can be used:

- Scoops and spoons
- Dredges (Ponar, Young)
- Coring Devices (tubes, Shelby tubes, Ogeechee Sand Pounders®, and augers)
- Vibracore® (Electronic Vibratory Core Tube Driver)

Regardless of the method used, precautions should be taken to insure that the sample collected is representative of the water body or conveyance. These methods are discussed in the following paragraphs.

4 Stainless Steel Scoops and Spoons

4.1 Wading

If the conveyance is dry or is a wadeable surface water body, the easiest way to collect a sediment sample is by using a stainless steel scoop or spoon. If the conveyance is dry, the sediment is accessed directly and is collected using either the stainless steel scoop or spoon. If the conveyance is a wadeable stream or other water body, the method is accomplished by wading into the surface water body and while facing upstream (into the current), scooping the sample along the bottom of the surface water body in the upstream direction. Excess water may be removed/drained from the scoop or spoon. However, this may result in the loss of some fine-grained particle size material associated with the substrate being sampled. Care should be taken to minimize the loss of this fine-grained material. Aliquots of the sample thus collected are then placed in a glass pan and homogenized according to the quartering method described in Section 2.4.

4.2 Bank/Platform Sampling

In surface water bodies that are too deep to wade, but less than eight feet deep, a stainless steel scoop or spoon attached to a piece of conduit can be used either from the banks, if the surface water body is narrow, or from a boat. Again, care should be taken to minimize the loss of the fine particle sizes. The sediment is placed into a glass pan and mixed according to the quartering method described in Section 2.4.

5 Dredges

5.1 General Considerations

Dredges provide a means of collecting sediment from surface water bodies that are too deep to access with a scoop and conduit. They are most useful when collecting softer, finer-grained substrates comprised of silts and clays but can also be used to collect sediments comprised of sands and gravel, although sample recovery in these materials may be less than complete.

Free, vertical clearance is required to use any of the dredges. Dredges, attached to ropes, are lowered vertically from the sampling platform (boat, bridge, etc.) to the substrate being sampled beneath the deployment point.

5.2 Ponar Dredge

The <u>Ponar</u> dredge has side plates and a screen on the top of the sample compartment and samples a 0.05 m² surface area. The screen over the sample compartment permits water to pass through the sampler as it descends thus reducing turbulence around the dredge. The Ponar dredge is easily operated by one person and is one of the most effective samplers for general use on most types of substrates.

The Ponar dredge is deployed in its open configuration. It is lowered gently from the sampling platform to the substrate below the platform. After the dredge lands on the substrate, the rope is tugged upward, closing the dredge and capturing the sample. The dredge is then hauled to the surface, where it is opened to acquire the sample.

5.3 Mini-Ponar Dredge

The Mini-Ponar dredge is a smaller, much lighter version of the Ponar dredge and samples a 0.023 m² surface area. It is used to collect smaller sample volumes when working in industrial tanks, lagoons, ponds, and shallow water bodies. It is a good device to use when collecting sludge and sediment containing hazardous constituents because the size of the dredge makes it more amenable to field cleaning. Its use and operation are the same as described in Section 5.2, Ponar Dredge, above.

5.4 Young Grab

The Young grab sampler is a stainless steel clamshell-type grab sampler similar to a Ponar dredge. It is a clamshell-type sampler with a scissors closing action typically used for marine and estuarine sediment sampling. The Young grab sampler is one of the most consistently performing grab sampling devices for sediment sampling in both offshore

marine sediments, as well as estuarine sediments. The Young sampler comes in two sizes, 0.1 m² and 0.04 m². The 0.1 m² is typically used when a larger volume of sediment is needed for chemistry and particle size. The 0.04 m² is typically used for marine benthic macroinvertebrate sampling and has become the standard grab sampler used by NOAA, USGS and USEPA.

The Young sampler is lowered to the substrate to be sampled with a cable or rope that has a catch that is released when tension is taken off the cable or rope. When the sample device is pulled up, the scissors action of the arms close the clamshell and grabs the sample.

The major difference in the Young grab sampler and other grab samplers is a square or rectangular frame attached to the device which prevents it from penetrating too deeply into soft sediments. In harder substrates, weights may be added to the frame in order to hold the grab in place to prevent collection of a "shallow" sample. A tripod frame can also be attached to the frame surrounding the Young grab sampler. The wire or rope that the grab is raised and lowered with passes through an opening in the top of the tripod and prevents the device from landing sideways or at an angle when there are strong currents or there is lateral movement of the sampling vessel during grab sampling operations.

The draw back to the Young grab sampler is that due to the weight and size of the frame, a ship with an "A" frame or a boat with a davit is required in order to raise and lower the sampler.

6 Sediment Coring Devices

6.1 General

Core samplers are used to sample vertical columns of sediment. They are particularly useful when a historical picture of sediment deposition is desired since they preserve the sequential layering of the deposit. They are also particularly useful when it is desirable to minimize the loss of material at the sediment-water interface. Many types of coring devices have been developed, depending on the depth of water from which the sample is to be obtained, the nature of the bottom material and the length of core to be collected. They vary from hand-driven push tubes to electronic vibrational core tube drivers. These methods are described below in the following sections.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed; the sample is withdrawn intact, permitting the removal of only those layers of interest; core liners manufactured of glass or Teflon® can be purchased, thus reducing possible sample interferences; and the samples are easily delivered to the lab for analysis in the tube in which they were collected.

The disadvantage of coring devices is that a relatively small surface area and sample size is obtained, often necessitating repetitive sampling in order to obtain the required amount of material for analysis. Because it is believed that this disadvantage is offset by the advantages, coring devices are recommended in sampling sediments for trace organic compounds or metals analyses.

6.2 Manually Deployed Push Tubes

In shallow, wadeable waters, or for diver-collected samples, the direct use of a core liner or tube manufactured of Teflon®, plastic, or glass is recommended for the collection of sediment samples. Plastic tubes are principally used for collection of samples for physical parameters such as particle size analysis and, in some instances, are acceptable when inorganic constituents are the only parameter of concern. Their use can also be extended to deep waters when SCUBA diving equipment is utilized. Teflon® or plastic is preferred to glass since they are unbreakable, reducing the possibility of sample loss or personal injury. Stainless steel push tubes are also acceptable and provide a better cutting edge and higher strength than Teflon®. The use of glass or Teflon® tubes eliminates any possible interference due to metals contamination from core barrels, cutting heads, and retainers. The tube should be approximately 12-inches in length if only recently deposited sediments (8 inches or less) are to be sampled. Longer tubes should be used when the depth of the substrate exceeds 8 inches. Soft or semi-consolidated sediments such as mud and clays have a greater adherence to the inside of the tube and thus can be sampled with larger

diameter tubes. Because coarse or unconsolidated sediments, such as sands and gravel, tend to fall out of the tube, a smaller diameter push tube is normally required to obtain a sample. In extreme cases, where sample retention in the tube is problematic, core-catchers or end caps made of Teflon® should be employed. A tube about two-inches in diameter is usually the best size. The wall thickness of the tube should be about 1/3-inch for Teflon® plastic, or glass. The inside wall may be filed down at the bottom of the tube to provide a cutting edge to facilitate entry of the liner into the substrate.

Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water (always work facing upstream and working from downstream up). The core tube is pushed into the substrate until four inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction. The top of the tube is then capped to provide suction and reduce the chance of losing the sample. A Teflon® plug or end cap, or a sheet of Teflon® held in place by a rubber stopper or cork may be used. After capping, the tube is slowly extracted with the suction and adherence of the sediment keeping the sample in the tube. Before pulling the bottom part of the tube and core above the water surface, it too should be capped. An alternative to the coring device is the Shelby tube. The Shelby tube has a gravity check valve at the top of the tube where an auger handle attaches. This check valve allows air and water to escape as the tube is advanced. Once the tube is to the desired depth, the check valve will close automatically forming suction on the tube; thus, holding the sample inside.

When extensive core sampling is required, such as a cross-sectional examination of a streambed with the objective of profiling both the physical and chemical contents of the sediment, complete cores are desirable. A strong coring tube such as one made from aluminum, steel or stainless steel is needed to penetrate the sediment and underlying clay or sands. To facilitate complete core collection and retention, it is recommended that the corer (like a Shelby tube) have a check valve built into the driving head which allows water and air to escape from the cutting core, thus creating a partial vacuum, helping to hold the sediment core in the tube. The corer is attached to a standard auger extension and handle, allowing it to be corkscrewed into the sediment from a boat or while wading. The coring tube is easily detached and the intact sediment core is removed with an extraction device.

Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by simply turning the core tube to its side, and gently pouring the liquid out until fine sediment particles appear in the waste liquid. The loss of some of the fine sediments usually occurs with this technique.

6.3 Ogeechee Sand Pounders® and Gravity Cores

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or a boat using different coring devices such as Ogeechee Sand Pounders®, gravity cores and vibrating coring devices. All three devices utilize a core barrel with a core liner tube system. The core liner can be removed from the core barrel and replaced with a clean core liner, as needed, after each sample. Liners are made of stainless steel, Teflon® or plastic. The type of core liner and its composition should be based on the contaminants to be evaluated.

Ogeechee Sand Pounders® and gravity cores are hand-held devices that use a standard size 2-inch diameter core barrel. The core tube and liner are interchangeable between the two units. The Ogeechee® uses a slide-hammer mechanism attached to the core head that allows the sampler to pound the core tube into the sediment. The Ogeechee® is good for sandy, more consolidated sediments. The gravity core uses a guiding fin mechanism with a built-in gravity-type check valve. The gravity core is placed in the water and released at the surface to free fall to the bottom. The fin mechanism keeps the core tube upright and free from spinning in the water column as it descends. The core tube stabs the bottom, forcing the sediment into the tube. Both coring devices are equipped with removable nose pieces on the core barrel and disposable core catchers for the liner tubes. The core catchers are designed to cap the liner tube to avoid loss of the core when retrieved from the bottom. The gravity core can be modified to attach a slide hammer mechanism, similar to the Ogeechee®, to further pound the core into the sediment further if deemed necessary.

Sediment cores collected from most hand operated coring devices can suffer from either spreading or compaction when driven into the sediment, depending on the softness of the sediment. Spreading occurs when the sediment is pushed or moved to the side during the advancement of the core tube. Compaction occurs when the sediment is being pushed downward as the core tube is advanced. Both phenomena can affect the physical integrity of the core sample. For instance, the core tube may be advanced through the sediment to a depth of 36 inches, but upon examination of the recovered core, there is only 24 inches of sediment in the core tube.

6.4 Vibratory Core Tube Drivers (Vibracore®)

Vibratory Core Tube Drivers (Vibracore®) facilitate sampling of soft or loosely consolidated, saturated sediments, with minimal compaction or spreading, using lined or unlined core tubes. It is designed for use with core tubes having nominal diameters ranging from 2-inches to 4-inches OD. The Vibracore® uses an electric motor to create vibration ranges from approximately 6,000 RPM to 8,000 RPM (100 Hz to 133 Hz) depending on the resistance afforded by the sediment; the greater the resistance, the higher the frequency. The actual vibrational displacement of the Vibracore® is on the order of a few tens of

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thousandths of an inch, so essentially no mixing of the sediment within the tube occurs. The vibrational energy tends to re-orient the sediment particles at the lower end of the core tube, causing them to move out of the way of the advancing wall of the core tube and into a more efficient (i.e. denser) packing. This action advances the core tube with minimal compaction of the sediment.

7 Diving

7.1 General

Sediment samples can also be obtained from large streams and open water bodies such as ponds, lakes, estuarine bodies and open ocean environments by divers. Using a variety of the above mentioned methods, divers can directly access the substrate and collect sediment samples. Depending upon the sampling methods used and the required analyses, the samples may be collected directly into the containers from the substrate or they may be returned, in bulk, to the bank or other sampling platform for processing and sample container allocation.

Appendix B – Sediment Sampling Form

EXAMPLE SEDIMENT SAMPLING FORM

Site Name: Name of Stream/Lake/River Sampled		
Sampling Point No.: Date/Time:		
Name of person(s) sampling:		
A. TYPE OF SAMPLING POINT		
Stream Paved Ditch Road Ditch River Drainage Ditch		
Other (describe):		
B. PURPOSE OF SAMPLING POINT		
Upstream Downstream On-Sitel Other (describe):		
C CAMDUING DOINT CONDITIONS & CEDIMENT CHAD ACTEDISTICS		
C. SAMPLING POINT CONDITIONS & SEDIMENT CHARACTERISTICS		
General description/condition of sampling point:		
Sediment Color:		
Sadiment Lithelemy, Percent Cravel / \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \		
Sediment Lithology: Percent Gravel () Percent Sand () Percent Silt () Percent Clay () Percent Organics ()		
Was monitoring point dw/2		
Was monitoring point dry? Yes No		
If yes, describe:		
Was sediment discolored? Yes No		
If yes, describe:		
Does sediment have odor? Yes No		
If yes, describe:		
Litter present? Yes No		
If yes, describe:		
Sample Coordinates: Longitude: Latitude: Latitude:		
D. FIELD MEASUREMENT		
Weather Conditions:		
PID/FID Reading (PPM):		
E. COMMENTS:		
MI OCHMANIA O		