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

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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 0 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 cores 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 the fine grained fraction analyzed for the elements of interest. For trace element analysis, the silt-clay fraction smaller than 63 µm will be used. For organics, sand and silt-clay fractions smaller than 2.0 mm will be used.

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.001155	NEON Training Plan
AD[05]	NEON.DOC.050005	Field Operations Job Instruction Training Plan
AD[06]	NEON.DOC.014051	Field Audit Plan
AD[07]	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 Acronyms

Acronym	Definition
cm	Centimeter
g	Gram
km^2	Square kilometer
L	Liter
µL	Microliter
µm	Micrometer
M	Metals

m	Meter
mL	Milliliter
mm	Millimeter
O	Organics
SS	Sediment Size
TE	Trace Elements

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 US 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). In non-wadeable streams, samples will be taken from two stations, each representing composited samples from between 5-10 depositional zones. Depositional zones represent the net outcome of multiple processes and flows. In a given depositional zone, samples will be taken at two or five points and composited with samples from other 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. If depositional zones are <1 m^2, 2-3 point within the zone will be sampled. Partially wetted zones (< 5 cm water) should only be sampled when no other sites are available, and sampling conditions should be documented in the field notes and data records as a potential outlier. Each sample will consist of the surficial 3 cm of bed sediment. Compositing samples allows for greater representativeness of mean concentrations for the area and results in smoothing of variability otherwise encountered between depositional areas.

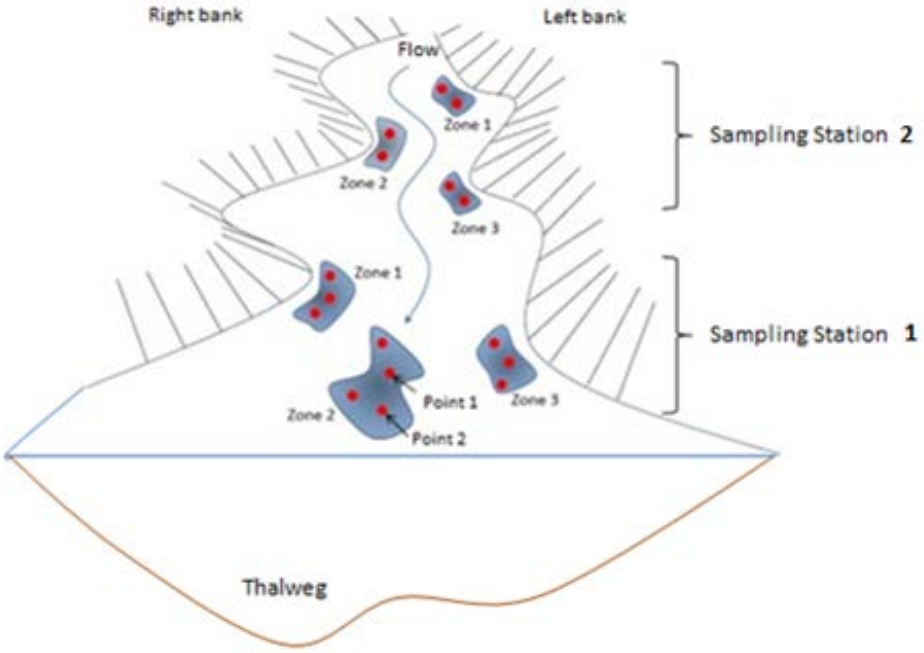

Figure 1. Identifying the location for sediment sampling in non-wadeable streams. The two sediment sampling stations should contain depositional zones that represent upstream influences and various flow regimes; left bank (looking downstream), right bank ((looking downstream), central channel, and different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

The coring systems used in non-wadeable streams are similar to those for lakes. For softer sediments a gravity corer will be used to extract the sediment samples with minimal impact on the surface sediments. For harder sediments a Petite ponar sampler will be used (Figure 3).

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. The same coring systems used in non-wadeable streams will be used in lakes and will depend on the sediment type. For softer sediments a gravity corer will be used to extract the sediment samples with minimal impact on the surface sediments. For harder sediments a Petite ponar sampler will be used (Figure 3). 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 sample container per type of sediment will be collected at each station, for a total of 4 containers per station. Two stations per lake or non-wadeable streams are sampled on each sampling date for a total of 8 sample containers per sampling date.

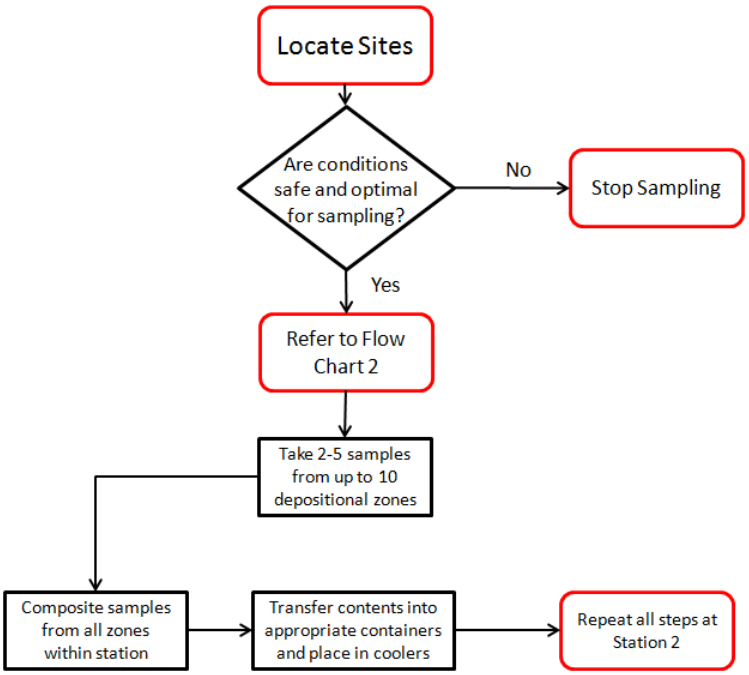

Figure 2. Decision flow chart 1 for determining the number of samples to be taken at each location

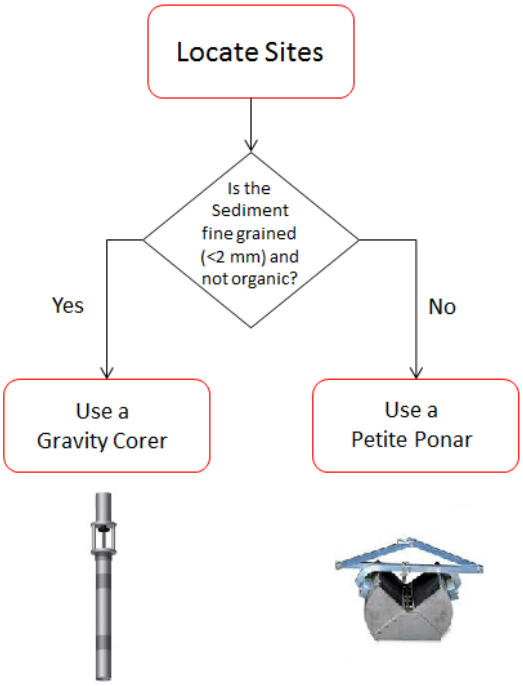

Figure 3. Decision flow chart 2 for determining appropriate sampler to use in the field

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[06]). 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[07]).

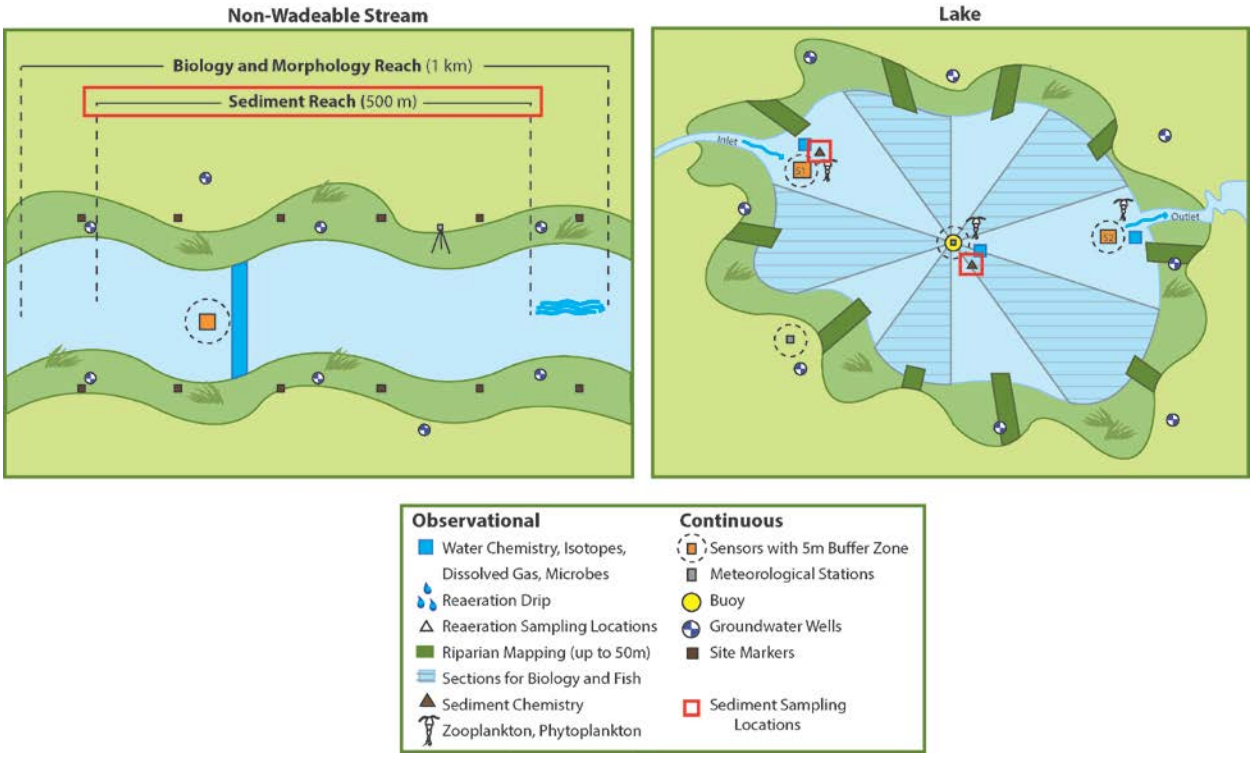

Figure 4. Generic non-wadeable stream and lake site layouts with sediment sampling locations

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. 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 (>25% change in flow within 30 minutes), allowing for the settlement of suspended sediments into depositional environments. Samples will be taken approximately every 2 months throughout the year. In lakes, samples should be taken every 2 months throughout the year but should capture the major turnover time in the spring and autumn, at relevant sites. 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, regardless of the time of day, +/- 2 hours of previous sampling date.

4.2 Criteria for Determining Onset and Cessation of Sampling

The NEON aquatic program will be sampling stream and lake sediments up to 5 times per year per site (other than metals that will be sampled 1 time per year during the summer base flow conditions). Sampling will occur 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 and preservation. All samples should be processed within 1 day from sampling.

Analysis:

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

4. Sediment samples collected for size analysis may be stored indefinitely prior to analysis.

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.	No adverse outcome.
	Should flow rates change (>25% baseflow) during sampling, stop work for the day and allow for flow conditions to stabilize.	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 poor.
2. All personnel using a boat are required to wear an approved personal flotation device.
3. When handling hazardous products (such as HCl) follow laboratory safety standards.
4. Wear gloves, a laboratory coat and protective eyewear.

5. Due to accessibility constraints at some sites, most sampling will have to take place from the boat, without dismounting from the vessel.
6. In areas with alligators, extra precaution must be taken by 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 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	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
	R	Plastic spatula	Collecting inorganic, nutrient and size analysis samples	1	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	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	4 L jug	Collecting native water	2	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	Bucket	Storing core tubes	1	N
	R	Gravity Corer	Sampling	1	N
	R	Petite ponar dredge	Sampling	1	N
	R	Core extruder	Removing sample from corer	1	Custom
MX100514	R	Multisonde	Measuring % DO, temperature and salinity	1	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	R	Sieve set, 6 part	Sieving samples before transferring into bottles	1	N
	R	Coolers, shipping, 1 gallon	Shipping	1	N
	R	Coolers, shipping, 5 gallons	Shipping	1	N
	R	Boots and/or hip waders	Safe wading	2	N
	R	First Aid Kit	Safety	1	N
	R	Camera	Photographing samples	1	N
	R	Sonar with GPS antenna	Navigating to sampling locations	1	N
Consumable items					
	R	Ice Pack	Keeping samples cool	6	N
	R	Latex gloves, in plastic bag	Not contaminating samples	Multiple	N
	R	Amber glass bottle, 1 L, for organics	Sample container	2	N
	R	Amber glass bottle, 1 L, for metals	Sample container	2	N
	R	Amber glass bottle, 1 L, for trace elements	Sample container	2	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	R	Plastic bottle, plastic, 1 L, for size analyses	Sample container	2	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	Teflon tape	Wrapping corer threads	1	N
	R	Electrical tape, roll	Securing the plug at bottom of core tube	1	N
	R	Masking tape, roll	Labeling core tubes	1	N
	R	Trash Bags 13 Gal	Transporting equipment	4	N
	R	Foam	Packing glass bottles	1	N
	R	Permanent Markers	Labeling samples	3	N
	R	Bottle labels	Labeling samples	14	N
	R	Amber glass bottles, 1 L, spares	Sample container	3	N
	R	HDPE bottle 500 mL	Sample container	1	N
	R	Vaseline bottle	Creating suction on the hand corer	1	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	R	Phosphate free detergent	Decontaminating equipment	1	N
	R	Nitric acid	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

Item No.	R/S	Description	Purpose	Quantity	Special Handling
	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					
	R	Plastic wash bottle	For detergent	1	N
	R	Teflon wash bottle	For methanol	1	N
	R	Container, waste, solvent, 2 gallon	Disposing chemicals	1	N
	R	Container, waste, acid, 2 gallon	Disposing chemicals	1	N

Item No.	R/S	Description	Purpose	Quantity	Special Handling
Consumable items					
	R	Deionized water (DI) (L)	Rinsing equipment	5	N
	R	Detergent, phosphate free, 0.2% (L)	Decontaminating equipment	4	N
	R	Methanol (CH_3OH) (L)	Decontaminating organic-contaminant equipment	1	Y
	R	Hydrochloric Acid (HCL) acid, 5% (L)	Decontaminating equipment	3	Y
	R	Nitric Acid (HNO_3), 5% (L)	Decontaminating trace-element equipment	3	Y

R/S=Required/Suggested

Table 5. 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
	R	Nylon Sieve Frame	Sieving samples	1	N
Consumable items					
	R	Nitex sieve material, 63 µm	Sieving samples	1	N
	R	Gloves, latex (box)	Not contaminating samples	1	N
	R	Kimwipes (box)	Cleaning	1	N
	R	Bottle labels	Labeling samples	8	N
	R	Forms, analytical request (TBD)	Shipping samples	Multiple	N
	R	Amber glass bottle, 500 mL, for organics	Sample shipping container	2	N
	R	Amber glass bottle, 500 mL, for metals	Sample shipping container	2	N
	R	Amber glass bottle, 500 mL, for trace elements	Sample shipping container	2	N
	R	Plastic bottle, plastic, 500 mL, for size analyses	Sample shipping container	2	N

R/S=Required/Suggested

6.2 Training Requirements

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

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

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 and 1-2 hours of laboratory work at the Domain Support Facility.

7 STANDARD OPERATING PROCEDURES

SOP A Preparing for Sampling

1. Ensure all equipment has been cleaned appropriately.
2. Check the sediment chemistry field sampling kit to make sure all supplies are packed.
3. Ensure GPS locations of sampling stations are entered into the GPS system (Figure 6).
4. Confirm type of sediment samples that will be collected and take the appropriate bottles and collection devices (Figure 3, Figure 5, Figure 8, Figure 9). Not all samples will be collected each trip.
5. Use a Sharpie to fill out bottle labels (Figure 7). **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 7) on the labels for each bottle.

Figure 5. Example of Amber bottles used for collecting organics, metals and trace elements and a Teflon bottle for sediment size samples

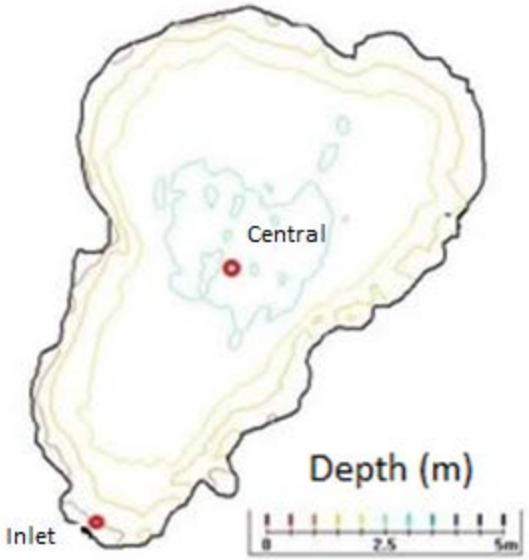

Figure 6. Identifying the Station for sediment sampling in lakes. The two sampling Stations (red circles) should denote depositional zones in the center of the lake (deepest section) and in a nearshore depositional environment

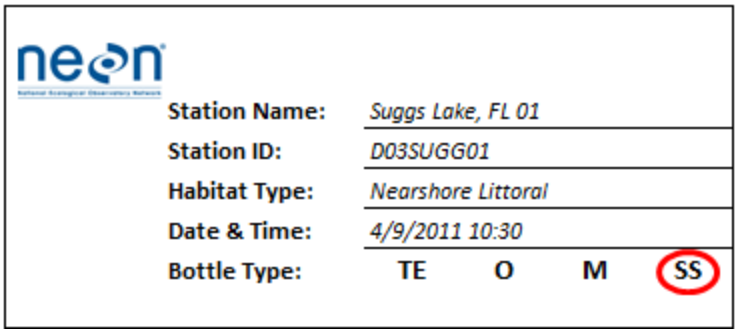

Figure 7. Example of NEON sediment chemistry bottle label. TE- Trace Elements; O- Organics; M- Metals; SS- Sediment Size

SOP B Field Sampling

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). These Stations can be identified from the site characterization lake bathymetric and morphologic maps (see NEON Bathymetric Mapping Protocol, (RD[09])(Figure 6).

Samples for non-wadeable streams will be taken from identified depositional zones as per the Sediment Chemistry Sampling in Wadeable Streams protocol (RD[10]). Identification of such zones can be aided by using the site characterization non-wadeable bathymetric and morphological maps. For non-wadeable streams, select two primary stations for sample collection. The zones should not interfere with the biological sampling Stations.

Before sampling:

1. Define the sediment sampling stations, zones and point locations based on bathymetric and sediment maps.
2. Place the equipment on the aluminum foil and bags. Be sure not to contaminate bags.
3. Rinse all equipment three times with native water prior to use.
4. Insert ice/ ice packs into each cooler.
5. Line each shipping cooler with a trash bag.

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. Calibrate the multisonde for dissolved oxygen (DO) according to equipment manual instructions.
4. Measure the % oxygen, temperature and conductivity 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 GPS position of the sampling zone on the field sampling sheet.
6. Repeat at each sediment sample zone right before taking samples.
7. Take sediment sample (see SOP B.3 and SOP B.4).
8. Proceed to next station and repeat steps 1-6.

B.2 Lakes

1. Ensure the General AQU Field Metadata Sheet (RD[06]) is completed.
2. Locate the deepest part of the lake (or thalweg for non-wadeable streams) 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. Calibrate the multisonde for dissolved oxygen (DO) (see instructions in ER [01]).
5. Measure the % oxygen, temperature and conductivity of the water about 50 cm above the sediment in each sampling zone and note data on the field sampling sheet (RD[05]).
6. Note the approximate GPS position of the sampling points on the field sampling sheet.
7. Repeat at each sediment sample zone right before taking samples.
8. Take up to 5 sediment samples (see SOP B.3 and SOP B.4).
9. Proceed to next station and repeat steps 1-7.

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

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.

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

1. Put on latex gloves.
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). At this point the slackening of the line activates the mechanism to close the jaws. Retrieve the sampler slowly to minimize the effect of turbulence (that might result in loss/disturbance of surface sediments).
5. Place glass bowl A beneath the sampler just as it breaks the surface of the water.

6. Open the doors on the top of the dredge and transfer the sample into the glass bowl. Use a scoop to subsample the middle portion of the sediment that was not in direct contact with the ponar and place into glass bowl B.

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 lake only after all samples have been successfully collected.

7. Immediately record observations regarding the appearance of the sediment (i.e., texture, color, odor, presence of biota, presence of detritus, and the depth of sediment sampled). Take a photo.
8. Repeat 1-7 the process at 5 points (or 5 per m^2) within each depositional zone.
9. Composite the samples from each of the zones in glass bowl B with a metal spatula ensuring approximately the same amount of sediment is contributing from each zone.
10. Use the funnel to distribute the composite sample into the 1 L collection bottles for the trace elements and organics.
11. If the sample contains many large pebbles and cobbles, sieve the sample prior to transfer to the sample bottle using the US-5 (2000 µm) mesh.
12. Repeat steps 1-11 using a plastic spatula and funnel to distribute the composite sample into the 1 L collections bottles for metals and sediment size.
13. Collect a total volume of approximately 2 times 4 L of wet sediment per Station (enough to fill one 1 L glass bottle for each of O, TE, and M (when measured) and one 1 L plastic bottle for SS), based on the tools used for extraction and homogenization.

NOTE: 1 sample container per analyte type of sediment and sediment size will be collected at each Station for a total of 4 containers per Station. Two Stations per lake or non-wadeable stream are collected on each sampling date for a total of 8 sample containers.

14. Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles.
15. Collect a 4 L jug of native water to take back to the lab for sample preparation.
16. Clean the Petite ponar sampler with the scrub brush.
17. Proceed to SOP B.5 and SOP E for sample treatment and shipping preparation in the field.

B.4 Sampling Lakes and Non-Wadeable Streams from a Boat with Gravity Corer

When the stream bed is composed of primarily fine sediment sizes (< sand) and sufficient depth of sediment is present (> 3 cm) the gravity corer should be used for collecting sediments (Figure 9).

Figure 9. Gravity corer sampler for use in lakes and non-wadeable streams

1. Put on latex gloves.
2. Ensure the top of the core tube (flat end, not tapered end) and the area where the plunger and corer housing meet have been sealed with Vaseline (~ once per coring day) (Figure 9). Ensure that the Vaseline is not contaminating other areas of the corer that will come into contact with the sediment.
3. Attach core tube to corer. Make sure flat end of corer is square against ring seal. Tighten hose clamp on tube housing using nut driver, do not over tighten. Core tube should not move within the housing. Raise plunger of corer to "loaded" position. Add the core cone when using in slightly harder sediments.

NOTE: If samples are being collected for Metal analysis, ensure the plastic tube is in place in the corer.

4. Test to see if corer is properly sealed by lowering it into water, triggering the corer, and lifting it to see if the tube is filled and holds water. (If water leaks out, re-check seals and Vaseline, and ensure hose clamp is tightened sufficiently around the core tube). Release water and re-set corer to "loaded" position.
5. Ensure that the rope is securely fastened to the sampler and that the other end is tied to the boat
6. Slowly lower corer into water until corer penetrates into sediment (max. speed =~ 0.5m/sec). It is important not to let the corer freefall, as this can substantially disturb surface sediments.
7. When the line slackens, send the messenger down to `trip' the release mechanism.
8. After plunger is triggered, raise slowly to the surface.

NOTE: NEVER let the bottom of the core tube exit the water until it has been plugged

9. While the core tube bottom is still submerged, insert the core plug (black or orange plug) to form a seal in bottom of the corer.
10. While holding the bottom of the core tube and plug to prevent plug from falling out, lift the corer slowly out of water.
11. Ensure the core is secured and loosen the hose clamp securing core tube to corer.
12. Detach the core tube from the corer, with one person holding the sampler in a vertical position while the second person dismantles the unit.
13. Secure the plug at bottom of core tube with electrical tape to prevent the plug from accidentally slipping. Add the cap to top of the core. Label core tube with masking tape and permanent ink. (lake name, site, date, person sampling).
14. Immediately record (in the field log book) observations regarding the appearance of the sediment (i.e., texture, color, odor, presence of biota, presence of detritus, and the depth of sediment sampled) (RD[05]).
15. Measure the length of the core to the nearest 0.1 cm and record on the Field sheet (RD[05]).
16. Place the core in the bucket while preparing extruder. If sediments got stirred while extracting the core, let the core sit for a while until sediments are settled riot to sampling.

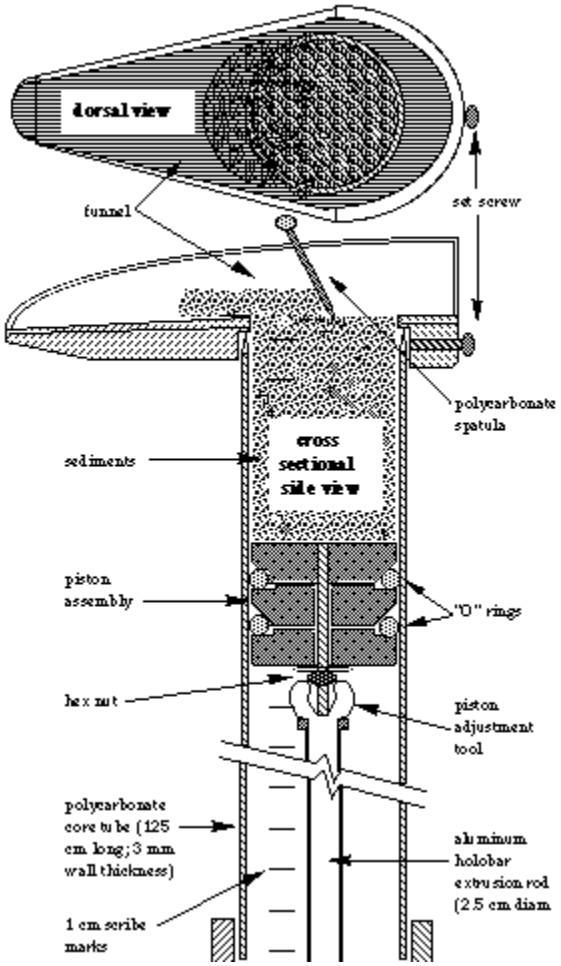

Figure 10. Set-up for sediment sub-sectioning and extrusion (from EPA, 2012)

17. Position the extruder under the corer tube plug at the base of the coring tube (Figure 10). Supporting both the core tube and the extruder in a vertical position, slowly lower the coring tube onto the extruder.
18. Using a syringe and tube decant the overlying water until about 5 cm above the sediment surface.
19. Continue to lower the corer on the extruder until the sediment is approximately 1 cm below the top of the tube (Figure 10).
20. Remove the remaining water from the top of the tube using the syringe with attached tubing and be sure to not disturb the surface sediments.
21. Secure the sectioning stage onto the top of the coring tube. Place the on the stage directly over the coring tube. Slowly extrude the sediment core in the sectioning tube until the top of the sediment reaches the 3 cm line on the sectioning tube.

22. Slide the sectioning tube onto the flat part of the stage and scrape the top 3 cm section of sediment into a clean glass bowl A ready to homogenize.
23. Repeat steps 1-22 until enough sediment is collected.
24. With a clean metal spatula spatula carefully stir the sediment to homogenize, then scoop an aliquot into the pre-labeled sediment sample bottle using the appropriate funnel (see Figure 5 for appropriate container).

NOTE: For samples that are to be analyzed for organics, the spatula and container must not be plastic (the container must be a glass bottle). For samples that are to be analyzed for metals, the spatula must not be metallic.

25. Transfer the homogenized sample to the appropriate bottles using the appropriate funnel.
26. Repeat steps 1-25 using a plastic spatula and funnel to distribute the composite sample into the 1 L collections bottles for metals and sediment size.
27. Collect a total volume of approximately 2 times 4 L of wet sediment per site (enough to fill one 1 L glass bottle for each of O, TE, and M (when measured) and one 1 L plastic bottle for SS), based on the tools used for extraction and homogenization.
28. Collect a 4 L jug of native water to take back to the lab for sample preparation.
29. Place the samples in a cooler with ice packs as soon as they are transferred to the pre-labeled bottles.
30. Proceed to SOP C 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 Sample Preservation

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

B.6 Ending the Sampling Day

1. Refreshing the sampling kit
   a. 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** equipment is 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.

c. Preparing equipment for **trace-element** sampling:
1) Rinse with 5% high purity nitric acid (HNO_3) solution.
2) Rinse three times with deionized water.
3) Allow to air dry.
4) Store in plastic bags.

d. Preparing equipment for **organic-contaminant** sampling:
1) Rinse with methanol.
2) Rinse with deionized water.
3) Allow to air dry.
4) Store in aluminum foil inside a plastic bag.

SOP C Laboratory Sampling and Analysis

Sediment samples for several types of analyses need to be processed prior to shipping to an external lab for analysis. For the purpose of analysis the samples will be processed within 12 hours of return to the Domain lab.

C.1 Sample Processing Timing

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

C.2 Sieving for Trace Elements

1. Stretch the 63 µm mesh nylon-sieve cloth over the plastic-sieve frame and attach retaining ring (Figure 11).
2. Assemble in series the 63 µm mesh nylon cloth sieve and the plastic funnel over a 500 mL plastic receiving bottle.
3. Decant the native water from the TE bottle and place in a wash bottle.
4. Place a small amount of composite sample from the TE bottle onto the 63 µm mesh nylon sieve with the spatula.
5. Pressure sieve the sample using native water in the wash bottle. The fine sediments pass through the sieve with a stream of water (pressure sieved) delivered by the wash bottle.
6. Work small amounts of bed material through the sieve at a time, discarding the material remaining on the sieve. It is not necessary to sieve all the material that is less than 63 µm in each aliquot.

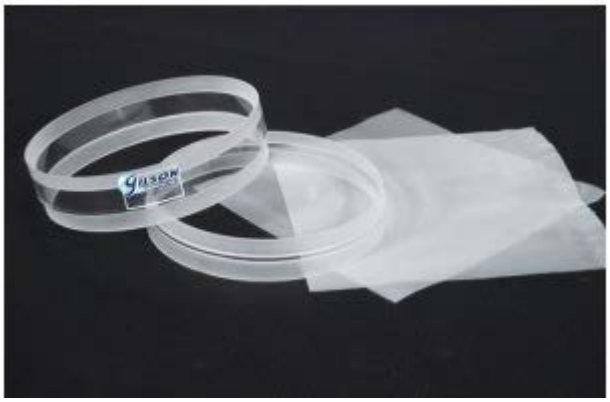

Figure 11. Sieve mesh and sieve frame for sediment sample processing

NOTE: Shaking the sieve will help separate the fines.

7. If additional wash water is needed, allow the sieved sediment/native water to settle several minutes and decant only the native water back into the wash bottle for reuse.
8. If not enough material is collected or if the liquid in the jar is clear or contains minimal fine sediments, filter through a 2000 µm mesh instead.
9. If sieving through a 63 µm mesh is difficult with high organic content, sieve through the 2 mm and then 150 µm mesh first.
10. Continue to reuse the native water until the necessary sample is obtained (a depth of approximately 5 cm in the receiving bottle). The analyses of inorganic constituents will require 10 g (dry weight) of sieved sediment.
11. If a large amount of native water remains in the sample with high fines content, decant. Follow instructions for decanting. If no decanting is necessary store and ship the samples according to SOP E.

C.3 Decanting Trace Element Samples

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

C.4 2 mm Sieving for Organic Contaminant Samples

1. Place the 2.0-mm stainless-steel sieve over a 500 mL glass jar.
2. Gently work aliquots of the sample through the sieve with a spatula. Do not use water. The bottom of the sieve may require periodic removal of the material that adheres to it, using the appropriate spatula.
3. Collect the sample in the jar until nearly full. 300 mL of wet sediment is necessary for analyses of organic contaminants.
4. If excess water is present, let the fines settle and decant as previously.

C.5 2 mm Sieving for Sediment Samples

1. Place the 2.0-mm stainless-steel sieve over a 500 mL Nalgene bottle.
2. Gently work an aliquot of the sample through the sieve with a Teflon policeman or spatula.
3. Collect 300 mL the sample in a 500 mL bottle. The bottle should be nearly full. 50 g of dry weight of material is needed.

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

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.

Information from the General Field Sampling Datasheet (RD[05]) should be entered into excel filed named 'DXX_SC_YYYYMMDD'.

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

1. Pack glass bottles in packing material (foam sleeves) for protection from breaking.
2. Place the O, M and SS bottles in 5 gallon cooler with ice packs.
3. Place the TE bottles in a 1 gallon cooler without ice packs.
4. Place the completed forms in a Ziploc bag and securely tape the bag to the cooler lid, which will help keep the forms dry. Make sure the time and date on the bottle(s) matches the time and date on the form(s) (RD[05]).

E.3 Timelines

Ship samples to the external Laboratory within 12 hours following processing and preservation. Ship samples "Priority Overnight."

E.4 Grouping/Splitting Samples

N/A

E.5 Return of Materials or Containers

Include a return shipping label with the address and account information so the Lab can return the cooler to NEON. Tie the garbage bag; tape the cooler shut and ship to address provided by NEON.

E.6 Shipping Inventory

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

E.7 Laboratory Contact Information and Shipping/Receipt Days

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

8 REFERENCES

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APPENDIX A DATASHEETS

The following datasheets are associated with this protocol:

Table 6. Datasheets associated with this protocol

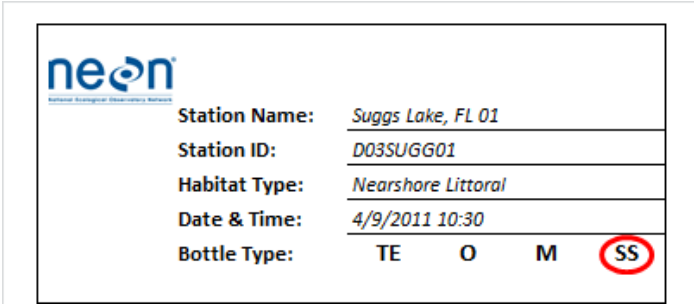
NEON Doc. #	Title
NEON.DOC.002435	Datasheets for AOS Protocol and Procedure: Sediment Chemistry Sampling in Lakes and Non-Wadeable Streams
NEON.DOC.001646	General AQU Field Metadata Sheet
NEON.DOC.002494	Datasheets for AOS Sample Shipping Inventory

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" * 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.

Step 6 – When the stream bed is composed of primarily fine sediment sizes (< sand) and sufficient depth of sediment is present (> 3 cm) the **gravity corer** should be used for collecting sediments.

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

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

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

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

Step 11 – Place the combined samples in a cooler with ice as soon as they are transferred to the pre-labeled bottles. DO NOT FREEZE.

Step 12 – Sieve the samples according to sample type, and transfer them to 500 mL bottles for shipping.

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…

- ☑ 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.
- ☑ When the stream bed is composed of primarily fine sediment sizes (< sand) and sufficient depth of sediment is present (> 3 cm) the **gravity corer** should be used for collecting sediments.
- ☑ For samples that are to be analyzed for organics, the spatula and container must not be plastic (the container must be a glass bottle).
- ☑ For samples that are to be analyzed for metals, 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.
- ☑ Decant any main excess of water (> 3 cm or 100 mL) from the container using a syringe and tube.
- ☑ 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: 1 sample container per analyte type of sediment and sediment size will be collected at each Station for a total of 4 containers per Station. Two Stations per lake or non-wadeable stream are collected on each sampling date for a total of 8 sample containers.**

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 NEON SEDIMENT COLLECTION DATA SHEET

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

NEON Sediment Collection

Lakes and Non-Wadeable Streams

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

Station ID:
N° of Zones:

Zone: Number of Point Samples:

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

Zone: Number of Point Samples:

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

Zone: Number of Point Samples:

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

Zone: Number of Point Samples:

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

Zone: Number of Point Samples:

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

APPENDIX G SEDIMENT COLLECTION CHARACTERIZATION DATA SHEET

NEON Sediment Collection

Lakes and Non-Wadeable Streams

Domain and Site: ______________________

Station ID: ______________________

Habitat Type: ______________________

Sate & Time: ______________________

Weather: ______________________

Collected by: ______________________

Station ID:

Zone:

	Photo N°	Corer Type	Core Length (cm)	Observations
1				
2				
3				
4				
5				

Zone:

	Photo N°		Core Length (cm)	Observations
1				
2				
3				
4				
5				

Zone:

	Photo N°		Core Length (cm)	Observations
1				
2				
3				
4				
5				

Zone:

	Photo N°		Core Length (cm)	Observations
1				
2				
3				
4				
5				

Zone:

	Photo N°		Core Length (cm)	Observations
1				
2				
3				
4				
5				

APPENDIX H SEDIMENT COLLECTION LABELS