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

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See configuration management system for approval history.

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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.

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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 chain. 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	Domain Chemical Hygiene Plan and Biosafety Manual
AD[04]	NEON.DOC.050005	Field Operations Job Instruction Training Plan
AD[05]	NEON.DOC.014051	Field Audit Plan
AD[06]	NEON.DOC.000824	Data and Data Product Quality Assurance and Control 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	NEON 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.001154	AOS Protocol and Procedure: Aquatic Decontamination
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. In non-wadeable streams samples will be collected from two stations, one downstream station and the other as an upstream station (Figure 1). At lake sites, sediment will also be collected from two stations, one at the inlet and other near the buoy (Figure 2). Samples should be collected and composited from a minimum of two depositional zones within the same station (Figure 1). The number of samples from each zone will be based on the surface area of each zone (i.e., the larger the area of the zone, the greater the number of subsamples collected). The sampling distribution 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, 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 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 for the area and results in smoothing of variability otherwise encountered between depositional areas. A Petite ponar sampler (SOP B.3) will be 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.

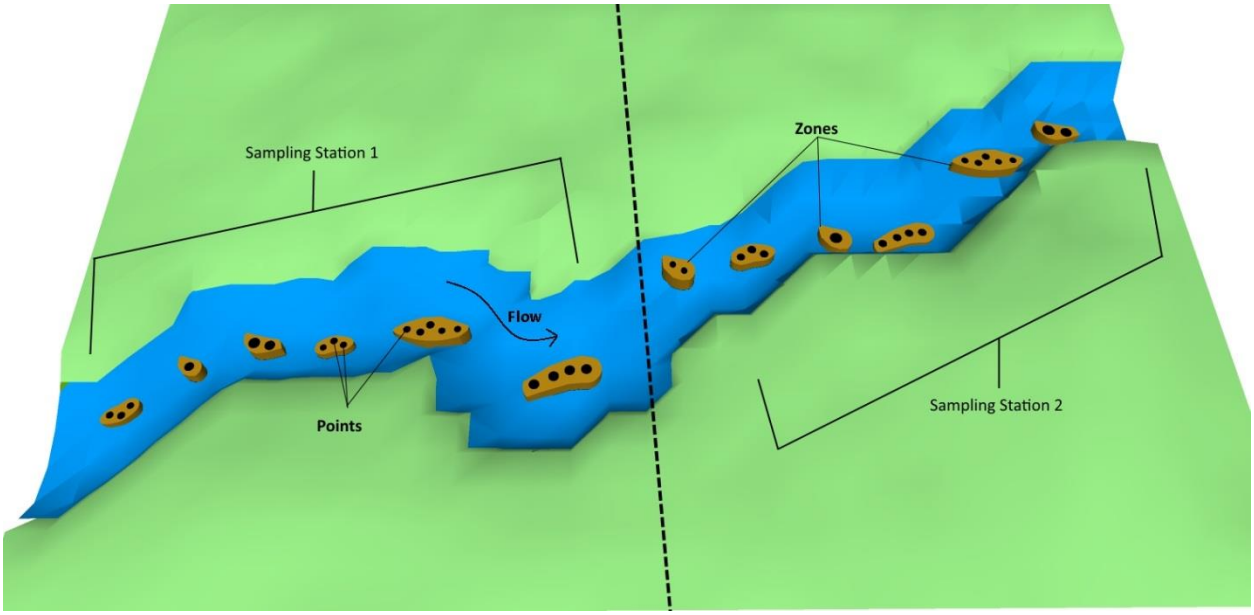

Figure 1. Identifying the location for sediment sampling in non-wadeable streams. The two sediment sampling stations should contain depositional zones that represent upstream influences and various flow regimes.

For lakes, 5-10 samples should be taken within a radius of 10 meters in the deeper depositional part of the lake will be taken and composited as described above. In addition, 5-10 samples from the nearshore depositional stations will be taken and composited. Where possible samples will be taken outside the main reach where biological sampling will take place in order to avoid disturbance of benthic habitats. Should this not be possible, samples will only be taken from depositional zones where no biological sampling will take place.

One 8 oz. (~250 mL) round glass jar will be used to collect samples for organic analyses, one 4 oz. (~125 mL) round glass jar for Total Organic Carbon and Total Carbon (TOC/TC) analyses, and another 8 oz. (~250 mL) jar will be used to collect sediments for inorganic analyses. An additional 1.0 U.S. gallon (~3.8 L) of sediment will be collected in two U.S. gallon plastic (Ziploc) sealable bags. Each bag will be filled half way, sealed, and placed in another sealable gallon-sized Ziploc 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. Two stations per lake or non-wadeable streams are sampled on each sampling date for a total of approximately 9.0 L of collected sediment.

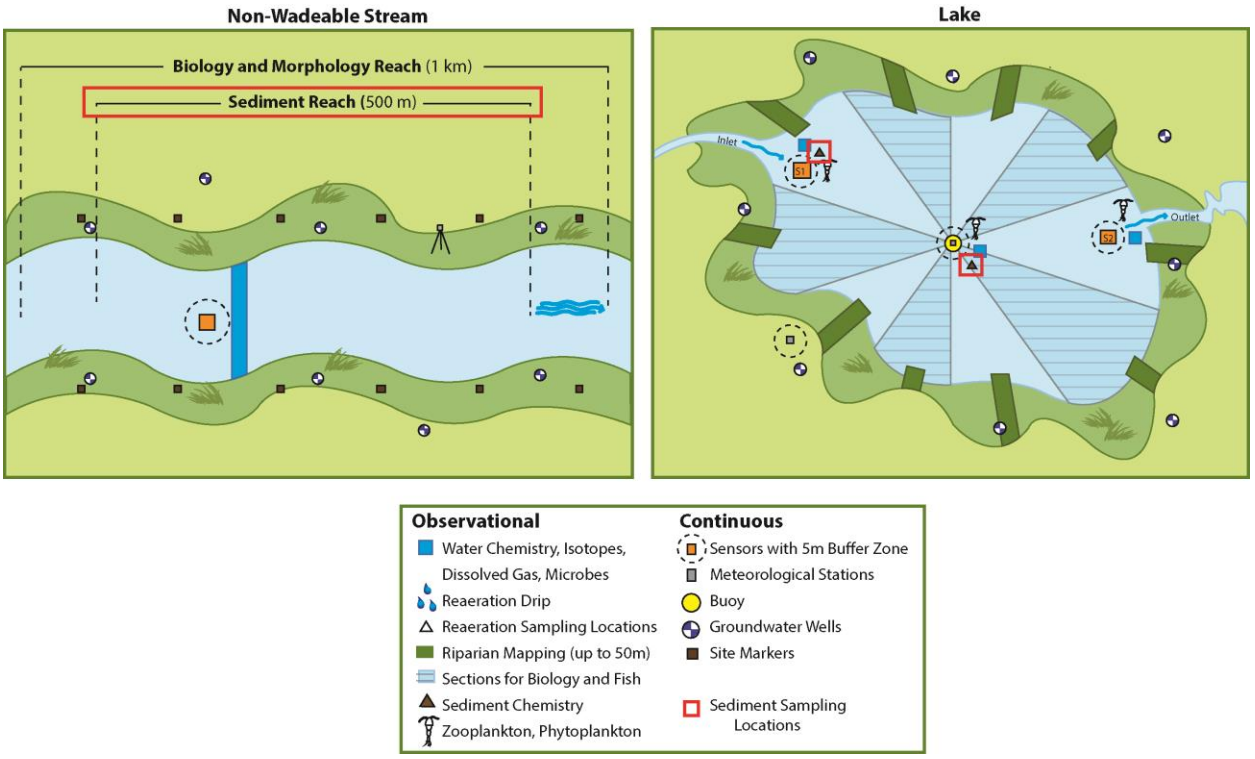

Figure 2. Generic non-wadeable stream and lake site layouts with sediment sampling locations

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 technicians **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 technicians 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 Data and Data Product Quality Assurance and Control Plan (AD[06]).

4 SAMPLING SCHEDULE

4.1 Sampling Frequency and Timing

Sampling for lake and non-wadeable stream bottom sediments shall occur up to 5 times per year at the determined sampling dates. Sediments will be collected during the three biological sampling bouts 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]).

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, allowing for the settlement of suspended sediments into depositional environments. Should a major event occur that disrupts sediments lake sediment samples should not be collected for at least 5 days following a major precipitation event. 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, +/- 2 hours of previous bout start date.

4.2 Criteria for Determining Onset and Cessation of Sampling

The NEON aquatic program will be sampling non-wadeable stream and lake sediments 3 times per year during the spring, summer and fall sampling bouts. Two additional sample events may be warranted following a major storm event that alters the morphology of the system. Sampling bouts will occur approximately every 2 months and during base flow and/or stable flow conditions to provide maximum direct access to the stream bed and to minimize seasonal streamflow variability. 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 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. Ship samples to the external Laboratory within 72 hours following processing. See Appendix E for a complete list of sediment chemistry analyses, storage requirements, and hold times. 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.	No adverse outcome.
	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 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. 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 (Mustang jackets) 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.	R/S	Description	Purpose	Quantity	Special Handling
Durable items					
	R	60 mL syringes	Syphoning water from samples	2	N
	R	Stopcocks	Syphoning water from samples	2	N
	R	50 cm of Tygon tubing 1/8” inner diameter	Syphoning water from samples	2	N
	R	Wash Bottle, plastic, 500mL	Rinsing sediment from samplers into buckets	1	N
	R	Wash Bottle, Teflon, 500mL	Decontaminating equipment with methanol	1	N
	R	Plastic Funnel 13 oz.	Collecting inorganic, nutrient and size analysis samples	1	N
	R	Stainless Steel Funnel 13 oz.	Collecting organic samples	1	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	R	Plastic spatula	Collecting inorganic, nutrient and size analysis samples	1	N
	R	Stainless steel spatula	Collecting organic samples	1	N
	R	Glass Bowl 4 qt.	Homogenizing samples	2	N
	R	Flexible forceps, featherweight	Removing debris from samples	1	N
	R	Tool Kit		1	N
	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
	R	Stainless Steel Bucket 5 gallons w/ lid	Homogenizing organic samples	1	N
	R	Plastic bucket 5 Gallons w/lid	Homogenizing inorganic, nutrient and size analysis samples	1	N
	R	Scoop, Polyethylene	Collecting inorganic, nutrient and size analysis samples	1	N
	R	Scoop, Stainless Steel	Collecting organic samples	1	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	R	Petite ponar	Sampling	1	N
MX100514	R	Multisonde	Measuring % DO, temperature and salinity	1	N
	R	4,000 µm sieve	Sieving samples before transferring into bottles	1	N
	R	Coolers	For shipping, provided by the external lab	1	N
	R	Boots and/or hip waders	Safe wading	2	N
	R	First Aid Kit	Safety	1	N
	R	Camera	Photographing sampling observations	1	N
	R	Sonar with GPS antenna	Navigating to sampling locations	1	N
Consumable items					
	R	Ice Pack	Keeping samples cool, provided by the external lab	Multiple	N
	R	Nitrile gloves, in plastic bag	Not contaminating samples	Multiple	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	R	Clear Boston-style round glass jar, 8 oz. (~250 mL), for organic and inorganic analyses	Sample container, provided by the external lab	4	N
	R	Clear Boston-style round glass jar, 4 oz. (~120 mL), for Total Organic Carbon/Total Carbon	Sample container, provided by the external lab	2	N
	R	Clear Ziploc-style 1-gallon plastic bag for sediment grain size analyses	Sample container, provided by the external lab	4	N
	R	Foil, aluminum, heavy duty, one roll	Storing equipment and avoiding contamination	1	N
	R	Equipment sealable bags, 5 gal	Transporting equipment	8	N
	R	Trash Bags 13 Gal	Transporting equipment	4	N
	R	Permanent Markers	Labeling samples	3	N
	R	Bottle labels	Labeling samples	14	N
MX100351	R	Phosphate free detergent	Decontaminating equipment	1	N
	R	Nitric acid	Decontaminating equipment	1	Y

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	R	Methanol	Decontaminating equipment	1	Y

R/S=Required/Suggested

Table 3. Equipment list – General boating equipment

Item No.	R/S	Description	Purpose	Quantity	Special Handling
Durable items					
	R	Boat		1	Y
	R	Anchor with rope		1	N
	R	Oars		2	N
	R	Trolling Electric Motor		1	Y
	R	Battery (12 volt)		1	Y
	R	Safety kit for boat (e.g., flares, bailer, float with rope)		1	Y
	R	First Aid Kit		1	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	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					
	R	Sieve set, 6 part	Sieving samples	1	N
Consumable items					
	R	Gloves, nitrile (box)	Not contaminating samples	1	N
	R	Kimwipes (box)	Cleaning	1	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	R	Bottle labels	Labeling samples	8	N
	R	Forms, analytical request (TBD)	Shipping samples	Multiple	N
	R	Clear Boston-style round glass jar, 8 oz. (~250 mL), for organic and inorganic analyses	Sample container, provided by the external lab	4	N
	R	Clear Boston-style round glass jar, 4 oz. (~120 mL), for Total Organic Carbon/Total Carbon	Sample container, provided by the external lab	2	N
	R	Clear Ziploc-style 1-gallon plastic bag for sediment grain size analyses	Sample container, provided by the external lab	2	N

R/S=Required/Suggested

6.2 Training Requirements

All technicians 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 technicians for 4-6 hours each sampling day plus travel to and from the site.

7 STANDARD OPERATING PROCEDURES

SOP A Preparing for Sampling

1. **Ensure all equipment has been cleaned and stored appropriately** (see SOP B.5).
2. Check the sediment chemistry field sampling kit to make sure all supplies are packed.
3. Ensure GPS locations of sampling stations are entered into the GPS system (Figure 3).
4. Confirm type of sediment samples that will be collected and take the appropriate bottles and collection devices (Figure 4).
5. Use NEON bottle labels; do not use labels provided by the external lab.
6. 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.** Circle the correct bottle type code (Figure 5) on the labels for each bottle.
7. Complete field data sheets on the personal digital assistant (PDA) associated with the sediment chemistry protocol. When the PDA is unavailable, fill out site information on the General Field Sampling Data Sheet (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 PDA or on the data sheets.

a. Record the Date (YYYYMMDD) and the time of day (use local, military time; ex. 13:46) that samples were collected on the stream Sediment Chemistry Sampling Datasheet (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 1 (station 1) or 2 (station 2). Indicate sample type with either "I" (inorganic), "O" (organic), "C" (TOC/TC), or "SS" (sediment size).

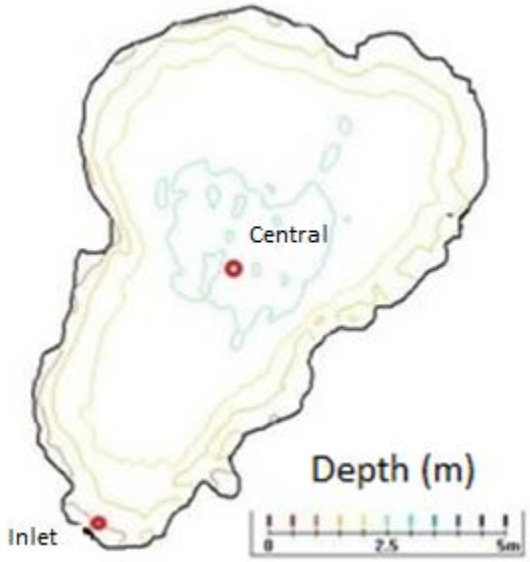

Figure 3. Identifying the Station for sediment sampling in lakes. The two sampling Stations (red circles) should denote depositional zones in the center of the lake (deepest section) and in a nearshore depositional environment

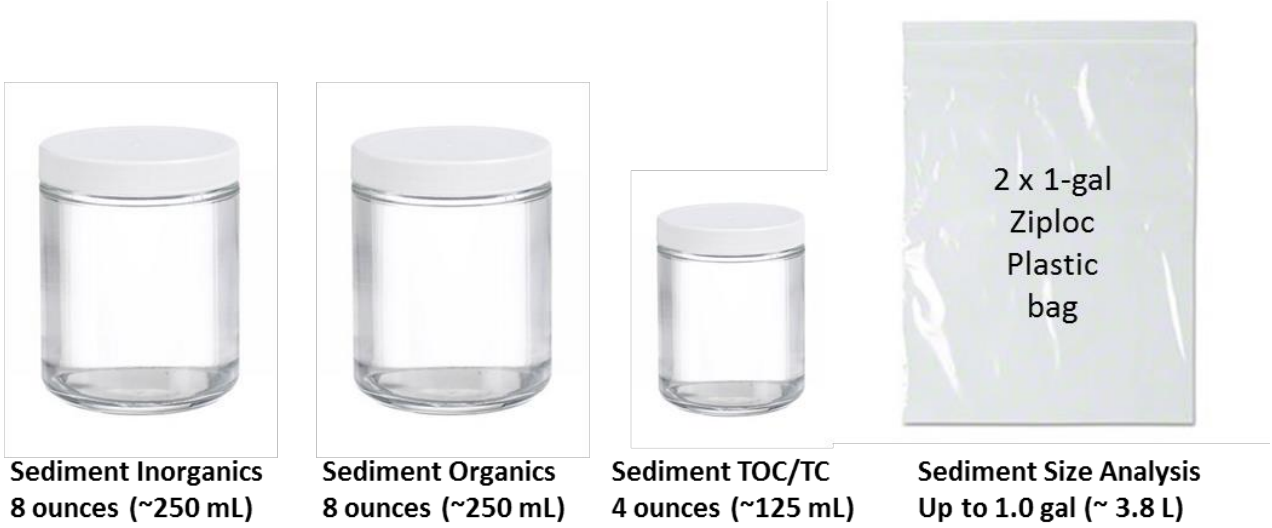

Figure 4. Example of clear Boston round glass jars with PTFE lined lid used for collecting sediment for inorganics, organics, and TOC/TC analyses. Sediments for grain size samples are collected by filling 2 1-gallon (~3.8L) plastic Ziploc bags about half-full.

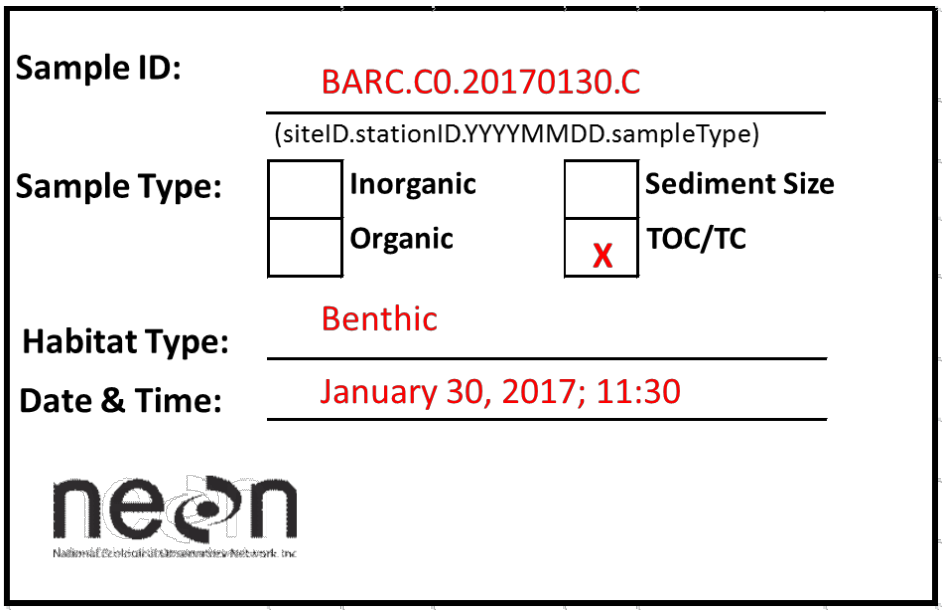

Figure 5. Example of NEON lake sediment chemistry bottle label. Habitat types in lakes may include littoral (inlet) or benthic (buoy). Non-wadeable habitats may include benthic.

SOP B Field Sampling

Before sampling:

1. Identify the suitable sediment sampling stations, zones, and point locations based on bathymetric and sediment geomorphology maps if available.
2. Place the equipment on the aluminum foil and bags. Be sure not to contaminate bags.
3. Insert ice/ ice packs into the cooler.
4. Line each shipping cooler with a trash bag.
5. Calibrate the multisonde for dissolved oxygen (DO) (see instructions in ER [01]).
6. When in the field, rinse all equipment three times with native water prior to use.

Lake sediment samples will be taken from two areas in the lake: the central and/or deepest part of the lake (representative of the maximum long-term accumulation), and from a nearshore depositional zone (representing an area of shorter term sediment transport and deposition; Figure 3). These Stations can be identified from the site characterization lake bathymetric and morphologic maps (see NEON Bathymetric Mapping Protocol, (RD[09]). The sampling zones must be 5 meters from aquatic sensors but no more than 10 meters beyond the sensor exclusion zone (Figure 6).

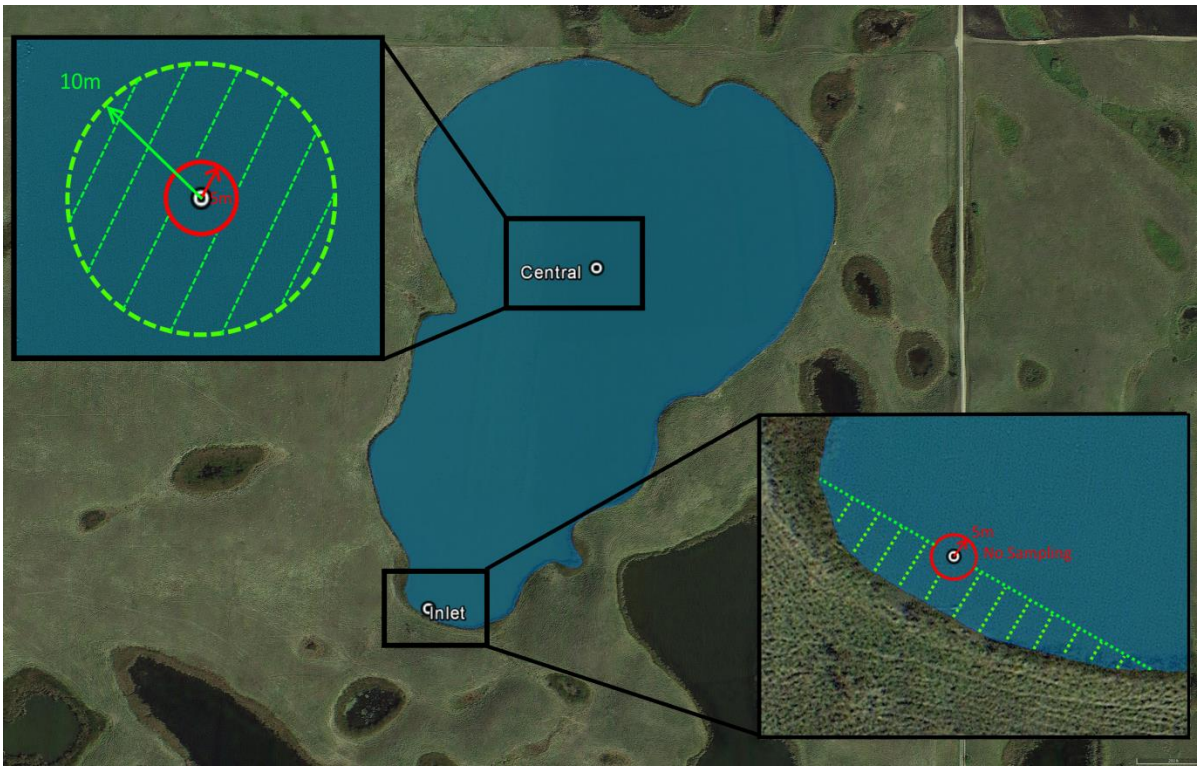

Figure 6. Example lake sediment collection stations with sensor exclusions areas (red) and sampling zones (green).

Non-wadeable stream sediment samples shall be collected from two identified sediment-sampling stations within the stream reach each covering up to 500 m of the AQU reach. The station divide is defined by the mid-way location between the top and the bottom of the AQU reach. At each of the 2 sampling stations, identify at least 5 depositional zones containing fine-grained particulate matter (Figure 7). 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 report or the stream morphology maps. If the site has no large depositional zones, take samples from depositional environments as per Figure 7b. Likewise, should the streambed be mostly sedimentary or organic in nature, then distribute the samples as per Figure 7c. The areal extent of each zone should be estimated and if possible quantified. Due to the affinity of metals and other nutrients to bind to smaller sized particles (<2 mm), this size fraction found in depositional zones better represents the potential quantification of sediment chemistry. The zones should not interfere with the biological sampling locations when possible; sediment sampling should occur 5 meters away from the instream sensor.

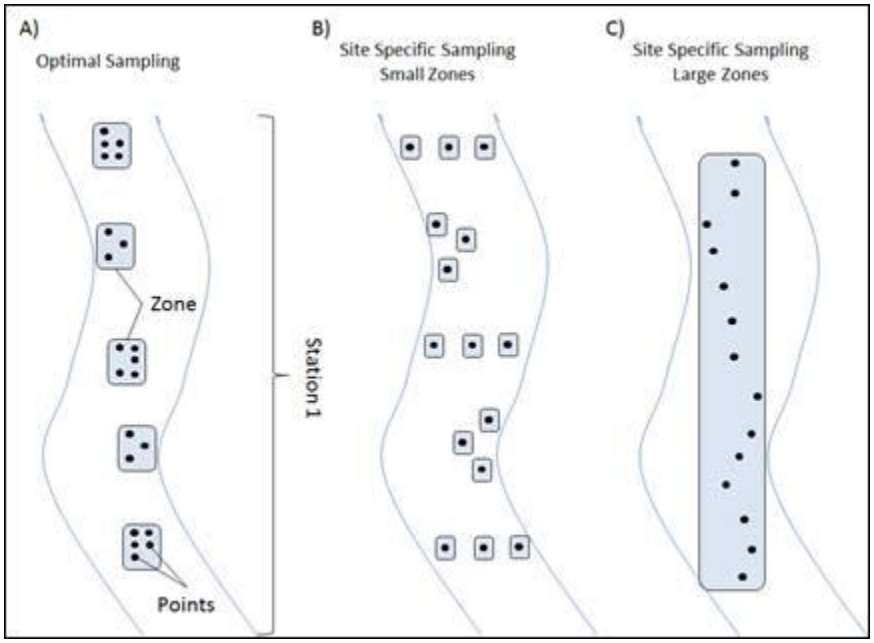

Figure 7. Diagram to exemplify determination of sampling zones in non-wadeable streams based on site differences. A) Ideal sampling set up based on availability of different depositional zones (~1 m^2); B) in sites with only random small pockets of sediment; and C) in sites where most of the stream bed is characterized by sedimentary or organic substrate.

B.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. Measure the % oxygen, temperature and specific conductivity (µS/cm) of the water about 50 cm above the sediment in each sampling zone and note data on the field sampling sheet (RD[05]).
4. Note the GPS position of the sampling zone on the field sampling sheet.
5. Repeat at each sediment sample zone right before taking samples.
6. Take sediment sample (see SOP B.3 and SOP B.4).
7. Proceed to next station and repeat steps 1-5.

B.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 upwind (or upstream) of the sampling site, and using an anchor line 3 times as long as the depth of the lake or 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 about 50 cm above the sediment in each sampling zone and note data on the field sampling sheet (RD[05]).
5. Note the approximate GPS position of the sampling points on the field sampling sheet.
6. Repeat at each sediment sample zone right before taking samples.
7. Take up to 5 sediment samples (see SOP B.3 and SOP B.4).
8. Proceed to next station and repeat steps 1-6.

B.3 Sampling Lakes and Non-Wadeable Streams with Petite Ponar Sampler

The ponar sampler is used to collect bed sediments regardless of grain size composition (Figure 8). For collecting nearshore or shallow sediments that are difficult to collect with the ponar a hand scoop may be used (see SOP B.4 below).

Figure 8. Petite ponar sampler for use in lakes and non-wadeable streams

1. Put on gloves (nitrile).
2. Set the grab sampling device with the jaws cocked open (Figure 8). Great care should be taken while handling the device while it is set; accidental closure can cause serious injuries.
3. Ensure that the rope is securely fastened to the sampler, and that the other end of the rope is tied to the boat.
4. 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.
5. 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. Retrieve the sampler slowly and steadily to minimize the effect of turbulence (that might result in loss/disturbance of surface sediments).
6. Place glass bowl A beneath the sampler just as it breaks the surface of the water.
7. Open the doors on the top of the sampler and transfer the sample into the glass bowl B. Use a stainless scoop for collecting organic samples; use the TOC/TC equipment for collecting those samples, and use a plastic scoop for inorganic samples to subsample the middle portion of the sediment that was not in direct contact with the ponar. 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).

NOTE: If the jaws were not closed completely, 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.

8. Immediately record observations regarding the appearance of the sediment, presence of detritus, and the depth of sediment sampled.
    a. Example of sediment observations could include texture (clumpy, watery, clay-like), color, odor (sulfur, earthy, sweet), and the presence of biota (invertebrates, plant material).
9. Repeat 1-7 the process at 3-5 points (or 5 per m^2) within each depositional zone.
10. Composite the samples from each of the zones in glass bowl B (or a bucket; stainless steel for organic samples and plastic for inorganic samples) with a spatula (metal for organic or TOC/TC samples; plastic for inorganic samples) ensuring approximately the same amount of sediment is contributed from each zone.
11. If the sample contains 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.

12. 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. Place a lid over the bowl or collection bucket to reduce sediment exposure to the atmosphere. Minimize the time that the plastic syringe or tubing is in contact with sediments sampled for organics.
13. Use the funnel (metal for organic samples; plastic for inorganic samples) to distribute the composited sample into the sample collections jar for the organics, organic contaminants, and TOC/TC analyses.
14. Repeat steps 1-12 using a plastic spatula and plastic funnel to distribute the composite sample from the glass bowl or plastic 5-gallon bucket into the 8 oz. (~250 mL) clear glass sample collection jar for inorganic sample analyses.
15. Collect enough sediment to fill 2 1-gallon plastic Ziploc bags at least halfway 1 gallon; ~3.8 L) with sediment for grain size analyses. Be sure to collect two or more sediment grabs with the sample even if one ponar grab provides enough material. Multiple composited samples from one location will provide a more representative chemical and physical characterization of the sediments.
16. Collect a total volume of approximately 4.5 L of wet sediment per Station using the appropriate tools for field collection and homogenization.

NOTE: Collected sediment material from each station will include one glass jar (8 oz., 250 mL) of organic sample, one glass jar (4 oz., 125 mL) of TOC/TC sample, one glass jar (8 oz., 250 mL) of inorganic sample, and one gallon (~3.8 L) of sediment in 2 1-gallon sized half-filled Ziploc-style plastic bags. The same number of samples and containers will be collected from the second station.

17. Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles.
18. Clean the Petite ponar sampler with the scrub brush and rinse with native water between sampling stations. Make sure any residual sediment is visibly removed from the sampler.
19. Proceed to SOP A and SOP D for sample treatment and shipping preparation in the field.

B.4 Sampling with a Scoop

Use the scoop sampler when collecting nearshore or shallow samples where the sediments are between plant roots or rocks (Figure 9).

Figure 9. Hand scoop sampler for use in the nearshore 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) whilst sampling in the stream in order to minimize the disturbance from entering and exiting the stream.
3. Collect samples from up to a maximum of 5 points per depositional zone greater than one square meter (Figure 3). If the depositional zones are less than 1 m^2, a minimum of 2 points within each depositional zone should be collected.
    a. Remove the top layer of fine sediment carefully (approximately 1-3 cm) by gently scooping in the upstream direction (ensure the scoop is metal when sampling for organics, organic contaminants, and TOC/TC; use Teflon when sampling for inorganics).
    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. For all inorganic samples, place sediment in the plastic bucket and proceed with the next sample. Composite samples from all zones ensuring that approximately the same amount of sediment is contributed from each zone. For organics use the stainless steel bucket and utensils. If fines or organics are left attached to the walls of the scoop, use a Nalgene wash bottle filled with native water to rinse down the sediments from the scoop into the bucket. Use the glass bowl to further composite samples if needed following settlement of suspended particles.
5. Repeat 1-4 the 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.
6. Using forceps, remove any debris or litter from the sample.
7. If the sample contains 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.

8. Use the funnel to distribute the composite sample into the collection bottle for the organics.
9. 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.
10. Repeat steps 1-8 using a plastic spatula and funnel to distribute the composite sample into the collections bottles for inorganics.
11. Collect a total volume of approximately 4.5 L of wet sediment per Station enough to fill one 8 oz. (~250 mL) glass jar for collecting sediment for organic analyses (including organic contaminants), one 4 oz. (~125 mL) glass jar for TOC/TC analyses, and one 8 oz. (~250 mL) glass jar for inorganic. Collect enough sediment to fill two 1-gallon (~3.8 L) plastic Ziploc bag at least halfway with sediment for grain size analyses.
12. Sediment for grain size analyses can be collected from either the plastic or metal buckets used for compositing the sample material; it is also acceptable to combine sediments from both buckets for the sediment grain size analysis sample.
13. Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles. DO NOT FREEZE.
14. Dispose of any excess sediment not collected for external lab analyses near the shore, away from depositional zones or areas frequently sampled (inlet or lake center).
15. 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.
16. Proceed to E 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.

B.5 Ending the Sampling Day

1. Refreshing the sampling kit
   a. Re-order sample kit (bottles and coolers) from 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 3.
2. Equipment maintenance, cleaning and storage
   a. Refer to NEON Aquatic Decontamination Protocol (RD[08]).
   b. Ensure all containers, tools, and equipment used for **inorganic, organic, TOC/TC, and sediment size sample collection** are cleaned prior to storage or reuse:
      1) Prepare a tub with 0.2% phosphate free detergent.
      2) Wash and soak the equipment for at least 30 minutes.
      3) Rinse thoroughly with tap water three times and then with de-ionized water three times using new water each time.
      4) Store glass bowls wrapped in foil.
   c. Preparing equipment for **inorganic** sampling:
      1) After the detergent soak described above, rinse with the containers/equipment with 5% high purity nitric acid (HNO_3).
         a) 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!**
         b) VERY IMPORTANT, consult the domain specific chemical hygiene plan for disposal of acid waste or other hazardous chemicals.
      2) Rinse three times with deionized water.
      3) Allow to air dry.
      4) 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.
   d. Preparing equipment for **organic** and **organic-contaminant** sampling:
      1) After the detergent soak described above, rinse containers and equipment with methanol from the Teflon wash bottle. Allow methanol rinse to air dry.
      2) 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.
   e. Preparing equipment for **Total Organic Carbon** and **Total Carbon (TOC/TC)** sampling:
      1) After the detergent soak described above, rinse containers and equipment with methanol from the Teflon wash bottle.
      2) Rinse three times with deionized water. Allow to air dry.
      3) 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.

B.6 Sample Processing Timing

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

SOP C 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 D 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.

D.1 Handling Hazardous Material

N/A

D.2 Supplies/Containers and Conditions

1. Place glass bottles in individual sealable plastic bag, place the gallon-size closeable bags in a second sealable plastic bag, and place in packing material (foam sleeves) for protection from breaking.
2. Place the round glass sample jars and gallon closeable plastic bags into the 5 gallon cooler with ice packs.
3. 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]).
4. Tie the garbage bag; security seals across the opening of the cooler and ship to address provided by NEON.

D.3 Timelines

Ship samples to the external Laboratory within 72 hours following field processing and preservation. Ship samples "Priority Overnight."

D.4 Grouping/Splitting Samples

N/A

D.5 Return of Materials or Containers

N/A

D.6 Shipping Inventory

Include sample shipment inventory (RD[11]). Email shipping inventory to external lab contact and copy the NEON CLA contact.

D.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 are associated with this protocol:

Table 5. Datasheets associated with this protocol

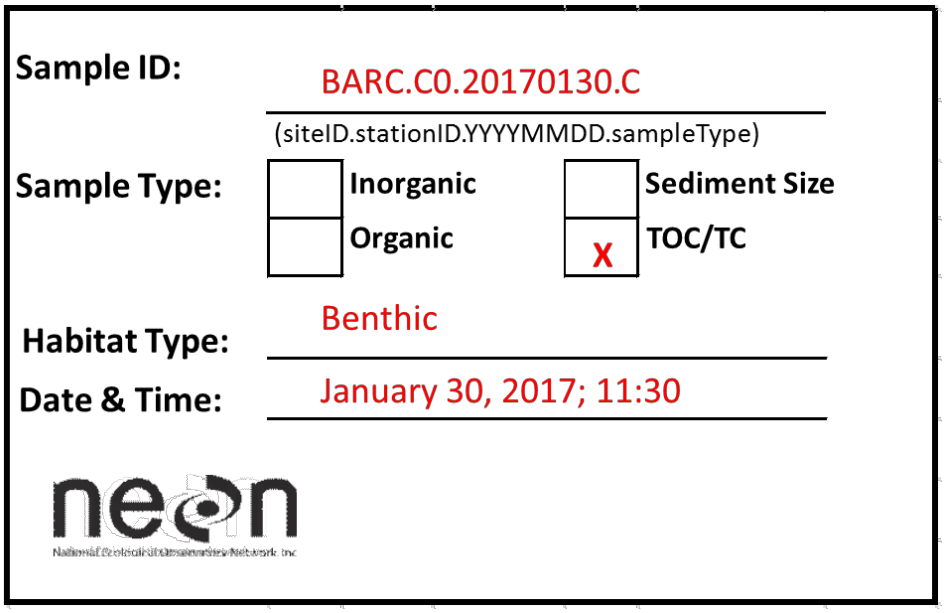
NEON Doc. #	Title
NEON.DOC.002435	Datasheets for AOS Protocol and Procedure: Sediment Chemistry Sampling in Lakes and Non-Wadeable Streams
NEON.DOC.001646	General AQU Field Metadata Sheet
NEON.DOC.002494	Datasheets for AOS Sample Shipping Inventory

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").



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 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 technicians 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: Collected sediment material from each station will include one glass jar (8 oz., 250 mL) of organic sample, one glass jar (4 oz., 125 mL) of TOC/TC sample, one glass jar (8 oz., 250 mL) of inorganic sample, and one gallon (~3.8 L) of sediment in 2 1-gallon sized half-filled Ziploc-style plastic bags. The same number of samples and containers will be collected from the second station.**

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 AQU's NEON 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