AOS PROTOCOL AND PROCEDURE:
SEDIMENT CHEMISTRY SAMPLING IN WADEABLE STREAMS

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Change Record

REVISION	DATE	ECO #	DESCRIPTION OF CHANGE
A	07/02/2014	ECO-01126	Initial release
B	11/05/2014	ECO-02271	Minor updates based on feedback from the field. Moved datasheets to NEON.DOC.002419.
C	01/22/2015	ECO-02632	Migration to new protocol template
D	02/25/2016	ECO-03503	Protocol updates based on current external lab arrangements and revisions following the technician review
E	03/09/2017	ECO-04465	CM updated with new template and changes based on feedback from FOPS. Added TOC/TC sample methods.
F	09/29/2017	ECO-05341	Major updates include: sediment sampling shall occur twice per year (bout 1 and bout 3). Organic samples for PCB/PAH and TOC/TC shall be collected once per year (bout 3). Expanded equipment cleaning instructions for TOC/TC and for glass equipment. Included additional guidance for using the mobile field device. Added barcode tracking instructions. No field datasheet updates. Equipment list updated.
G	01/03/2019	ECO-05969	Reorganized the sediment sampling, equipment types, and cleaning instructions throughout the SOP. Wadeable stream sampling stations and sampling equipment figures updated. Clarified using multiple equipment types as needed. Included 8 oz. jars for carbon samples; half-filled. Equipment list includes glass spatula and glass jar with PTFE lid to composite carbon samples. Provided a statement acknowledging mitigation of contamination risks from equipment and cleaning residue. Added “Sediment Sampling by Analyte Type” and “Equipment Maintenance, Cleaning, and Storage” sections in the SOP with clear instructions for sampling and cleaning equipment by material and analyte type. Improved cooler packing steps in SOP C and E.2. Water quality only measured once per depositional zone. Added sampling impractical and reach conditions fields. Updated figures.

TABLE OF CONTENTS

1 OVERVIEW 3

1.1 Background 3

1.2 Scope 4

1.2.1 NEON Science Requirements and Data Products 4

1.3 Acknowledgments 5

2 RELATED DOCUMENTS AND ACRONYMS 5

2.1 Applicable Documents 5

2.2 Reference Documents 5

2.3 External References 5

2.4 Acronyms 6

2.5 Definitions 6

3 METHOD 7

4 SAMPLING SCHEDULE 11

4.1 Sampling Frequency and Timing 11

4.2 Criteria for Determining Onset and Cessation of Sampling 11

4.3 Timing for Laboratory Processing and Analysis 11

4.4 Sampling Timing Contingencies 12

5 SAFETY 12

6 PERSONNEL AND EQUIPMENT 14

6.1 Equipment 14

6.2 Training Requirements 20

6.3 Specialized Skills 20

6.4 Estimated Time 20

7 STANDARD OPERATING PROCEDURES 20

SOP A PREPARING FOR DATA CAPTURE 20

SOP B PREPARING FOR SAMPLING 21

SOP C FIELD SAMPLING 25

SOP D DATA ENTRY AND VERIFICATION 34

SOP E SAMPLE SHIPMENT 34

8 REFERENCES 36

APPENDIX A DATASHEETS 37

APPENDIX B QUICK REFERENCES 38

APPENDIX C REMINDERS 41

APPENDIX D ESTIMATED DATES FOR ONSET AND CESSATION OF SAMPLING 42

APPENDIX E SITE-SPECIFIC INFORMATION 43

APPENDIX F SEDIMENT CHEMISTRY ANALYSES, STORAGE CONDITIONS, AND HOLD TIMES 44

LIST OF TABLES AND FIGURES

Table 1. Contingent decisions 12
Table 2. Equipment list – Field equipment list 14
Table 3. Datasheets and mobile applications associated with this protocol 37

Figure 1. Examples of depositional zones in a wadeable stream. 4
Figure 2. Sediment sampling stations at a wadeable stream site. Samples collected from Station 1 "Upstream" represent sediments upstream of the mid-way point of the reach. Station 2 "Downstream" samples are collected downstream of the mid-way point of the reach. 8
Figure 3. Identifying the location for sediment sampling in wadeable streams within the sediment reach. At each station, 5-10 depositional zones (brown blobs) are sampled. Within each depositional zone, up to 5 point samples are collected (black dots). 8
Figure 6. Example of clear Boston round glass jars with PTFE lined lid used for collecting sediment for inorganics, organics (PAH/PCB), and carbon (TOC/TC) analyses. Sediments for grain size samples are collected by filling 2 1-gallon (~3.8L) plastic Ziploc bags about half-full. 21
Figure 7. Sediment collection instruments by sample analyte type. When collecting sediments for inorganics with the hand corer, insert the plastic nosepiece. Note that the stainless steel sieve can be used for any analyte to remove sediments and other material larger than 2 mm. Sediment size samples can be collected from any sampler type. 23
Figure 11. Diagram to exemplify determination of sampling zones based on site differences. A) Ideal sampling set up based on availability of different depositional zones (~1 m^2); B) in sites with only random small pockets of sediment; and C) in sites where most of the stream bed is characterized by sedimentary or organic substrate. 26
Figure 14. Scoop samplers composed of metal, plastic or glass are used for collecting sediment in wadeable streams 29

1 OVERVIEW

1.1 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. As such, sediments provide information on various processes such as sedimentation, water dynamics, sediment contaminant interaction, sediment-organism interaction and historical indicators (IAEA, 2003). Under certain environmental 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 web. While this may provide essential nutrients and energy for organisms, it also potentially poses a threat to the aquatic systems.

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 and typically are found at the inside bend of a stream, riffle, pool lip, downstream from obstacles or simply shallow waters near the shore (USGS, 1994) (Figure 1). Depositional zones represent the net outcome of multiple processes and flows.

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, 1994). Surface sediment is considered to range from 1 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 inorganics and to enhance the comparability of data among sites, bed-sediment samples will be sieved so that the fine grained size fraction is analyzed for the elements of interest.

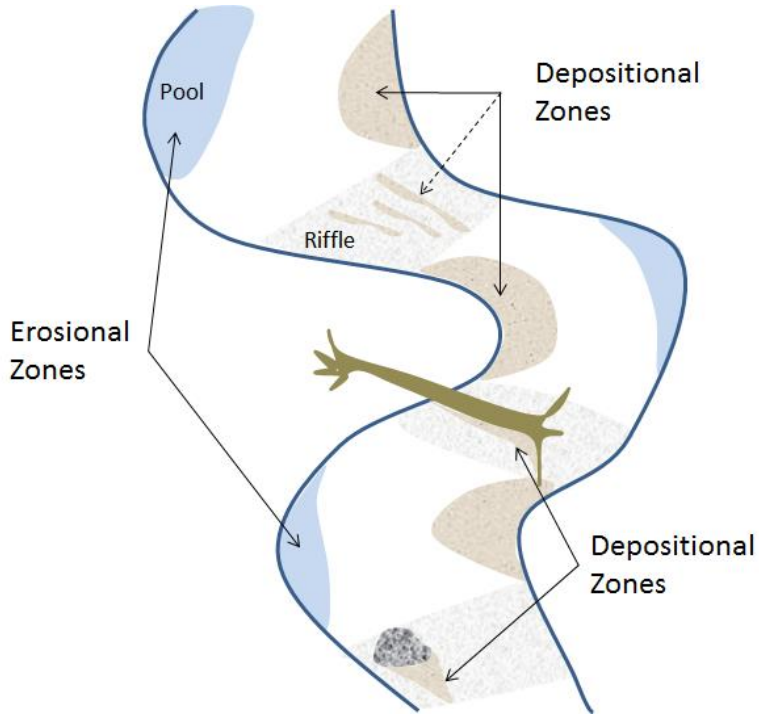

Figure 1. Examples of depositional zones in a wadeable stream.

1.2 Scope

This document provides a change-controlled version of Observatory protocols and procedures. 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.

1.2.1 NEON Science Requirements and Data Products

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.

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, and are documented in the NEON Scientific Data Products Catalog (RD[03]).

1.3 Acknowledgments

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

2 RELATED DOCUMENTS AND ACRONYMS

2.1 Applicable Documents

Applicable documents contain higher-level information that is implemented in the current document. Examples include designs, plans, or standards.

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	Chemical Hygiene Plan and Biosafety Manual
AD[04]	NEON.DOC.050005	Field Operations Job Instruction Training Plan
AD[05]	NEON.DOC.014051	Field and Laboratory Procedures Quality Assurance Plan for Field Operations Activities
AD[06]	NEON.DOC.004104	NEON Science Performance QA/QC Plan

2.2 Reference Documents

Reference documents contain information that supports or complements the current document. Examples include related protocols, datasheets, or general-information references.

RD[01]	NEON.DOC.000008	NEON Acronym List
RD[02]	NEON.DOC.000243	NEON Glossary of Terms
RD[03]	NEON.DOC.005003	NEON Scientific Data Products Catalog
RD[04]	NEON.DOC.001271	AOS/TOS Protocol and Procedure: Manual Data Transcription
RD[05]	NEON.DOC.002419	Datasheets for AOS Protocol and Procedure: Sediment Chemistry Sampling in Wadeable Streams
RD[06]	NEON.DOC.001646	General AQU Field Metadata Sheet
RD[07]	NEON.DOC.001152	NEON Aquatic Sample Strategy Document
RD[08]	NEON.DOC.004257	NEON Standard Operating Procedure: Decontamination of Sensors, Field Equipment, and Field Vehicles
RD[09]	NEON.DOC.001153	AOS Protocol and Procedure: Wadeable Stream Morphology Mapping
RD[10]	NEON.DOC.002494	Datasheets for AOS Sample Shipping Inventory

2.3 External References

ER[01]	YSI Inc. 1998. Handheld Oxygen, Conductivity, Salinity and Temperature System, Operations Manual. ITEM # 038503, Revision E http://www.enviroequipment.com/rentals/PDF/YSI-85-Manual.pdf.

2.4 Acronyms

Acronym	Definition
cm	Centimeter
g	Gram
km^2	Square kilometer
L	Liter
µL	Microliter
µm	Micrometer
m	Meter
mm	Millimeter

2.5 Definitions

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 METHOD

This protocol follows closely USGS (1994).

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). At wadeable streams, samples are collected from an upstream station (Station 1) located above the mid-way point of the reach (Figure 2). A downstream station (Station 2) is located below the mid-way location of the reach. Each station representing composited samples from between 5-10 depositional zones (Figure 3). Compositing samples allow for greater representativeness of mean chemical and physical attributes of each station and smooths the variability of these features.

In a given depositional zone, samples are taken at a minimum of two to a maximum of five points and composited with samples from other depositional zones within the same station (Figure 3). 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 should follow approximately 5 samples per square meter; a minimum of two locations will be sampled with up to five points composited at each station. 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 1-3 cm of bed sediment. See Figure 4 and Figure 5 for a work flow chart for collecting sediment.

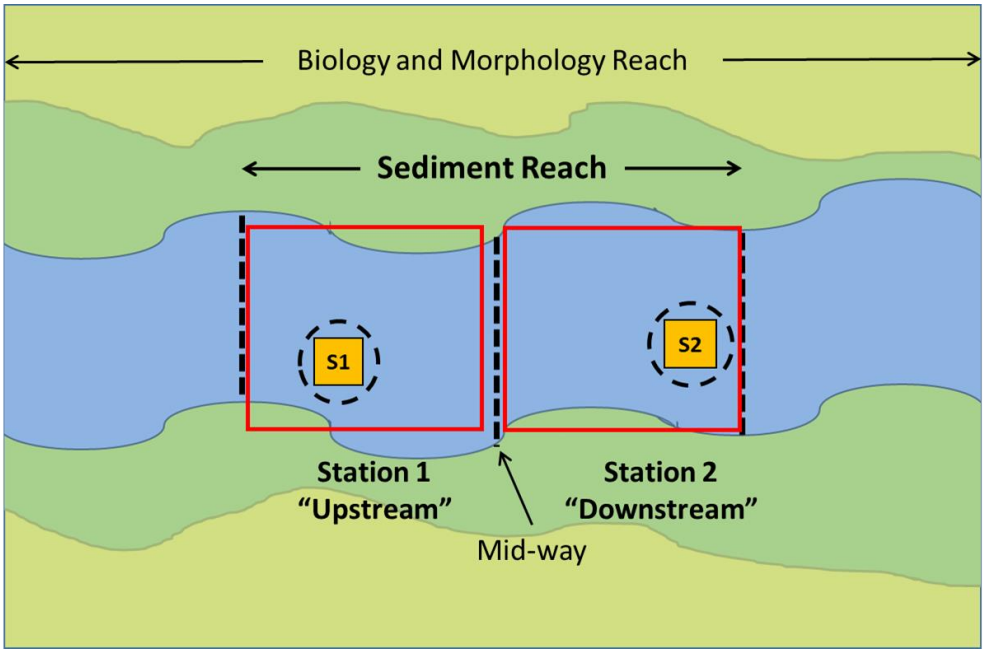

Figure 2. Sediment sampling stations at a wadeable stream site. Samples collected from Station 1 "Upstream" represent sediments upstream of the mid-way point of the reach. Station 2 "Downstream" samples are collected downstream of the mid-way point of the reach.

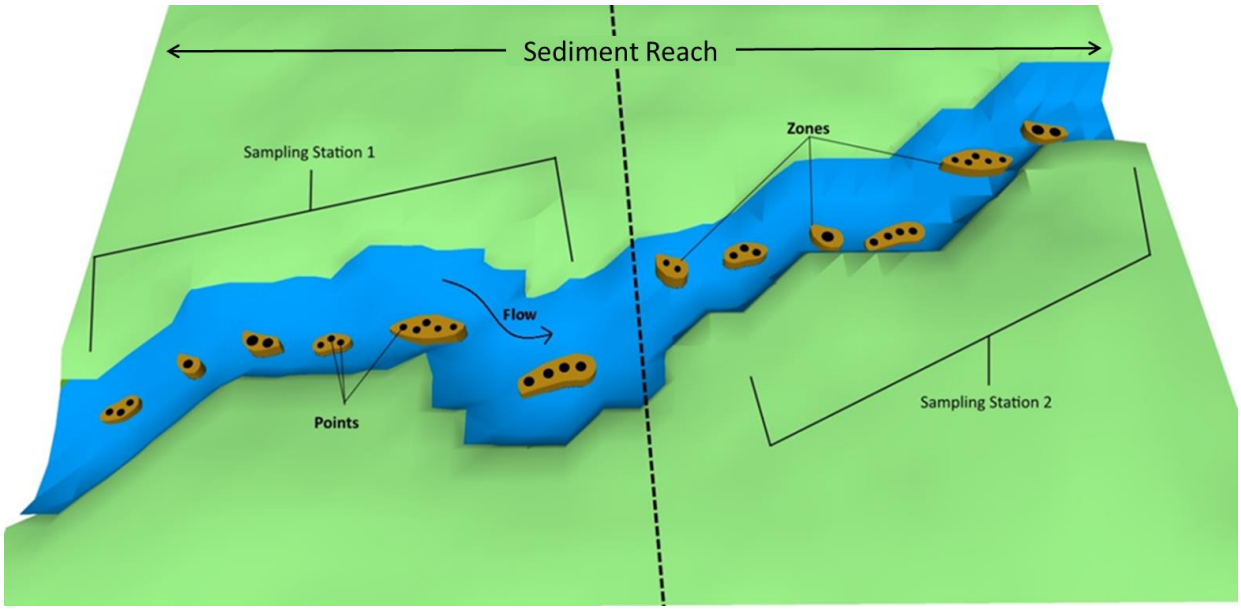

Figure 3. Identifying the location for sediment sampling in wadeable streams within the sediment reach. At each station, 5-10 depositional zones (brown blobs) are sampled. Within each depositional zone, up to 5 point samples are collected (black dots).

The sampling method used in wadeable streams depends on the sediment type. For softer fine sediments (< 2 mm), rich with deep organic material, a hand corer is used to extract the sediment samples with minimal impact on the surface sediments. For harder and shallow substrate with more coarse sediments (> 2 mm) a scoop sampler is used (Figure). Both the hand corer and the scoop can be used throughout the reach within the sampling bout. For example, if sampling in a riffle, the scoop is used to sample coarse sediments, then in a stream run, the hand corer can be used to sample finer sediment material. Be sure to document which tool or tools were used during sample collection. Sediment samples are collected after most of the biological sampling activities, except fish sampling in order to avoid disturbance of benthic habitats.

Two stations per wadeable stream are sampled ***twice per year***; during bout 1 and bout 3 (spring and fall).

Bout 1: Samples are collected for inorganic and sediment size analyses only. Organic contaminants (PAH/PCB) or carbon (TOC/TC) samples are **NOT collected during bout 1.**

- Sample kits will include (one kit per station):
    - One 8 oz. (~250 mL) glass jar for inorganics
    - Four 1-gallon (~3.8 L) plastic Ziploc bags to fill two halfway with sediment (and double bag) for grain size analyses.

Bout 3: Samples are collected and analyzed for **all analytes** including inorganics, sediment size, organic contaminants (PAH/PCB), and carbon (TOC/TC).

- Sample kits will include (one kit per station):
    - One 8 oz. (~250 mL) glass jar for inorganic analyses
    - Four 1-gallon (~3.8 L) plastic Ziploc bags to fill two halfway with sediment (and double bag) for grain size analyses.
    - One 8 oz. (~250 mL) glass jar for collecting sediment for organic analyses (including organic contaminants).
    - One 8 oz. (~250 mL) glass jar for carbon (TOC/TC) analyses **filled half way.**

At each station, when collecting sediments for organic analyses/organic contaminants (PAH/PCB) fill one 8 oz. (~250 mL) round glass jar with sediments and fill another 8 oz. jar for inorganic analyses. When sampling for Total Organic Carbon and Total Carbon (TOC/TC) analyses, fill an 8 oz. glass jar at least half-way with sediment. Fill two-1.0 U.S. gallon (~3.8 L) sealable (Ziploc-style) plastic bags half-way with sediment for physical size analyses. Each bag will be sealed and placed in another sealable gallon-sized plastic bag. Place both bags into a single bag (large garbage bag) and place the sample label and barcode for sediment size samples on the single bag. This method is recommended to prevent completely filling a single gallon-size plastic bag which could open and spill during transport or sample shipping.

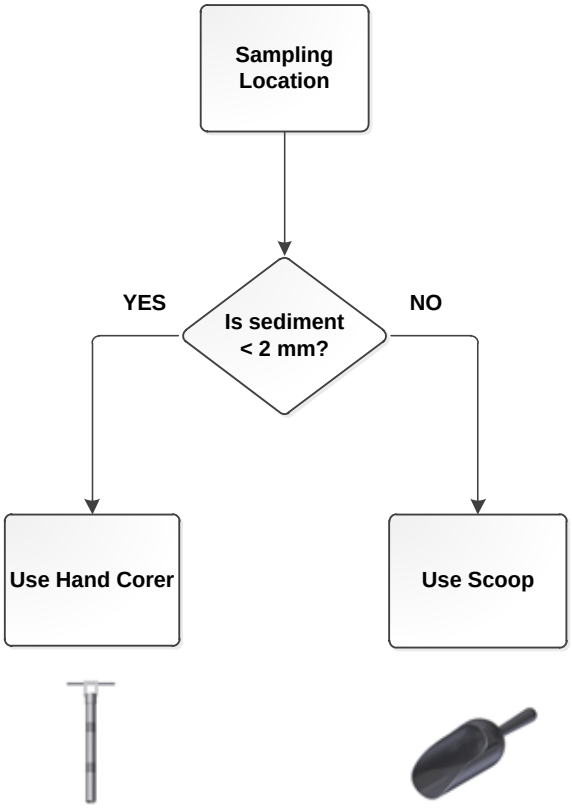

Figure 4. Decision flow chart for determining appropriate sampler to use in the field. **Note:** if the sediments depth is shallow (< 3 cm) the scoop is the more appropriate collection tool.

Standard Operating Procedures (SOPs), in Section 7 of this document, provide detailed step-by-step directions, contingency plans, sampling tips, and best practices for implementing this sampling procedure. To properly collect and process samples, field scientists **must** follow the protocol and associated SOPs. Use NEON's problem reporting system to resolve any field issues associated with implementing this protocol.

The value of NEON data hinges on consistent implementation of this protocol across all NEON domains, for the life of the project. It is therefore essential that field personnel carry out this protocol as outlined in this document. In the event that local conditions create uncertainty about carrying out these steps, it is critical that field scientists document the problem and enter it in NEON's problem tracking system.

The procedures described in this protocol will be audited according to the Field Audit Plan (AD[05]). Additional quality assurance will be performed on data collected via these procedures according to the NEON Science Performance QA/QC Plan (AD[06]).

4 SAMPLING SCHEDULE

4.1 Sampling Frequency and Timing

Sampling for wadeable stream bottom sediments shall occur two times per during the spring and fall sampling bouts. Sediments will be collected during the biological sampling bouts one and three at most sites. In some cases, additional sediment samples may be collected following a large disturbance event (e.g. flood or drought) or other shift in site conditions (e.g. chemical spill). Sampling shall occur within +/- 2 weeks of the given dates. The timing of these samples shall follow the procedures outlined in the NEON Aquatic Sample Strategy Document (RD[07]).

4.2 Criteria for Determining Onset and Cessation of Sampling

The timing of the sampling is flow dependent in wadeable streams. Should a major event occur that disrupts sediments (flooding), wadeable stream samples should not be collected for at least 5 days following a major flow event, allowing for the settlement of suspended sediments into depositional environments and when safe conditions return. All samples should be taken at the same time each day and within +/- 2 hours of the collection time form the previous bout collection time.

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. 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 (>3 times median discharge for the preceding year) to allow time for the settlement of suspended sediments into depositional environments.

Additional sample events may be warranted following a major storm event that alters the morphology of the system. The timing of the sampling is flow dependent. Sediment samples can only be taken when velocity in the wadeable streams is below 0.93 m^2/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[07]).

4.3 Timing for Laboratory Processing and Analysis

For the purpose of analysis, the samples will be processed within 12 hours of return to the Domain lab. It is recommended that the samples are shipped to the external Laboratory within 72 hours following processing. However, if it is expected to take longer to ship the samples, be sure to store them in a refrigerator between 0-6°C (DO NOT FREEZE) until the samples can be shipped. Samples must be shipped to the external lab on ice between 0-6°C but not frozen and arrive within 7 days of sample collection. Ship samples “Priority Overnight.” DO NOT send them “FedEx First Overnight.” If samples are shipped on Friday, send “Priority Overnight with Saturday Delivery.” Be sure to mark the Saturday

delivery box on the FedEx form. It is recommended that samples collected before a major holiday should be stored (refrigerated) at the DSF and shipped after the holiday to avoid shipping delays. Appendix E for a complete list of sediment chemistry analyses, storage requirements, and hold times. Some Domains will ship via UPS and on a recommended range of days (e.g. Monday – Thursday). Refer to the Domain-specific shipping instructions provided by CLA.

4.4 Sampling Timing Contingencies

Table 1. Contingent decisions

Delay/ Situation	Action	Outcome for Data Products
Hours	If sampling stirred up sediments or added chemical constituents to the stream (i.e., gas additions) within the past hour, allow the water to clear and disturbance to pass, sample upstream/upwind of the disturbance. Should flow rates change (>3 times median discharge for the preceding year) during sampling, stop work for the day and allow for flow conditions to stabilize. Discard all samples.	No adverse outcome.
5 or More Days	Following a major precipitation event and resulting high flow, samples will be taken at least 5 days following a major flow event when the stream is at median discharge.	No adverse outcome.

5 SAFETY

This document identifies procedure-specific safety hazards and associated safety requirements. It does not describe general safety practices or site-specific safety practices.

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:

1. Activities in streams should only be performed when flow conditions are safe. Do not attempt to wade a stream where velocity x depth is ≥ 10 ft^2/s (0.93 m^2/s).
2. When handling hazardous products (such as nitric acid) follow laboratory safety standards and have Safety Data Sheet (SDS) readily available to review prior to handling any chemicals. Wear gloves, a laboratory coat and protective eyewear.

3. Personal flotation devices (PFDs) shall be worn when wading in deep streams (per USGS, A Guide to Safe Field Operations). Depth requirements may vary in different regions.
4. In areas with alligators or other submerged wildlife dangers, extra precaution must be taken. The crew will be required to not put hands and feet in the water and to make sure a safe distance is kept from alligators.
5. At extreme cold water sites, additional safety training may be required (example Oksrukuyik Creek) and include the use of special equipment (e.g. floatation jacket or PFD) for added safety.

6 PERSONNEL AND EQUIPMENT

6.1 Equipment

The following equipment is needed to implement the procedures in this document. Equipment lists are organized by task. They do not include standard field and laboratory supplies such as charging stations, first aid kits, drying ovens, ultra-low refrigerators, etc.

Table 2. Equipment list – Field equipment list

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
Durable items							
MX108989	Grainger, W.W.	21TR61	R	Scoop, Polyethylene	Collecting inorganic and size analysis samples	1	N
MX102978	Grainger, W.W.	1YPC3	R	Scoop, Stainless Steel	Collecting organic samples	1	N
	Amazon Capital Services Inc.	B00KMNZ57I B00IPL4FQQ	R	Scoop, Glass (1 oz, 5 oz)	Collecting organic or organic and TOC/TC samples	1	N
MX100309	Ben Meadows Co., Inc.	223583	R	Hand Corer	Collecting samples	1	N
MX108907	Global Equipment Company	WGB548988	R	Stainless Steel Bucket 5 gallons w/ lid	Homogenizing organic samples	1	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
MX100526	Grainger, W.W.	34A216	R	Plastic bucket 5 Gallons w/ lid	Homogenizing inorganic and size analysis samples	1	N
MX102975	Amazon Capital Services Inc.	B0000CF41U B00004SZ7N	R	Glass Bowl 4 qt.	Homogenizing samples	2	N
			R	Glass amber jar with PTFE lid (1 – 4 liter)	Homogenizing samples	1	N
MX100554	Thomas Scientific, Inc.	8939D81	R	60 mL syringes	Syphoning water from samples	2	N
MX101261	Fisher Scientific Company	NC0823056	R	Stopcocks	Syphoning water from samples	2	N
MX100364	Thomas Scientific, Inc. Cole-Parmer Fisher Scientific Company	1207W05 EW0640776 14171212	R	50 cm of Tygon tubing 1/8" inner diameter	Syphoning water from samples	2	N
MX100308	Fisher Scientific Company	0340910E	R	Wash Bottle, plastic, 500mL	Rinsing sediment from samplers into buckets	1	N
MX107128	Fisher Scientific Company	0340912E	R	Wash Bottle, Teflon, 500mL	Decontaminating equipment with methanol	1	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
MX103251	Fisher Scientific Company	1050010	R	Plastic Funnel 16 oz.	Collecting inorganic, nutrient and size analysis samples	1	N
MX107413	Grainger, W.W.	4NCR9	R	Stainless Steel Funnel 64 oz.	Collecting organic samples	1	N
MX100371	VWR	82027472	R	Plastic spatula	Collecting inorganic, nutrient and size analysis samples	1	N
MX100356	VWR	82027502	R	Stainless steel spatula	Collecting organic samples	1	N
			R	Glass spatula	Collecting organic and TOC/TC samples	1	N
MX102975	BioQuip Products Inc.	4748	R	Flexible forceps, featherweight	Removing debris from samples	1	N
MX105823	Forestry Suppliers, Inc.	71112	R	Measuring stick, foldable, plastic	Measuring the water depth at sampling locations	1	N
			R	Field documentation forms and field books	Documenting notes in the field	3	N
			R	Brush, scrub, soft nonmetallic	Cleaning samplers	1	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
MX100514	Thomas Scientific, Inc.	1185K52 15177622	R	Handheld YSI Pro 2030	Measuring % DO, temperature and salinity	1	N
	External Lab		R	Coolers	For shipping, provided by the external lab	1	N
MX100491 MX100494 MX107505	Ben Meadows Co., Inc. Grainger, W.W. Forestry Suppliers, Inc. Cabela's		R	Boots and/or hip waders	Safe wading	2	N
MX102603	Grainger, W.W.	11C657	R	First Aid Kit	Safety	1	N
MX110075 MX102739	Forestry Suppliers, Inc. Cabela's Inc. Recreational Equipment Inc.	39481 IK-270217 895022	R	GPS	Navigating to sampling locations	1	N
MX111388	CDW-G	4452963	R	Mobile field data recording device (Tablet)	Recording data	1	N
Consumable items							

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
	External lab		R	Ice Pack	Keeping samples cool, provided by the external lab	Multiple	N
			R	Nitrile gloves, in plastic bag	Not contaminating samples	Multiple	N
	External Lab		R	Clear Boston-style round glass jar, 8 oz. (~250 mL), for organic, inorganic, and Total Organic Carbon/Total Carbon analyses	Sample container, provided by the external lab	6	N
MX104844	Grainger, W.W.	5LH30	R	Clear Ziploc-style 1-gallon plastic bag for sediment grain size analyses	Sample container, provided by the external lab	4	N
MX100589	Grainger, W.W.	6CHG5	R	Foil, aluminum, heavy duty, one roll	Storing equipment and avoiding contamination	1	N
MX102002	Grainger, W.W.	1JU51	R	Permanent Markers	Labeling samples	3	N
MX103942	Ben Meadows Co., Inc. Forestry Suppliers, Inc.	010510-1 49247	R	Bottle labels	Labeling samples	14	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
			S	Adhesive barcode labels	Labeling sample bottles with barcode-readable labels	1 sheet	N
MX100351	Fisher Scientific Company	435826	R	Phosphate free detergent	Decontaminating equipment	1	N
MX108128	Fisher Scientific Company	A200212	R	Nitric acid	Decontaminating equipment	1	Y
MX100352	Fisher Scientific Company	BPA4084	R	Methanol	Decontaminating equipment	1	Y

R/S=Required/Suggested

6.2 Training Requirements

All field scientists must complete protocol-specific training for safety and implementation of this protocol as required in Field Operations Job Instruction Training Plan (AD[04]).

Personnel are to be trained in wadeable stream sediment chemistry measurements and safe working practices for stream work. All personnel that wade to collect sediments shall review the USFWS Wader Safety Video as required by the NEON safety training program. The safety video is available with CSP2202-OLT Electrofishing Safety.

6.3 Specialized Skills

N/A

6.4 Estimated Time

The time required to implement a protocol will vary depending on a number of factors, such as skill level, system diversity, environmental conditions, and distance between sample plots. The timeframe provided below is an estimate based on completion of a task by a skilled two-person team (i.e., not the time it takes at the beginning of the field season). Use this estimate as framework for assessing progress. If a task is taking significantly longer than the estimated time, a problem ticket should be submitted.

We estimate wadeable stream sediment chemistry sampling requires 2 field scientists for 4-6 hours of field work each sampling day plus travel to and from the site. Preparing the samples for shipment requires up to 2 hours for one field scientist.

7 STANDARD OPERATING PROCEDURES

SOP A Preparing for Data Capture

Mobile applications are the preferred mechanism for data entry (SOP D). Data should be entered into the protocol-specific application as they are being collected, whenever possible, to minimize data transcription and improve data quality. For detailed instructions on protocol-specific data entry into mobile devices, see the NEON Internal Sampling Support Library (SSL). Mobile devices should be synced at the end of each field day, where possible; alternatively, devices should be synced immediately upon return to the Domain Support Facility. However, given the potential for mobile devices to fail under field conditions, it is imperative that paper datasheets are always available to record data. Paper datasheets should be carried along with the mobile devices to sampling locations at all times.

SOP B Preparing for Sampling

This protocol requires careful attention to the equipment type used to collect aquatic sediment samples for various chemical and physical analyses described in the Methods, Section 3. *It is also very important to clean and store all equipment according to the intended use while sampling in the field* (SOP C.4). *There is no perfect sampler type for collecting sediments which completely eliminate the risk of cross contamination from the sampler or the residues on the equipment from cleaning. For example, in some cases metals (sieve and ponar) will come into contact with sediments that will be analyzed for trace elements (inorganics). In another example, samples collected for TOC/TC may come into contact with instruments cleaned with methanol. The objective is to minimize the risk of cross contamination and to keep the equipment and cleaning methods consistent with every sediment chemistry bout across NEON.*

1. **Ensure all equipment has been cleaned and stored appropriately** (see SOP C.4).
2. Mark the hand corer/liner with 1 cm increments using a waterproof marker to help define the sampling depth.
3. Refresh the sampling kit. Ensure, sample kits (bottles and coolers) are ordered from the external lab at least one week prior to the next scheduled sampling. Restock the sampling kit (shipping cooler) with new sediment chemistry sampling bottles (with new labels attached), and other equipment and consumables in Table 2.
4. Ensure GPS locations of sampling stations are entered into the GPS system.
5. Confirm type of sediment samples that will be collected and take the appropriate bottles and collection devices (Figure and Figure 7).
6. Review the Equipment list in Section 6.1, Table 2 to gather all of the materials needed for sampling sediments.

Figure 4. Example of clear Boston round glass jars with PTFE lined lid used for collecting sediment for inorganics, organics (PAH/PCB), and carbon (TOC/TC) analyses. Sediments for grain size samples are collected by filling 2 1-gallon (~3.8L) plastic Ziploc bags about half-full.

2. Review Section 6.1 Equipment, Table 2 for a complete list of materials needed to complete this protocol. The general material types used to collect sediments for each respective analyte type (Figure 7) follows:
   a. Inorganics: use plastic (polyethylene) equipment including hand scoop or hand corer (with plastic nosepiece), funnel, and spatula. Collect and composite in a plastic bucket. This equipment must be cleaned following SOP C.6.2.
   b. Organics: use stainless steel equipment including hand scoop or hand corer **without** plastic nosepiece, funnel, and spatula. Collect and composite in a stainless bucket. This equipment must be cleaned following SOP C.6.3.
   c. Carbon: use glass equipment including hand scoop (if using hand corer remove plastic nosepiece), and spatula. Collect and composite in a glass bowl or glass jar with PTFE lid. This equipment must be cleaned following SOP C.6.4.
   d. Sediment size: this material can be collected and composited using any equipment material type.
   e. If using glass equipment for sampling for all analytes, follow the cleaning procedures for Carbon in SOP C.6.4.

Note: It is very important to thoroughly mix collected sediments in order to minimize the inherent environmental variability within sediments.

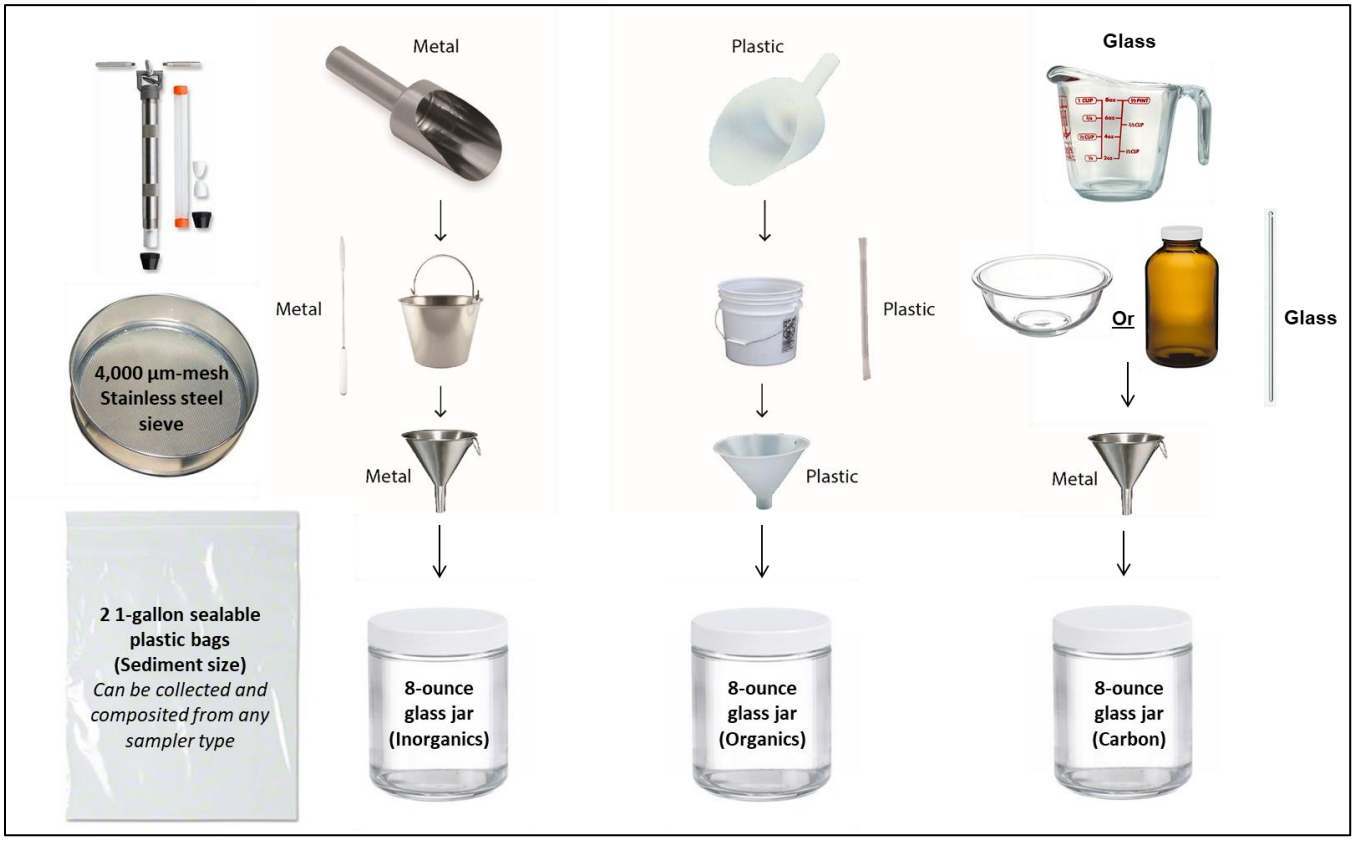

Figure 75. Sediment collection instruments by sample analyte type. When collecting sediments for inorganics with the hand corer, insert the plastic nosepiece. Note that the stainless steel sieve can be used for any analyte to remove sediments and other material larger than 2 mm. Sediment size samples can be collected from any sampler type.

3. Use NEON bottle labels; do not use labels provided by the external lab (Figure 8).
4. Use a Sharpie to fill out bottle labels (Figure 8). Labels are waterproof but should be filled out before getting wet to ensure ink will stick to the labels. Mark the correct sample type code (Figure 8) on the labels for each bottle.
    a. When the system is available, adhesive Type I barcode labels (Figure 9) will be added to the sample containers and scanned by the mobile application (Figure 10).
    b. Keep a human-readable sample labels on each bottle with a minimum of the sample ID printed to assist with organization and shipping.
    c. Be sure to attach barcodes and sample labels to jars before filling them with sediment.

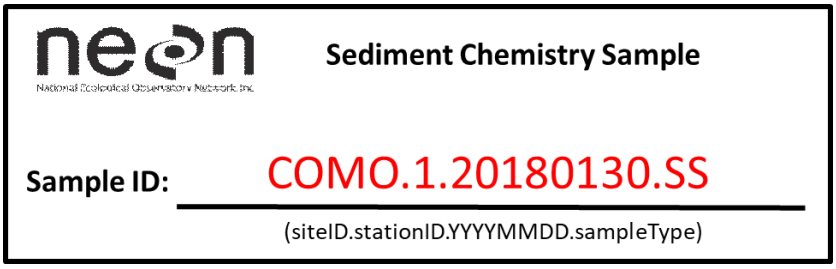

Figure 8. Example of NEON sediment chemistry bottle label. Record the dominant habitat type for wadeable streams that may include riffle, run, pool, or step pool.

Figure 9. Example of adhesive Type I barcode label.

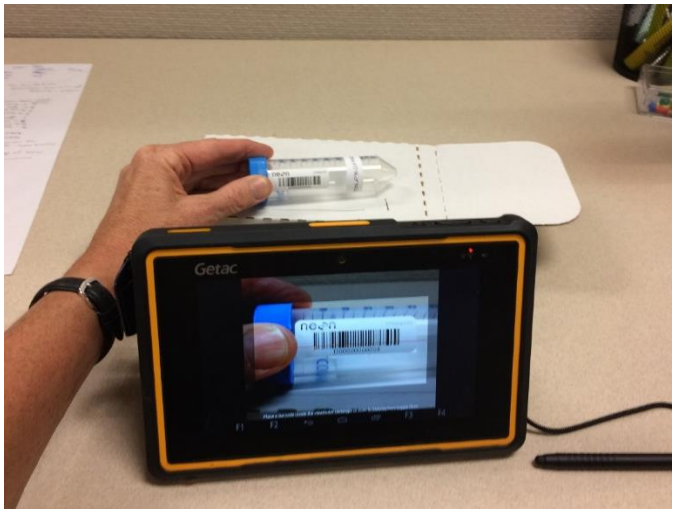

Figure 10. Barcode label scanning.

5. Complete data collection on the mobile device or the *Sediment Chemistry Field Datasheets* associated with the sediment chemistry protocol (RD[05]). Also include site information on the *General Field Sampling Datasheet* (metadata; RD[06]) and the additional sample collection

datasheets (RD[05]). The General Field Sampling Data Sheet only needs to be collected once per day either using the mobile device or the *Sediment Chemistry Field Datasheets.*

a. Record the Date (YYYYMMDD) and the time of day (use local, military time; ex. 13:46) that samples were collected on the mobile device or the *Sediment Chemistry Field Datasheets* (RD[05]).

b. NOTE: Use the same time for all bottles filled at the same sampling station during each sampling event (i.e., the time the sediments were collected from the stream).

c. The Sample ID, Date (YYYYMMDD), and Time must match the sediment chemistry label (**Error! Reference source not found.**). Station ID is either Station 1 (upstream) or Station 2 (downstream). Indicate sample type with either "I" (inorganic), "O" (organic), "C" (TOC/TC), or "SS" (sediment size).

SOP C Field Sampling

Before sampling:

1. For each station, identify suitable depositional zones for sampling based on bathymetric and geomorphology maps if available.
2. Protect the sampling equipment from contamination (aluminum foil, plastic bags, metal or plastic buckets).
3. Prepare ice packs (freeze 8-10 ice packs); have ready for shipping in cooler.
4. Line each shipping cooler with a trash bag.
5. Prepare and have on hand all required paperwork for shipping (chain of custody, permits, manifest, and receipt forms)
6. Calibrate the handheld YSI Pro 2030 for dissolved oxygen before sampling (see instructions in ER [01]).
7. When in the field, rinse all equipment three times with native water prior to use.

Stream sediment samples shall be collected from two identified sediment-sampling stations within the 500 m sediment sampling reach (Figure 2). Each station covers up to 250 m or half of the 500 m sediment reach and may or may not include the S1 and S2 sensors. The dividing point between the two sediment sampling stations is defined by the mid-way point between the top and bottom of the biology and morphology reach (Figure 3). At each of the 2 sampling stations, identify at least 5 wadeable depositional zones containing fine-grained particulate matter (Figure 11a). The number of depositional zones will be dependent on stream morphology (see RD[09]). Identification of depositional zones can be aided by using the site characterization stream morphology maps if available. If the site has no large depositional zones, take samples from depositional environments as per Figure 11b. Likewise, should the streambed be mostly sedimentary or organic in nature, then distribute the samples as per Figure 11c.

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. The zones should not interfere with the biological sampling locations when possible. At sites that are limited by depositional zone areas or sediment substrate size, follow the instructions in Figure 11 and Figure 12. The priority for these sites is to collect sufficient material for chemical and physical analyses. Indicate if the reach is inaccessible by recording **sampling impractical** (dry, frozen, snow, other) and document if the **reach condition** could affect the data collection (normal flow, segmented pools, partially wetted sediments, low flow, high flow, heavy vegetation, skipped station, other). If multiple reach conditions affect data collection, document the most impactful condition.

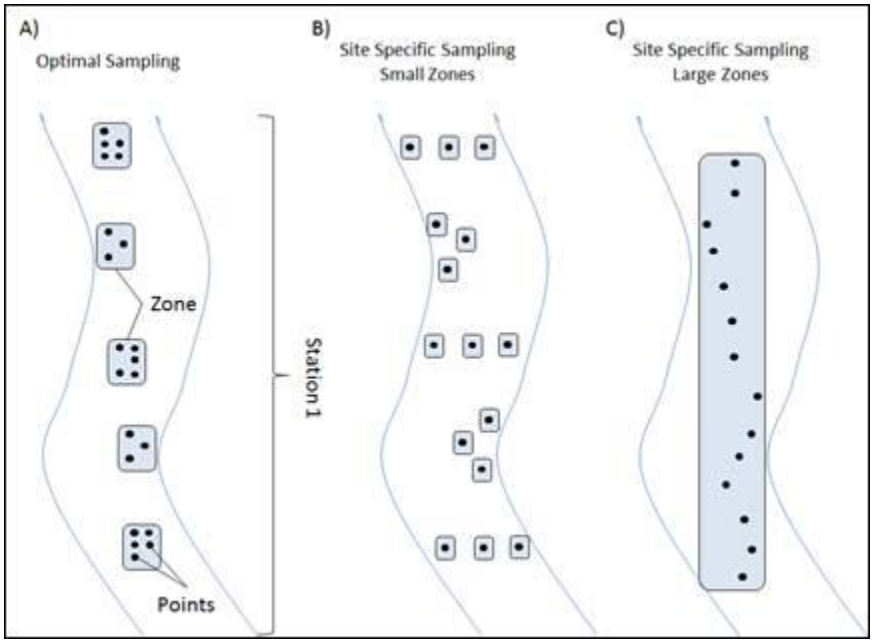

Figure 6. Diagram to exemplify determination of sampling zones based on site differences. A) Ideal sampling set up based on availability of different depositional zones (~1 m^2); B) in sites with only random small pockets of sediment; and C) in sites where most of the stream bed is characterized by sedimentary or organic substrate.

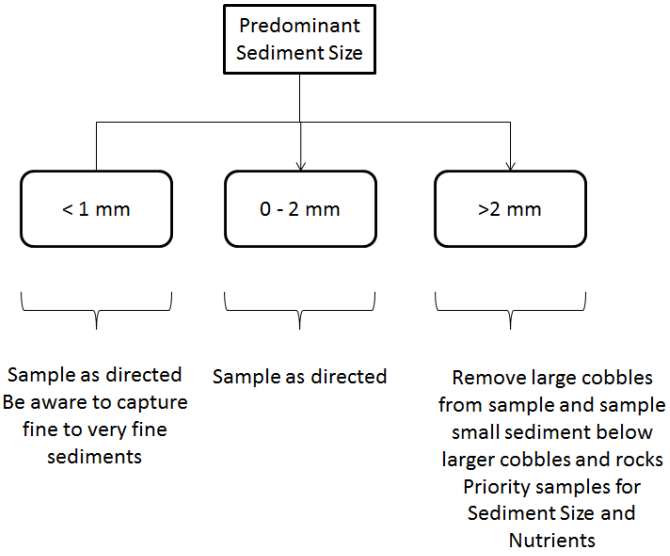

Figure 12. Decision flow chart for sampling in streams with stream bed materials that are limited in quantity or type.

C.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[06]) is completed.
3. Measure the % oxygen, temperature and specific conductivity (µS/cm) of the water above each depositional zone and record one entry at the Sample Point level in the mobile device or the *Sediment Chemistry Field Datasheets* (RD[05]).
    a. Measure water quality using the handheld temperature and conductivity device (YSI Pro 2030) holding it about 10 cm above the sediment in the water column.
    b. For zones in less than 10 cm water depth, measure water quality half-way between the water surface and the sediment.
    c. Rinse all equipment with native water three times before use.
4. Sediments should be collected and composited from depositional zones throughout the sediment sampling reach for an even representation of the chemical and physical attributes of each station (Figure 11).
    a. When sampling sediments that are smaller than 2 mm use a Hand Corer (SOP C.2) and use a Scoop when sampling coarser sediments greater than 2 mm (SOP C.3).
    b. Collect sediments from several points from depositional zones distributed across the sampling station (Figure 11a) and record the water quality and a GPS location on the mobile device or the *Sediment Chemistry Field Datasheets*.

c. For depositional zones less than 5 square meters collect water quality and a GPS point from one center location (Figure 11b).
d. For depositional zones greater than 5 square meters collect water quality and a GPS point from the downstream and upstream locations (Figure 11c).

5. Using a folding measuring stick, measure the water depth at each sampling point within each zone on the field sampling sheet (RD[05]).
6. Repeat steps 1 – 4 above at each sediment sample zone when taking samples.
7. Proceed to next station and repeat steps 1-5.

C.2 Sampling with a Hand Corer

When the bed sediment is composed of primarily smaller sediment sizes (<2 mm) use the hand corer for collecting sediments (Figure 13).

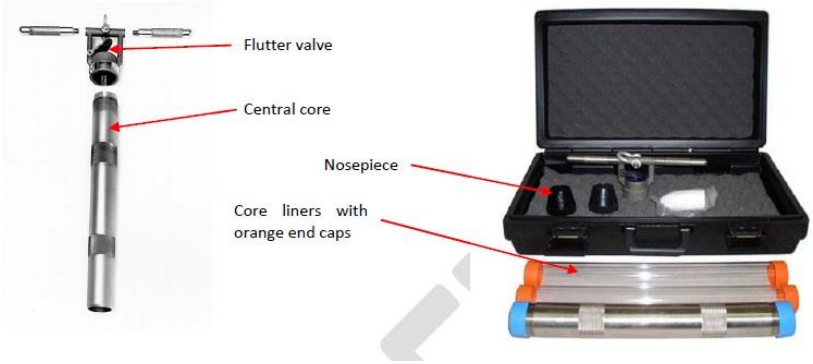

Figure 13. Hand corer for use in wadeable streams with fine sediments

1. Put on gloves (nitrile).
2. Assemble the hand coring sampler: if sampling for inorganics insert a core liner with the plastic nosepiece; if sampling for organics do not insert a liner.
3. Take the appropriate sediment collection buckets (stainless steel, plastic, or glass) whilst sampling in the stream in order to minimize the disturbance from entering and exiting the stream.
4. To operate the hand corer:
    a. Insert sampler into the bed up to 3 cm.
    b. Use a metal spatula for organics, a plastic spatula for inorganics, or a glass spatula for carbon samples to cap the end of the corer before the sampler breaks to surface of the water to prevent sediment from falling out.
    c. Only collect the top 3 cm of the bed material from the exterior of the core.
    d. Ensure the upper flutter valve is closed and lift the corer above the surface maintaining it as vertical as possible. It may require using native water to keep the flutter valve wet for a good seal. Inspect for adequate fine material; if not appropriate, discard near the bank downstream of the depositional zone and take another sample.

e. To retain collected sediments in the corer, gently remove the nosepiece and replace with an orange core cap if sampling for inorganics. Otherwise simply cap end of corer with blue cap. Using a spatula to retain sample material is also effective. Be sure to use the appropriate spatula type when handling inorganic, organic, and carbon samples.
f. Move to the processing area on the stream bank.

5. Deposit all sediment samples into the appropriate bucket and proceed with the next sample.
6. Clean the sampler with the scrub brush.
7. Proceed to SOP E for sample treatment and shipping preparation in the field.

C.3 Sampling with a Hand Scoop

1. Put on gloves (nitrile).
2. Take the appropriate sediment collection buckets (stainless steel, plastic, or glass) whilst sampling in the stream in order to minimize the disturbance from entering and exiting the stream.
3. To operate the hand scoop:
   a. Remove the top layer (approximately 1-3 cm) of fine sediment carefully by gently scooping in the upstream direction.
      1) Ensure the scoop is plastic when sampling for inorganics (Figure 14).

Figure 7. Scoop samplers composed of metal, plastic or glass are used for collecting sediment in wadeable streams

      2) Use the metal scoop when sampling for organics.
      3) A glass scoop is used when sampling for carbon.
      4) The sediment size sample can be collected with any scoop type.
   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.
4. Deposit all sediment samples into the appropriate bucket and proceed with the next sample.

5. Clean the sampler with the scrub brush and rinse with native water between sampling stations. Make sure any residual sediment is visibly removed from the sampler.
   Proceed to SOP E for sample treatment and shipping preparation in the field.

C.4 Sediment Sampling by Analyte Type

Sediment can be collected with either the plastic hand scoop or hand corer and composited in a single plastic (polyethylene) bucket or glass bowl when sampling just for inorganic and sediment size analyses (bout 1). However, when collecting sediments for the full suite of analyses (inorganic, organic, carbon, and sediment size; bout 3) collect and composite material collected at either non-wadeable of lake sites (SOP C.1 and SOP C.2) with equipment constructed with the appropriate material (plastic for inorganics, metal for organics, or glass) and cleaned following Section C.7. When collecting sediment with the hand corer (SOP C.2; Figure 13) collect samples from up to a maximum of 5 points per depositional zone greater than one square meter (Figure 11). If the depositional zones are less than 1 m^2, a minimum of 2 points within each depositional zone should be collected. When collecting sediments for the full suite of analytes (bout 3) ensure that each depositional zone is sampled equally for inorganic, organic, carbon (TOC/TC), and sediment size samples. When insufficient sediment depth and/or larger sediment size (>2mm) is encountered, particularly in rocky streams and rock bottomed streams, use the hand scoop sampler (SOP C.3; Figure 14).

1. Collecting and compositing samples by bout and analyte type. To minimize cross contamination, it is recommended to sample sediments following the order below.
   a. **Bout 1:** Sediment can be collected and composited in a single plastic bucket when sampling just for inorganic and sediment size analyses.
      i. **Inorganics:** Collect in a plastic (polyethylene) bucket, use plastic hand corer with plastic nosepiece or plastic hand scoop.
         1. Composite the material using a plastic spatula then transfer into the sample jar. Fill an **8 oz. (250 mL)** glass collection jar with PTFE lid.
         2. **Note:** inorganic sampling using the stainless steel sieve will knowingly affect trace metals analyses. Use the sieve sparingly when processing inorganic samples.

      ii. **Sediment size:** Collect, composite, and distribute these samples using any sampler and bucket type. **Fill half of two separate 1-gallon Ziploc-type plastic bags.**

   b. **Bout 3:** When sampling sediments for the full suite of analyses collect and composite material as follows:

i. **Carbon (TOC/TC):** Collect in a glass bowl or glass jar, use glass hand scoop (if using the hand corer, remove the plastic nosepiece and triple rinse the end of the corer tube (5 cm) with native water).
    1. Use a glass spatula when homogenizing and transferring into the carbon sample jar. Fill **half** of an **8 oz. (250 mL)** glass jar with PTFE lid for a collected volume of 4 oz. (125 mL).
    2. **Note:** If using glass to collect and composite the carbon sample, use the amber glass jar with a PTFE screw-on lid described in Section 6.1, Equipment, Table 2. Also, it is recommended that glass bowls or jars are stored in a plastic bucket when carrying over rocky or uneven terrain. This will help protect the bowl while transporting and containing any glass should the bowl break in the field.

ii. **Organics:** Collect in a stainless steel bucket, use stainless steel hand corer or hand scoop.
    1. Use stainless steel funnel and spatula (Figure 7) for compositing and transferring into the organic sample jar. Fill an **8 oz. (250 mL)** glass collection jar with PTFE lid.

iii. **Inorganics:** Collect in a plastic (polyethylene) bucket, use plastic hand corer with plastic nosepiece or plastic hand scoop.
    1. Composite the material using a plastic spatula then transfer into the sample jar. Fill an **8 oz. (250 mL)** glass collection jar with PTFE lid.

    2. **Note:** inorganic sampling using the stainless steel sieve will knowingly affect trace metals analyses. Use the sieve sparingly when processing inorganic samples.

iii. **Sediment size:** Collect, composite, and distribute these samples using any sampler and bucket type. A separate bucket for sediment size samples is not needed. **Fill half of two separate 1-gallon Ziploc-type plastic bags.**

2. 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).
3. For light (sparse) debris > 4 mm, use forceps to remove litter from the sample.
4. If the sample contains heavy debris, many large pebbles and cobbles (> 4mm), place collected material in the bucket to composite then either pick out or sieve the debris and pebbles/cobbles out of the sample prior to transfer to the sample bottle using the US-5 (4,000 µm mesh). Coarse rock and debris may be discarded on the shore away from other depositional zones. The external lab will screen these composited samples to < 2 mm at their facility. It is VERY IMPORTANT that the majority of the sample (> 50%) contain sediment (not water) in order to

provide the external lab with a sufficient volume of material to run all of the required analytical parameters.

5. Let the sample settle for up to five minutes then gently decant any excess water (> 3 cm or 100 mL) from the container by gently pouring off the liquid layer taking care to prevent disposing of the fine sediment size fraction. It may be helpful to remove the liquid layer using a syringe and tube (or just the syringe) to suction off remaining water. For samples with excessive suspended sediment, it may be necessary to move onto the next sampling station and let the sediments settle prior to decanting. Minimize the time that the plastic syringe or tubing is in contact with sediments sampled for organics.
6. Use the appropriate funnel to distribute the composited sample into the collection jar for the specific analyte. Use the stainless steel funnel for carbon (TOC/TC) samples (Figure 7).
7. Repeat steps SOP C.4 1-9 using the appropriate equipment to composite the sample into the collections bottles. Collect a sufficient volume of sediment material with the appropriate equipment to fill the sample collection jars.
8. Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles. DO NOT FREEZE.
9. Dispose of any excess sediment not collected for external lab analyses near the shore, away from depositional zones.
10. **Note:** At the end of sediment sampling, collected sediments from both stations will result in the following contents for packing and shipping:
    a. **Bout 1:** (only inorganic and sediment size samples)
        i. 2 glass jars (8 oz., 250 mL) inorganic samples
        ii. 4 half-filled one gallon Ziploc bags (~3.8 L) sediment size
    b. **Bout 3:** (Full suite of samples, inorganic, sediment size, organic, and carbon)
        i. 2 glass jars (8 oz., 250 mL) inorganic samples
        ii. 4 half-filled one gallon Ziploc bags (~3.8 L) sediment size
        iii. 2 glass jars (8 oz., 250 mL) organic samples
        iv. 2 half-filled glass jars (8 oz., 250 mL) carbon samples

C.5 Ending the Sampling Day

1. Decontaminate all equipment according to the NEON Standard Operating Procedure: Decontamination of Sensors, Field Equipment, and Field Vehicles (RD[08]) before using at another site. For equipment specific cleaning see SOP C.6 below.
2. At a minimum, after returning from the field, clean the sampling equipment as described in SOP C.7, Step 1 below.

C.6 Equipment Maintenance, Cleaning, and Storage

1. Ensure all containers, tools, and equipment used for inorganic, organic, carbon, and sediment size sample collection are cleaned prior to storage or before initiating sampling:
   a. Rinse equipment and containers to remove obvious residual sediments (dump rinsate into an appropriate waste container; large sediments could clog lab sink drains over time).
   b. Prepare a tub with 0.2% phosphate-free detergent.
   c. Wash containers/equipment in the detergent bath.
   d. Soak the containers/equipment for at least 30 minutes.
   e. Rinse thoroughly with de-ionized water three times using new water each time.
   f. For containers and sampling equipment intended for inorganic and organic sediment collection, see steps b and c below.
   g. For all remaining equipment, allow to air dry.

2. Preparing **plastic (polyethylene)** equipment for **inorganic** sediment sampling:
   a. After the detergent soak described above, rinse with the containers/equipment with 5% high purity nitric acid (HNO_3).
      1) Here is an example for making 1.0 L of 5% HNO_3 with 69.1% nitric solution. Mix 72.36 mL of HNO_3 with 927.64 mL tap water. **ALWAYS add acid to water!**
      2) VERY IMPORTANT, consult the Chemical Hygiene Plan and Biosafety Manual (AD[03]) for disposal of acid waste or other hazardous chemicals.
   b. Rinse three times with deionized water.
   c. Allow to air dry.
   d. Store in plastic bags or within the decontaminated plastic compositing bucket and covered with plastic wrap or the plastic bucket lid. Avoid exposing inorganic sampling equipment to metal.
3. Preparing the **stainless steel** equipment (including the metal sieve) for **organic (PAH/PCB)** sampling:
   a. After the detergent soak described above, rinse containers and equipment with methanol from the Teflon wash bottle.
   b. Do not rinse with DI water.
   c. Allow methanol rinsed equipment to air dry.
   d. Store in aluminum foil or within the decontaminated metal compositing bucket and covered with foil or the metal bucket lid. Avoid exposing the organic sampling equipment to plastic materials.
4. Preparing **glass** equipment for **Carbon (TOC/TC)** sampling:
   a. After the detergent soak described above, rinse containers and equipment with methanol from the Teflon wash bottle.
   b. Rinse three times with deionized water. Allow to air dry.

c. Store in aluminum foil or within the decontaminated metal compositing bucket and covered with foil or the metal bucket lid. Avoid exposing the organic sampling equipment to plastic materials and methanol. Label these tools for TOC/TC sampling.

5. When preparing **glass collection bowls or jars with PTFE lids** and the **metal sieve** for initial sediment collection and homogenization follow the **carbon (TOC/TC)** sampling equipment directions. **Note:** sample kits provided by the lab have been pre-cleaned including sample jars and bags.

C.7 Sample Processing Timing

Samples will be processed within 12 hours of return to the Domain lab. It is recommended that the samples are shipped to the external Laboratory within 72 hours following processing. However, if it is expected to take longer to ship the samples, be sure to store them in a refrigerator between 0-6°C (DO NOT FREEZE) until the samples can be shipped. Samples must be shipped to the external lab on ice between 0-6°C but not frozen and arrive within 7 days of sample collection.

SOP D Data Entry and Verification

As a best practice, field data collected on paper datasheets should be digitally transcribed within 7 days of collection or the end of a sampling bout (where applicable). However, given logistical constraints, the maximum timeline for entering data is within 14 days of collection or the end of a sampling bout (where applicable). See RD[04] for complete instructions regarding manual data transcription.

SOP E Sample Shipment

Information included in this SOP conveys science-based packaging, shipping, and handling requirements, not lab-specific or logistical demands. For that information, reference the CLA shipping document on CLA's NEON intranet site.

E.1 Handling Hazardous Material

N/A

E.2 Supplies/Containers and Conditions

General steps for packing the cooler with sediment samples collected from 2 stations for the full suite of analytes:

1. Make sure all labels are readable, intact, and securely attached to each jar.
2. Line the cooler with a large plastic bag.
3. Place the glass jars in the bubble wrap provided in the sample kit. Group them by analyte type and place the wrapped jars in a plastic bag provided in the sample kit.

4. Place the two half-filled sediment size gallon bags from Station 1 into a single plastic bag with label. Do this for the Station 2 sediment size samples as well.
5. Place the plastic bags with sample jars and bags into the cooler. Add the ice packs throughout the cooler between the bags.
6. Use extra bubble wrap or newspaper to fill any gaps in the cooler.
7. Complete and include the shipping label, chain of custody (COC) form, and shipping inventory. Also, include a copy of the appropriate sediment shipping permit. 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) (RD[05]).

8. Tie the garbage bag liner. Place security seals across the opening of the cooler and ship to address provided by NEON.

E.3 Timelines

Samples will be processed within 12 hours of return to the Domain lab. It is recommended that the samples are shipped to the external Laboratory within 72 hours following processing. However, if it is expected to take longer to ship the samples, be sure to store them in a refrigerator between 0-6°C (DO NOT FREEZE) until the samples can be shipped. Samples must be shipped to the external lab on ice between 0-6°C but not frozen and arrive within 7 days of sample collection. Ship samples “Priority Overnight.” DO NOT send them “FedEx First Overnight.” If samples are shipped on Friday, send “Priority Overnight with Saturday Delivery.” Be sure to mark the Saturday delivery box on the FedEx form. It is recommended that samples collected before a major holiday should be stored (refrigerated) at the DSF and shipped after the holiday to avoid shipping delays. Appendix E for a complete list of sediment chemistry analyses, storage requirements, and hold times. Sampling Timing Contingencies.

E.4 Grouping/Splitting Samples

N/A

E.5 Return of Materials or Containers

N/A

E.6 Shipping Inventory

Include sample shipment inventory (RD[10]). Ensure that the auto-generated email with the shipping inventory is sent to the external lab contact and copy is sent to the NEON CLA contact.

E.7 Laboratory Contact Information and Shipping/Receipt Days

See the CLA shipping document on CLA’s NEON intranet site.

8 REFERENCES

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APPENDIX A DATASHEETS

The following datasheets and mobile device applications are associated with this protocol:

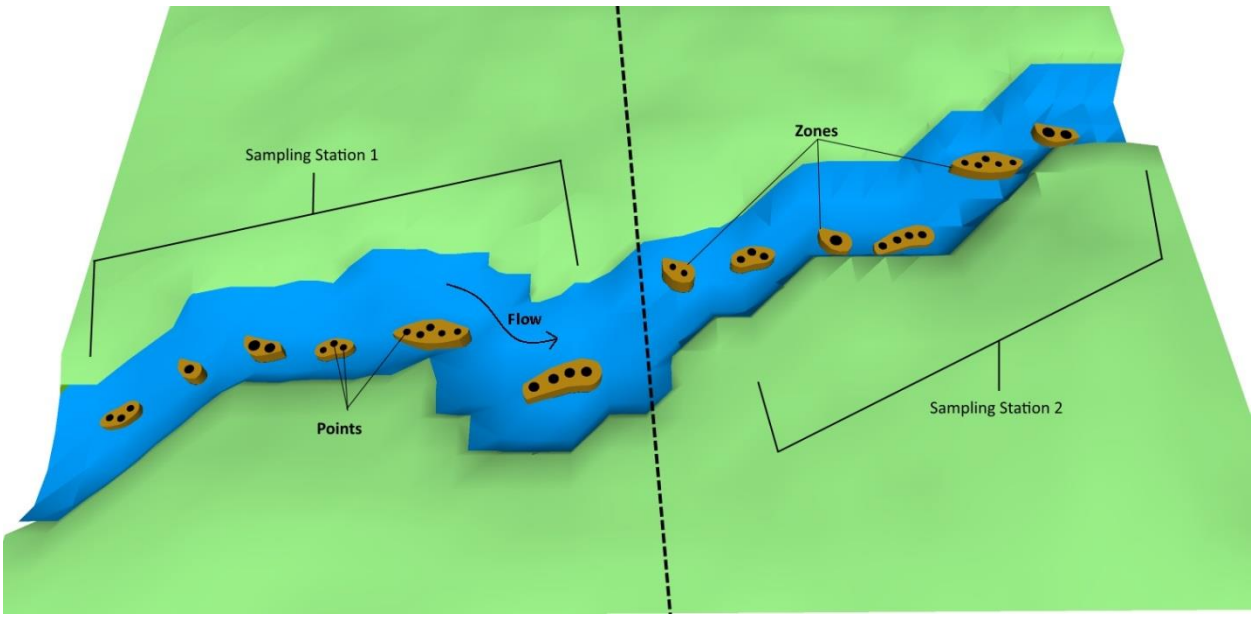
Table 3. Datasheets and mobile applications associated with this protocol

NEON Doc. #	Title	Mobile Application
NEON.DOC. 002419	Datasheets for AOS Protocol and Procedure: Sediment Chemistry Sampling in Wadeable Streams	(AOS) Sediment [PROD]
NEON.DOC.001646	General AQU Field Metadata Sheet	(AOS) Field Metadata [PROD]
NEON.DOC.002494	Datasheets for AOS Sample Shipping Inventory	Shipping Application [PROD]

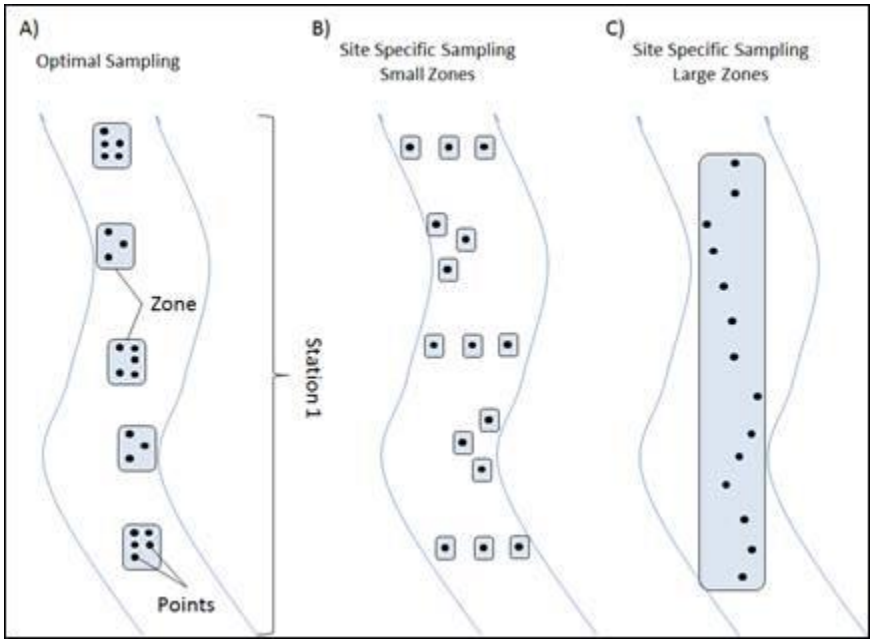
These datasheets can be found in Agile or the NEON Document Warehouse.

APPENDIX B QUICK REFERENCES

B.1 Diagrams for Identifying the Locations of Sediment Sampling

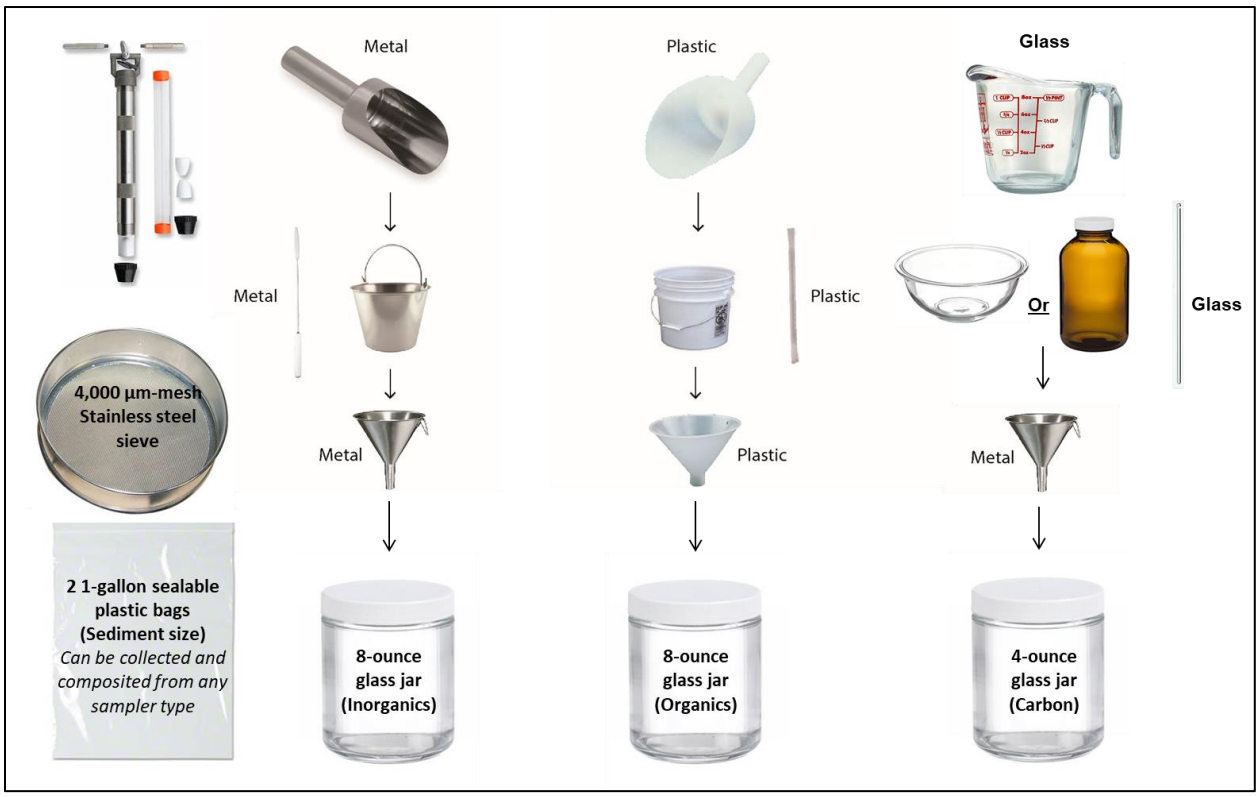


B.2 Diagram of Sample Collection Zones



B.3 Flowchart of Sample Collection

Hand scoops and the hand corer can be used to collect sediments for specific analytes as described in SOP C above. The metal sieve should be used if collected sediments have large pieces of rock or debris > 2 mm. Use the appropriate sampler and equipment type for the specific analyte as described in the figure below. Sediment for size analyses can be collected suing any of the sampler and equipment types.

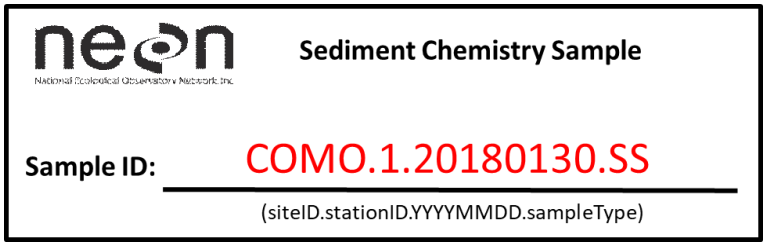


B.4 Steps for Sediment Chemistry Sampling

Step 1 – Check the sediment chemistry field sampling kit to make sure all supplies are packed.

Step 2 – Prepare labels (2" x 4").

1. When the system is available, adhesive barcode labels (Figure 9) will be added to the sample containers and scanned by the mobile application (Figure 10).
2. Keep a human-readable label on each bottle with a minimum of the sample ID printed to assist with organization and shipping.



Step 3 – Ensure the General AQU Field Metadata Sheet (RD[06]) is completed per field site visit.

Step 4 – Begin sampling at the most downstream zone.

Step 5 – When insufficient sediment depth and larger sediment size (>2mm) is encountered, particularly in rocky streams and rock bottomed streams, use the **scoop sampler.**

Step 6 – When the bed sediment is composed of primarily smaller sediment sizes (<2 mm) use the **hand corer**.

Step 7 – Collect samples from 2 to 3 locations (or 5 per m^2) within each depositional zone.

Step 8 – For all inorganic and sediment size samples, place sediment in the plastic bucket and proceed with the next sample. Composite samples from all zones ensuring approximately the same amount of sediment is contributed from each zone.

Step 9 – For organics and organic contaminant samples use the stainless steel bucket and utensils. If fines or organics are left attached to the walls of the sampler, use a Nalgene wash bottle filled with native water to rinse down the sediments from the corer into the bucket.

Step 10 – Repeat the collection process at 5 points (or 3-5 per m^2) within each depositional zone. Ensure that a representative sample for organics is collected from each depositional zone.

Step 11 – Place the combined samples into the appropriate pre-labeled container and then place the containers into a cooler with ice. DO NOT FREEZE.

APPENDIX C REMINDERS

Before heading into the field: Make sure you…

- ☑ Collect and prepare decontaminated equipment including labels.
- ☑ Pre-print labels (and barcode labels when available) on waterproof paper.
- ☑ Fill out the labels before they get wet.

Sample collection: Be sure to…

- ☑ When insufficient sediment depth and larger sediment size (>2mm) is encountered, particularly in rocky streams and rock bottomed streams, use the **scoop sampler.**
- ☑ When the bed sediment is composed of primarily smaller sediment sizes (<2 mm) use the **hand corer**.
- ☑ For samples that are to be analyzed for organics and organic contaminants, the spatula and container must not be plastic (the sample must be collected and shipped in a glass bottle).
- ☑ For samples that are to be analyzed for metals (inorganic), the spatula must not be metallic. Note: only a metal sieve is available at this time and may be used when removing coarse material even with inorganic samples.
- ☑ Do not sample anywhere you or other field scientists have walked in the reach, or locations that appear recently disturbed. Wait for disturbance to pass.
- ☑ Use caution when sampling as items can easily fall into stream while bending to sample.
- ☑ Let the sample settle for up to five minutes then gently decant any excess water (> 3 cm or 100 mL) from the container. Use a syringe and tube (or just the syringe) to suction off remaining water if helpful. For samples with excessive suspended sediment, it may be necessary to move onto the next sampling station and let the sediments settle prior to decanting. Minimize the time that the plastic syringe or tubing is in contact with sediments sampled for organics. Also, minimize the volume of water applied from plastic wash bottles when rinsing organic sample material.
- ☑ Ensure the sediments in the water have settled prior to decanting. If necessary move onto the next sampling and let the sediments settle prior to decanting.
- ☑ Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles.
- ☑ DO NOT FREEZE samples.

Note: At the end of sediment sampling, collected sediments from both stations will result in the following contents for packing and shipping:

Bout 1: (only inorganic and sediment size samples)

- 2 glass jars (8 oz., 250 mL) inorganic samples
- 4 half-filled one gallon Ziploc bags (~3.8 L) sediment size

Bout 3: (Full suite of samples, inorganic, sediment size, organic, and carbon)

- 2 glass jars (8 oz., 250 mL) inorganic samples
- 4 half-filled one gallon Ziploc bags (~3.8 L) sediment size
- 2 glass jars (8 oz., 250 mL) organic samples
- 2 half-filled glass jars (8 oz., 250 mL) carbon samples

APPENDIX D ESTIMATED DATES FOR ONSET AND CESSATION OF SAMPLING

See the Site Specific Sampling Strategy Document on AQU's NEON intranet site.

APPENDIX E SITE-SPECIFIC INFORMATION

See the Site Specific Sampling Strategy Document on the FOPS AOS intranet site.

APPENDIX F SEDIMENT CHEMISTRY ANALYSES, STORAGE CONDITIONS, AND HOLD TIMES

Sample Type	Required Analyses	Target Minimum Quantity (g dry weight)	Required Method	Storage Conditions	Hold time
Carbon (samples with ".C" suffix)	Total organic carbon	5	ASA No.9 29-2.2.4	Refrigerate 0-6 °C	28 days from collection
	Total carbon				
Organic (samples with ".O" suffix)	PAHs	90	M3540/8270C	Refrigerate 0-6 °C	14 days from collection
	PCBs		M3540/8082		
Inorganic (samples with ".I" suffix)	Aluminum, total Antimony, total Barium, total Beryllium, total Cadmium, total Calcium, total Chromium, total Copper, total Iron, total Lead, total Magnesium, total Manganese, total Molybdenum, total Nickel, total Phosphorus, total Potassium, total Sodium, total Tin, total Titanium, total Zinc, total	10	M 3050/6010b or 6020	Refrigerate 0-6 °C	Digested within 6 months of collection/analyzed within 6 months of digestion
	Mercury by Direct Combustion AA	3	M7473		28 days from collection
	Conductivity, Saturated Paste	50	SM2510B		28 days from collection
	pH, Saturated Paste		EPA 600/2-78-054 section 3.2.2		28 days from collection
	Total Alkalinity Bicarbonate as CaCO3 Carbonate as CaCO3 Hydroxide as CaCO3	50	SM2320B		Analysis within 12 days of extraction
	Total Nitrogen	5	M351.2		Analysis within 28 days of extraction
	Nitrate as N, soluble (water)	50	Calculate (NO3 NO2) – NO2		Analysis within 28 days of extraction

Sample Type	Required Analyses	Target Minimum Quantity (g dry weight)	Required Method	Storage Conditions	Hold time
	Nitrate/Nitrite as N, soluble (water)		M353.2		Analysis within 28 days of extraction
	Nitrite as N, soluble (water)		M353.2		Analysis within 28 days of extraction
	Nitrogen, ammonia (water)		M350.1		Analysis within 28 days of extraction
	Grain size	100	ASA no.9 15-4.2.2		Indefinitely
Size analysis (samples with ".SS" suffix)	Soil texture analysis	300	ASTM D 422	Room temperature	Indefinitely