AOS PROTOCOL AND PROCEDURE: SEDIMENT CHEMISTRY SAMPLING IN LAKES AND NON-WADEABLE STREAMS

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Change Record

REVISION	DATE	ECO #	DESCRIPTION OF CHANGE
A	06/02/2014	ECO-01125	Initial release
B	01/22/2015	ECO-02632	Migration to new protocol template
C	02/25/2016	ECO-03503	Protocol updates based on current external lab and technician edits
D	03/09/2017	ECO-04465	CM updated with new template and changes based on feedback from FOPS. Added TOC/TC sample methods.
E	03/13/2018	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 (fall). 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.
F	01/03/2019	ECO-05969	Reorganized the sediment sampling, equipment types, and cleaning instructions throughout the SOP. Added sampling equipment figure. 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. Clarified use of glass bowl A and B. 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.

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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 lakes and non-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 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. 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 NAWQA, 1996). In lakes, deposition zones are often delimited as the deepest zone of the lake and areas with low gradients adjacent to high erosional and/or inflow regions.

Sediment deposition patterns are a direct consequence of the flow characteristics of a water body. 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. In lakes, the deepest part of the lake is considered the equivalent to the depositional zones of a stream, since most sediment is transported to deeper zones over time through wind and current induced turbulence, a process known as focusing. However, caution must be exercised, since areas in a lake typified by large inflows and aeration also represent important depositional environments and areas of higher oxygen exchange, and should be sampled accordingly.

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 several depositional zones in order to smooth local scale variability and represent average conditions of the site. In lakes, samples are composited from 5-10 samples taken in the deepest part of the lake and again at another nearshore depositional environment. In non-wadeable streams, samples shall be taken from 5-10 depositional zones within the two stations. To increase the probability of detecting trace elements 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. 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.

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) National Field Manual for the Collection of Water--Quality Data (U.S. Geological Survey TWRI Book 9, Chapter A4, Version 2.0, 9/2006), the United States Geological Survey (1994), Guidelines for Collecting and Processing Samples of Stream bed Sediment for Analysis of Trace Elements and Organic Contaminants for the National Water-Quality Assessment Program., Larry R. Shelton and Paul D. Capel, U.S. GEOLOGICAL SURVEY Open-File Report 94-458, Sacramento, California and the U.S. Environmental Protection Agency (1994), Technical Standard Operating Procedure SOP #EH-02 Sediment Sampling (Adapted from ERT/REAC SOP #2016 Rev 0.0), 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.002435	Datasheets for AOS Protocol and Procedure: Sediment Chemistry Sampling in Lakes and Non-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.001197	AOS Protocol and Procedure: Bathymetry and Morphology of Lakes and Non-Wadeable Streams
RD[10]	NEON.DOC.001193	AOS Protocol and Procedure: Sediment Chemistry Sampling in Wadeable Streams
RD[11]	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
m	Meter
Mm	Millimeter

3 METHOD

Sediment chemistry sampling strategies in lakes and non-wadeable streams are based on modified versions of United States Geological Survey (2007), National Parks Service (2008), and U.S. Environmental Protection Agency (2012).

The spatial distribution of sediment characteristics depends on processes such as current, turbulence, lake or 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). Depositional zones represent the net outcome of multiple processes and flows. At non-wadeable streams and lakes, sediment samples are collected from depositional zones within two stations throughout the sediment sampling reach (Figure 1). At non-wadeable streams, samples are collected from an upstream station (Station 1) located above the mid-way point of the reach. A downstream station (Station 2) is located below the mid-way location of the reach. At lakes, sediment samples are collected from the inlet (Station IN) and at the buoy (Station C0; Figure 2).

The sampling distribution should follow approximately 5 samples per square meter; however, it is usually difficult to estimate the size of depositional zones in non-wadeable streams and lakes due to water depth and turbidity. Therefore, at each non-wadeable stream and lake station, 2 to 5 point samples will be collected and composited at each station. More point samples may need to be collected in order to acquire a sufficient volume of sediment for each sample type.

Partially wetted sediment depositional zones (< 5 cm water) should only be sampled when no other submerged depositional zones are available. Sampling conditions should be documented in the field notes and field metadata as a potential outlier. Each sample will consist of the surficial 1-3 cm of bed sediment. Compositing samples allow for greater representativeness of mean concentrations from each station and results in smoothing out the variability otherwise encountered between depositional areas. A petite Ponar sampler (SOP C.3) is used to collect sediments in lakes and non-wadeable streams (Figure 2). When collecting sediment along the shoreline of lakes or non-wadeable streams, a hand scoop may be used to collect material between rocks or other tight spaces.

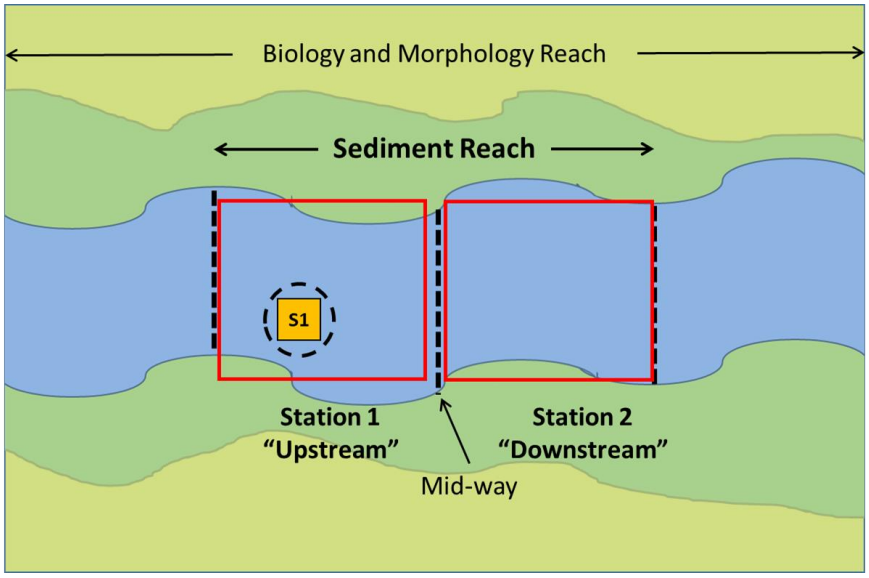


Figure 1. Identifying the location for sediment sampling in non-wadeable stream stations (red boxes). Station 1"Upstream" sediments are sampled in lower half of the upstream section of the reach. Station 2 "Downstream" sediments are sampled in the upper half of the downstream reach. Avoid sampling within the 5 m sensor buffers at the buoy and S1 locations.

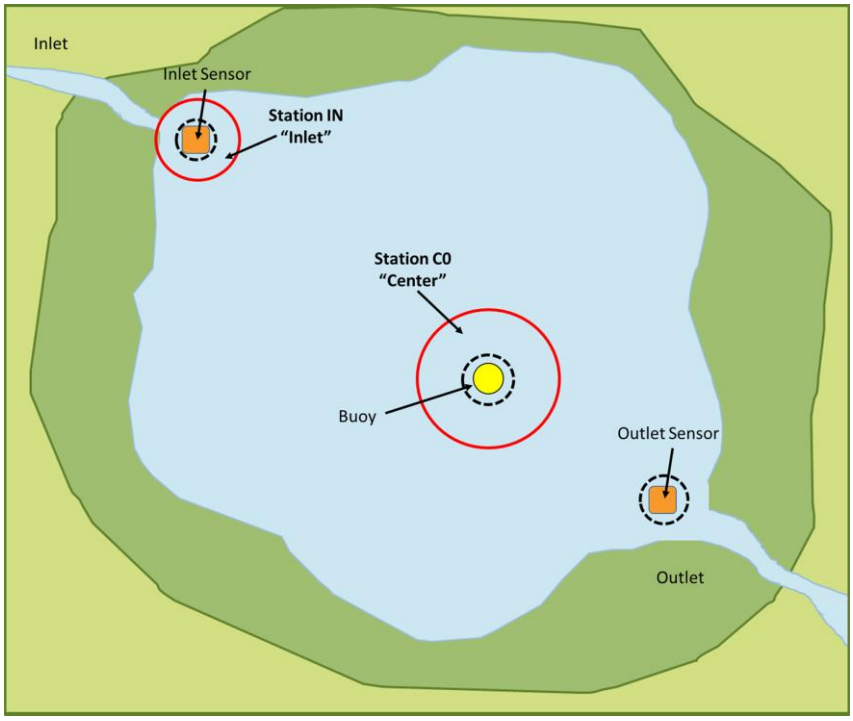


Figure 2. Lake sampling stations are illustrated with red circles. Station IN "inlet" samples are collected within 10 m of the 5 m sensor buffer at the inlet. Sediment sampling at Station C0 "center" occurs 10 m outside of the 5 meter buoy buffer. Avoid sampling within the 5 m sensor buffers at the buoy and S1 locations. No sediment samples are collected at the outlet.

Two stations per lake and non-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.

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 and Laboratory Procedures Quality Assurance Plan for Field Operations Activities (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 lake and non-wadeable stream bottom sediments shall occur two times per during the spring and fall sampling bouts. Sediments will be collected during 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 non-wadeable streams and event (precipitation) dependent in lakes. Should a major event occur that disrupts sediments, non-wadeable stream samples should not be collected for at least 5 days following a major flow event (flooding) and when safe conditions return. Should a major event occur that disrupts lake sediment samples should not be collected for at least 5 days following a major event and when safe conditions return. The conditions should be calm (winds <9 km/hr.) in order to ensure no sediment re-suspension in the nearshore area at the river confluence or shallow littoral areas of the lakes. 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.

Additional sample events may be warranted following a major storm event that alters the morphology of the system. 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 and leaf- off. The timing of such samples should reflect the hydrologic nature of the non-wadeable stream or lake and the temporal variability of the system. Sampling should not take place for at least 5 days after major events that disrupt the sediments in order to allow for the settlement of the sediments. 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/lake (i.e., gas additions) within the past hour, allow the water to clear and disturbance to pass, sample upstream/upwind of the disturbance. The conditions should be calm (winds <9 km/hr.) on the lakes. Should wind speed increase to where noticeable white caps are present, stop sampling immediately and proceed to the closest shoreline.	No adverse outcome.
5 or More Days	Following a major precipitation events and resulting high flow, samples will be taken at least 5 days following a major flow event.	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 Scientist 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. If the sampling trip involves the use of a boat (lakes and non-wadeable streams), then the weather forecast or marine conditions should be obtained prior to departure to the field. The sampling trip should be rescheduled to a later date when conditions are suitable for working on or near water.
2. All personnel using a boat are required to wear an approved personal flotation device.

3. When handling hazardous products (such as nitric acid) follow laboratory safety standards and have Safety Data Sheet (SDS) readily available for review prior to handling the chemical. Wear gloves, a laboratory coat and protective eyewear.
4. Due to accessibility constraints at some sites, most sampling will have to take place from the boat, without dismounting from the vessel.
5. 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.
6. At extreme cold water sites, additional safety training may be required (example Toolik Field Station cold water boater safety training) and include the use of special equipment (e.g. floatation jacket or PFD) for added safety.
7. When operating the petite Ponar, take care to handle it when the boat is properly balanced to avoid dropping the sampler on people (feet), the boat, or equipment. Also, follow the safety guidelines below when opening, closing, deploying, and lifting the Ponar to prevent injuries to fingers and hands.
    a. When not deploying the Ponar, place the safety pin through both locking holes.
    b. During Ponar deployments make sure that the boat is steady and that the sampler is deployed in a controlled manner but with enough velocity to penetrate the sediments and that the jaws close.
    c. Take care when retrieving the sampler when it is filled with sediments. Pull the line up in a steady, even motion. Avoid twisting or jerky motions to prevent back and shoulder injuries. It may be helpful for two or more people to pull up the sampler.

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							
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 13 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	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
MX107412	BioQuip Products Inc.	4748	R	Flexible forceps, featherweight	Removing debris from samples	1	N
			R	Tool Kit		1	N
MX105823	Forestry Suppliers, Inc.	71112	R	Measuring stick, foldable, plastic	Measuring the water depth at sampling locations	1	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
			R	Field documentation forms and field books	Documenting notes in the field	3	N
			R	Brush, scrub, soft nonmetallic	Cleaning samplers	1	N
MX108907	Global Equipment Company	WGB548988	R	Stainless Steel Bucket 5 gallons w/ lid	Homogenizing organic samples	1	N
MX100526	Grainger, W.W.	34A216	R	Plastic bucket 5 Gallons w/lid	Homogenizing inorganic, nutrient and size analysis samples	1	N
MX108989	Grainger, W.W.	21TR61	R	Scoop, Polyethylene	Collecting inorganic, nutrient 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 and TOC/TC samples	1	N
MX100303	Ben Meadows Co., Inc.	223578	R	Petite Ponar	Sampling	1	N
MX100514	Thomas Scientific, Inc.	1185K52 15177622	R	Handheld YSI Pro 2030	Measuring % DO, temperature and salinity	1	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
MX104789	Fisher Scientific Company	048841AA	R	4,000 µm sieve	Sieving samples before transferring into bottles	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
MX107144	B&H Photo Corp	PADMCTS30BL	R	Camera	Photographing sampling observations	1	N
			R	Sonar with GPS antenna	Navigating to sampling locations	1	N
MX111388	CDW-G	4452963	R	Mobile field data recording device (Tablet)	Recording data	1	N
Consumable items							
	External lab		R	Ice Pack	Keeping samples cool, provided by the external lab	Multiple	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
			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
			R	Equipment sealable bags, 5 gal	Transporting equipment	8	N
MX102717	Uline	S-5106	R	Trash Bags 13 Gal	Transporting equipment	4	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
			S	Adhesive barcode labels	Labeling sample bottles with barcode-readable labels	1 sheet	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
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

Table 3. Equipment list – General boating equipment

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
Durable items							
			R	Boat		1	Y
MX107097	Amazon Capital Services Inc.	B003ZZG5EM	R	Anchor with rope		1	N
MX100457			R	Oars		2	N

Item No.	Supplier	Supplier ID	R/S	Description	Purpose	Quantity	Special Handling
MX100458	West Marine Products, Inc.	13487178	R	Trolling Electric Motor		1	Y
MX100899	Grainger, W.W.	2UKJ5	R	Battery (12 volt)		1	Y
MX100435	Amazon Capital Services Inc.	B003QKQ4V0	R	Safety kit for boat (e.g., flares, bailer, float with rope)		1	Y
MX102603	Grainger, W.W.	11C657	R	First Aid Kit		1	N
			R	Personal Flotation Devices (PFDs)		1 per person	N
Consumable items							
				(none)			

R/S=Required/Suggested

Table 4. Equipment list – Equipment maintenance and storage

Item No.			R/S	Description	Purpose	Quantity	Special Handling
Durable items							

Item No.			R/S	Description	Purpose	Quantity	Special Handling
				(none)			
Consumable items							
			R	Gloves, nitrile (box)	Not contaminating samples	1	N
MX100642	Thomas Scientific, Inc.	2904F24	R	Kimwipes (box)	Cleaning	1	N
MX103942	Ben Meadows Co., Inc. Forestry Suppliers, Inc.	010510-1 49247	R	Bottle labels	Labeling samples	8	N
			R	Forms, analytical request (TBD)	Shipping samples	Multiple	N

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 lake and non-wadeable stream sediment chemistry measurements and safe working practices for lake work. All personnel required to operate a boat shall be trained through the NEON boater safety training program. Where applicable, personnel will be licensed to operate a boat and be able to safely handle a motor and operate a boat while working.

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 lake and non-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. 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.5). *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.5).
2. 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.
3. Ensure GPS locations of sampling stations are entered into the GPS system (Figure 2 and Figure 3).

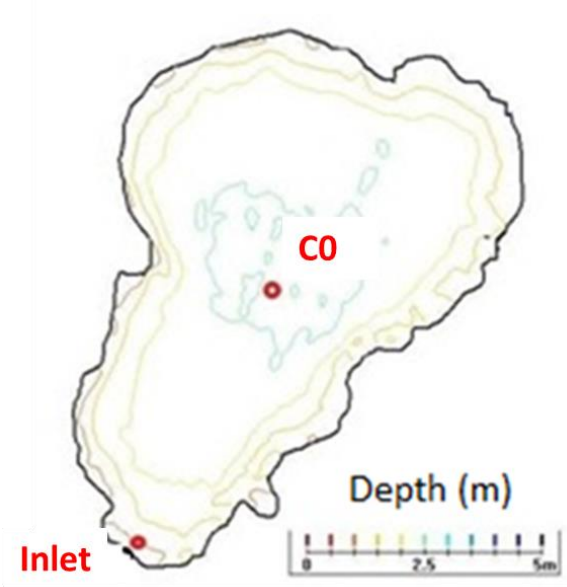


Figure 3. Identifying the Station for sediment sampling in lakes. The two sampling Stations (red circles) should denote depositional zones in the center (C0) of the lake (deepest section) and in a nearshore depositional environment near the inlet.

4. Confirm type of sediment samples that will be collected and take the appropriate bottles and collection devices (Figure 4).
5. 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.

6. The following equipment should be used to collect sediments for each respective analyte type (Figure 5):
   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.7.2.
   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.7.3.
   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.7.4.
   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.7.4.
7. Use NEON bottle labels; do not use labels provided by the external lab.

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

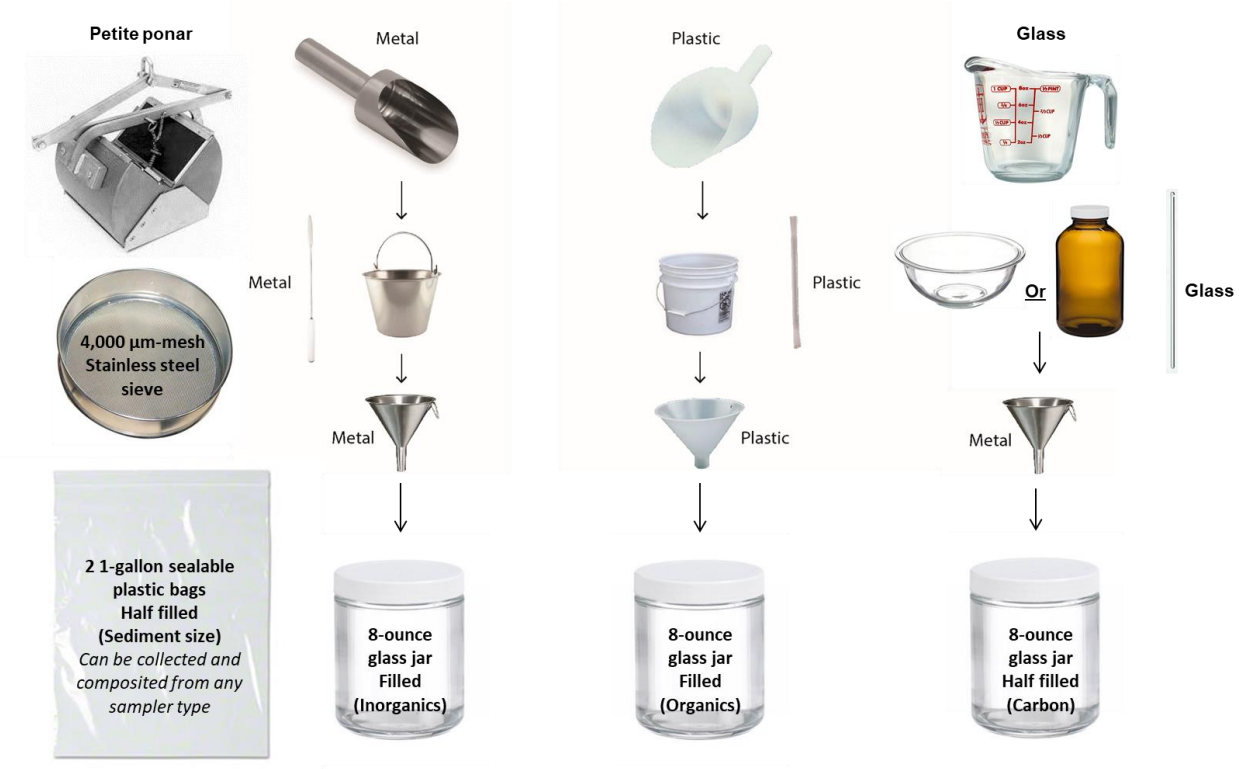


Figure 55 Note, the petite Ponar and stainless steel sieve can be used to collect and screen sediments for any analyte Sediment size samples can be collected from any sampler type.

8. Use a Sharpie to fill out bottle labels. **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 6) on the labels for each bottle.
   a. When the system is available, adhesive Type I barcode labels (Figure 7) will be added to the sample containers and scanned by the mobile application (Figure 8). Be sure to attach barcodes to jars before filling them with sediment.
   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.

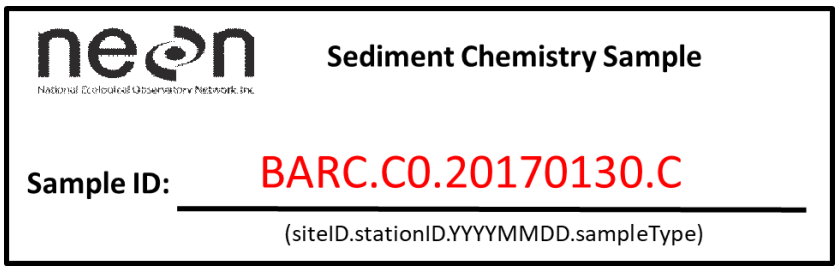


Figure 6. Example of NEON lake sediment chemistry bottle label. Record the dominant habitat type in lakes as either littoral (inlet) or benthic (buoy). Non-wadeable habitat types may include benthic.

Figure 7. Example of adhesive Type I barcode label.

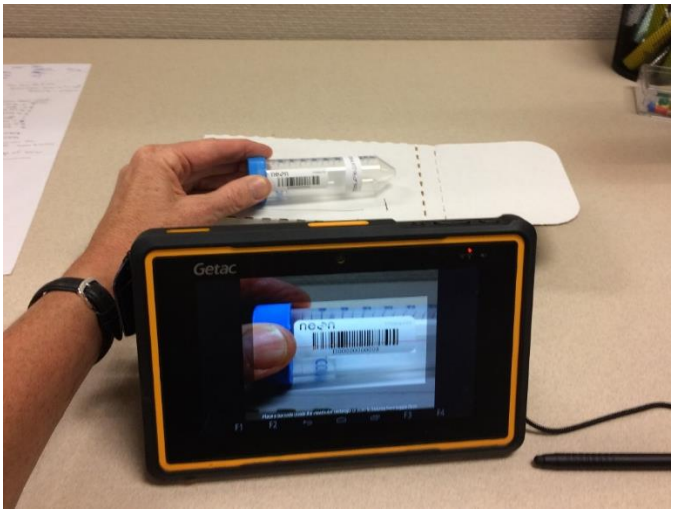


Figure 8. Barcode label scanning.

9. 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 *General AQU Field Metadata Sheet.*

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. Station ID for lakes is either IN (inlet) or C0 (Center); StationID for non-wadeable streams 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.

Non-wadeable stream sediment samples shall be collected from two previously identified sediment-sampling stations within the reach (Figure 1). The station divide is defined by the mid-way location between the top and the bottom of the aquatic reach. At each of the 2 sampling stations, identify at least 5 depositional zones containing fine-grained particulate matter (Figure 9). The number of depositional zones will be dependent on stream morphology (see RD[09]). Identification of depositional zones can be aided by using the most recent bathymetric map. If the site has no large depositional zones, take samples from depositional environments as per Figure 9b. Likewise, should the streambed be mostly sedimentary or organic in nature, then distribute the samples as per Figure 9c. 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; sediment sampling should occur 5 meters away from the instream sensor.

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.

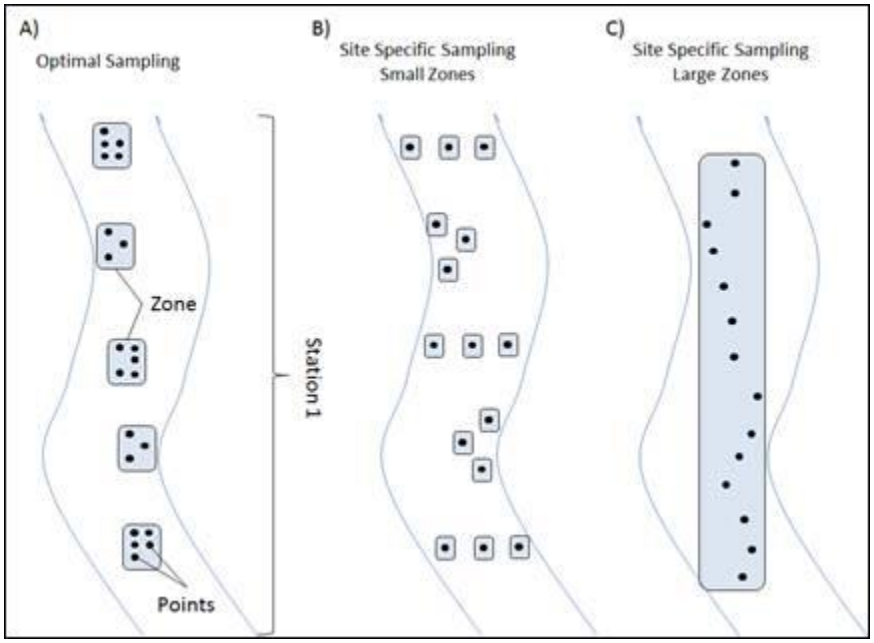


Figure 9. Diagram to exemplify determination of sampling zones in non-wadeable streams based on site differences. A) Ideal sampling set up based on availability of different depositional zones (~1 m^2); B) in sites with only random small pockets of sediment; and C) in sites where most of the stream bed is characterized by sedimentary or organic substrate.

Lake sediment samples will be collected from two stations: Station C0 (central; near the buoy) near the deepest part of the lake which represents the maximum long-term accumulation of sediment. Station IN includes nearshore sediments near the lake inlet, which represents an area of shorter term sediment transport and deposition (Figure 2). The sampling zones must be 5 meters from aquatic sensors (inlet sensor and buoy) but no more than 10 meters beyond the sensor exclusion zone (Figure 10).

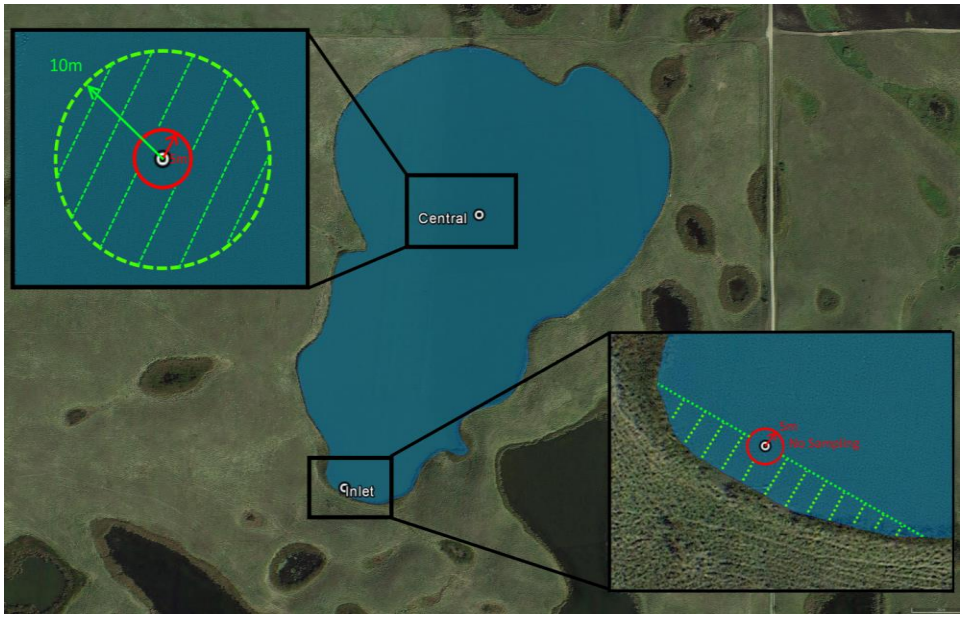


Figure 10. Example lake sediment collection stations with sensor exclusions areas (red) and sampling zones (green).

C.1 Non-Wadeable Streams

1. Ensure the General AQU Field Metadata Sheet (RD[06]) is completed.
2. Work from downstream to upstream starting at the most downstream zone in order to minimize sediment disruption. If sediments are disrupted, wait until the area has cleared before sampling.
3. Position the boat at the appropriate site location and lower the anchor gently so as not to suspend any sediments. Such contamination may be minimized by anchoring the boat downstream of the sampling site, and using an anchor line 3 times as long as the depth of the non-wadeable stream If sediments are disrupted, wait until the area has cleared before sampling.
4. Measure the % oxygen, temperature and specific conductivity (µS/cm) of the water column, 50 cm above each sampling zone. 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. Rinse all equipment with native water three times before use.
5. Note the GPS position of the sampling zone on the field sampling sheet.
6. Repeat steps 1- 5 at each sediment sample zone right before taking samples.
7. Take sediment sample (see SOP C.3 and SOP **Error! Reference source not found.**).
8. Proceed to next station and repeat steps 1-7.

C.2 Lakes

1. Ensure the General AQU Field Metadata Sheet (RD[06]) is completed.
2. Locate the deepest part of the lake using the GPS coordinates and the site map provided.
3. Position the boat at the appropriate site location and lower the anchor gently so as not to re-suspend any sediments. Such contamination may be minimized by anchoring the boat downwind of the sampling site, and using an anchor line 3 times as long as the depth of the lake If sediments are disrupted, wait until the area has cleared before sampling.
4. Measure the % oxygen, temperature and specific conductivity (µS/cm) of the water column, 50 cm above each sampling zone. 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 50 cm above the sediment in the water column.
    b. Rinse all equipment with native water three times before use.
5. Note the approximate GPS position of the sampling points on the field sampling sheet.
6. Repeat steps 1-5 at each sediment sample zone right before taking samples.
7. Proceed to next station and repeat steps 1-6.

C.3 Sampling with a Petite Ponar Sampler

The Ponar sampler is used to collect bed sediments regardless of grain size composition (Figure 11). For collecting nearshore or shallow sediments that are difficult to collect with the Ponar a hand scoop may be used (see SOP C.4 below).

Figure 6. Petite Ponar sampler for use in lakes and non-wadeable streams

1. Put on gloves (nitrile).
2. To operate the Ponar sampler:
    a. Set the grab sampling device with the jaws cocked open (Figure 11). Replace the straight safety pin with the spring loaded pin for deployment.
        1) **Note:** great care should be taken while handling the device while it is set; accidental closure can cause serious injuries.
    b. Ensure that the rope is securely fastened to the sampler, and that the other end of the rope is tied to the boat.

c. Lower the sampler until it is resting on the sediment (its own weight is adequate to penetrate soft sediments). Be sure that the sampler does not descend too fast to avoid suspending the fine sediments and so that the sampler only collects the top 3 cm of material.
d. At this point the slackening of the line activates the mechanism to close the jaws. It is also recommended to use a weighted "messenger" to ensure that the sampler closes. Attach the messenger to the rope and push it down the line to trigger the closure of the sampler. Make sure the rope is taut to ensure the messenger doesn't lose its effectiveness.
e. Retrieve the sampler slowly and steadily to minimize the effect of turbulence (that might result in loss/disturbance of surface sediments).

3. Place glass bowl A beneath the sampler just as it breaks the surface of the water.
4. Slide out the screens on the top of the sampler and transfer the liquid sample into the glass bowl A. Set the glass bowl A aside and proceed to the next step.
5. From the top of the Ponar scoop sediments out from the center taking care to avoid removing material that is touching the metal internal surface of the Ponar. Place this material into glass bowl B.
   a. **Note:** When removing any residual sediment material clumped in the Ponar, collect from the middle portion of the sampler not in direct contact with the metal interior. Place sediments into glass bowl B or into a 5 gallon bucket (use a stainless steel bucket for collecting organic samples; plastic for inorganic samples; glass for carbon samples).
6. Return to glass bowl A and see how much fine sediment has settled out.
   a. If a measureable amount of sediment has settled out, decant off the top layer of water and transfer the solid material into glass bowl B to composite.
   b. If only a trace amount of sediment has settled out or the water in the bowl remains too turbid to see settle accumulations of sediment; discard the sample in the field.
7. 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.
8. Proceed to **Error! Reference source not found.** for sample treatment and shipping preparation in the field.

NOTE: If the jaws were not closed completely enough to trap a sufficient volume of sediment, the sample must be discarded. Discard the sample into a bucket if the second collection attempt is made from the same general area. Dump the unwanted sample back into the water only after all samples have been successfully collected.

C.4 Sampling with a Hand Scoop

Use the scoop sampler when collecting nearshore or shallow samples where the sediments are between plant roots or rocks (Figure 12).

Figure 72. Hand scoop samplers composed of metal, plastic or glass are used at the nearshore station where roots and rocks make Ponar sampling difficult

1. Put on gloves (nitrile).
2. Take the 5-gallon sediment collection buckets (stainless steel and plastic) and glass bowl whilst sampling in the field from the boat to capture sediments collected form the Ponar or hand scoop (nearshore).
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 12).
      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.
6. Proceed to **Error! Reference source not found.** for sample treatment and shipping preparation in the field.

NOTE: Many lake sediment samples are anoxic and a number of chemical changes will take place if the samples are exposed to atmospheric oxygen. If samples are to be retained with as low oxygen as possible to minimize the chemical and microbial transformations, they will need to be packed inside multiple airtight containers.

C.5 Sediment Sampling by Analyte Type

1. Sediment can be collected with either the plastic hand scoop or Ponar 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. Using either the petite Ponar (SOP C.3; Figure 11) or the hand scoop (SOP C.4; Figure 12) collect samples from up to a maximum of 5 points per depositional zone greater than one square meter (Figure 10). 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 along rocky or vegetated nearshore habitats, use the hand scoop sampler.
    a. **Note:** it may not be possible to see the size of depositional zones at lake and non-wadeable sites because of the water depth and turbidity. It is recommended that the boat is moved a few meters within the sampling station to collect and composite material across a wider area.
2. 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 (Figure 5).
        i. **Inorganics:** Collect in a plastic (polyethylene) bucket, use the Ponar 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 metal Ponar and stainless steel sieve will knowingly affect trace metals analyses. Care should be taken when scooping sediment from the Ponar for inorganic analyses. When collecting sediments for inorganic analysis, take from the center of the Ponar, furthest from the material that has contacted the inner surface of the device. Also, use the sieve sparingly.

        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 (Figure 5) as follows:

i. **Carbon (TOC/TC):** Collect in a two glass bowls (A and B), use the Ponar or glass hand scoop.
    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 collecting from a boat or 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 Ponar or stainless steel hand scoop.
    1. Use a stainless steel funnel and spatula 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 the Ponar or plastic hand scoop.
    1. Use a plastic funnel and spatula for compositing and transferring into the inorganic sample jar. Fill an **8 oz. (250 mL)** glass collection jar with PTFE lid.
    2. **Note:** inorganic sampling using the metal Ponar and stainless steel sieve will knowingly affect trace metals analyses. Care should be taken when scooping sediment from the Ponar for inorganic analyses. When collecting sediments for inorganic analysis, take from the center of the Ponar, furthest from the material that has contacted the inner surface of the device. Also, use the sieve sparingly.

iv. **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.**

3. 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).
4. For light (sparse) debris > 4 mm, use forceps to remove litter from the sample.
5. 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.

6. For very watery samples let the material 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.
7. 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 5).
8. Repeat steps SOP C. 1-7 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.
9. Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles. DO NOT FREEZE.
10. Dispose of any excess sediment not collected for external lab analyses near the shore, away from depositional zones.
11. **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.6 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.7 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 equipment** (collection bowls, jars with PTFE lids and scoops) 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.8 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[11]). 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

Ashley, G. M. 1995. Glaciolacustrine Environments. In: J. Menzies (ed.), Modern Glacial Environments - Processes, Dynamics and Sediments. Butterworth-Heinemann Ltd., Oxford, pp. 417- 444.

Golterman, H.L., P.G. Sly, R.L. Thomas. 1983. Study of the relationship between water quality and sediment transport. 231pp., Techn. Papers in Hydrology, UNESCO, Paris.

Håkansson, L. and M. Jansson. 1983. Principles of Lake Sedimentology. Springer Verlag, Berlin.

International Atomic Energy Agency. 2003. Annual Report, Collection and preparation of bottom sediment samples for analysis of radionuclides and trace elements.

Keith, L.H. 1991. Environmental sampling and analysis: A practical guide: 143 pp. Lewis Publ., CRC Press, Boca Raton, Florida

Kumke, T., A. Schoonderwaldt and U. Kienel. 2005. Spatial variability of sedimentological properties in a large Siberian lake. Aquat. Sci. 67: 86-96

National Parks Service. 2008. Water Quality Monitoring Protocol for Inland Lakes. Natural Resource Report NPS/GLKN/NRTR—2008/109

Nevers, M.B., and R.L. Whitman. 2007. United States Geological Survey, USGS Lake Monitoring Field Manual.

United States Geological Survey. 2006. National Field Manual for the Collection of Water--Quality Data (U.S. Geological Survey TWRI Book 9, Chapter A4, Version 2.0, 9/2006).

United States Geological Survey. 1994. Guidelines for Collecting and Processing Samples of Stream bed Sediment for Analysis of Trace Elements and Organic Contaminants for the National Water-Quality Assessment Program., Larry R. Shelton and Paul D. Capel, U.S. GEOLOGICAL SURVEY Open-File Report 94-458, Sacramento, California

U.S. Environmental Protection Agency. 1994. Technical Standard Operating Procedure SOP #EH-02. Sediment Sampling (Adapted from ERT/REAC SOP #2016 Rev 0.0).

U.S. Environmental Protection Agency. 2012. National Lakes Assessment Program, Field Operations Manual.

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.

APPENDIX A DATASHEETS

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

Table 5. Datasheets and mobile applications associated with this protocol

NEON Doc. #	Title	Mobile Application
NEON.DOC.002435	Datasheets for AOS Protocol and Procedure: Sediment Chemistry Sampling in Lakes and Non-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]

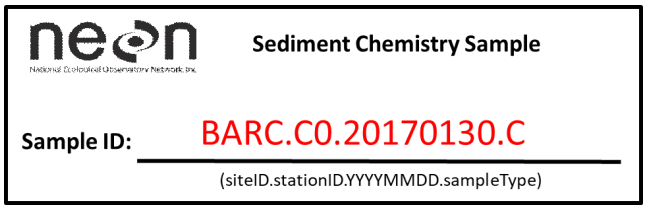
These datasheets can be found in Agile or the NEON Document Warehouse.

APPENDIX B QUICK REFERENCES

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 6) will be added to the sample containers and scanned by the mobile application (Figure 7).
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 – If working in a non-wadeable stream, begin sampling at the most downstream zone. If working in a lake, begin sampling in the deepest part of the lake using the GPS coordinates and the site map provided.

Step 5 – When the bed sediment is composed of primarily larger sediment sizes (sand and >2 mm) or organics the **Ponar sampler** should be used for collecting sediments. The Ponar is also used to collect loosely consolidated sediments.

Step 6 – When the nearshore stream or lake sediment is composed of primarily fine sediment sizes (< sand) and between roots or rocks the **hand corer** should be used for collecting sediments.

Step 7 – Collect samples from 2-5 locations (points) 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 are contributed from each zone.

Step 9 – For organics and organic contaminant samples use the stainless steel bucket and utensils. Use the TOC/TC equipment for those samples. 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 Ponar into the bucket.

Step 10 – Repeat the collection process at 2-5 locations (points) 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 all 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…

- ☑ Use the **Ponar sampler** to collect all deep water samples.
- ☑ Use the **hand scoop** to collect nearshore sediments that are difficult to grab using the Ponar sampler. This would include sediments between rocks and roots.
- ☑ For samples that are to be analyzed for organics and organic contaminants, the spatula and container must not be plastic (the container must be a glass bottle). Use TOC/TC equipment for those samples.
- ☑ For samples that are to be analyzed for metals (inorganic), the spatula must not be metallic.
- ☑ Do not sample anywhere you or other field scientists have walked, 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.
- ☑ 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 Total carbon	5	ASA No.9 29-2.2.4	Refrigerate 0-6 °C	28 days from collection
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