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Toxic Substances Hydrology Program

Standard Operating Procedure for Collection of Soil and Sediment Samples for the Sediment-bound Contaminant Resiliency and Response (SCoRR) Strategy Pilot Study

Open-File Report 2015–1188B

U.S. Department of the Interior
U.S. Geological Survey

Cover. Breach in the coastline at Rodanthe, North Carolina, caused by Hurricane Irene in August 2011. Photograph by Karen Morgan, U.S. Geological Survey.

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SALLY JEWELL, Secretary

U.S. Geological Survey
Suzette M. Kimball, Acting Director

U.S. Geological Survey, Reston, Virginia: 2015

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Conversion Factors and Datums

Multiply	By	To obtain
	Length	
centimeter (cm)	0.3937	inch (in.)
millimeter (mm)	0.03937	inch (in.)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
kilometer (km)	0.5400	mile, nautical (nmi)
meter (m)	1.094	yard (yd)
	Volume	
liter (L)	33.82	ounce, fluid (fl. oz)
milliliter (mL)	0.03382	ounce, fluid (fl. oz)
liter (L)	0.2642	gallon (gal)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:

$$°F = (1.8 \times °C) + 32$$

Vertical coordinate information is referenced to the North American Vertical Datum of 1988 (NAVD 88).

Horizontal coordinate information is referenced to the North American Datum of 1983 (NAD 83).

Supplemental Information

This Standard Operating Procedure (SOP) document is project specific, meant to be used in conjunction with the U.S. Geological Survey National Field Manual for the Collection of Water-quality Data, Book 9 (a USGS Techniques of Water-Resources Investigations [TWRI] publication). However, this SOP can be adopted for sampling efforts specific to storm-derived change for local and national programs outside of the SCoRR study.

Abbreviations

EPA	U.S. Environmental Protection Agency
GPS	Global positioning system
HDPE	High-density polyethylene
NOAA	National Oceanic and Atmospheric Agency
NWIS	National Water Information System
ODK	Open Data Kit
SCoRR	Sediment-bound Contaminant Resiliency and Response
SOP	Standard operating procedure
USGS	U.S. Geological Survey

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Abstract

An understanding of the effects on human and ecological health brought by major coastal storms or flooding events is typically limited because of a lack of regionally consistent baseline and trends data in locations proximal to potential contaminant sources and mitigation activities, sensitive ecosystems, and recreational facilities where exposures are probable. In an attempt to close this gap, the U.S. Geological Survey (USGS) has implemented the Sediment-bound Contaminant Resiliency and Response (SCoRR) strategy pilot study to collect regional sediment-quality data prior to and in response to future coastal storms. The standard operating procedure (SOP) detailed in this document serves as the sample-collection protocol for the SCoRR strategy by providing step-by-step instructions for site preparation, sample collection and processing, and shipping of soil and surficial sediment (for example, bed sediment, marsh sediment, or beach material). The objectives of the SCoRR strategy pilot study are (1) to create a baseline of soil-, sand-, marsh sediment-, and bed-sediment-quality data from sites located in the coastal counties from Maine to Virginia based on their potential risk of being contaminated in the event of a major coastal storm or flooding (defined as Resiliency mode); and (2) respond to major coastal storms and flooding by reoccupying select baseline sites and sampling within days of the event (defined as Response mode). For both modes, samples are collected in a consistent manner to minimize bias and maximize quality control by ensuring that all sampling personnel across the region collect, document, and process soil and sediment samples following the procedures outlined in this SOP. Samples are analyzed using four USGS-developed screening methods—inorganic geochemistry, organic geochemistry, pathogens, and biological assays—which are also outlined in this SOP. Because the SCoRR strategy employs a multi-metric approach for sample analyses, this protocol expands upon and reconciles differences in the sample collection protocols outlined in the USGS "National Field Manual for the Collection of Water-Quality Data," which should be used in conjunction with this SOP. A new data entry and sample tracking system also is presented to ensure all relevant data and metadata are gathered at the sample locations and in the laboratories.

Introduction

Enhanced dispersion and concentration of contaminants such as trace metals and organic pollutants through storm-induced disturbances and sea-level rise are major factors that adversely impact the health and resilience of communities and ecosystems (Reilly and others, 2015). Infrastructure damage and chemical releases to adjacent waters can become associated with suspended sediments that are then deposited on shoreline soils, on beaches, and in the sediment of marshes and estuarine beds. Furthermore, pathogen outbreaks are often associated with sediment resuspension events, and extreme storm events can lead to food-web disruption and niche displacement (Plumlee and others, 2014). Overall, the presence of toxins and pathogens in contaminated soils and sediments can have environmental health implications in exposed populations of wildlife and humans.

The impacts to human and ecological health resulting from contaminants released during a major coastal storm or flooding event are difficult to measure without historical data and an established protocol for responding soon after the event. The U.S. Geological Survey (USGS) Sediment-bound Contaminant Resiliency and Response (SCoRR) strategy pilot study has been developed to demonstrate a sample-collection strategy to link the chemical, geological, and biological elements of a site. This means that a representative sample being collected from a site be handled according to the various sampling requirements of the respective analytical methods.

Sample collection conflicts that arise need to be reconciled from the perspective of data quality and should consider protocol complexity, staff time, reproducibility, preservation and shipping, cross contamination, and cost. The USGS

"National Field Manual for the Collection of Water-Quality Data" (U.S. Geological Survey, variously dated [b]) provides guidance on proper collection and handling of sediment samples, and procedures are focused on collection for organic or inorganic chemistry analyses with a standard shipping procedure. Considerations for the collection of data for genetics, bioassays, and inorganic and organic geochemistry from a single split sample require the various sampling procedures to be compatible so that the sample collection process is not onerous or introduces contamination. For example, the use of a Teflon-coated stainless steel spoon allows for a single tool to be used to sample for organic and inorganic geochemistry samples. Furthermore, samples are sieved by personnel at the receiving laboratories, which eliminates the need for two sieves in the field (stainless steel and plastic) and thus lessens the burden on the field crew (particularly when responding to a major storm or flooding event).

Standardizing the operating procedures for the USGS Water Science Centers (WSC) participating in SCoRR will provide consistent data quality and control throughout the study region. Techniques and methods from current Federal programs, such as the USGS National Water Quality Assessment and the U.S. Department of Agriculture (USDA) Soil Survey, are referenced throughout this standard operating procedure (SOP) document. This SOP provides a communication tool for the current field crew of SCoRR but can be modified for sampling efforts specific to storm-derived change assessment and used or adopted for local and national programs outside of the SCoRR study.

Background

As part of the response to Hurricane Sandy, USGS collected data on the effects of contaminant source disturbance and dispersion (Buxton and others, 2013). A major limitation of understanding the effects of Hurricane Sandy on contaminant transport and retention was the lack of regionally consistent baseline and trends data in locations proximal to potential contaminant sources and mitigation activities, sensitive ecosystems, and recreational facilities where human and ecological exposures are probable. As a result, the SCoRR strategy was implemented to compare current baseline conditions (defined as Resiliency mode) to conditions observed shortly after episodic stressors like a major coastal storm (defined as Response mode). A four-tiered approach has been developed to demonstrate the linkage between geospatial analyses used to identify potential sources of contamination (Tier 1) and subsequent qualitative (Tier 2) and quantitative (Tier 3) analytics used to document baseline (Resiliency mode) and event-based (Response mode) environmental conditions (Reilly and others, 2015). Repeated Resiliency mode sampling and analysis allow for evaluation of incremental changes anticipated to occur as a result of sea-level rise and land-use changes. Subsequent biological uptake, fate, transport, and exposure studies (Tier 4) can be designed and prioritized based upon SCoRR strategy findings.

Sampling locations are identified using a decision support tool (from a geographic information system [GIS]) that considers the potential contaminant threat and vulnerability proximal to a potential station and stakeholder input (see Reilly and others, 2015, for description of site identification under Tier 1). Following site prioritization, a composite of surficial samples is collected at each site based on the matrix (soil, surfacial sediment, or bed sediment) of interest in the area. The current study area is the coastal counties (as defined by National Oceanographic and Atmospheric Administration [NOAA]) from Maine to Virginia, with USGS Water Science Centers from each State within the area participating in the sample acquisition during Resiliency mode and specialized teams during Response mode.

All samples collected under SCoRR are screened at USGS laboratories to qualitatively assess the inorganic geochemistry (including mineralogy, major ions, and metals), organic geochemistry (for attributes such as molecular structure and spectrophotometric response), ecological effects (including biological assays), and pathological composition (presence/absence and quantification of over 40 bacteria and virus populations). Results from this screening (Tier 2) inform the process of selecting a subset of samples to be assessed more rigorously (for example, chemical compounds identified and quantified) as part of Tier 3. In Resiliency mode, a fraction of total samples will be subjected to Tier 3 analysis, whereas all samples collected in Response mode will be subjected to analysis under Tiers 2 and 3. Many of the screening methods being selected for this study were evaluated during the regional sediment-quality reconnaissance conducted as part of the USGS response to Hurricane Sandy; references to these methods can be found in Fischer and others (2015). The following summarizes the analyses conducted as a part of Tiers 2 and 3.

Inorganic Geochemistry

Screening methods will evaluate metals and trace elements at concentrations in the hundredths of parts per million (ppm) for qualifying leachable metals and major ions associated with soil and sediment, as well as those that comprise the media, and are used to classify the soil and identify any potential sources of contamination from anthropogenic sources. Instruments used for these analyses include x-ray fluorescence (XRF) to determine the relative percentage concentration of metals and x-ray diffraction (XRD) to determine the relative percentage mineralogy. Total carbon, total organic carbon, and total inorganic carbon concentrations are measured, as is the particle-size distribution of the materials. Changes to the properties of deionized water, simulated lung fluid, and simulated seawater solutions upon introduction of the sample are tested—in particular, change in pH and concentration of metals and major ions in solution.

Organic Geochemistry

Organic compounds associated with anthropogenic contaminant sources (such as those common to industrial, commercial, and residential land uses) are identified and discerned from naturally occurring organics. Methods used for screening sediment for organic compounds include attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR), liquid chromatography ultraviolet/fluorescence spectroscopy. Quantification of organic compounds (and respective degradates) identified in Tier 2 analyses is conducted using liquid chromatography ultraviolet/fluorescence spectroscopy, time-of-flight mass spectroscopy (LC/UV-Vis fluorescence/TOF-MS) under Tier 3.

Biological Assays

Enzyme- and whole cell-based assays are employed to determine potential adverse environmental health outcomes associated with sediment-bound contaminants. Endpoints include toxicity, Microtox genotoxicity and mutagenicity, nuclear receptor transactivation/translocation, metallothionein gene transcription, and enzyme inhibition. Bioassay screening is performed on sediment extracts per Ciparis and others (2012), Knapen and others (2007), and Stavreva and others (2012).

Pathogens

While naturally occurring pathogens are common in sediments, elevated levels of human-borne pathogens can be indicative of contributions from failed wastewater treatment facilities, aging septic systems, or combined sewer systems. A presence/absence real-time polymerase chain reaction assay (PCR) is used to screen samples for select pathogenic viruses and antibiotic-resistance genes (ARG). Digital PCR analysis is used to quantify detected pathogens and ARG. For Tier 3 analyses, select samples (those containing multiple pathogens or ARG) are further screened using genetic chip-based analyses, which will allow the identification of a comprehensive list of select bacterial, fungal and viral pathogens, and general ecological community composition. Following review of the data, results are interpreted as they relate to this demonstration of the SCoRR strategy and will also augment an ongoing nationwide assessment of pathogens and how they relate to the various soil and associated chemical composition (Griffin and others, 2014).

Purpose and Scope

In order to ensure a consistent sampling protocol throughout the SCoRR study area, this SOP has been created to describe, in detail, the manner in which samples are to be obtained, including all aspects of field preparation, safety, sample collection, and proper sample storage and shipping. This SOP serves to outline and describe the procedures and protocols for sampling soil, sand, marsh sediment, and bed sediment for the field crews supporting SCoRR in order to provide consistent data quality and control throughout the study region. This SOP can also provide a guide for other USGS projects and programs.

Sampling Methods

Field manuals for a variety of sampling procedures exist within the USGS and elsewhere; however, this SOP was created to provide instructions on sampling soil, sand, marsh sediment, and bed sediment for four suites of analyses, each with differing methods. In order to reconcile differences of sampling methods for inorganic geochemistry (including analysis for methylmercury), organic geochemistry, pathogens, and bioassay techniques, samples are split into a stainless steel bowl and a plastic bowl using a single, Teflon-coated stainless steel spoon. Samples are collected as composites across various coastal landscapes, thus techniques for choosing the proper sample pattern are described. Also employed for this pilot study is an app-based electronic field form and the use of barcoding, both of which were designed to minimize data loss, transcription error, and time in the field.

Techniques and methods adopted from current Federal program manuals, such as the guidelines for the USGS National Water Quality Assessment program (Shelton and Capel, 1994), the USGS “National Field Manual for the Collection of Water-Quality Data” (U.S. Geological Survey, variously dated), the U.S. Environmental Protection Agency (EPA) and USGS “Sample Collection Protocol for Bacteria Pathogens in Surface Soil” (Bowling and others, 2014), and the “Soil Survey Program” by the U.S. Department of Agriculture (2015), are referenced throughout the report. Selected procedures described in following sections are summarized in Appendix 1 to provide reference during sampling.

Personnel Safety

General Field Safety

Field crews are to collect samples from a site only if the area is secure and it is safe to do so (that is, personnel are not put in harm’s way because of a situation beyond those encountered during normal operations)—particularly when sampling during Response Mode (post storm). Like all environmental samples, the material collected may contain chemicals and (or) pathogens of known and unknown toxicity to humans that are present as a result of adjacent land use and (or) storm damage and should be treated accordingly. Field personnel should be familiar with the USGS “Occupational Safety and Health

Program Requirements Handbook" (U.S. Geological Survey, variously dated [a]), the safety protocols and procedures outlined in chapter A9 of the USGS "National Field Manual for the Collection of Water-Quality Data" (Lane and Fay, 1997), as well as any activity- or site-specific training offered by USGS. Only staff with the appropriate sample collection and field safety training should be engaged in field work associated with SCoRR.

Per USGS requirements, a Job Hazard Analysis document outlining the potential risks associated with sampling must be completed and kept on file at each field office for personnel collecting data for SCoRR and should be available during sample collection. Standard safety protocols are to be followed when sampling from a boat (for example, dedicated captain and at least one person to sample with all wearing approved personal floatation devices), and a USGS Float Plan should be filed with the USGS WSC Safety Officer prior to departure each day or as required by the WSC. Sampling in, over, or in close proximity to water requires appropriate training and the use of an approved personal flotation device at all times. Guidelines in the U.S. Department of Labor Occupational Safety and Health Administration (OSHA) training should also be followed when working in disaster areas, such as following landfall of a major storm where significant damage has occurred (https://www.osha.gov/dte/outreach/disaster/index.html).

Sample Handling and Processing

Sample sites may be contaminated with harmful chemicals or pathogens, particularly as a result of infrastructure damage following a major storm. It is important that the field crew is conscious of this potential and uses the appropriate safety and personal protective equipment for a given threat. An OSHA-approved face mask and safety glasses or goggles should be worn when dust or fine particles are suspended in the air. At a minimum, all personnel collecting or handling samples should be wearing two pairs of nitrile gloves—one as a base layer and one that gets changed. Ideally, two personnel are onsite to collect each sample and follow the USGS Clean Hands/Dirty Hands procedure outlined in chapter A4 of the USGS "National Field Manual for the Collection of Water-Quality Data" (U.S. Geological Survey, 2006). For this procedure, one sampler acting as Clean Hands handles only the sample supplies that come in direct contact with the sample, and the other sampler acting as Dirty Hands handles the supplies that could come in contact with potential sources of contamination (U.S. Geological Survey, 2006). The USGS personnel collecting samples should be properly trained on sample collection through one of the approved courses offered by the USGS National Water Quality Laboratory.

As the biological activity and chemical composition of sediment samples will be assessed, care should be taken as to not introduce contaminants to the samples being collected. This includes but is not limited to volatile organic compounds from fuels and solvents; nicotine and other products from cigarettes and chewing tobacco; caffeine from beverages; surfactants and fragrances from personal care products; and microbes from coughing, sneezing, breathing, or stepping on the samples or in the sample area, as described in chapter A4 of the USGS "National Field Manual for the Collection of Water-Quality Data" (U.S. Geological Survey, 2006). Refer to the "Quality Control During Sample Collection" section for additional details on the collecting of an uncompromised sample.

Preparation

Depending on the study area, the collection of a soil sample may require a research or sample-collection permit from local, State, or Federal agencies or permission from land owners. Soil collection and transport also may require permits issued by the USDA, which outlines guidelines for the proper handling and shipment of soils from areas with the potential for spreading invasive species (check with local USDA officer or visit the USDA Web site [referenced in the "Selected References" section] for a list of quarantined States). The USDA soil permits are typically issued in conjunction with the respective State health or environmental agency and are on file at all USGS laboratories currently involved with the SCoRR study.

In addition to this SOP, personnel should be familiar with the bottom-material (that is, bed sediment) collection protocol outlined in Radtke (2005) and Shelton and Capel (1994) for sampling for chemical analyses, and Bowling and others (2014) for sampling for biological analyses. This SOP also references "The Environmental and Medical Geochemistry of Potentially Hazardous Materials Produced by Disasters" (Plumlee and others, 2014) and the "Sample Collection of Ash and Burned Soils from the October 2007 Southern California Wildfires" (Hoefen and others, 2009), as well as the USGS Hurricane Sandy, Theme 4, marsh- and bed-sediment sampling standard operating procedures (Irene Fisher, USGS, written commun., 2013), which was followed for collecting samples during the USGS Hurricane Sandy ecological and human health assessment in 2013 (Fischer and others, 2015).

During Resiliency mode, sample collection can be spread out over several months in order to accommodate field crew schedules, stakeholder needs, and other considerations. The entire network should be sampled within a single season for consistency among sites and should be sampled in the same season for the entire study region (for example, the northeast region). The collection of samples also should be scheduled from Monday to Thursday to ensure overnight delivery (laboratories used for the SCoRR project do not accept samples on Saturday). However, during Response mode, stations should be identified as soon as possible and routes to each site should be planned as crews mobilize. Samples of soil and sediment should be collected as soon as possible (when safe) following a major storm and (or) flooding event in order to preserve and capture the potential changes that have occurred at the

site (Plumlee and others, 2014). The collection of samples under Response mode should be scheduled from Monday to Wednesday to ensure overnight delivery if shipping to and (or) from disaster-affected areas. A list of alternative stations should be in place for the Resiliency and Response modes in the event that a site is not accessible because of traffic, roadway damage, or police closure. If a site is temporarily not accessible, crews should be prepared to visit the site later or on a different day. If a site is permanently blocked, or the required amount of sample cannot be collected from the immediate area, crews should be prepared to proceed to an alternate station.

Overnight shipping facilities close to the sample sites should be located before departing for the field. Ice supply should also be considered for field trips requiring one or more overnights. The frozen gel packs supplied for shipping pathogen samples should be kept in ice when traveling and would be better stored in a room freezer (if available) overnight.

Site Evaluation and Sampling Patterns

The location and sample media dictate the method for sample collection used at each site. Samples of soil and surficial sediment (for example, marsh sediment and beach sand) are typically collected without special equipment, whereas collection of bed sediment in areas with water overlying the sediment requires samples be collected by wading in on foot or from a bridge, dock, or boat. At each site, multiple grab samples are collected to create a composited sample. The distribution of the individual composite samples will depend on the extent and accessibility of each location. A decision should be made prior to arrival at the site as to which sample pattern should be employed (grid, spoke, or transect) using satellite images offered by online mappers, a reconnaissance trip, or institutional knowledge of the site; however, a different pattern and (or) method may need to be used by the field crew upon arrival if the site has been recently disturbed (for example, construction) or covered by water (for example, a tidal marsh).

Figures 1–3 provide examples of the three sample site patterns that can be employed for collecting a composite sample. The distances depicted in figures 1–3 are examples and can be as short as 10 feet (ft) apart for this study, depending on desired extent of sample area, and the fewest number subareas from which composites are collected is five. Ultimately, professional judgement is required for determining the number of subareas to be sampled at a site to ensure a representative sample for the sample area (Radtke, 2005; Shelton and Capel, 1994). Per the USGS National Field Manual for the Collection of Water-Quality Data, chapter A8 (Radtke, 2005), subareas are selected at each SCoRR study site by using either a systematic regular interval for establishing a grid, a transect pattern based on site characteristics, accessibility, and the total desired area to be sampled (for example, an entire shoreline field or a particular stretch of public beach bordering homes), or a nonstatistical deterministic method for spokes pattern or bed sediment sampling (Radtke, 2005).

Sample collection using a grid pattern is employed in uniform fields or a coastal community (developed area) where soil (or other media) can be collected near the center of each box (fig. 1). The boxes that comprise the grid can vary in size and shape depending on the total amount of sampling area and desired extent of sample area away from the prioritized station. Figure 2 illustrates the spoke diagram, which is well suited for sampling in marshes because each sample away from the centroid can extend out to capture or avoid particular features, such as mosquito ditches or pannes. The lengths of each spoke (that is, the distance from the centroid) shown in figure 2 should remain the same at a given site, but can vary from site to site. The number of composite points (or subareas) should also be evenly divided to get equal angles from the centroid. The transect pattern shown in figure 3 is useful when the desired sample area is a long, narrow strip of land (for example, a beach) or from the coast inland. A transect should start at the station coordinates identified and proceed in a single direction, with samples for the composite collected at set increments.

Sampling Equipment and Supplies

Equipment and supplies needed for sample collection are listed in Appendix 2. It is important to ensure all sampling equipment is functioning properly, free from rust and corrosion, and thoroughly cleaned (and sanitized if needed for pathogen sampling) prior to leaving for the field. Thermometer(s) used in the field (for air and soil) should be checked for accuracy at the beginning of the sampling season and calibrated every 12 months using a National Institute of Science and Technology traceable as described in the USGS Field Manual (Wilde, variously dated). Multiparameter water-quality sensors should be checked and calibrated using approved standards prior to use in the field with calibration records properly documented (for example, in a USGS logbook).

The Garmin (Olathe, Kansas) Monterra™ was selected for use in this study because it is an all-in-one handheld device that provides the ability to acquire position through Global Positioning System (GPS) satellites (as opposed to cellular-based triangulation), take pictures of sampling locations, scan barcodes on sample containers, and the functionality to allow for field data to be entered electronically. (A handheld GPS, camera, and laptop/paper field forms can be used if a Monterra™ is not available.) The GPS data are collected using the North American Datum of 1983 (NAD 83), and an accuracy of plus or minus 10 ft should be achieved before accepting the location coordinates. The GPS device should be turned on and actively searching for satellites for as long as possible before accepting the location (a GPS accuracy of 10 ft or less is acceptable). Therefore, it is best to turn the device on upon arrival at a site (or earlier).

The SCoRR field form (framework of the electronic version shown in Appendix 3, and a printable [back-up] version

Figure 1. Example of a grid created for collecting soil samples in wide areas that are uniform in soil and sediment consistency and potential for contamination, such as a field in a riverside park near Annapolis, Maryland.

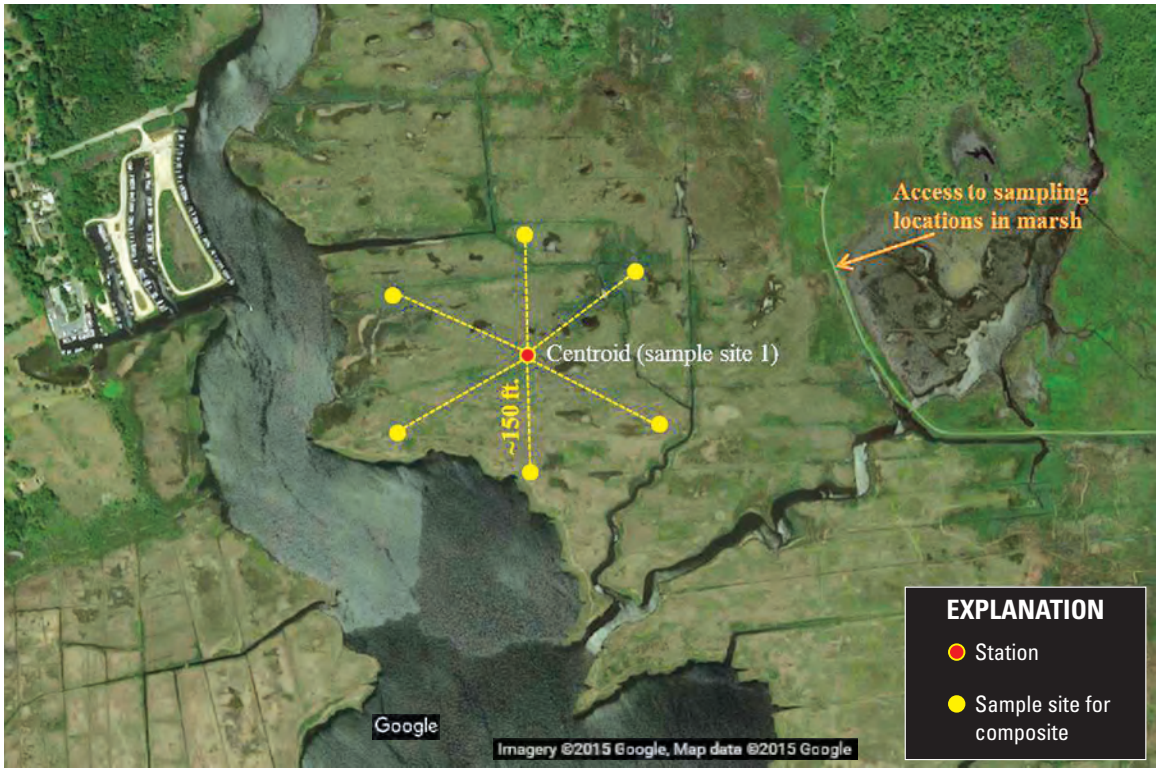

Figure 2. Example of a spoke pattern for collecting marsh sediment samples in a large sample area with variable sediment conditions, such as the saltwater marsh at Wertheim National Wildlife Refuge, Brookhaven, New York.

Figure 3. Example of a transect pattern created for collecting a beach sediment sample from a long, narrow sample area, such as a beach at Cape Cod, Massachusetts.

is shown in Appendix 4) must be completed and submitted electronically in order to process a sample and generate a reference number. The electronic field form application was created using a free, open-source program developed by Open Data Kit (ODK) (https://opendatakit.org/), which uploads all acquired data to the *formhub.org* server. A database of the identified sampling stations is provided by the SCoRR network coordinator prior to start of the sampling effort (in Resiliency mode and when Response mode is activated). Each field crew should download the prioritized stations set for their region for the field form to query; instructions are provided to the lead sampler in advance of the sampling effort. The field form requires a basic description of the location, latitude and longitude, a representative picture of the sampling location, and a date/time. Depending on the device (if not using the Monterra™), most of these fields are populated automatically by clicking on the "auto fill" box; otherwise, additional equipment (GPS and camera) will be required and uploaded separately following sample collection, but prior to upload.

The default barcode reader application will be activated by the electronic field form (the number is also printed on the label should manual entry be necessary). Sampling containers are to be pre-labelled with a barcode (this study uses the standard Code 39 symbology) that has a sample-set-specific serial number, with the last two numbers representing the container code of the eight sample containers in sequential order (01–08). Figure 4 is an example of the sample container label indicating the state ("NY") from which the sample was collected, the six-digit unique sample-set identifier (123456) that will be automatically attributed to the data entered into the field form, and the two-digit analysis code that is designated by the container type (02). Note that the hyphens used to break up the alphanumeric serial number displayed on the label are not coded into the barcode (that is, label text NY–123456–02 is stored as "NY12345602" in the database). As codes are sample, not station, specific, any sample set can be used (within the state identified) at a site.

The barcode serves to automate the data transfer process and will document the chain of custody through the sampling and analyses steps associated with this project. Once samples are received by the analyzing laboratories, the containers will be scanned to automatically generate an entry in the laboratory

Figure 4. Example of barcode label used on sample vessels for the Sediment-bound Resiliency and Response (SCoRR) pilot study.

results database, into which data from each analysis will be linked to the field forms when queried. The inorganic and organic geochemistry laboratories will conduct chemical extractions of the sediment per their respective methods and send extracts to the bioassays laboratory for analysis—no samples are shipped from the field directly to the bioassays laboratory. For field blanks submitted to the pathogen laboratory, a sample set with an additional vial coded 01B should be used; alternatively, a spare label can be used as long as the sample set along with 01B is written on the label (see the section "Quality Control During Sample Collection" for more information about field blanks). Vials containing extracts from the original sample jars will be labeled by the organics and inorganics laboratories with a barcode encoded with the sample identity and an "E1" for "extract [number] 1"; subsequent extracts from the same container (if necessary) will have sequential numbers following the "E."

A standard cooler should be filled with enough ice to ensure samples remain chilled in the field, frozen ice packs remain frozen, and shipping coolers can be filled properly (if shipping from the field). In addition to the set of frozen gel packs placed in the small foam shipping cooler to keep the vials for pathogen analysis chilled in the field, sets of frozen gel packs equal to the number of small shipping coolers expected to be shipped should be packed is a large zip-top bag and kept in ice if shipping from the field. Arrangements should be made prior to departing for long field days or multiple-day field trips to ensure availability of ice necessary for the aforementioned uses.

Field Measurements

Prior to collecting samples, the field crew should position themselves at the first sampling point (station) in the pattern to complete the electronic field form, which includes recording GPS data, collecting field parameters, and taking at least two field photographs. Physical parameters of soil, air, and water (if applicable) are to be collected at all sample sites using a thermometer and multiparameter water-quality sonde (Appendix 2). The minimum required parameters are listed in table 1 and on the field form. (Additional measurements may be collected and documented along with their respective USGS parameter codes in the "Notes" section of the field form.)

Sample Collection

Sample collection protocol involves collecting eight samples, each with their own collection requirements. One container is directly filled with sediment, and other containers require the composite sample from multiple subareas in a pattern from a site to be homogenized and then distributed into containers. The steps listed below outline these collection requirements and are critical for the site preservation and quality assurances for the laboratories.

Collecting soil or surficial sediment and filling sample containers can be done by an individual sampler, but a crew of two qualified sampling personnel is optimal. When two sampling personnel are onsite, one crew member is responsible for preparing the site, organizing the sampling materials, and keeping the sample containers and labels clean and free of contaminants. The other crew member should focus on collecting the sample, keeping track of the amount of sample collected, and ensuring that the sample site does not become contaminated (U.S. Geological Survey, 2006).

Barcodes for the jars should be scanned prior to being filled, and care should be taken to not get the sample on the label while filling because it could interfere with barcode reading in the laboratory. If a jar cannot be scanned, the code can be entered manually using the keypad; type in the sample jar serial number below the barcode (without hyphens). If a container is lost or broken during sample collection, fill a replacement jar and write the sample set code and container number on a new waterproof label and attach to the jar (if original jar

Table 1. Minimum required physical parameters and observations at each sample location.

[Physical parameters associated with the sample area are to be documented prior to collecting samples. Observations of weather, sky, and wind at sample location are qualitative with basic responses outlined on the field form. USGS, U.S. Geological Survey; °C, degrees Celsius; --, not applicable; ft, feet; µS/cm, microsiemens per centimeter at 25 degrees Celsius; mg/L, milligrams per liter]

Media	Parameter	Units	USGS parameter code	Reference figure
Soil or sediment	Soil temperature	°C	--	Figure 5
Air	Air temperature	°C	00020	Figure 6
	Weather	--	--	
	Sky	--	--	
	Winds	--	--	
Water (at sample depth)	Depth to bottom	ft	81903	Figure 7
	Water temperature	°C	00010	
	Specific conductance	µS/cm	00095	
	pH	pH units	00400	
	Dissolved oxygen	mg/L	00300	

Figure 5. The collection of soil temperature data.

Figure 6. The collection of air temperature data.

Figure 7. The collection of water parameters.

was lost), or carefully remove the label from the old jar and adhere it to the replacement (only attempt to remove label from a broken jar if there is no risk of being cut by broken glass; otherwise, create a new label). A spare set of sample containers should always be included with field equipment in case of breakage or loss.

Sampling Soil or Surficial Sediment

1. Identify the area from which soil or surficial sediment is to be sampled and follow the pre-determined sample pattern (examples of sample patterns are shown in figs. 1–3) chosen for the site. If site is on the shoreline, locate the high tide mark (or rack line); samples are to be collected upland from this mark (fig. 8A). Also, make a plan that includes the path for approaching and exiting each subarea for collecting the composite samples. Once a site is cleared and prepared, your foot pattern must avoid collection points.

2. Put on disposable shoe covers; take care not to step on or otherwise compromise the point within each subarea identified for sample collection. (Note that shoe covers are not needed on a boat or may not be practical in a marsh, in which case, take extra precautions not to step on or too close to the sampling points.)

3. During sample collection, it is important to avoid grasses, stones, and pieces of debris larger than 2 millimeters (mm); if present, **gently** clear grasses, stones, and debris using gloved hands or a clean garden tool without removing the top layer of soil or sediment (fig. 8B).
4. Place a new plastic dropcloth around the sampling point for resting bowls and other sampling equipment (fig. 9A). The same plastic dropcloth may be used at each point as long as there is a clean place to rest bowls and equipment.
5. Put on a pair of nitrile gloves.
6. Collect sample for pathogens analysis.
   A. Select an undisturbed area that is free from debris within the sample pattern.
   B. Follow the "Filling Sample Containers—Pathogens" section.
7. Collect samples for inorganic and organic geochemistry analyses and chemistry archives.
   A. Put on second pair of nitrile gloves.
   B. Collect the top 2 centimeters (cm) of soil or sediment, or top 5 cm of beach sand, from each point within the pattern using a Teflon-coated stainless steel spoon, and place equal portions of soil in the stainless steel bowl and the plastic bowl (figs. 9B and 9C). The volume collected in each bowl from a point should be roughly proportional to 1 liter (L) divided by the number of subareas chosen for the site (for example, a transect with five composite sites would have about 200 mL of soil collected in each bowl [400 milliliters (mL) total] per point). Note: If excess amounts of grasses, stones, or other debris cannot be easily brushed aside or dissociated from the soil, sand, or marsh sediment, gently sieve the material through a stainless steel sieve (for media sieved into the stainless steel bowl) and a plastic sieve (for media sieved into the plastic bowl) each with a 2-mm pore size and make note of the sieve use in the field form "Notes" section. Keep bowls covered (aluminum foil for stainless steel bowl and plastic for plastic bowl) between subareas (fig. 9D).

Figure 8. Examples of *A*, sample locations at sample types, and *B*, sample point preparations.

C. Continue until all points in the pattern have been sampled and at least 1 L of soil has been collected in each bowl (fig. 9E).

D. Use Teflon-coated stainless steel spoon to gently homogenize sample in each bowl, taking care not to scrape the edge of the bowls (figs. 9F and 9G).

E. Remove outer gloves and put on new pair of nitrile gloves.

F. Using the Teflon-coated stainless steel spoon, fill jars for inorganic and organic geochemistry and chemistry archive samples per the respective subsection in the "Filling sample containers" section.

Sampling Bed Sediment

For bed sediment sample, two sampling personnel are necessary.

1. Determine which sampler to use:

   A. If water depth is 4 ft or less, sampling can be conducted with the Ekman sampler with the handle attached.

   B. If the water is greater than 4 ft deep, choosing between the Ekman and Petite Ponar samplers is dependent upon the substrate and the intensity of the waves or current. The Ekman sampler is the best

Figure 9. Steps for collecting and homogenizing soil in stainless steel and plastic bowls for organic and inorganic geochemistry analysis and archives.

choice for finely divided muck, mud, ooze, submerged marl, and fine peaty materials that are free from vegetation, such as sticks and decayed leaves (or with short, erect vegetation only) as well as intermixtures of sand, stones, and other coarse debris. The Ekman sampler is not recommended for rocky or sandy bottoms or moderate macrophyte growth because small pebbles or macrophyte stems prevent proper jaw closure. The Petite Ponar sampler is better suited for sampling compacted material, locations with debris (for example, sticks, pebbles, shells) and, because it is heavier than the Ekman sampler, locations where current/wave action is a factor. If unsure of the substrate, the Petite Ponar sampler should be used.

2. [Clean Hands] Put on a new pair of nitrile gloves.

3. [Dirty Hands] Deploy and retrieve sampler (figs. 10A and 10B).

A. Place sampler on clean surface and remove screens (Petite Ponar sampler) or open doors (Ekman sampler) to ensure that an appropriate sample was collected (fig. 10C). If a representative sample was not collected (for example, only shells or seaweed was grabbed), reset sampler and redeploy.

B. [Clean Hands] Allow sample to rest for 1 minute to allow flocculate to settle. Remove overlying water (if necessary) from sample containers using gentle suction with the peristaltic pump or disposable syringe; immerse tubing or inlet just below water surface so as to not come in contact with sediment or floc.

C. [Clean Hands] Remove the upper 2 cm with Teflon-coated stainless steel spoon and place equal amounts of the sediment into the stainless steel bowl and the plastic bowl (fig. 10D).

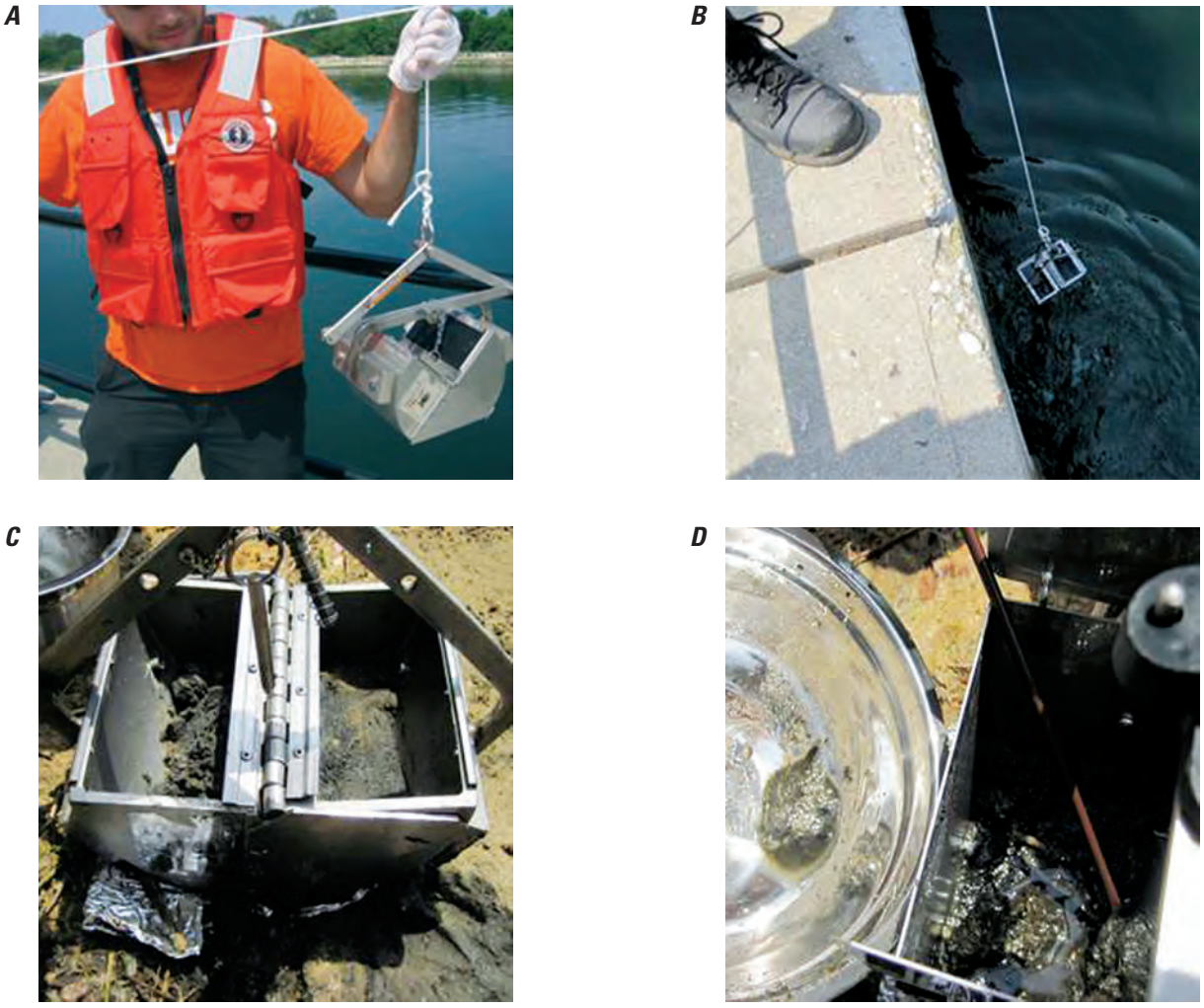

Figure 10. Steps for collecting bed sediment samples using a Petite Ponar sampler.

D. Multiple grab samples will be required to collect the total amount of sediment required for all analyses, and it is important to not collect from the same spot (thus collecting the deeper sediment). Move over slightly to collect a grab from a different spot. Note: When sampling from a boat, the boat should be anchored at the station before attempting to sample. Drifting tends to occur (even when anchored) and will usually help position the sampler for a different grab at each drop. However, if after the initial grab, it is not easy to discern between disturbed sediment and fresh sediment (for example, there is no clear distinction between the oxic and anoxic layers after removing the top 2 cm where in previous samples there was), move the boat forward one boat length to increase probability that a sample is not collected at the same point.

E. Collect sample for pathogen analysis from one (or more, if necessary) grab: Prior to collecting for chemistry analyses for that grab, follow the "Filling Sample Containers—Pathogens" section.

4. Repeat step 3A-D until 1 L of sediment has been collected in each bowl. Note: If excessive biota or debris (greater than 2 mm) is present, collect additional sediment in sample containers 02, 05, 06, 07, and 08.

5. [Clean Hands] Use Teflon-coated stainless steel spoon to homogenize sample in each bowl.

6. [Clean Hands] Remove top gloves and put on a new pair of nitrile gloves.

7. Collect samples for inorganic and organic geochemistry analyses and chemistry archives.

A. Using the Teflon-coated stainless steel spoon, fill jars for organics and chemistry archive (for organics) samples per the respective subsection in the "Filling Sample Containers" section.

B. Using the Teflon-coated stainless steel spoon, fill jars for inorganics and chemistry archive (for inorganics) per the "Filling Sample Containers" section.

Filling Sample Containers

All soil, surficial sediment, and bed sediment collected from multiple subareas or grabs at a site are to be homogenized in the stainless steel bowl for organic geochemistry analyses and a plastic bowl for inorganic geochemistry analyses and distributed to the appropriate prelabeled containers for inorganic and organic screenings (with the exception of samples for pathogen screening, which are collected separately [without homogenization]) by scrapping the top 2 cm of single composite point or grab. Barcodes should be scanned (if device is capable) or entered manually (numbers below barcode) in the field. The field information will be stored in the database and sent along with the barcode information to all laboratories. The following list describes the sampling vessel filling procedure for each suite of analyses and also is summarized in table 2:

1. Pathogens

Note: this sample is **not** taken from the bowl used to homogenize material for organic and inorganic analyses.

A. Put on a second pair of nitrile gloves in addition to the base pair.

B. Using the sterile 50-mL centrifuge (container code 01)

I. Remove the cap—DO NOT put the cap down, keep in hand away from other objects and out of the wind and precipitation.

II. Carefully skim the surface of the soil or sediment (with vial label pointing up) or use a sterile disposable plastic spatula to help scoop samples into vial filling the tube to at least the 40-mL mark (figs. 11A and 11B).

III. Tap side of vial gently to shake loose sample stuck on the threaded opening.

IV. Cap tightly; do not overtighten (fig. 11C).

V. Wipe vial of excess soil or sediment and moisture with a clean laboratory wipe.

VI. Wrap cap with precut Parafilm® (figs. 11D and 11E).

VII. Place tube in small zip-top bag (fig. 11F).

VIII. Place in small foam shipping cooler with frozen gel packs to chill (fig. 11G).

C. Remove outer pair of gloves.

2. Inorganic Geochemistry

A. Inorganics sample

I. Using the Teflon-coated stainless steel spoon, fill the 250-mL plastic high-density polyethylene (HDPE) jar (container code 02) until three-fourths full with soil or sediment from the plastic bowl (fig. 12A).

II. Gently clean rim of jar with a clean laboratory wipe (fig. 12B).

III. Cap tightly; do not overtighten (fig. 12C).

IV. Place jar in small zip-top bag (fig. 12D).

Table 2. Sample requirements for soil and sediment collection.

[Each analytical method requires specific sample containers, amount of material required, and preservation. Container code represents the two-digit suffix on the barcode labels. Maximum holding time, which is the acceptable duration of time between sample collection and shipment; anything with a holding time of less than 24 hours should be shipped the day of collection ("ship same day") via the selected courier's overnight service; mL, milliliters; HDPE, high-density polyethylene; °C, degrees Celsius]

Analytical method	Sample container	Container code	Fill amount	Field preservation method	Storage preservation method	Maximum holding time under storage	Shipping preservation	Destination	Contingency field storage
Pathogens	50 mL sterile plastic centrifuge tube	01	40 mL	Frozen gel packs	None, ship same day	12 hours	Frozen gel packs	St. Petersburg, Florida	Triple bag in small zip-tops; keep cold on ice in cooler
Inorganic Geochemistry	250 mL plastic HDPE jar	02	¾-full	Chill on ice	None, ship same day	48 hours	Water ice	Denver, Colorado	Keep cold on ice in cooler or in freezer
Soil Moisture	50 mL sterile plastic centrifuge tube	03	50 mL	Chill on ice	None, ship same day	48 hours	Water ice	Denver, Colorado	Keep cold on ice in cooler or in freezer
Methylmercury	50 mL sterile plastic centrifuge tube	04	20 mL	Chill on ice	Freeze at -20 °C, ship all vials when sampling complete	6 months	Water ice	Middleton, Wisconsin	Keep cold on ice in cooler or in refridgerator
Organic Geochemistry 1	250 mL baked amber jar with Teflon-lined cap	05	¾-full	Chill on ice	None, ship same day	24 hours	Water ice	Lawrence, Kansas	Keep cold on ice in cooler or in freezer
Organic Geochemistry 2	250 mL baked amber jar with Teflon-lined cap	06	¾-full	Chill on ice	None, ship same day	24 hours	Water ice	Lawrence, Kansas	Keep cold on ice in cooler or in freezer
Chemistry Archive (Inorganics)	250 mL plastic HDPE jar	07	¾-full	Chill on ice	Freeze at -20 °C	6 months	Water ice	Lawrence, Kansas	Keep cold on ice in cooler or in freezer
Chemistry Archive (Organics)	125 mL baked amber jar with Teflon-lined cap	08	¾-full	Chill on ice	Freeze at -20 °C	6 months	Water ice	Lawrence, Kansas	Keep cold on ice in cooler or in freezer

Figure 11. Steps for collecting samples for pathogens analysis (sample container 01).

B. Soil moisture sample

I. Using the Teflon-coated stainless steel spoon, fill the 50-mL centrifuge tube (container code 03) with 50 mL of soil or sediment from the plastic bowl (fig. 13A).

II. Gently clean rim of jar with a clean laboratory wipe (fig. 13B).

III. Cap tightly; do not overtighten (fig. 13C).

IV. Place vial in the same small zip-top bag as the inorganic screening sample jar (fig. 13D).

V. Place bag in cooler on ice to chill (fig. 13E).

C. Methylmercury sample

I. Using the Teflon-coated stainless steel spoon, fill the 50-mL centrifuge tube (container code 04) with 20 mL of soil or sediment from the plastic bowl (fig. 14A).

II. Clean rim of jar with a clean laboratory wipe (fig. 14B).

III. Cap tightly; do not overtighten (fig. 14C).

IV. Place wrapped jar in a small zip-top bag (fig. 14D).

V. Place bag in cooler on ice to chill (fig. 14E).

Figure 12. Steps for collecting samples for inorganics screening analysis (sample container 02).

3. Organic Geochemistry

   A. Using the Teflon-coated stainless steel spoon, fill two 250-mL baked amber glass jars (container codes 05 and 06) until three-fourths full with soil or sediment from the stainless steel bowl (fig. 15A).

   B. Clean rim of each jar with a clean laboratory wipe (fig. 15B).

   C. Cap each jar tightly with Teflon-lined cap; do not overtighten (fig. 15C).

   D. Wrap each jar with the large bubble-wrap protectors (fig. 15D).

   E. Place both wrapped jars in the same large zip-top bag (fig. 15E).

   F. Place bag in cooler on ice to chill (fig. 15F).

4. Chemistry Archive

   A. Inorganics

      I. Using the Teflon-coated stainless steel spoon, fill the 250-mL plastic (HDPE) jar (container code 07) until three-fourths full with soil or sediment from the plastic bowl (fig. 16A).

      II. Clean rim of jar with a clean laboratory wipe (fig. 16B).

      III. Cap tightly; do not overtighten (fig. 16C).

      IV. Place jar in a large zip-top bag (fig. 16D).

   B. Organics

      I. Using the Teflon-coated stainless steel spoon, fill the 125-mL baked amber glass jar (container code

Figure 13. Steps for collecting samples for soil moisture analysis (sample container 03).

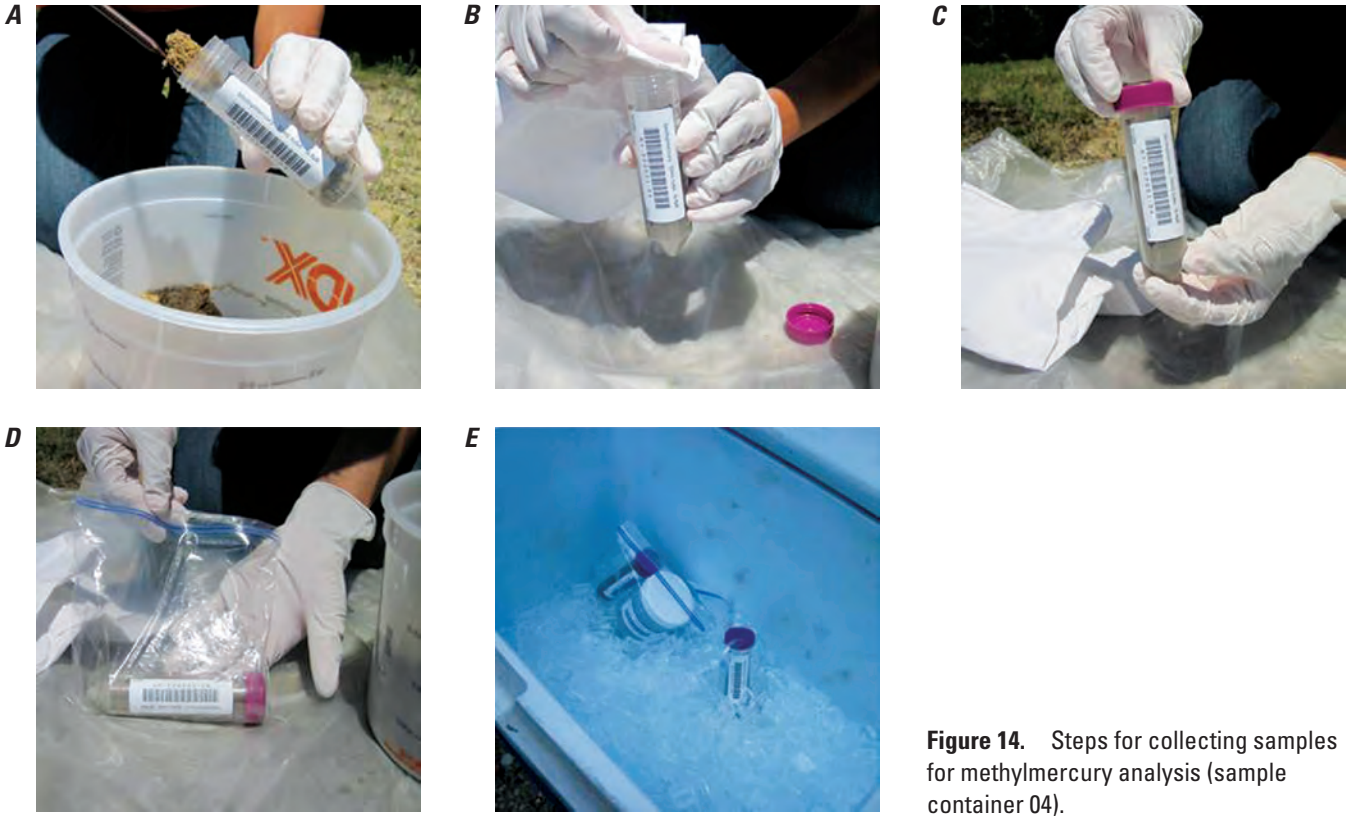

Figure 14. Steps for collecting samples for methylmercury analysis (sample container 04).

Figure 15. Steps for collecting samples for organics screening analysis (sample containers 05 and 06).

08) until three-fourths full with soil or sediment from the stainless steel bowl (fig. 17A).

II. Clean rim of jar with a clean laboratory wipe (fig. 17B).

III. Cap tightly—do not overtighten (fig. 17C).

IV. Wrap jar with small bubble-wrap protector (fig. 17D).

V. Place wrapped jar in the same large zip-top bag as the inorganics geochemistry archive jar (fig. 17E).

VI. Place bag in cooler on ice to chill (fig. 17F).

Sequential replicates are to be collected at the same time as the environmental sample for stations where a replicate (quality control) sample is planned. The purpose of collecting a sequential replicate sample is to identify and characterize the variability in the sampling process and the sample site. A sequential replicate is collected by filling two sets of bowls (plastic and stainless steel) with the predetermined volume from the same subareas as close to the sample points as possible before moving to the next subarea. Two sets of prelabeled containers are then filled (there is nothing on the labels to indicate a replicate, and though the use of sets with sequential sample-set identifiers is preferred, it is not required). The first sample will be stored in the database as REGULAR environmental sample (USGS National Water Information System [NWIS] Sample Type code 9), and the second sample will be stored in the database as the sequential REPLICATE (NWIS Sample Type code 7).

To collect a field blank for the pathogens method, follow the "Filling Sample Containers—Pathogens" section except use the sterile soil provided to fill vial 01B directly, or use a new sterile disposable spatula. When transferring sterile soil, be careful not to expose the blank sample to precipitation or surfaces (other than the sterile spatula, if needed). The blank sample should be shipped in the same cooler as the environmental sample from the same site (vials will have the same sample-set identifier). The pathogen blank sample will be stored in the database as the sequential FIELD BLANK (NWIS Sample Type code 2).

It is important that the sample site is left minimally disturbed. Once sampling at a site is complete, repair divots in the soil (by covering or adding nearby soil) and replace any objects moved prior to collecting a sample. Collect all gloves, booties, masks, used laboratory wipes, plastic dropcloths, and other used disposables and trash, place it in a trash bag, and dispose of back at the office.

Cleaning and Sanitizing Equipment

All equipment should be thoroughly cleaned using the steps outlined below in a clean environment (such as a field preparation laboratory) and away from running vehicles or other sources of contamination. Thoroughly cleaning and sanitizing (if necessary) equipment in the field is only required if the same equipment is to be used for another site in the same outing; otherwise, equipment can be rinsed with tap water and cleaned in a field preparation laboratory or other appropriately equipped space.

1. Put on a pair of nitrile gloves.
2. Set up a clean working space using aluminum foil and clean laboratory wipes for resting cleaned equipment.
3. Rinse equipment (spoons, bowls, sampler, and sieves [if used]) with tap water.
4. Use nylon brush to remove particles; be sure to clear any material stuck in the moveable and removable parts of the sampler (for example, the screens of the Ponar sampler), and rinse with tap water.
5. In the field between sites, gently clean equipment with nylon brush and a dilute solution of Liquinox™ soap. Rinse thoroughly with tap water, followed by deionized (DI) water. Follow step 6 below for sanitization if necessary. Wrap in aluminum foil, and place in a bag to keep clean between sites. Upon returning to the office, equipment should be thoroughly cleaned and placed on a clean surface to air dry (away from any potential sources of airborne or waterborne contaminants) before wrapping in aluminum foil for storage or next use.
6. Sampling equipment that is not presterilized and is used before or during the collection of a pathogen

Figure 16. Steps for collecting samples for inorganics chemistry archive (sample container 07).

sample (samplers [such as the Ponar] and the stainless steel trowel [in the absence of a disposable sterile spatula]) should be sanitized at least 30 minutes prior to use according to the process outlined by Bowling and others (2014).

A. Put on a new pair of nitrile gloves.

B. Pick up one piece of equipment at a time.

C. Wipe down equipment using a 70-percent alcohol wipe.

D. Wipe down equipment using a 10-percent bleach wipe.

E. Place bleached equipment in zip-top bag (must remain in bag with bleach residue for at least 30 minutes; this can be traveltime between sites).

F. After 30 minutes, put on a new pair of nitrile gloves to remove the equipment from the bag.

Quality Control During Sample Collection

Samples are collected in accordance with the USGS and EPA guidelines outlined in Radtke (2005) and Bowling and others (2014), each of which describes proper quality-control procedures during a sampling event. Procedures such as multiple glove exchanges during sampling, proper sample handling, and following the cleaning/sanitizing protocol emphasized in this SOP will minimize the potential for contamination of the sample by personnel and from cross contamination across sites. Sequential replicate samples collected in the field for analyses represent 10 percent of the total sample set. Sterile soil that is transported and transferred to a sample vial in the field (that is, a field blank) is used for quality control by the field crew collecting samples for pathogen analyses and represents 10 percent of the total sample set. Laboratory quality controls specific to each analytical method (such as spikes, blanks, and analysis of reference materials) are documented in respective methods documentation.

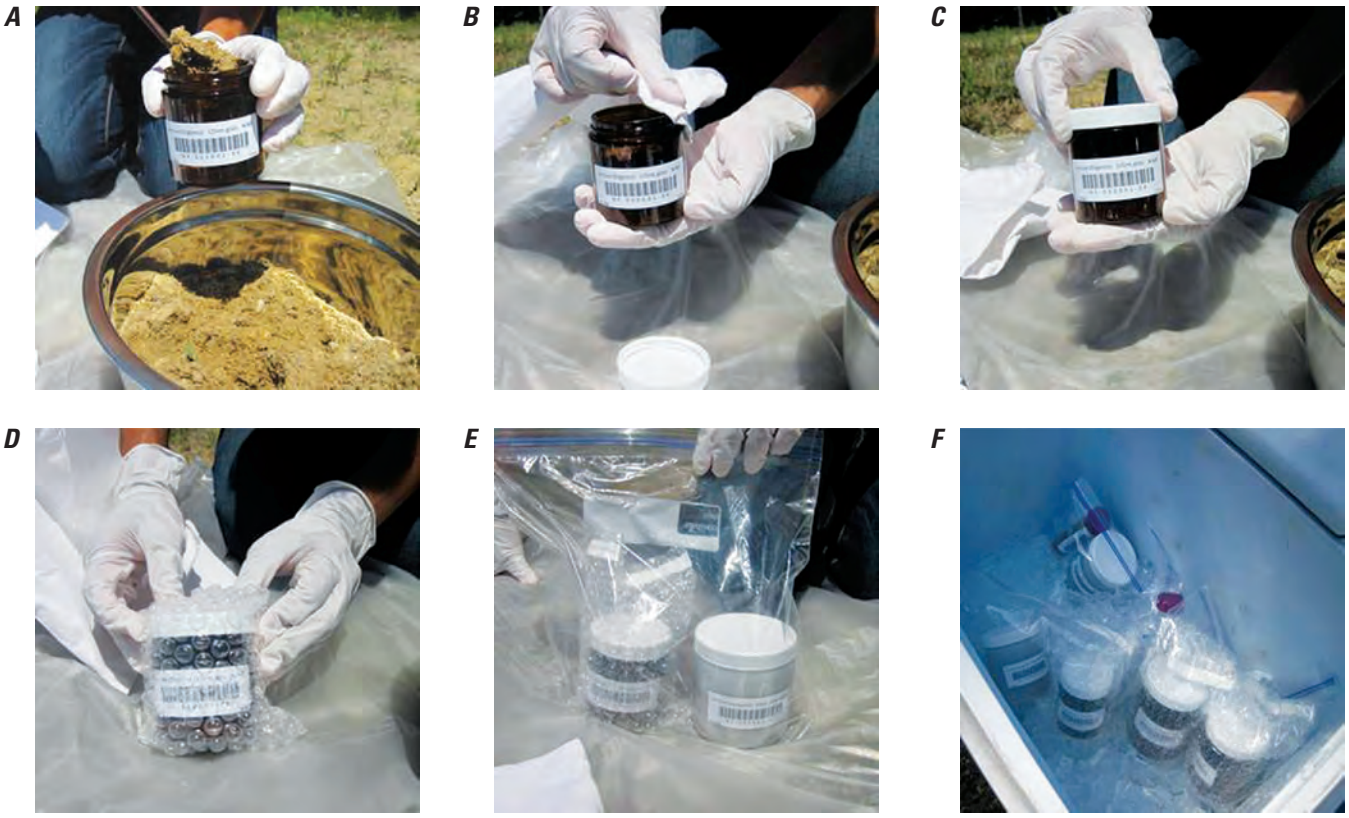

Figure 17. Steps for collecting samples for organics chemistry archive (sample container 08).

Post Sample-Collection Procedure

Uploading Field Data

Data collected in the field using electronic field forms (Appendix 3) are uploaded to the formhub.org server the same day that the overnight samples are shipped to ensure the chain-of-custody sequence is maintained. If a paper version of the field form (Appendix 4) is used, the information and data must be transferred/entered into an electronic field form as soon as possible. Data from the field form are synchronized with the databases in each of the USGS laboratories participating in the SCoRR study by way of the online server and a laboratory ODK form, which will be completed by laboratory staff receiving the samples. Information from the field form, such as field parameters, collection date and time, and field photographs, will be available to laboratory staff as they log in samples.

Shipping Samples

The foam shipping coolers supplied to field crews can hold two sample sets and the appropriate amount of ice (or frozen gel packs). Ideally, two samples would be collected per field trip; however, if two cannot be sampled in a day, it is important to note the maximum time each container can be stored in order to not compromise the analytical results (table 2 outlines the shipping requirements for each sample container). Before the box is sealed, a SCoRR Cooler Inventory Form (Appendix 5) is completed and included with the shipment. The cooler boxes are prelabeled with overnight shipping return labels, indicating the respective laboratories and required overnight shipping; boxes can be shipped from any of the contracted courier's staffed location or using their pick-up service.

Figure 18 portrays the chain of custody for samples collected for the SCoRR project, and table 3 lists the shipping addresses for each laboratory to which samples are being

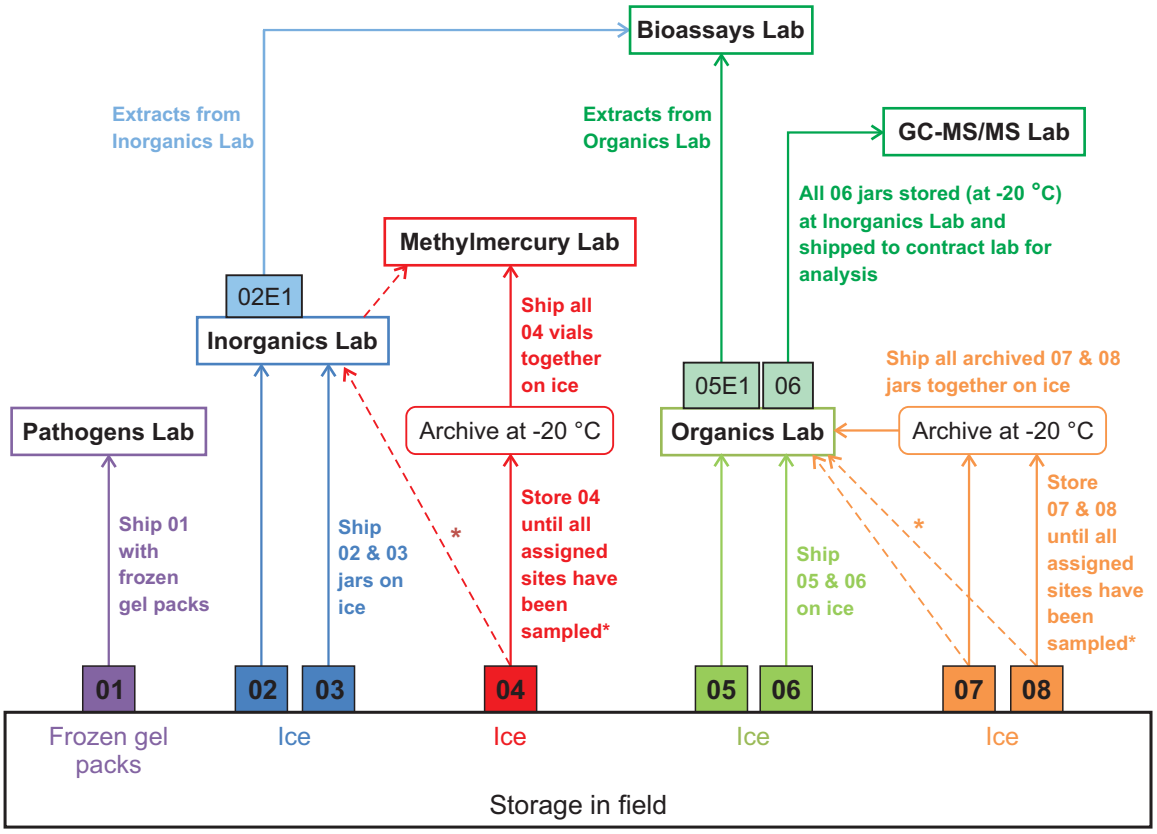

Figure 18. Diagram showing chain of custody for samples collected for the Sediment-bound Contaminant Resiliency and Response (SCoRR) study. (*, If -20 °C storage at the field office is not possible, ship sample container 04 with containers 02 and 03 in same cooler overnight to the Colorado laboratory and the chemistry archive samples (containers 07 and 08) with containers 05 and 06 in same cooler overnight to the Kansas laboratory.)

Table 3. Shipping destinations and preservation for samples collected under the Sediment-bound Contaminant Resiliency and Response (SCoRR) study.

[ml, milliliters; HDPE, High-density polyethylene]

Analytical method	Sample container	Container code	Destination
Pathogens	50 milliliter (mL) sterile plastic centrifuge tube	01	Dale Griffin U.S. Geological Survey 600 4th Street South St. Petersburg, FL 33701
Inorganics	250 mL plastic HDPE jar	02	William Benzel U.S. Geological Survey Denver Federal Center Bldg. 810, ENT E-11, MS-973 Denver, CO 80225
	50 mL sterile plastic centrifuge tube	03	
Methylmercury	50 mL sterile plastic centrifuge tube	04	U.S. Geological Survey Methylmercury Research Laboratory 8505 Research Way Middleton, WI 53562
Organics	250 mL baked amber jar with Teflon-lined cap	05	Keith Loftin U.S. Geological Survey Organic Geochemistry Research Lab 4821 Quail Crest Place Lawrence, KS 66049
	250 mL baked amber jar with Teflon-lined cap	06	
Chemistry archive	250 mL plastic HDPE jar	07	Keith Loftin U.S. Geological Survey Organic Geochemistry Research Lab 4821 Quail Crest Place Lawrence, KS 66049
	125 mL baked amber jar with Teflon-lined cap	08	

submitted for the 2015–16 sampling season. Samples being sent to the USGS pathogens laboratory in St. Petersburg, Florida, have a short hold time (12 hours) and must be shipped by overnight transport on the same day they are collected. When shipping these vials, use two frozen gel packs (one on top and one on bottom), insert foam cover, close box, and tape seams with packing tape (fig. 19A). In the event, gel packs cannot be kept frozen (for example, multiple-day field trips without access to a freezer), ice may be placed in a large zip-top bag, bagged again, and placed on top of the double-bagged sample vial(s) for shipping.

Samples being sent to the USGS laboratories in Denver, Colorado, and Lawrence, Kansas, for inorganic and organic geochemistry analyses, respectively, should be shipped by overnight transport on the same day of collection but may be stored at -20 degrees Celsius (°C) for up to 24 hours if additional samples are to be collected and shipped together the following day. When shipping these containers, line the inside of the large foam shipping coolers with a plastic bag, place jars in the appropriate box (sets of containers 02 and 03 should be in the same bag and are shipped together to Denver, Colorado; sets of containers 05 and 06 should be shipped together to Lawrence, Kansas), fill with ice to just below cooler edge, tie off the bag, insert foam cover (the lid must be closed completely and fit snug), close box, and tape seams with packing tape (fig. 19B).

Should shipment be delayed and a freezer is not available, it will be necessary to fill the standard cooler being used to transport the samples with ice. Samples collected for inorganic and organic geochemistry analyses, as well as chemistry archives, are to be kept in a cooler with an abundance of ice or stored in a freezer set to a constant temperature of at least -20 °C until they can be shipped (except for soil moisture sample vial (container 03), which can be kept in refrigerator or on ice). For samples collected for pathogen analyses, vials should be triple-bagged with zip-top bags and kept completely surrounded by ice; DO NOT freeze pathogens vial (container 01).

All sample containers labeled 06 will be stored at -20 °C at the organics laboratory until sampling is complete, at which time the jars will be forwarded to a contracted laboratory for gas chromatography tandem mass spectroscopy analyses (GC–MS/MS) (fig. 18). Samples collected for methylmercury analysis (container 04) at the USGS Middleton, Wisconsin, laboratory are to be stored at -20 °C until all sampling has

Figure 19. Examples of foam shipping coolers packed to efficiently utilize space and minimize breakage for *A*, pathogen samples, and *B*, organic geochemistry samples (use the same method for inorganic geochemistry samples [not shown]).

been completed by the field office. The vials should then be shipped, on ice, in a single cooler by overnight transport along with an inventory form found on the laboratory website (http://wi.water.usgs.gov/mercury-lab/forms.html). Similarly, samples collected for chemistry archive (containers 07 and 08) should be stored at -20 °C until all sampling has been completed by the field office, then the jars can be packed in coolers, on ice, and shipped to the USGS organics laboratory in Lawrence, Kansas, by overnight transportation. If -20 °C storage at the field office is not possible, ship sample container 04 with containers 02 and 03 in same cooler overnight to the Colorado laboratory and the chemistry archive samples (containers 07 and 08) with containers 05 and 06 in same cooler overnight to the Kansas laboratory. Including the additional containers will result in only having room for one sample set per foam shipping cooler.

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Glossary

Bed sediment Sediment covered by water (referred to as bottom material in USGS National Field Manual).

Composite A sample made up of multiple grab samples from subareas.

Homogenize To mix thoroughly such that all media from composites are uniformly distributed.

Site An area around the station from which samples are collected using determined sampling pattern.

Station A specific GPS location. Stations are given unique station identification numbers and are the reference location for the sample site.

Subarea The divided area based on a sample pattern from which a single composite sample will be collected.

Surficial sediment Sediment exposed to the atmosphere (not covered by water), for example, beach sand.

Appendixes 1–5

Appendix 1. SCoRR Standard Operating Procedure quick reference guide

Field Measurements

- Prior to sampling, position crew at desired coordinates to complete electronic field form.
  - Multi-parameter QW: Water temp., SC, pH, DO mg/L, DO %-saturation, Turbidity.
  - NIST Certified Thermistor: Soil Temperature (if applicable), Air Temperature.
  - Depth to bottom (if applicable).
  - Alternate coordinates, if necessary, or if proposed coordinates are incorrect.
  - Note weather: wind, precipitation, cloud cover, and other environmental conditions near sampling site.
  - Include a minimum of three photos of the site/sampling location.

Media	Parameter	Units	USGS parameter code	Reference figure
Soil or sediment	Soil temperature	°C	--	Figure 5
Air	Air temperature	°C	00020	Figure 6
	Weather	--	--	
	Sky	--	--	
	Winds	--	--	
Water (at sample depth)	Depth to bottom	ft	81903	Figure 7
	Water temperature	°C	00010	
	Specific conductance	µS/cm	00095	
	pH	pH units	00400	
	Dissolved oxygen	mg/L	00300	

Sampling Pattern Options

GRID

SPOKE

TRANSECT

Sample Collection

- **Soil or Surficial Sediment**
  - Identify the desired area of sampling making sure not to disrupt or destroy the immediate area.
  - Avoid grasses, stones, and debris. If present, carefully remove with gloved hands or with a clean garden tool without removing top layer of soil or sediment.
  - Sample for pathogens at first station of composite sample.
  - Only sample top **2 cm** of soil or sediment and top **5 cm** of sand.
  - If large particulates and debris cannot be easily removed, use 2mm pore size sieves.
    - Stainless steel sieve for stainless bowl, plastic sieve for plastic bowl.
  - Continue at each composite station until **1 L** of soil is collected per homogenizing bowl.
  - Follow ***Filling Containers*** protocol to complete sample collection.

- **Bed Sediment**
  - Determine whether to use an Ekman (<4' - fine sediment) or Petite Ponar (>4' - coarse sediment); if unknown, Petite Ponar should be used.
    - **CH** = Clean Hands, **DH** = Dirty Hands.
  - DH: Deploy and retrieve sampler
  - CH: Allow flocculent to settle for one minute. Remove overlying water, if necessary.
  - CH: Collect pathogens on first grab; collect from additional grabs if more substrate is needed. The remainder of the top **2 cm/5 cm**, depending on material type, of grab can be distributed to homogenizing bowls.
  - DH: Continue to collect grabs from different locations to avoid re-sampling.
  - CH: Remove upper **2 cm** for sediment or **5 cm** of sand of each grab with Teflon-coated stainless steel spoon until there is **1 L** of sediment in each bowl.
  - Follow ***Filling Containers*** protocol to complete sample collection.

Filling Containers

1. **Pathogens** (01=Pathogens)
   a. 01: Skim 50 mL centrifuge to at least **40 mL** from single grab
      i. Use disposable plastic spatula
      ii. Clean vial and wrap in Parafilm®
      iii. Small zip-top bag → Small foam shipping cooler, Frozen gel packs
2. **Inorganic Geochemistry** (02=Screening, 03=Soil Moisture, 04=Methylmercury)
   a. 02: Fill 250 mL HDPE jar until **¾-full** from **PLASTIC BOWL**
      i. Fill using Teflon coated stainless steel spoon
      ii. Clean rim, close jar
      iii. Large zip-top bag
   b. 03: Fill 50 mL centrifuge **full** from **PLASTIC BOWL**
      i. Fill using Teflon coated stainless steel spoon
      ii. Clean rim, close vial
      iii. Same zip-lock bag as 02 → Cooler chill
   c. 04: Fill 50 mL centrifuge to **20 mL** from **PLASTIC BOWL**
      i. Fill using Teflon coated stainless steel spoon
      ii. Clean rim, close vial
      iii. Small zip-top → Cooler, Ice
3. **Organic Geochemistry** (05 & 06=Organic Geochemistry)
   a. 05 & 06: Fill **two** 250 mL amber glass jars **¾-full** from **STAINLESS STEEL BOWL**
      i. Fill using Teflon coated stainless steel spoon
      ii. Clean rims, close jars
      iii. Place each jar with bubble-wrap sleeve
      iv. Both jars in same large zip-top → Cooler, Ice
4. **Chemistry Archive** (07=Inorganics, 08=Organics)
   a. 07: Fill 250mL HDPE jar **¾-full** from **PLASTIC BOWL**
      i. Fill using Teflon coated stainless steel spoon
      ii. Clean rim, close jar
      iii. Large zip-top bag w/ 08 → Cooler, Ice
   b. 08: Fill 125mL amber glass jar **¾** from **STAINLESS STEEL BOWL**

i. Fill using Teflon coated stainless steel spoon
ii. Clean rim, close jar
iii. Place in bubble-wrap sleeve
iv. Same zip-top bag as 07 → Cooler, Ice

Cleaning and Sanitizing Equipment

- Rinse all equipment at site with tap water to loosen large debris and put in plastic bag to be cleaned later at controlled laboratory environment.
- Wearing nitrile gloves, set up a clean working space using aluminum foil and clean laboratory wipes.
- Rinse all equipment (spoons, bowls, sampler, and sieves) with **Tap** water.
- Using a nylon brush, tap water, and dilute Liquinox™, gently clean surfaces of residual material.
- Rinse with tap water → Rinse with DIW → Wrap in aluminum foil and into a plastic bag to dry.
- **Full sanitization is only needed** for equipment in contact with soil/sediment used to collect a sample for pathogen analysis and should occur prior to use at the office or in the field between sites.
    - Once dry, Wipe all surfaces with **70% Alcohol Wipe** → Wipe all surfaces with **10% Bleach Wipe**.
    - Put equipment in bag with bleach residue (dampness) for at least **30 minutes**.
    - Remove from bag just prior to use.

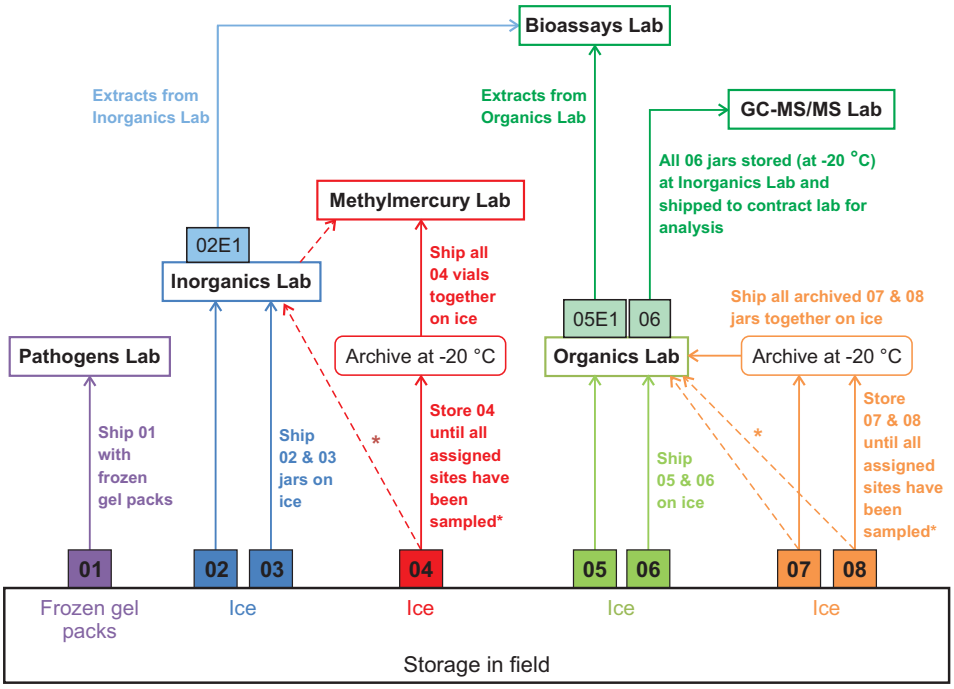
Uploading Field Data

- All field data are to be uploaded the same day as sample collection.
    - Data needs to be available to laboratories while logging samples into the database.
        - This includes field measurements, photos, and any updated information regarding the sample collection or sampling location.
- Upload data directly to the ***formhub.org*** Server.

Shipping

- Used supplied foam shipping containers and appropriate method of chilling/preservation; ice vs. frozen gel.
- ***Be aware of sample holding times!!!** Pathogens with 12-hour maximum holding time.

Analytical Method	Sample Container	Container Code	Destination	Maximum Holding Time	Shipping Preservation
Pathogens	50 mL sterile plastic centrifuge tube	01	St. Petersburg, FL	12 hours	Frozen gel packs
Inorganics	250 mL plastic HDPE jar 50 mL sterile plastic centrifuge tube	02 03	Denver, CO	48 hours 24 hours	Water ice
Methylmercury	50 mL sterile plastic centrifuge tube	04	Middleton, WI	6 months	Water ice
Organics	250 mL baked amber jar with Teflon-lined cap 250 mL baked amber jar with Teflon-lined cap	05 06	Lawrence, KS	24 hours	Water ice
Chemistry Archive	250 mL plastic HDPE jar 125 mL baked amber jar with Teflon-lined cap	07 08	Lawrence, KS	6 months	Water ice



- **01 → St. Petersburg, FL** ship on frozen gel packs day of sampling.
- **02, 03 → Denver, CO** ship on ice together day of sampling.
- **04 → Middleton, WI** stored together at -20°C until sampling complete.
- **05, 06 → Lawrence, KS** ship on ice together day of sampling.
- **07, 08 → Lawrence, KS** stored together at -20°C until sampling complete.

- Ideally sample two sites per field day. If additional day needed to submit two samples together, preserve samples in ice/freezer depending on sample's needs, and ship both sites' samples together the following day. **Do not freeze** pathogens (**01**) vial—triple-bag in additional zip-top bags and keep on ice or in refrigerator until next day.
- Foam shipping boxes are pre-labeled (with contracted courier labels) to respective laboratories; be sure that containers are placed in the proper shipping boxes.
- Line inside of large foam shipping coolers with plastic bag, small layer of ice on bottom, then bagged samples, then more ice to fill bag while allowing the coolers to shut snugly. Tie off plastic liner.
- Include a **SCoRR Cooler Inventory Form** (app. 5) in each cooler to be sent.
- Close cooler lid and seal foam coolers with packing tape along entire seal of cooler lid.
- Seal cooler box and drop off or pick-up from any staffed or scheduled contracted courier location.

Appendix 2. Equipment and Supplies Checklist

All field kits should include the following items (at a minimum):

Personal Protection
- □ Nitrile gloves, non-powdered
- □ Eye protection [goggles or glasses]
- □ Dust masks
- □ Ear plugs
- □ Disposable shoe covers [booties]
- □ Waders
- □ Personal floatation device

Sample-Collection Supplies [per site]
- □ (1) Teflon-coated stainless spoons
- □ (1) Stainless steel trowel, thin blade
- □ (1) Sterile spatula [disposable]
- □ Stainless steel bowl [with 2-liter capacity or larger]
- □ Plastic bowl [with 2-liter capacity or larger]
- □ (2) 250 mL wide-mouth HDPE jars
- □ (2) 250 mL baked amber glass jars, Teflon-lined cap
- □ (1) 125 mL baked amber glass jars, Teflon-lined cap
- □ (3) 50 mL sterile plastic centrifuge tube
- □ Spare set of the 8 sample containers
- □ Spare set of the sample containers labels

Field Equipment
- □ Field device [Garmin Monterra™] with built-in camera, satellite-based GPS, and the SCoRR electronic field form preloaded
- □ Thermometer [for air temp]
- □ Soil thermometer [NIST calibrated]
- □ Handheld GPS [if not built into field device]
- □ Digital camera [if not built into field device]
- □ Paper field forms (for back-up) [Appendix 3]
- □ Engineers ruler
- □ Tape measure [25 ft. or greater]
- □ Measuring wheel
- □ Stainless steel sieve [2 mm pore size]
- □ Plastic sieve [2 mm pore size]
- □ Multiparameter water-quality sonde [YSI 6-series or comparable] equipped with DO, Temp/Cond, and pH sensors for samples overlain by water

Field Equipment for bed sediment sampling
- □ Standard Ekman benthic grab sampler assembly
  - □ (2) Messengers [1 + 1 spare]
  - □ Handle
  - □ Spare trip head
  - □ Spare cable assembly (pair)
  - □ Spare spring hubs (pair)
- □ Petite Ponar benthic grab sampler assembly
  - □ (2) Messengers [1 + 1 spare]
  - □ Carabiner
  - □ (2) Spare release pins
  - □ Spare screens (pair)
- □ Braided line [double the length of the deepest site]
- □ Battery-operated peristaltic pump with tubing
- □ Disposable syringe (at least 60 mL)

Field Supplies
- □ Zip-top bags, small [roughly 6 in. x 6 in.]
- □ Zip-top bags, large [roughly 11 in. x 11 in.]
- □ (1) Bubble wrap sleeve, small [for 125 mL jar]
- □ (2) Bubble wrap sleeve, large [for 250 mL jars]
- □ Plastic drop cloth
- □ Nylon brush [for cleaning]
- □ Carboy of deionized (DI) water
- □ Squeeze bottle for DI water
- □ Carboy of tap water
- □ Alcohol wipes [70% ethanol]
- □ Bleach wipes [10%]
- □ Parafilm® strips [1.5 in. x 3 in.]
- □ Solution of Liquinox™ [0.2% by volume **max**]
- □ Aluminum foil [wide enough to cover bowl]
- □ Waterproof pens
- □ Laboratory wipes
- □ Cooler, large with ice
- □ Sterile soil for blanks
- □ Trash bags

Shipping Supplies [per site]
- □ (3) Shipping Forms, printed (Appendix 4)
- □ (2) Foam shipping coolers, large
- □ (2) Plastic coolers bags [for large foam coolers]
- □ Foam shipping cooler, small
- □ (2) Frozen gel packs
- □ Packing tape

****ALWAYS BRING A SPARE SET OF SAMPLE CONTAINERS IN THE FIELD****

Appendix 3. SCoRR Field Form—electronic version template

[Electronic field form created using Open Data Kit (*https://opendatakit.org/*) and installed on the Android-based Gar Monterra™.]

State	Preloaded, select from drop-down
SCoRR ID	Preloaded, select from drop-down
Station Name	Preloaded, loads when SCoRR ID is selected (NWIS or stakeholder Site Name)
Station ID	Preloaded, loads when Station Name is selected (NWIS IDs [if applicable])
Contact info for site access/permission	Preloaded and displayed along with Station ID and Station Name based on selected station
Sampling Mode	Choose from Resiliency, Response
Field Team Leader	Pre-loaded, select from drop-down with list of WSC project field coordinators
Sampling Personnel (initials)	Text box for initials those in the sampling party
Latitude	Acquired automatically from built-in GPS—standardize; Decimal Degrees
Longitude	Acquired automatically from built-in GPS—standardize; Decimal Degrees
GPS Accuracy	Acquired automatically from built-in GPS—in feet
GPS Datum	Acquired automatically from built-in GPS—should be set to NAD83
Observed Land Use in Vicinity	Entered via drop-down per the Anderson Land-use classification system
Sample Location description	Enter at first visit—subsequently loads when Station Name is selected
Photo(s)	Tap box to activate built-in camera. Require 2: close up of a sample point with reference ruler, and a general location pic. Allow up to 3 more field photos per site.
Sample Medium	Choose from Soil, Sediment—have another field in database automatically populated with NWIS Sample Medium codes (SO or SB)
Sample Collection Method	Choose from Grid, Transect, Spoke, Boat grabs
--if Method = Grid	Prompt for Individual Grid Size (sq. ft.) and Grid Length (ft.)—Range limit "300" for length
--if Method = Transect	Prompt for Distance between Sampling Points (ft.)—Range limit "50"
--if Method = Spoke	Prompt for Distance from Centroid (ft.)—Range limit "150"
--if Method = Boat grabs	Prompt for Sample Area Covered (sq. ft.)
Number of Subsamples Collected for Composite	Range limit ">0"
QC at site?	Choose from None, Replicate—have another field in database automatically populated with NWIS Sample Type codes (9 or 7). If QC at site = Y, duplicate record in database but use Sample Medium code SOQ or SBQ
Air Temperature (°C) [00020]	Range limit "-20–50"
Soil Temperature (°C)	Range limit "-10–50"
Weather	Choose from Clear, Rain, Fog, Snow, Sleet
Sky	Choose from Sunny, Partly Cloudy, Overcast
Wind	Choose from Calm, Breezy, Windy
Surface water overlaying sample?	Choose from Y/N ["Y" adds 6 required fields indicated by +]
+ Depth to bottom (ft.) [81903]	Range limit "lower limit 0"
+ Water Temperature (°C) [00010]	Range limit "-10 to 50"
+ Specific Conductance (µS/cm) [00095]	Range limit "0–50000"
+ pH [00400]	Range limit "1–14"
+ Dissolved oxygen (mg/L) [00300]	Range limit "0–20"
+ Sediment Sampler Type	Choose from Ponar, Ekman, other
Sample Container ID number	Scan or type-in each bottle ID(s) before filling in field with option to enter manually; Two sets of numbers to denote site and analyses (error will display if bottle Serial numbers don't match); Example: XXXXX–01, XXXXX–02 where 'XXXXX" is the sample suite's Serial number and container 01, etc. is the analyses code (01 = Pathogens, 02 = Inorganic Geochemistry, etc.)
Sample Date	Acquired automatically from computer when Containers are scanned
Sample Time	Acquired automatically from computer
Field Notes	Text box to enter field notes—entry not required
Date and Time Field Form uploaded	Acquired automatically from computer when finalized and uploaded to server

Appendix 4. SCoRR Field Form—manual entry template

U.S. GEOLOGICAL SURVEY
Sediment-bound Contaminant Resiliency and Response (SCoRR)
Field Form—Manual Entry
PLEASE COMPLETE ENTIRE FORM

Station Name _____

Station ID _____ **Location** _____

Latitude _____ **Longitude** _____ **GPS accuracy (ft.)** _____

GPS Datum _____ **Observed Land Use (circle one):** Undevel. Ag. Res. Comm. Ind.

Observed Land Use Density (circle one): None H M L **Sampling Personnel** _____

Sample Date _____ **Sample Time** _____ ("Standard" time—account for Daylight Savings)

Sample Medium (circle one): Soil Sand Bed Sed. Marsh Sed. **Total Sample Area (sq. ft.)** _____

Sample Method (circle one): Grid Transect Spoke Grab Other **Number of Subsamples** _____

QC at site? None Replicate Field Spike **Sampling Mode:** Resiliency Response

Physical Parameters

Air Temperature (°C) [00020] _____ **Weather (circle one):** Dry Rain Fog Snow

Sky (circle one): Clear Partly cloudy Overcast **Winds (circle one):** Calm Breezy Windy

If water overlays sample, collect water parameters:

Depth to bottom (ft.) [81903] _____

Water Temperature (°C) [00010] _____

Specific Conductance (µS/cm) [00095] _____

pH [00400] _____

Dissolved oxygen (mg/L) [00300] _____

For Soil, Sand, and Marsh Sediment samples:

Soil Temperature (°C) _____

For Bed Sediment samples:

Sediment Sampler Make/Model _____

Sediment Sampler Type _____

Sample Container ID _____**-XX** **Filled:** □ 01 | □ 02 | □ 03 | □ 04 | □ 05 | □ 06 | □ 07 | □ 08

Photo(s) file name _____

Field Notes _____

_____

Transpose this form into the SCoRR Electronic Field Form application as soon as possible (and retain for records)

Appendix 5. SCoRR Cooler Inventory Form

Contents of Shipping Cooler
USGS Sediment-bound Contaminant Resiliency and Response (SCoRR) Pilot Study

Select Lab/Contact:

□ **Pathogens** | Dale Griffin □ **Inorganics** | William Benzel □ **Organics** | Keith Loftin □ **Archives** | Keith Loftin

Sample-set Identifier	Container(s) Identifier	SCORR ID	Sample Date (yyyymmdd)	Sample Time (hhmm)	Soil Temperature, field (°C)	Shipped with
ex: NY-999999	*02 & 03*	*NY-03246*	*20150701*	*1300*	*21.0*	*ice*

Chain of Custody
WSC Point of Contact: _____ phone: _____ email: _____

Sampling Personnel: _____

Signature: _____ **Date Shipped:** _____

Lab log-in: _____ phone: _____ email: _____

Signature: _____ **Date Received:** _____

Version 1 (2015)

For additional information:
U.S. Geological Survey
Toxic Substances Hydrology Program
12201 Sunrise Valley Drive
Reston, Virginia 20192

Or visit the U.S. Geological Survey Environmental Health Web site at:
http://www.usgs.gov/envirohealth/

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