Standard Operating Procedure for the Collection, Processing, and Analysis of Sediment Samples

OKLAHOMA WATER RESOURCES BOARD
WATER QUALITY PROGRAMS DIVISION
3800 NORTH CLASSEN
OKLAHOMA CITY, OK 73118

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STANDARD OPERATING PROCEDURE FOR THE COLLECTION, PROCESSING, AND ANALYSIS OF SEDIMENT SAMPLES

1.0 Introduction

The purpose of this document is to provide an outline of sediment sample collection and processing procedures used by the Water Quality Programs Division of the Oklahoma Water Resources Board (OWRB) for all projects that are part of the Lakes Monitoring Program and wetlands studies. The following techniques for sampling will be outlined in this document; an experienced staff member will conduct further training on an as-needed basis. An example of the chain of custody form for the Soil, Water and Forage Analytical Laboratory (SWAFL) at Oklahoma State University (OSU) is attached.

2.0 Definitions/Terms

3.0 Safety

Upon reaching the sampling location, site safety determinations should be made before proceeding. Please refer to the OWRB safety manual for information on boat safety, trailering, and operating on boats (OWRB, 2017).

4.0 Quality of the Measurement

A variety of Quality Assurance/Quality Control (QA/QC) samples are routinely collected to assure that environmental samples meet the Data Quality Objectives (DQO's) outlined in the controlling Quality Assurance Project Plan (QAPP). QA/QC sampling is designed to control each step of the sampling process. The project QAPP should be consulted before trip planning to ensure the appropriate samples are collected. Duplicate samples may be collected to ensure that composite samples are properly homogenized and processed. Replicate samples may be collected to ensure that the sampling methodology employed is collecting a representative, repeatable sample. Spike or known samples may be submitted to test the efficacy of the analytical laboratory.

5.0 Personnel and Equipment

5.1 Personnel

Principal investigators for the OWRB are required to have bachelor's degrees and/or experience with biological or other applicable sciences. Each sampling trip will have a designated crew leader along with other investigators. In all instances, the collection of sediment samples from lakes or wetlands requires at least a two-person field crew. Investigators must be familiar with OWRB standard operating procedure (SOP) documents concerning sediment collections and measurements, as well as habitat assessments and biological collections. Training will follow the methods outlined in these documents; additional training will be provided when new SOPs are developed. Field crew training will be accomplished through dry-run exercises in the laboratory to familiarize crews with sample collection, sample preservation, instrument operation, calibration, and maintenance. In addition, when new personnel are hired or new methods developed, qualified staff will train them on sample collection, measurement, and field analysis methods via side-by-side field trips to familiarize staff with SOP requirements. When training is considered adequate, a qualified staff member will audit field staff for adherence to SOPs.

5.2 Equipment

In addition to regular sampling bottles, a clean and clearly labeled sample container should be included for each site and for each QA code where sediment is collected. For nutrient and/or bulk density samples from lake sites or bulk density samples from wetlands sites, this should be a clean, 4 oz wide mouth glass jar. For composite nutrient samples from wetland sites, this should be a 1-L wide mouth HDPE bottle.

Additionally, for bulk density collections, the 4 oz. glass jar must be pre-weighed on the Optima OPD-A503 precision balance scale located in the OWRB Water Quality Laboratory. These weights must be recorded and labeled on the corresponding jar.

5.2.1 Lakes Collection Equipment

Lake sediment samples are collected using a gravity corer, core tube, and a sectioning apparatus consisting of a stage and a sectioning tube. The Gravity corer kit should include, at a minimum, the following:

Gravity Corer Head (with cable and messenger)
Core Tubes (68 mm diameter, 60 cm length)
Sectioning Tube (with line marked 5 cm from bottom of tube)
Sectioning Stage
Extruder Rod
Core plug
Screwdriver or bit driver
Gloves (latex/nitrile, non-powdered)

An illustration of the gravity corer setup is shown in **Figure 1**

5.2.2 Wetlands Collection Equipment

A hand-driven butyrate tube is used to collect soil cores for nutrient analysis when the target soil is submerged or sufficiently loose or saturated. A hand-driven disposable soil core sampling syringe is used to collect bulk density samples in these same conditions. A standard graduated syringe may be modified by removing the end of the barrel if necessary.

Butyrate Tube (68 mm diameter, 2 m length)
Tube cap
Core plug
Extruder Rod
Sectioning Stage
Sectioning Tube

For soils that cannot be easily collected via hand-driving, a hammer-driven or auguring soil corer with an inserted butyrate core tube should be used for the collection of soils for either nutrient analysis or bulk density.

Handle (fixed or ratcheting, or hammer-drive)
Extensions
Core tube(s)
Corer Head
End attachments (auguring or driving)

6.0 Collection of Lakes Sediment Samples

Collection of sediment samples from lakes using a gravity corer will be conducted according to the methods outlined in the Environmental Protection Agency (EPA) National Lakes Assessment (NLA) Field Operations Manual (USEPA, 2017) and are as follows:

Use of gravity corer:

1. Wear surgical gloves at all times during sample collection to protect yourself from any potential contaminants in the sediments, and to prevent contamination of the sample from trace contaminants on the skin of the sampling crew.
2. If the bottom has been disturbed during the initial depth determination or for any other reason, move at least 5 m to take the core. **It is critical the corer strikes undisturbed surface sediments**.
3. Insert the core tube into the sample housing apparatus and tighten the hose clamp screws to secure the tube. Only tighten/untighten the lower pair of clamps, which secure the sample tube to the apparatus; the upper pair of clamps must remain tightened to the rest of the apparatus. Ensure that the messenger is attached to sampler line. Set the release mechanism.
4. Slowly lower the corer through the water column until the bottom of the core tube is just touching the sediment surface. Raise the corer 0.5 to 1 m and, while maintaining a slight tension on the line, lower the corer, allowing it to settle into the bottom substrate. Immediately after the corer drops into the sediment, maintain line tension to prevent the corer from tilting and disturbing the core sample. Aim to obtain a core at least 6 cm in depth.
5. Trip the corer by releasing the messenger weight so that it slides down the line. Keeping the line vertical and keeping light tension on the line will help ensure that the messenger trips the mechanism.
6. Slowly raise the corer back to the surface, keeping the bottom of the core tube under the water.
7. While keeping the bottom of the core tube under water, reach under the surface with a gloved hand and plug the bottom of the corer with a corer tube plug. Do not tilt the corer more than 45 degrees to accomplish this.
8. Keeping your hand under the corer tube plug, raise the corer into the boat in a vertical position. Stand the corer in a large tub or bucket to prevent contaminating the boat with sediment material.

Sample collection from corer:

1. Detach the core tube from the corer. One crew member should hold the sampler in a vertical position while the other loosens the lower pair of hose clamps to release the core tube from the rest of the device.
2. Position the extruder under the corer tube plug at the base of the coring tube. Supporting both the core tube and the extruder in a vertical position, slowly lower the coring tube onto the extruder until the sediment is approximately 1 cm below the top of the tube. This operation is best done with the core tube standing in a bucket or tub to catch the discarded water.
3. Remove the remaining water above the sediment core by using a syringe with tube (or pipette) so that the surface sediments are not disturbed. Wait a few minutes for flocculent matter to settle, when possible.
4. Secure the sectioning stage onto the top of the coring tube. Place the Plexiglas sectioning tube (marked with a line 5 cm from the bottom) on the stage directly over the coring tube. Slowly extrude the sediment core into the sectioning tube until the top of the sediment reaches the 5 cm line on the sectioning tube. Slide the sectioning tube onto the flat part of the stage and use spatula or spoon to scrape the top 5 cm section of sediment into a clean 4 oz glass jar.

For bulk density samples, follow the same process, ensuring that the sample jar is pre-weighed, and the collected volume of sediment is recorded. **It is important to be as precise as possible with the collected volume.**

6.1 Lake Sediment Sample Processing

All sediment samples will be labeled on the top and side of the jar and immediately placed on ice for preservation. The site, date, time of collection, and volume of sediment collected for each sample will be recorded on a field sheet. Nutrient samples should be delivered to SWAFL with an accurately completed chain of custody as soon after collection as possible for analysis. Bulk density samples should begin the drying process as soon as possible.

7.0 Collection of Wetland Sediment Samples

7.1 Collection of Wetland Nutrient Samples

If target sediments are under water that is too deep for wading, a gravity corer may be employed as described above.

For sediments that are fully saturated or under wadable water use a clean butyrate tube approximately 2 meters in length (or long enough that the top will remain above the water surface) with a ring clearly marked 10 cm from the top and bottom.

1. Cover the top of the tube with an airtight seal.
2. Carefully place the bottom of the tube vertically down into **undisturbed** sediment and gently press down. Remove the top seal once the tube is settled into the sediment.
3. Drive the tube down gently to a depth of at least 10 cm if possible. Particularly dense or clayey soils may prevent this. If only shorter cores can be obtained, measure and record the depth of each core collected.
4. Twist the tube slightly to break the core away, and gently pull the tube up to extract the sample.
5. Once removed, use a core tube plug and extruding rod to push the sample to the other end of the tube.
6. Remove the remaining water above the sediment core by using a syringe so that the surface sediments are not disturbed. Similarly, if excessive amounts of leaf litter or other detritus is present on top of the soil, carefully remove as much as possible without removing any soil from the sample.
7. Extract the top 10 cm of the core (or all, if less than 10 cm) into a clean, labeled 1-L wide mouth sample bottle.
8. Clean the tube and repeat steps 1-7 two times in equally spaced, undisturbed sites around the wetland and composite the cores into the same bottle.

For collecting drier soils with soil coring device:

1. Assemble the coring device with a clean core tube, and either the auguring tip and ratcheting handle, or the conical tip and hammer-drive handle, depending on the soil type.
2. Drive or auger the device to slightly past 10 cm over the point where the tip attaches.
3. Gently remove the device from the soil and disassemble it to retrieve the core tube.
4. Use a core tube plug and extruding rod to extract the top 10 cm of the core into a clean, labeled 1-L wide-mouth sample bottle.
5. Clean the core tube, and repeat steps 1-4 two times in equally spaced, undisturbed sites around the wetland and composite the cores into the same bottle.

7.2 Collection of Wetland Bulk Density Samples

During trip setup, pre-weigh glass jars (without lid) for every sample to be taken. Record that number and assign an ID to that jar.

To collect a bulk density sample:

1. Remove the plunger of a soil core sampling syringe and drive the barrel into the soil.
2. Twist gently, then pull the barrel out with the soil sample.

3. Carefully drain standing water (if present) from the top of the cylinder. Similarly, if excessive amounts of leaf litter or other detritus is present on top of the soil, carefully remove as much as possible without removing any soil from the sample. Replace the plunger.
4. If the collected core is greater than the marked volume, carefully remove any excess.
5. Expel sample into pre-weighed glass jar, noting the volume of the sample collected.

7.3 Processing of Wetland Sediment Samples

All sediment samples will be labeled on the top and side of the jar and immediately placed on ice for preservation. The site, date, time of collection, and volume of sediment collected for each sample will be recorded on a field sheet. Nutrient samples should be delivered to SWAFL as soon after collection as possible for analysis. Bulk density samples should begin the drying process as soon as possible.

8.0 Bulk Density Sample Processing

The bulk density of a soil sample, usually expressed in grams per cubic centimeter (g/cc), is determined by dividing the mass of the sample (once all moisture has been removed from it) by the volume of the sample collected. Moisture removal is accomplished via drying the sample in the lab oven.

To dry bulk density samples:

1. Remove lid and weigh sample jar. Record this value as initial weight.
2. Place all samples in lab oven set to 105°C.
3. Allow to dry in oven for duration of workday, or overnight.
4. Turn oven off, allow about 10 minutes for glass to cool for handling. It is best to weigh the sample as soon as possible to avoid absorption of water from the air.
5. Weigh sample again and record weight. Calculate the percent difference between the new weight and the initial weight.
6. Repeat the drying process until the percent difference between weights is negligible (preferably less than 1%). At this point the sample can be considered completely dried.
7. Obtain the final mass of the sample and divide by the volume of the collected sample to obtain bulk density.

9.0 Measurement of Physical Parameters Using Soil Probes

Instruments such as the Hanna Instruments HI98331 conductivity meter and the HI981030 soil pH tester may be used to measure physical characteristics of soils directly either in the field or with a sample in the lab. Prior to taking any measurements, soil probes should be inspected, cleaned, and calibrated.

9.1 Conductivity and Temperature

The Groline HI98331 directly measures soil conductivity and temperature by inserting the probe into soil.

To calibrate the instrument:

1. Press the ON/OFF button to turn the meter ON
2. Enter calibration mode by holding the ON/OFF button until “OFF” is replaced by “CAL.” Release the button.
3. Submerge the probe tip in 1413 µS/cm calibration solution. This can be in a freshly opened sachet of solution, or in a beaker. If in a beaker, the probe tip should be kept about one inch away from the bottom or sides

4. The meter will automatically recognize the solution. “REC” will be displayed until the reading is stable and the calibration is accepted. “Stor” will be displayed upon successful calibration. “---WRNG” will be displayed if there is an error.

To take measurements with the instrument:

1. Press the ON/OFF button to turn the meter ON
2. Insert the probe to the desired depth. Recommended and maximum immersion levels are shown in **Error! Reference source not found.**.
3. Saturate the soil with deionized water if needed.
4. Once the reading is stable, record the measurement on a field sheet. The conductivity value is automatically compensated for temperature, and is given in mS/cm, rather than µS/cm.
5. Rinse and dry the probe between measurements and after use.

9.2 pH

The Groline HI981030 directly measures soil pH by inserting the probe into soil. Unlike the HI98331 conductivity meter, it should not be directly driven into the soil before preparation or be used in stony soils as probe tip is glass and is fragile.

To calibrate the instrument:

1. Press the ON/OFF button to turn the meter ON
2. Enter calibration mode by holding the ON/OFF button until “OFF” is replaced by “CAL.” Release the button
3. When “7.01” is displayed, place the tip of the electrode in pH 7.01 buffer. When the reading is stable the stability icon will disappear.
4. When “4.01” is displayed, place the tip of the electrode in pH 4.01 buffer. When the reading is stable the stability icon will disappear.
5. When “Sto” is displayed, the calibration has been saved and the meter will return to measurement mode.

To take measurements with the instrument:

1. Press the ON/OFF button to turn the meter ON and remove the protective cap from the electrode tip.
2. Use a small auger (**Error! Reference source not found.**) or similar tool to make a hole in the soil about 20 cm deep. Depth should be consistent between measurements.
3. Add some deionized water to the hole; the soil should be damp but not saturated.
4. Insert the electrode into the hole. Recommended and maximum immersion levels are shown in **Error! Reference source not found.**
5. Once the reading is stable, record the measurement on a field sheet.
6. Rinse the probe between measurements and after use. Replace the protective cap.

10.0 Forms

10.1 Chains of Custody

Chains of custody are documents remitted to the analytical laboratory documenting characteristics and metadata for each lake or wetland sample. These forms are used for several purposes; they act as a legal document to show proper delivery of samples occurred and they make a general list of the parameters that should be analyzed. They are a data sheet and a legal document and should be treated with the responsibility these dictate. The date, time, weight, and volume for each sample collected must be included and the form should be legible, accurate, and complete. Additional

information such as bottle info or lot numbers should also be included if available. All forms should be signed and dated by field collector and laboratory receiving personnel at the time of delivery. For guidance on proper procedure to complete the chains of custody, refer to a supervisor and/or crew leader. All samples are pre-logged in advance of sampling by means of an approved request for acquisition (RFA) for the cost of all analyses to be performed, and a chain of custody document from SWAFL (**Figure 2**), which is available as a fillable PDF.

11.0 Data Storage

When weather permits, the electronic lakes field sheet should be completed on a laptop and saved in the format "Sample ID Lake Name Season Year." If the paper copy is used it should be transcribed into an electronic field data sheet as soon as possible and stored in the appropriate binder. The data from the electronic field sheet should be transferred to the network upon arrival to the office for review and upload to the Ambient Water Quality Monitoring System (AWQMS). Each sample should be maintained electronically in the database under its unique sample number.

For bulk density sampling, an excel sheet is used to track ID and empty weight for every sample jar, and then the initial weight and the dried weight for each sample as well as to calculate the percent difference in weight between dryings. Once the drying is complete the final bulk density of the sample will be calculated and recorded here.

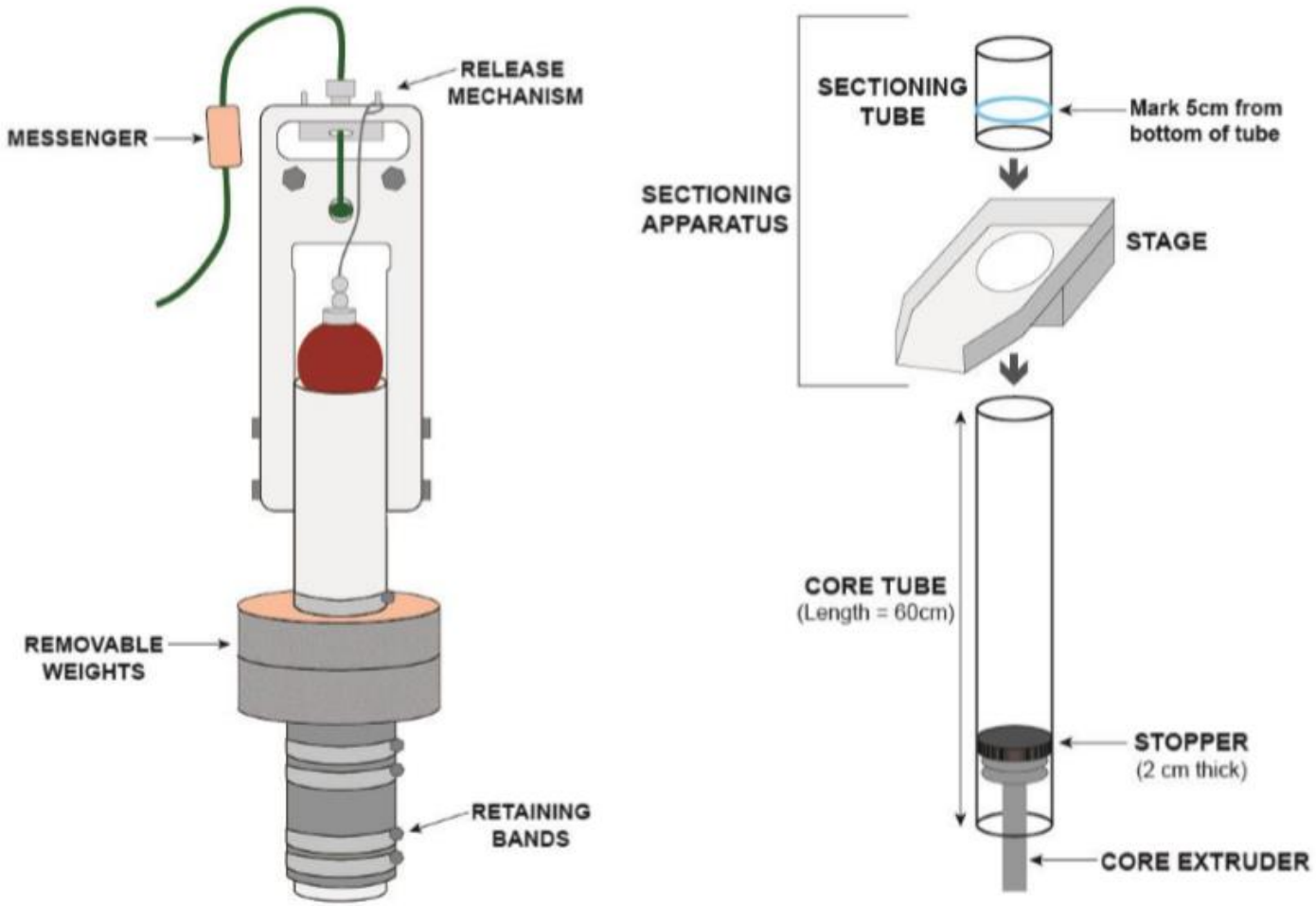
12.0 References

Hanna Instruments Inc. (2016). Instruction Manual - Groline HI98331 Direct Soil Conductivity & Temperature Meter.

Hanna Instruments Inc. (2019). Instruction Manual - Groline HI981030 Soil pH Tester.

OWRB. (2017). *Office and Field Safety Manual.* Oklahoma City: OWRB.

USEPA. (2017). *National Lakes Assessment 2017. Field Operations Manual. EPA 841-B-16-002.* Environmental Protection Agency: Washington, DC.



• **Figure 1 - Gravity corer setup (left), and core extruders setup (right)**

Chain of Custody

Soil, Water & Forage Analytical Laboratory
045 Agricultural Hall, Stillwater, OK 74078

www.soiltesting.okstate.edu email:soiltesting@okstate.edu (405)744-6630

Customer Code:	**Customer Name:** Address: Phone #: Email:	**Project Name:**
Samplers Name (print)		**Samplers (signature):**

		Sample Analysis								
Sample #	Sample Identification (Date and Time, etc.)	Irrigation Water	Household Water	Livestock Water	Comprehensive Salinity	Salinity Mgmt	Routine Fertility	Other Request:	Lab ID (Lab USE)	NOTES:

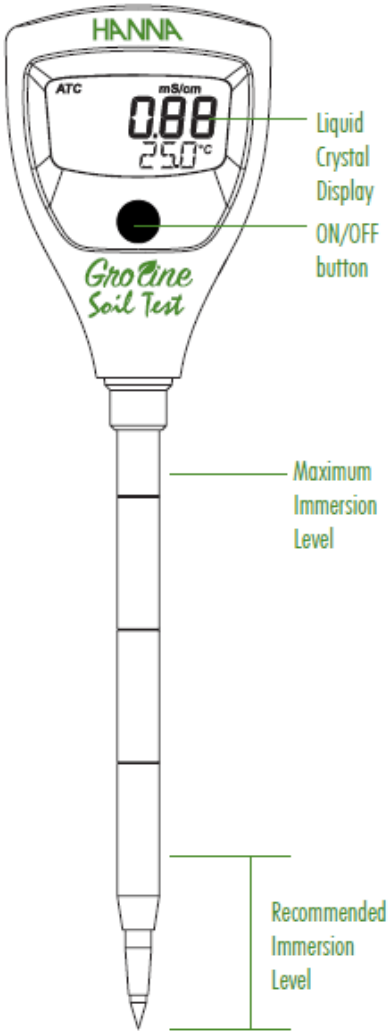
RELINQUISHED BY: (Signature)	**TIME**	**DATE**	**RECEIVED BY: (Signature)**	**TIME**	**DATE**
RELINQUISHED BY: (Signature)	**TIME**	**DATE**	**RECEIVED BY: (Signature)**	**TIME**	**DATE**

• **Figure 2 - SWAFL chain of custody**

Jar	Empty weight	Site	Date/time	Volume collected	Initial weight	Weight after 1st dry	% difference	Weight after second dry	% difference	Final bulk density
#	g			cc	g	g		g		g/cc
1	142.2	Wet - 35	5/17/20 @ 1320	60	187.3	168.3	10.14	168.1	0.12	0.43
2										
3										

- **Figure 3 - Example data-tracking sheet for bulk density.**

Operation



• Figure 4 - HI98331 Groline Soil Conductivity Meter

• Figure 5 - HI981030 Groline Soil pH Tester

- Figure 6 - Soil auger for use with pH probe.