

<i>Title:</i> AOS Protocol and Procedure: Sediment Chemistry Sampling in Wadeable Streams		<i>Date:</i> 07/02/0214
<i>NEON Doc. #:</i> NEON.DOC.001193	<i>Author:</i> C. Roehm	<i>Revision:</i> A

## AOS PROTOCOL AND PROCEDURE: SEDIMENT CHEMISTRY SAMPLING IN WADEABLE STREAMS

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## 1 DESCRIPTION

### 1.1 Purpose

The primary purpose of this document is to provide a change-controlled version of Observatory protocols and procedures. This document provides the content for training and field-based materials for NEON staff and contractors. Documentation of content changes (i.e. changes in particular tasks or safety practices) will occur via this change-controlled document, not through field manuals or training materials.

This document is a detailed description of the field data collection, relevant pre- and post-field tasks, and safety issues as they relate to this procedure and protocol.

### 1.2 Scope

This document relates the tasks for a specific field sampling or laboratory processing activity and directly associated activities and safety practices. This document does not describe:

- General safety practices
- Site-specific safety practices
- General equipment maintenance

It does identify procedure-specific safety hazards and associated safety requirements.

### 1.3 Acknowledgments

This protocol is based on modified versions of the United States Geological Survey (2006), United States Geological Survey (1994), and U.S. Environmental Protection Agency (1994).

## 2 RELATED DOCUMENTS AND ACRONYMS

### 2.1 Applicable Documents

Applicable documents contain information that shall be applied in the current document. Examples are higher level requirements documents, standards, rules and regulations.

AD [01]	NEON.DOC.004300	EHS Safety Policy and Program Manual
AD [02]	NEON.DOC.004316	Operations Field Safety and Security Plan
AD [03]	NEON.DOC.000724	Domain Chemical Hygiene Plan and Biosafety Manual
AD [04]	NEON.DOC.001155	NEON Training Plan
AD [05]	NEON.DOC.050005	Field Operations Job Instruction Training Plan

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## 2.2 Reference Documents

Reference documents contain information complementing, explaining, detailing, or otherwise supporting the information included in the current document.

RD[01]	NEON.DOC.000008	NEON Acronym List
RD[02]	NEON.DOC.004300	EHS Safety Policy and Program Manual
RD[03]	NEON.DOC.005003	NEON Scientific Data Products Catalog
RD[04]	NEON.DOC.014051	Field Audit Plan
RD[05]	NEON.DOC.001152	NEON Aquatic Sample Strategy Document
RD[06]	NEON.DOC.000824	Data and Data Product Quality Assurance and Control Plan
RD [07]	NEON.DOC.001153	AOS Protocol and Procedure: Stream Morphology Mapping
RD [08]	NEON.DOC.001154	NEON Aquatic Decontamination Protocol

## 2.3 External References

External references contain information pertinent to this document, but are not NEON configuration-controlled. Examples include manuals, brochures, technical notes, and external websites.

ER [01]	
ER [02]	
ER [03]	

## 2.4 Acronyms

Acronym	Definition
Cm	Centimeter
G	Gram
km <sup>2</sup>	Square kilometer
L	Liter
μL	Microliter
mL	Milliliter
Mm	Micrometer
Mm	Millimeter
M	Meter
O	Organics
TE	Trace Elements
M	Metals
SS	Sediment Size

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## 2.5 Definitions

A **protocol** is a formal summary description of a procedure and its related rationale, and includes information on knowledge and resources needed to implement the procedure. A **procedure** is a set of prescribed actions that must take place to achieve a certain result, and can also be called a method. It differs from a science design in that science designs provide a more complete description of the rationale for selecting specific protocols. It differs from a training manual in that training manuals provide materials in support of skills acquisition in the topic areas including information on how to best train staff rather than detailing only the steps of the procedure.

Decant: To draw off (a liquid) without disturbing the sediment or the lower liquid layers.

Depositional Zone: Area where sediments settle and accumulate on the stream bottom.

Flow Regime: A stream's naturally occurring changes in water flow through the course of a year.

Pool: A stretch of a stream in which water velocity is low and the water depth is above average.

Reach: A stretch of a stream which includes riffles, pools and runs. NEON defines this area as the permitted reach for sampling.

Riffle: A stretch of a stream denoted as shallow and coarse bedded where water flows at a higher velocity and turbulence.

Run: A stretch of a stream where water flows smoothly.

Stage: The water level above some arbitrary point in the stream.

Thalweg: Deepest part of the stream or channel, usually the line of fastest water flow.

## 3 BACKGROUND AND OBJECTIVES

### 3.1 Background

Sediment is a naturally occurring material that originates from the weathering and erosion of rock. It can be subsequently transported and redistributed by wind, water and ice. Sediments in aquatic environments and as defined for this protocol constitute the bed material of wadeable streams. Sediments are defined as the fraction smaller than 2 mm in grain size.

Sediments are sinks for elemental cycles in aquatic systems and are recognized as one of the largest sources of pollutants. Therefore, sediments provide information on various processes such as sedimentation, water dynamics, sediment contaminant interaction, sediment-organism interaction and historical indicators (IAEA, 2003). Under certain conditions, sediments can be re-suspended within the water column and transported downstream or elsewhere in the water body. This mechanism allows for trace and other elements and compounds to re-enter the food chain. While this may provide essential nutrients and energy for organisms, it also potentially poses a threat to the aquatic systems.

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The variation in the composition of bed surface sediments results from the inherent heterogeneity of the surrounding watershed and airshed. Many metals and other elements are most likely to be concentrated in sediments typified by fine particle size samples and high organic matter content. This association is largely dependent on the sorptive capacity of fine sediments and organic matter imparted by their surface charges. Hence, even though element concentrations in the water column may be low, suspended sediments and bed sediments can contain large elemental concentrations. Fine bed sediments are most likely to be collected from depositional zones. A depositional zone is defined as the area within a river where the energy regime is low. Such areas are found at the inside bend of a stream, pool or river, downstream from obstacles or simply shallow waters near the shore (USGS, 1996).

Sediment deposition patterns are a direct consequence of the flow characteristics of a waterbody. The appropriate season and hydrologic conditions for sampling stream bed sediment are determined by current and antecedent discharge conditions. Access to the sampling site can be limited during seasonal high-flow conditions. Unusually high flows can wash out, redistribute, or bury substantial parts of sediment deposits; therefore, sampling should be delayed following major discharge events to allow fresh sediment to deposit. When sampling for bed sediment during summer or autumn, low-flow conditions are recommended to provide maximum direct access to the stream bed and to minimize seasonal streamflow variability.

The sample strategy for sediment analysis focuses on fine-grained surficial sediments from natural depositional zones during low-flow conditions (USGS, 1996). Surface sediment is considered to range from 0 to 3 cm in depth (Golterman et al., 1983; Keith, 1991). Samples are composited from between 5-10 depositional zones within a station in the stream reach in order to smooth local scale variability and represent average conditions of the reach. To increase the probability of detecting trace elements and to enhance the comparability of data among sites, bed-sediment samples will be sieved so the fine grained fraction analyzed for the elements of interest. For trace element analysis, the silt-clay fraction smaller than 63 µm will be used. For organics, sand and silt-clay fractions smaller than 2.0 mm will be used.

**3.2 NEON Science Requirements**

This protocol fulfills Observatory science requirements that reside in NEON’s Dynamic Object-Oriented Requirements System (DOORS). Copies of approved science requirements have been exported from DOORS and are available in NEON’s document repository, or upon request.

**3.3 NEON Data Products**

Execution of this protocol procures samples and/or generates raw data satisfying NEON Observatory scientific requirements. These data and samples are used to create NEON data products, which are documented in the NEON Scientific Data Products Catalog, available on the NEON website.

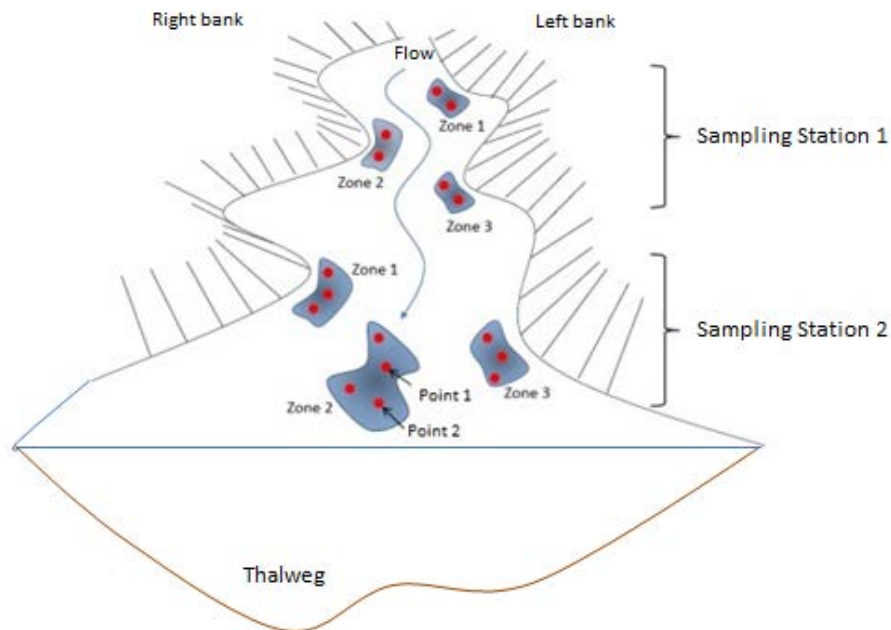


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#### 4      **PROTOCOL**

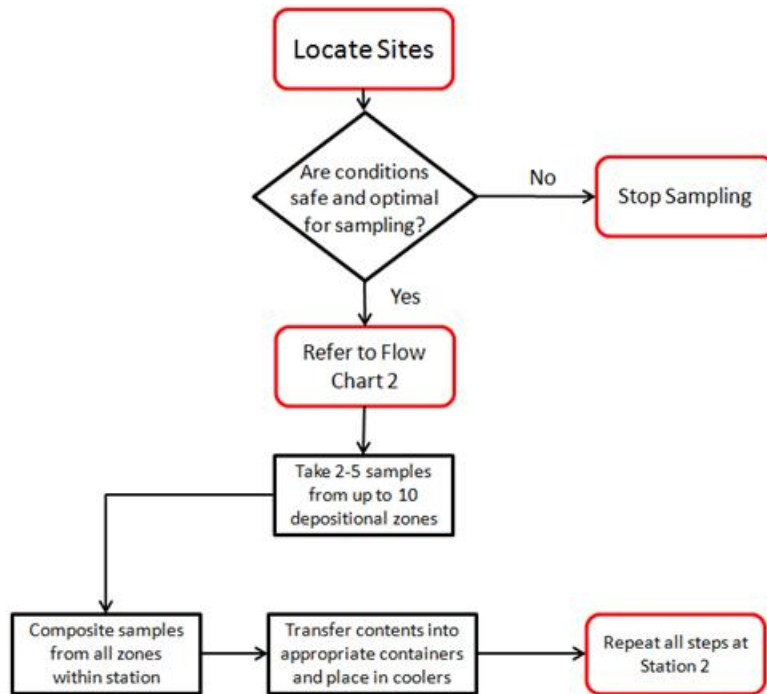
This protocol follows closely USGS (1996).

The spatial distribution of sediment characteristics depends on processes such as flow, turbulence, stream morphology, chemical precipitation and turbidity, on physical factors and on catchment characteristics such as underlying geology, the number and size of inflows and land use (Håkanson and Jansson, 1983; Ashley, 1995; Kumke et al., 2005). In wadeable streams, samples are taken from two stations, each representing composited samples from between 5-10 depositional zones. Depositional zones represent the net outcome of multiple processes and flows. In a given depositional zone, samples are taken at two or five points and composited with samples from other depositional zones within the same station (Fig. 1). The number of samples from each zone will be based on the surface area of each zone (i.e., the larger the area of the zone, the greater the number of subsamples collected). The sampling distribution follows approximately 5 samples per square meter (Fig. 2). If depositional zones are <1 m<sup>2</sup>, 2-3 locations within the zone are sampled. Partially wetted zones (< 5 cm water) should only be sampled when no other sites are available, and sampling conditions should be documented in the field notes and data records as a potential outlier. Each sample consists of the surficial 3 cm of bed sediment. Compositing samples allows for greater representativeness of mean concentrations for the area and results in smoothing of variability otherwise encountered between depositional areas.



**Figure 1.** Identifying the location for sediment sampling in wadeable streams. The two sediment sampling stations should contain depositional zones that represent upstream influences and various flow regimes; left bank (looking downstream), right bank (looking downstream), central channel, and different depths of water.

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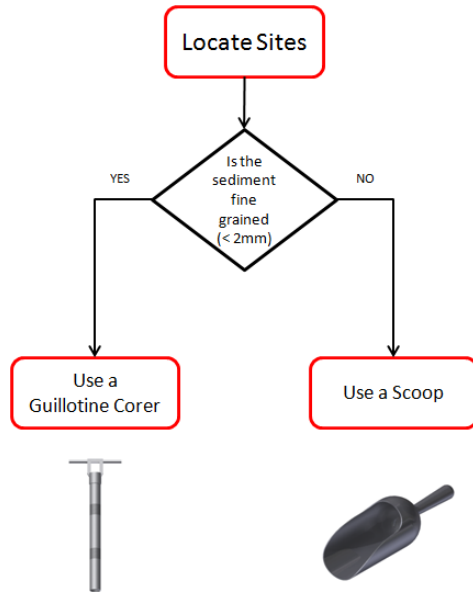


**Figure 2.** Decision flow chart 1 for determining the number of samples to be taken at each location.

The coring systems used in wadeable streams depends on the sediment type. For softer sediments a guillotine corer is used to extract the sediment samples with minimal impact on the surface sediments. For harder sediments a scoop sampler is used (Fig. 3). Where possible samples are taken outside of areas where biological sampling will take place in order to avoid disturbance of benthic habitats. Should this not be possible, samples will only be taken from depositional zones where no biological sampling will take place or following biological sampling.

One sample container per analyte type of sediment is collected at each station, for a total of 4 containers per station. Two stations per wadeable stream are sampled on each sampling date for a total of 8 sample containers per sampling date.

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**Figure 3.** Decision flow chart for determining appropriate sampler to use in the field.

## 5 QUALITY ASSURANCE AND CONTROL

The procedures associated with this protocol will be audited according to the Field Audit Plan (RD[04]). Additional quality assurance will be performed on data collected via these procedures according to the NEON Data and Data Product Quality Assurance and Control Plan (RD[06]).

- 1) Should flow rates change (>25% baseflow) during sampling, stop work for the day and allow for flow conditions to stabilize. Discard all samples.
- 2) Following a major precipitation events and resulting high flow, samples will be taken at least 5 days following a major flow event.
- 3) Samples for organic analysis should be extracted within 14 days.
- 4) Samples for metals, except for mercury, must be analyzed within six months.
- 5) Sediment samples for mercury and nutrients must be analyzed within 28 days.

The following field implementation guidance must be followed to ensure quality standards are met:

- 1) Has there recently (<1 day) been any previous sampling that occurred within the sampling site.
  - a) If YES, go to 2.
  - b) If NO, continue sampling.
- 2) Did any of the previous work stir up sediments or added chemical constituents to the water?
  - a) If Yes, (either or)
    - I) Allow water to clear and disturbance to pass
    - II) Sample in a different location to the disturbance
  - b) If NO, continue sampling.

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## 6 SAFETY

Personnel working at a NEON site must be compliant with safe field work practices as outlined in the Operations Field Safety and Security Plan (AD[02]) and EHS Safety Policy and Program Manual (AD[01]). Additional safety issues associated with this field procedure are outlined below. The Field Operations Manager and the Lead Field Technician have primary authority to stop work activities based on unsafe field conditions; however, all employees have the responsibility and right to stop their work in unsafe conditions.

In addition, the following safety requirements are sought:

- Activities in streams should only be performed when flow conditions are safe. Do not attempt to wade a stream where velocity x depth is  $\geq 10 \text{ ft}^2/\text{s}$  ( $0.93 \text{ m}^2/\text{s}$ ).
- Acid must be stored in acid-safe containment cabinets in compliance with site-specific Chemical Hygiene and Biosafety Manual (AD[06]). Acid vials can be transported to the field in the containment boxes received from the lab, as a means of secondary containment. Wear protective nitrile gloves when dispensing acid preservatives into the appropriate water samples. Used acid preservative vials and gloves should be placed in the appropriate acid waste containers.

## 7 PERSONNEL REQUIREMENTS

We estimate wadeable stream sediment chemistry sampling requires 2 technicians for 4-6 hours each sampling day plus travel to and from the site and 1-2 hours of laboratory work at the Domain Support Facility.

## 8 TRAINING REQUIREMENTS

All technicians must complete required safety training as defined in the NEON Training Plan (AD[04]). Additionally technicians complete protocol specific training for safety and implementation of protocol as required in Field Operations Job Instruction Training Plan (AD[05]).

Personnel are to be trained in wadeable stream sediment chemistry measurements and safe working practices for stream work.

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**9 FIELD STANDARD OPERATING PROCEDURE**

**9.1 Sampling Frequency and Timing**

Sediment chemistry sampling occurs up to 5 times per year at each NEON site. Sediment samples can only be taken when velocity in the wadeable streams is below 0.93 m<sup>2</sup>/s (USGS, 2008). The specific times will be determined using multivariate statistics and site specific historical information provided in the NEON Aquatic Sample Strategy Document (RD[05]).Criteria for Determining Sampling Dates

Sample timing should reflect the influence of chemical and biological inputs and varying chemical conditions. Hence the temporal strategy for NEON samples will include sampling dates reflective of key times of leaf – on, leaf- off and peak greenness. The timing of such samples should reflect the hydrologic nature of the stream and the temporal variability of the system.

The NEON aquatic program will be sampling stream sediments up to 5 times per year per site (other than metals that will be sampled 1 time per year during the summer base flow conditions). Sampling will occur during base flow and/or stable flow conditions to provide maximum direct access to the stream bed and to minimize seasonal streamflow variability. The timing of the sampling is flow dependent. Should a major event occur that disrupts sediments, wadeable stream samples should not be collected for at least 5 days following a major flow event (>25% change in flow within 30 minutes), allowing for the settlement of suspended sediments into depositional environments.

**9.1.1 Sampling Frequency**

Sampling for wadeable stream bottom sediments shall occur up to 5 times per year at the determined sampling dates.

**9.1.2 Sampling Timing Parameters**

Sampling shall occur within +/- 2 weeks of the given dates.

All samples should be taken at the same time each day, regardless of the time of day, +/- 2 hours of previous sampling date.

## 9.2 Equipment and Materials

**Table 1.** Field equipment list.

Maximo Item No.	Item Description	Quantity	Habitat-Specific	Special Handling
	Scoop, Polyethylene	1		
	Scoop, Stainless Steel	1		
MX100309	Hand Corer	1		
	60 mL syringes	2		
	Stopcocks	2		
	50 cm of Tygon tubing 1/8" inner diameter	2		
	Wash Bottle, plastic, 500mL	1		
	Wash Bottle, Teflon, 500mL	1		
	Plastic Funnel 13 oz	1		
	Stainless Steel Funnel 13 oz	1		
	Plastic spatula	1		
	Stainless steel spatula	1		
	Glass Bowl 4 qt.	2		
	4 L jug	1		
	Measuring stick, foldable, plastic	1		
	Field documentation forms and field books	3		
	Ice Pack	6		
	Latex gloves, in plastic bag	multiple		
	Amber glass bottle, 1 L, for organics	2		
	Amber glass bottle, 1 L, for metals	2		
	Amber glass bottle, 1 L, for trace elements	2		
	Plastic bottle, plastic, 1 L, for size analyses	2		
	Foil, aluminum, heavy duty, one roll	1		
	Equipment sealable bags, 5 gal	8		
	Teflon tape	1		
	Electrical tape, roll	1		
	Masking tape, roll	1		
	Trash Bags 13 Gal	4		
	Foam for glass bottles	1		
	Permanent Markers	3		
	Bottle labels	14		
	Amber glass bottles, 1 L, spares	3		
	HDPE bottle 500 mL	1		
	Vaseline bottle	1		
	Brush, scrub, soft nonmetallic	1		
MX100512	Multisonde	1		
	Sieve set, 6 part	1		
	Coolers, shipping, 1 gallon	1		

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Maximo Item No.	Item Description	Quantity	Habitat-Specific	Special Handling
	Coolers, shipping, 5 gallons	1		
	Boots and/or hip waders	2		
	First Aid Kit	1		
	Camera	1		
	GPS, high accuracy	1		

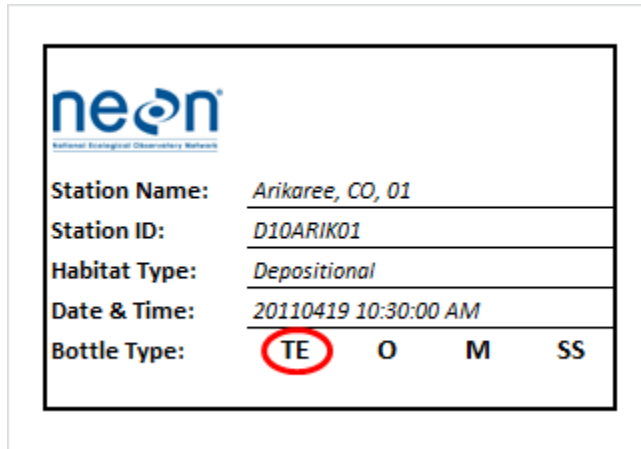
### 9.3 Preparation

- 1) Ensure all equipment has been cleaned appropriately.
- 2) Check the sediment chemistry field sampling kit to make sure all supplies are packed.
- 3) Ensure GPS locations of sampling stations are entered into the GPS system.
- 4) Confirm type of sediment samples that will be collected and take the appropriate bottles and collection devices (Figures 3 and 4). Not all samples will be collected each trip.
- 5) Attach pre-printed labels (Figure 5) to bottles (Figure 4).
- 6) Define the sediment sampling stations, zones and locations based on morphological and sediment maps.
- 7) Place the equipment on the aluminum foil and bags. Be sure not to contaminate bags.
- 8) Insert ice into each cooler.
- 9) Line each shipping cooler with a trash bag.
- 10) Fill out Site information on the forms in Appendix A and the General Field Metadata sheet. Make sure that “Station ID,” “State,” and “Station Name” are correctly filled out on the form. Station ID should be in the form of DXX, where DXX is the NEON Domain number. Form Station ID, Date (YYYYMMDD) and Time must match the chemistry label (Figure 5).



**Figure 4.** Example of Amber bottles used for collecting organics, metals and trace elements and a Teflon bottle for sediment size samples.

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**Figure 5.** Example NEON sediment chemistry label. TE- Trace Elements; O- Organics; M- Metals; SS- Sediment sample

## 9.4 Sample Collection in the Field

Stream sediment samples shall be collected from two identified sediment-sampling stations within the stream reach (Fig. 1). At each of the 2 sampling stations, identify at least 5 wadeable depositional zones containing fine-grained particulate matter. The number of depositional zones will be dependent on stream morphology (see RD[07]). Identification of depositional zones can be aided by using the site characterization stream morphology maps. The areal extent of each zone should be estimated and if possible quantified. Due to the affinity of metals and other nutrients to bind to smaller sized particles (<2 mm), this size fraction found in depositional zones better represents the potential quantification of sediment chemistry. Within each identified depositional zone, up to 5 samples should be taken. If the depositional zones are large (i.e. >1 m<sup>2</sup>), sediment from 5 sample locations per meter squared should be collected. The zones should not interfere with the biological sampling locations when possible.

### 9.4.1 Sampling in Wadeable Streams

- 1) Start at the most downstream station and zone working from downstream to upstream in order to minimize sediment disruption. If sediments are disrupted, wait until the area has cleared before sampling.
- 2) Ensure the General AQU Field Metadata Sheet (RD [10]) is completed.
- 3) Calibrate the multisonde for dissolved oxygen (DO) (see the link to the YSI Manual in the Reference Section).
- 4) Measure the % oxygen, temperature and salinity of the water about 10 cm above the sediment in each sampling zone and note data on the field sampling sheet (Appendix B).
- 5) Note the GPS position of the sampling zone on the field sampling sheet.
- 6) Using a folding measure stick, measure the water depth at the sampling locations within each zone and note the average for each zone on the field sampling sheet (Appendix B).
- 7) Repeat at each sediment sample zone right before taking samples.



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- 8) Rinse all equipment three times with stream water prior to use.
- 9) Take sediment sample (see Sections 9.4.2 and 9.4.3).
- 10) Proceed to next station and repeat steps 1-7.

#### 9.4.2 Sampling with Hand Corer

When the bed sediment is composed of primarily smaller sediment sizes (<2 mm) use the Hand (Hand) corer for collecting sediments.

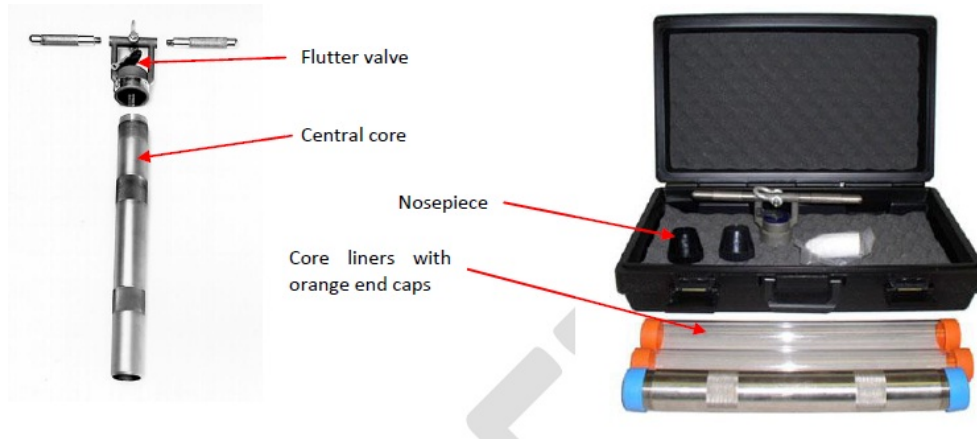


Figure 6. Hand corer for use in wadeable streams with fine sediments.

- 1) Put on latex gloves.
- 2) Approximate the size of the depositional zone and note this on the field sheet.
- 3) Assemble the hand coring sampler: if sampling for metals or sediment size insert a core liner; if sampling for trace elements or organics do not insert a liner.
- 4) Collect samples from 2 to 3 locations (or 5 per m<sup>2</sup>) within each depositional zone.
  - a) Insert sampler into the bed 4 to 5 cm.
  - b) Gently clear the bed material from the exterior of the core.
  - c) Ensure the upper flutter valve is closed and lift the corer above the surface maintaining it as vertical as possible. It may require maintaining the flutter valve wet or add a small amount of Vaseline around the rubber gasket.
  - d) Inspect for adequate fine material; if not appropriate, discard and take another sample.
  - e) Move to the processing area on the stream bank.
  - f) Gently remove the nosepiece and replace with an orange core cap if sampling for metals and sediment size. Otherwise simply cap end of corer with blue cap.
  - g) Release the valve and remove the core liner from the sampler and cap with another orange cap (if using a liner) if not simply unscrew the top of the corer and cap with another blue cap.
- 5) Immediately record observations regarding the appearance of the sediment (i.e., texture, color, odor, presence of biota, presence of detritus, and the depth of sediment sampled). Take a photo.

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- 6) Repeat 1-5 the process at 5 points (or 5 per m<sup>2</sup>) within each depositional zone.
- 7) Composite the samples from each of the zones in glass bowl B with a metal spatula ensuring approximately the same amount of sediment is contributing from each zone.
- 8) Use the funnel to distribute the composite sample into the 1 L collection bottles for the major and trace elements and organics.
- 9) If the sample contains many large pebbles and cobbles, sieve the sample prior to transfer to the sample bottle using the US-5 (4000 μm) mesh.
- 10) Repeat steps 1-9 using a plastic spatula and funnel to distribute the composite sample into the 1 L collections bottles for metals and sediment size.
- 11) Collect a total volume of approximately 2 L of wet sediment per Station (enough to fill one 1 L glass bottle for each of O, TE, or M (when measured) and one 1 L plastic bottle for SS), based on the tools used for extraction and homogenization.
- 12) Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles.
- 13) Collect a 4 L jug of native water to take back to the lab for sample preparation.
- 14) Clean the sampler with the scrub brush.
- 15) Proceed to section 9.5 and 9.6 for sample treatment and shipping preparation in the field.

**NOTE: One sample container per analyte type of sediment will be collected at each site other than sediment size, for a total of 4 containers per site. Two sites per wadeable stream are collected on each sampling date for a total of 8 sample containers.**

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### 9.4.3 Sampling Wadeable Streams with a Scoop

When insufficient sediment depth and larger sediment size (>2mm) is encountered, particularly in rocky streams and rock bottomed streams, use the scoop sampler.



**Figure 7.** Scoop sampler for use in wadeable streams.

- 1) Put on latex gloves.
- 2) Approximate the size of the depositional zone and note this on the field sheet.
- 3) Take the glass bowl whilst sampling in the stream in order to minimize the disturbance from entering and exiting the stream.
- 4) Collect samples from 2 to 3 locations (or 5 per m<sup>2</sup>) within each depositional zone.
  - a) Remove the top layer of fine sediment carefully (approximately 1-3 cm) by gently scooping in the upstream direction (ensure the scoop is metal when sampling for trace elements and organics and Teflon when sampling for metals).
  - b) Slowly pour off most of the overlying water over one of the BACK corners of the scoop. Make sure that the top layer of fine sediment is not discarded.
  - c) Inspect for adequate fine material; if not appropriate, discard.
  - d) Place the sediments into the glass compositing bowl.
- 5) Repeat 1-4 the process at 5 points (or 5 per m<sup>2</sup>) within each depositional zone.
- 6) Composite the samples from each of the zones in the glass bowl with a metal spatula ensuring approximately the same amount of sediment is contributing from each zone.
- 7) Use the funnel to distribute the composite sample into the 1 L collection bottles for the major and trace elements and organics.
- 8) If the sample contains many large pebbles and cobbles, sieve the sample prior to transfer to the sample bottle using the US-5 (4000 μm) mesh.
- 9) Repeat steps 1-8 using a plastic spatula and funnel to distribute the composite sample into the 1 L collections bottles for metals and sediment size.

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- 10) Collect a total volume of approximately 2 L of wet sediment per Station (enough to fill one 1 L glass bottle for each of O, TE, or M (when measured) and one 1 L plastic bottle for SS), based on the tools used for extraction and homogenization.
- 11) Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles.
- 12) Collect a 4 L jug of native water to take back to the lab for sample preparation.
- 13) Clean the sampler with the scrub brush.
- 14) Proceed to section 9.5 and 9.6 for sample treatment and shipping preparation in the field.

**NOTE: 1 sample container per analyte type of sediment will be collected at each site other than sediment size, for a total of 4 containers per site. Two sites per wadeable stream are collected on each sampling date for a total of 8 sample containers.**

**9.5 Sample Preservation**

Sediment/native-water samples that need to be processed in the Domain lab (Section 10.3) should be placed on ice (do not freeze) for transport to the local laboratory for further processing. Following processing in the Domain lab these samples should follow the shipping procedures outlined below.

**9.6 Sample Shipping**

Ship samples to the external Laboratory within 12 hours following processing and preservation:

- 1) Pack glass bottles in packing material (foam sleeves) for protection from breaking.
- 2) Place the O, M and SS bottles in 5 gallon cooler with ice packs.
- 3) Place the TE bottles in a 1 gallon cooler without ice packs.
- 4) Place the completed forms in a Ziploc bag and securely tape the bag to the cooler lid, which will help keep the forms dry. Make sure the time and date on the bottle(s) matches the time and date on the form(s) (Appendix A).
- 5) Include a return shipping label with the address and account information so the Lab can return the cooler to NEON. Tie the garbage bag; tape the cooler shut and ship to address provided by NEON.
- 6) Ship samples “Priority Overnight.”

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## 9.7 Data Handling

- 1) Information from the General Field Sampling Datasheet should be entered into excel filed named 'DXX\_WC\_YYMMDD.' .
- 2) All photos, data and notes shall be uploaded and transcribed into the database.

## 9.8 Refreshing the Sampling Kit

Restock the sampling kit (shipping cooler) with new sediment chemistry sampling bottles (with new labels attached), and other equipment and consumables in Table 1.

## 9.9 Equipment Maintenance, Cleaning, and Storage

Decontaminate all equipment according to NEON Aquatic Decontamination Protocol, (RD[08]) before using at another site. For equipment specific leaning see below.

- 1) Ensure **all** equipment is cleaned prior to storage or reuse:
  - a) Prepare a tub with 0.2% phosphate free detergent.
  - b) Wash and soak the equipment for at least 30 minutes.
  - c) Rinse thoroughly with tap water three times and then with de-ionized water three times using new water each time.
- 2) Preparing equipment for **trace-element** sampling:
  - a) Rinse with 5% high purity nitric acid (HNO<sub>3</sub>) solution.
  - b) Rinse three times with deionized water.
  - c) Allow to air dry.
  - d) Store in plastic bags.
- 3) Preparing equipment for **organic-contaminant** sampling:
  - a) Rinse with methanol.
  - b) Rinse with deionized water.
  - c) Allow to air dry.
  - d) Store in aluminum foil inside a plastic bag.

## 10 LABORATORY STANDARD OPERATING PROCEDURE

Sediment samples for several types of analyses need to be processed prior to shipping to an external lab for analysis. For the purpose of analysis the samples will be processed immediately upon return to the Domain lab.

### 10.1 Sample Processing Time

Samples for organic analysis should be extracted within 14 days. Samples for metals, except for mercury, must be analyzed within six months. Sediment samples for mercury and nutrients must be analyzed within 28 days.

Ship samples to the external Laboratory within 72 hours following processing and preservation. All samples should be processed within 1 day from sampling.

### 10.2 Equipment and Materials

Table 2. Laboratory equipment list.

Maximo Item No.	Item Description	Quantity	Habitat-Specific	Special Handling
	Deionized water (DI) (L)	5		
MX100351	Detergent, phosphate free, 0.2% (L)	4		
MX100352	Methanol (CH <sub>3</sub> OH) (L)	1		
MX100581	Hydrochloric Acid (HCL) acid, 5% (L)	3		
MX105734	Nitric Acid (HNO <sub>3</sub> ), 5% (L)	3		
	Plastic wash bottle, for detergent	1		
	Teflon wash bottle, for methanol	1		
	Container, waste, solvent, 2 gallon	1		
	Container, waste, acid, 2 gallon	1		
	Sieve set, 6 part	1		
MX102986	Nitex sieve material, 63 μm	1		
MX100357	Nylon Sieve Frame	1		
	Gloves, latex (box)	1		
	Kimwipes (box)	1		
	Bottle labels	8		
	Forms, analytical request (TBD)	Multiple		
	Amber glass bottle, 500 mL, for organics	2		
	Amber glass bottle, 500 mL, for metals	2		
	Amber glass bottle, 500 mL, for trace elements	2		
	Plastic bottle, plastic, 500 mL, for size analyses	2		

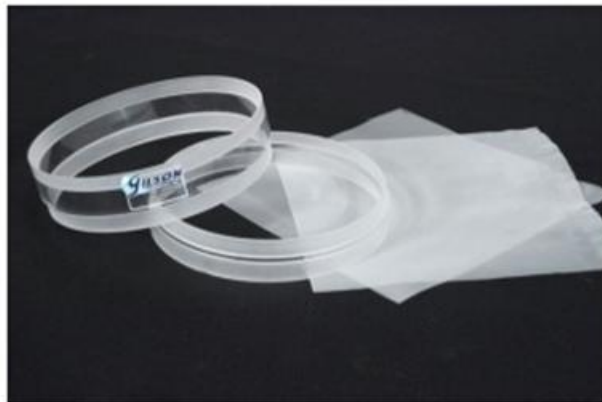
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### 10.3 Sample Processing in the Lab

Wear latex gloves and thoroughly mix (homogenize) the composite sample in the glass bowl.

#### Sieving for Trace Elements

- 1) Stretch the 63  $\mu\text{m}$  mesh nylon-sieve cloth over the plastic-sieve frame and attach retaining ring (Figure 8).
- 2) Assemble in series the 63  $\mu\text{m}$  mesh nylon cloth sieve and the plastic funnel over a 500 mL plastic receiving bottle.
- 3) Decant the stream water from the TE bottle and place in a wash bottle.
- 4) Place a small amount of composite sample from the TE bottle onto the 63  $\mu\text{m}$  mesh nylon sieve with the spatula.
- 5) Pressure sieve the sample using stream water in the wash bottle. The fine sediments pass through the sieve with a stream of water (pressure sieved) delivered by the wash bottle.
- 6) Work small amounts of bed material through the sieve at a time, discarding the material remaining on the sieve. It is not necessary to sieve all the material that is less than 63  $\mu\text{m}$  in each aliquot.



**Figure 8.** Sieve mesh and sieve frame for sediment sample processing.

**NOTE: Shaking the sieve will help separate the fines.**

- 7) If additional wash water is needed, allow the sieved sediment/native water to settle several minutes and decant only the native water back into the wash bottle for reuse.
- 8) If not enough material is collected or if the liquid in the jar is clear or contains minimal fine sediments, filter through a 2 mm mesh instead.
- 9) If sieving through a 0.6  $\mu\text{m}$  mesh is difficult with high organic content, sieve through the 2 mm and then 150  $\mu\text{m}$  mesh first.
- 10) Continue to reuse the native water until the necessary sample is obtained (a depth of approximately 5 cm in the receiving bottle). The analyses of inorganic constituents will require 10 g (dry weight) of sieved sediment.

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- 11) If a large amount of native water remains in the sample with high fines content, decant. Follow instructions for decanting. If no decanting is necessary store and ship the samples according to section 10.4.

### 2 mm Sieving for Organic Contaminant Samples

- 1) Place the 2.0-mm stainless-steel sieve over a 500 mL glass jar.
- 2) Gently work aliquots of the sample through the sieve with a spatula. Do not use water. The bottom of the sieve may require periodic removal of the material that adheres to it, using the appropriate spatula.
- 3) Collect the sample in the jar until nearly full. 500 mL of wet sediment is necessary for analyses of organic contaminants.

### 2 mm Sieving for Sediment Size

- 1) Place the 2.0-mm stainless-steel sieve over a 500 mL Teflon bottle.
- 2) Gently work an aliquot of the sample through the sieve with a Teflon policeman or spatula.
- 3) Collect the sample in a 500 mL bottle. The bottle should be nearly full. 50 g of dry weight of material is needed.

**NOTE 1 cm of fine grained sediment in the bottle will yield approximately 10 g of dry weight sample.**

### Decanting Major and trace Element Samples

- 1) Following sieving, store the sample in a refrigerator and allow the sediments to settle until water is clear. This process could take 2 to 3 days, but no longer than 1 week.
- 2) Decant the liquid to approximately 1 cm above the sediment/water interface with a syringe.
- 3) Centrifuging the sample might be necessary if the fine sediment has not settled within a week.
- 4) Discard the decanted liquid.
- 5) Prepare samples for shipping as per section 10.4.

### 10.4 Sample Shipping

Ship samples to the external Laboratory within 72 hours following processing and preservation:

- 1) Pack glass bottles in packing material (foam sleeves) for protection from breaking.
- 2) Place the O, M and SS bottles in 5 gallon cooler with ice packs.
- 3) Place the TE bottles in a 1 gallon cooler without ice packs.
- 4) Place the completed forms in a Ziploc bag and securely tape the bag to the cooler lid, which will help keep the forms dry. Make sure the time and date on the bottle(s) matches the time and date on the form(s) (Appendices A-C).



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## APPENDIX A FIELD DATA SHEETS

The following field data sheets serve as a backup procedure for times when electronic data collection devices (PDA) are not available.

**General Field Sampling Data Sheet**

**Domain ID:** \_\_\_\_\_

**Site ID:** \_\_\_\_\_

**Date (YYYYMMDD):** \_\_\_\_\_

**Collectors' Name(s):** \_\_\_\_\_

**Team Lead:** \_\_\_\_\_

**Arrival Time (ex. 13:26 MST):** \_\_\_\_\_

**Departure Time:** \_\_\_\_\_

**\*Be sure to include the time zone and standard or daylight savings time, e.g., MST**

**Initial stage (m):** \_\_\_\_\_

**End stage (m):** \_\_\_\_\_

---

**Weather**

**Wind (mph):**    Calm    Breezy    Windy    Gusty

**Sky:**    Clear    Partly Cloudy (25, 50, 75%)    Overcast

**Precipitation:**    None    Mist/Fog    Light Rain    Rain    Heavy Rain    Sleet    Snow

**Previous rain event within 48 hours (Y/N):** \_\_\_\_\_

**Evidence of previous rain event:** \_\_\_\_\_

**Air Temperature (F):**    30s    40s    50s    60s    70s    80s    90s    >100

**Water Color/Clarity:**    Clear    Blue    Green    Brown    Gray

**Riparian Phenology:**    no leaves    breaking buds    leaves    increasing leaf size  
    colored leaves    falling leaves

**General Comments:**

**Post Field Work Observations (Check and comment if observed in channel):**



## NEON Sediment Collection

Wadeable Streams

Domain and Site: \_\_\_\_\_  
 Station ID: \_\_\_\_\_  
 Habitat Type: \_\_\_\_\_  
 Sate & Time: \_\_\_\_\_  
 Weather: \_\_\_\_\_  
 Collected by: \_\_\_\_\_

Station ID: \_\_\_\_\_

N<sup>o</sup> of Zones: \_\_\_\_\_

Zone: \_\_\_\_\_ Number of Point Samples: \_\_\_\_\_

	GPS	Water Depth (m)	DO (mg/L)	DO (%)	Conductivity (uS/cm)	Temperature (°C)
1						
2						
3						
4						
5						

Zone: \_\_\_\_\_ Number of Point Samples: \_\_\_\_\_

	GPS	Water Depth (m)	DO (mg/L)	DO (%)	Conductivity (uS/cm)	Temperature (°C)
1						
2						
3						
4						
5						

Zone: \_\_\_\_\_ Number of Point Samples: \_\_\_\_\_

	GPS	Water Depth (m)	DO (mg/L)	DO (%)	Conductivity (uS/cm)	Temperature (°C)
1						
2						
3						
4						
5						

Zone: \_\_\_\_\_ Number of Point Samples: \_\_\_\_\_

	GPS	Water Depth (m)	DO (mg/L)	DO (%)	Conductivity (uS/cm)	Temperature (°C)
1						
2						
3						
4						
5						

Zone: \_\_\_\_\_ Number of Point Samples: \_\_\_\_\_

	GPS	Water Depth (m)	DO (mg/L)	DO (%)	Conductivity (uS/cm)	Temperature (°C)
1						
2						
3						
4						
5						



## NEON Sediment Collection

Wadeable Streams

Domain and Site: \_\_\_\_\_  
 Station ID: \_\_\_\_\_  
 Habitat Type: \_\_\_\_\_  
 Sate & Time: \_\_\_\_\_  
 Weather: \_\_\_\_\_  
 Collected by: \_\_\_\_\_

Station ID: \_\_\_\_\_

Zone: \_\_\_\_\_

	Photo N <sup>o</sup>	Sampler Type	Core Length (cm)	Observations
	1			
	2			
	3			
	4			
	5			

Zone: \_\_\_\_\_

	Photo N <sup>o</sup>	Sampler Type	Core Length (cm)	Observations
	1			
	2			
	3			
	4			
	5			

Zone: \_\_\_\_\_

	Photo N <sup>o</sup>	Sampler Type	Core Length (cm)	Observations
	1			
	2			
	3			
	4			
	5			

Zone: \_\_\_\_\_

	Photo N <sup>o</sup>	Sampler Type	Core Length (cm)	Observations
	1			
	2			
	3			
	4			
	5			

Zone: \_\_\_\_\_

	Photo N <sup>o</sup>	Sampler Type	Core Length (cm)	Observations
	1			
	2			
	3			
	4			
	5			