## STANDARD OPERATING PROCEDURE FOR COLLECTION OF SEDIMENT SAMPLES IN WETLANDS



# WATER QUALITY

State of Utah Department of Environmental Quality Division of Water Quality

> Revision 2.0 Effective May 2021

#### Foreword

Utah Division of Water Quality (DWQ) Standard Operating Procedures (SOPs) are adapted from published methods, or developed by in-house technical experts. This document is intended primarily for internal DWQ use. This SOP should not replace any official published methods.

Any reference within this document to specific equipment, manufacturers, or supplies is only for descriptive purposes and does not constitute an endorsement of a particular product or service by DWQ. Additionally, any distribution of this SOP does not constitute an endorsement of a particular procedure or method.

Although DWQ will follow this SOP in most instances, there may be instances in which DWQ will use an alternative methodology, procedure, or process.

The methodology detailed below is the protocol followed by DWQ's monitoring staff and verified by DWQ's Quality Assurance officer.

Signature:	Benjamin R. Brown Benjamin R. Brown (Jul 6, 2021 16:15 MDT)
Email:	brbrown@utah.gov

**Benjamin R. Brown** *Monitoring Section Manager*  Date

Signature: Toby Hooker er (Jul 6, 2021 16:23 MDT) Email: tobyhooker@utah.gov

**Toby Hooker** *Quality Assurance Officer*  Date

## **REVISION PAGE**

Date	Revision #	Summary of Changes	Sections	Other Comments
09/10/2011	1	applicable v s t		Adapted from GSL wetlands field manual and put into new standardized format, began document control/revision tracking
12/4/2020	2.0	Changed name to SOP_Wetlands_Sedimen t_2021_v0	all	Previous name: SOP Sediment Wetlands_09102011_W etL
1/22/21	2.0	Removed diatoms from SOP	2.0, 9.0	Diatom SOP available in Archive
1/22/21	2.0	Title changed from GSL Impounded Wetland 2012 Monitoring Activities	Title	
1/27/21	2.0	Removed Section 10.0 Laboratory Analysis	10.0	Added section on sample handling in 9.0
1/29/21	2.0	Updated language, grammar, structure	All	Clarified and revised sentence structure and grammar throughout the entire document.

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## 1.0 SCOPE AND APPLICABILITY

This document presents the standard operating procedure (SOP) for the collection of sediment samples in Utah's wetlands. Previous versions of this SOP included sampling procedures for sediment diatoms and for analysis of total sediment metals and nutrient concentrations. However, the sediment-diatom procedure is no longer included here - more detailed information on diatom sampling can be found in project-specific SAPs or the earlier, version 1.0 of the SOP for Wetlands Sediment. This SOP details procedures for the collection of soil nutrient extract using a KCl solution and solids for available nutrient and metal analysis. Additional extract types can be collected, such as 0.5M NaHCO3 (Olsen-P), or 0.5 M K2SO4 (microbial biomass-C, -N, and -P), and others, but this SOP just includes KCl extracts.

Sediment chemistry plays an important role in wetlands ecosystems. Numerous studies (summarized in Hoven and Miller 2007) have shown that submergent and emergent vegetation in wetlands primarily derive their nutrient requirements from sediments rather than from the water column. Nutrients and chemical contaminants can be trapped or immobilized in wetland soils and sediments, and may be stored for long periods, altering both local and downstream water chemistry (Johnstone 1991). For many chemical parameters temporal variability in concentrations is lower in sediments, especially from composited samples, than concentrations obtained from the water column, so sediment samples may provide a more integrative measure of background chemical conditions than water chemistry alone.

This SOP has been created for Utah DWQ wetland monitoring purposes and is a modification of procedures described in *National Lakes Assessment 2017: Field Operations Manual* (EPA, 2017). This SOP applies to all DWQ field staff, DWQ cooperators, and volunteer monitors.

## 2.0 SUMMARY OF METHOD

Each sediment extract and soil sample consists of a composite of 5 core samples (10 cm of each core is retained). Core samples are collected using a modified KB coring device (see **Figure 2**). In fringe wetlands and non-inundated soils the sampling device may be simplified and need assistance of a wooden block and rubber mallet to drive the core tube.

The five core samples used in the composite are collected at five points along a 100 m transect. The samples are combined into a stainless-steel mixing container and mixed vigorously to homogenize the sediment. Once homogenized, sediment is scooped into zip-lock bags (most analytes) and/or 100 ml specimen cups with KCl extract.

## **3.0 DEFINITIONS**

**DI water:** Deionized water is prepared at DWQ's lab and/or the state lab. It is tested by the state lab to ensure it is analyte-free

FRNG:	fringe wetland
IW:	impounded wetland(s)
KCl:	Potassium chloride

PVC:	polyvinyl chloride
SAP:	Sampling and Analysis Plan
triple rinse:	an EPA verified procedure proven

**triple rinse:** an EPA verified procedure proven to remove and/or dilute contamination. It is important that every piece of reusable equipment that encounters site water is rinsed.

## 4.0 HEALTH AND SAFETY WARNINGS

Hazardous conditions potentially exist at every waterbody. If unfavorable conditions are present at the time of sampling, it is recommended that the sampling be rescheduled. If hazardous conditions arise during sampling, such as lightning, high winds, rising water, or flash flood warning, personnel should cease sampling and move to a safe location.

When working in Utah and other warm climates, take steps to avoid heat induced illnesses such as heat stroke or heat exhaustion.

Use caution when working in waders as drowning hazards exist.

Take appropriate precautions when operating equipment and working on, in, or around water, as well as possibly steep and unconsolidated banks, bridges, or edges of ponds/lagoons. All field crews should follow DWQ health and safety procedures and be equipped with safety equipment such as proper wading gear, personal flotation devices (PFDs), gloves, first aid kits, cellular phone, etc.

Use caution when sampling from a bridge or boat and take appropriate actions to make the situation as safe as possible; suspend the sampling if conditions are unsafe.

## 5.0 CAUTIONS

Be sure to push the core greater than 10 cm to ensure a complete sample is collected, especially when a root base is present. In addition, when sampling inundated or fully-saturated soils, the sampler should take care to ensure that the sample is not compacted as the core is inserted into the sediment; a compacted sediment sample will be apparent by the level of the sediment inside the core being much (> 1 cm) lower than that outside the core.

## 6.0 INTERFERENCES

Disturbance of the sediment by sampler's footsteps may cause collection of a non-representative core sample (sediment thickness and organic layer may be altered). It is critical that the core tube strikes minimally disturbed surface sediments.

Care should be taken to not place the core tube into water that has a sediment plume caused by the sampler walking to the site. Also, the sampler should attempt to avoid trapping submerged aquatic vegetation while coring.

This method may not be suitable in wetlands with highly organic or rocky soils because the PVC core tube cannot cut through roots or rocks well. In such conditions a steel core (or auger) can be used instead.

## 7.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

DWQ personnel performing wetland sediment sampling must be familiar with sampling techniques, safety procedures, proper handling, and record keeping. Samplers are responsible for attending refresher meetings held each spring/summer to review procedures and techniques. New staff will be trained in the field by DWQ trained personnel.

Cooperators are required to read this SOP annually and acknowledge they have done so via a signature page that will be kept on-file at DWQ along with the official hard copy of this SOP (see **Appendix 1**).

## 8.0 EQUIPMENT AND SUPPLIES

- $\Box$  Copy of this SOP
- □ Tablet/phone loaded with correct field forms
- □ Field sheets, field notebook, pens, pencils and permanent Sharpies
- □ Plastic, high-sided utility sled or float tube (fishing type) for toting equipment
- □ zip-lock bags for homogenized samples
- 100 mL specimen cup with 2 M KCl solution (KCl-extract nitrogen sampling) (See Appendix 2 for details on how to make the solution, ample solutions are generally prepared at the start of the season.)
- □ 6.35 cm-diameter modified KB sediment corer (**Figure 2**) core tube, stage, plunger/core extractor, rubber stopper, sectioning tube
- □ rubber mallet and wooden block (may be necessary on fringe wetlands)
- □ Stainless steel mixing bucket/container
- □ Stainless steel scoop
- □ bristle brush with attached rope (to clean core tube)
- DI water for decontamination
- □ PVC meter stick marked in centimeters for measuring water depth
- □ Sample labels (Figure1)
- □ KCl sample tracking form (see **Appendix 3**)
- □ Rubber mallet and wood block (for fringe and non-inundated soils)

#### For processing KCl extract samples at the shop, the following supplies are also needed

- □ Geopump
- □ Whatman #1 Filter either 150mm or 47mm
- □ Filter apparatus, including vacuum flask and membrane filter stage. See Figure 5.

## 9.0 PROCEDURE

#### 9.1 Sediment Sample Collection

See diagram in **Figure 2** for equipment names for the modified KB coring device and pictures detailing its use.

*Note: this sampling method may not be appropriate for wetlands with (saturated but not inundated) organic soils, or rocky soils.* 

- 1. Load sled with modified KB-core device, tablet/phone, stainless steel mixing bucket/container and PVC meter stick.
- 2. Determine the five sampling points along a 100m transect. Refer to the project-specific SAP for details on how to set up the transect and select five locations.
- 3. Prior to sampling, triple rinse the sampling tools with site water. Avoid disturbing sediments near the sample site.
- 4. At the first sample point, measure and record water depth to the nearest 0.1 m with the metered PVC stick and record depth on the SAV Field form (see **Appendix 4**).
- 5. Push the core tube into the surface sediment past the specified sampling depth, deep enough to ensure that the sampling tube and the entire sediment sample can fully be recovered. Refer to the project-specific SAP to determine the specified depth.
- 6. Place a rubber stopper on the top of the core tube to maintain suction inside the sampling tube.

Note: If sampling in dry soils, you may not need the stopper and may use a rubber mallet and wood block to assist in driving the core tube.

- 7. Tilt and rotate the core tube to break the soil core free from the surrounding soil, and pull the core tube to the surface.
- 8. To remove the sediment core, place the core extruder in the bottom of the core tube and remove the rubber stopper from the top of the core tube.
- 9. Slowly but firmly, push the core extruder through the core tube until the excess water and vegetation overflow leaving only the top of the soil sample at the upper end of the core tube.
- 10. Place the clean stage on the top of the core tube.
- 11. Place the sectioning tube (marked with a line 10 cm from the bottom) on the stage directly over the core tube. Slowly compress the core tube and core extruder, maintaining vertical alignment of the tube, such that the sediment sample enters the sectioning tube and the top of the sediment reaches the desired length (depth) of the sample.

- 12. Slide the sectioning tube towards the opening on one side of the stage to separate the sample from the core tube.
- 13. Transfer the soil sample into the stainless-steel container for compositing.

Note: In some cases, soil samples may be composited directly in a polyethylene bag, so long as the bag is sturdy enough to not rupture during homogenization, large enough to easily include all samples, and is free from any cross-contamination.

- 14. Rinse the core tube and components with site water (at the point just sampled) to prevent carryover of sediment from one sampling point to another.
  - a. To aid in cleaning the core tube between sampling points along the transect, use a bristle brush to pull through the core tube, rinsing with site water.
- 15. Repeat Steps 2 through 13 to sample the four remaining sampling points, compositing each individual sample into the stainless-steel container.
- 16. Return to the staging area after collecting the composite samples. Use a metal spoon to fully homogenize the sample.

Note: Option to place samples directly in a polyethylene bag in the field and dump into the stainless-steel container at the truck for compositing. This may alleviate any concerns about the sample oxidizing or drying between first and last sample collections. If using this method, be careful not to puncture the bag.

#### For KCL extraction samples (only if included on the project-specific SAP):

- 17. Place 10-15 grams (approximately one heaping tablespoon) of homogenized soil in a cup containing 100-mL (2M) KCl solution.
- 18. Shake vigorously but briefly (30 to 60 sec) to ensure that the soil and KCl solution are well mixed.
- 19. Ensure the label is filled out and place the KCl extract sample in a dark cooler.

#### For remaining soil sample:

- 20. Place the remaining homogenized soil sample (at least two cups) in zip-lock bag(s) and label the bags with the DWQ site ID and date. (*Samples may need to be divided into multiple bags if more than one lab is being used for analysis*).
- 21. Store the soil samples in the open zip-lock bags on a shelf to dry and process the KCl extract samples following the instructions below.

Note: The above procedure can be modified to collect different depths of soil. For example, for some analysis, 1 cm of sample is collected from each site. If this is the case, follow the above

procedure, but when sectioning off the soil sample, push the sample to the 1 cm line instead of 10 cm.

#### 9.2 KCl Extract Sample Processing

These steps are completed back at the shop. See **Appendix 2** for preparation of KCl soil-extract cups. These should be prepared in advance of the sample collection.

- 1. Samples should be shaken for 1-hour. This can be one hour in the shop or if samples are transported directly from the field they only need to be shaken for 30 minutes, assuming a bumpy ride.
- 2. After shaking, for 1 hour, let the sediment settle to the bottom.

Note: If time is an issue, or if the geopump approach leaves a filtrate that is too dirty, then sample-extracts can be allowed to settle overnight in a fridge, and then filtered the next day...better to not leave the extracts for more than 24 hours before filtering, if possible. The filtering procedure will go much faster if the bulk of solids are allowed to settle.

- 3. Weigh and record sample weight on a datasheet to ensure that at least 10 grams of soil was gathered. See **Appendix 3** for datasheet.
- 4. Set up filter in a clean, empty cup
  - a. Gravity option fold large Whatman #1 filter into quarters, open like a funnel and pour solution into the filter (avoid pouring sediment into the filter as much as is possible, it will make filtering slow). See **Figure 4**
  - b. Geo pump option place small Whatman #1 filter into a vacuum flask, hook tubing up to suck water out of the full cup and send filtrate into the clean, empty vacuum flask. Rinse flask between samples. See Figure 5
- 5. Filter at least 50 ml (more is great) into the clean cup
- 6. Label the filtrate with site name and date
- 7. Put filtrate in the freezer until samples are taken to the lab
- 8. Clean the specimen cup with Liquinox® and DI water.

#### 9.3 Sample Handling and Preservation

Refer to Table 1 or the project-specific SAP for additional sample handling requirements.

Description	Parameters	Details
Sediment Nutrients (IW & FRNG)	PO4,Total N, Total and Organic C	Five 0-10 cm cores (composited); Stored in 1-gallon zip bag, air dried
		Sent to USU Analytical Lab
Sediment Total Metals (IW & FRNG)	Aluminum, Arsenic, Barium, Cadmium, Cobalt, Copper, Iron, Mercury, Lithium, Manganese, Nickel, Lead, Selenium, and	Five 0-10 cm cores (composited); Stored in 1-gallon zip bag, air dried Sent to USU Analytical Lab
Sediment Nutrient Extracts (IW &	Zinc Nutrient Extracts: NH4, NO3/NO2	10-15 grams soil to 100 mL KCl solution; shake, filter, and freeze
FRNG)		Sent to USU Analytical Lab

*Table 1.* Sampling parameters and handling.

Sediment samples are typically analyzed for nutrients, metals, and/or trace elements using EPA or equivalent methods, depending on specific project goals. The specific methodology and quality control samples run for these analyses can be obtained from the analyzing laboratory.

Sediments intended for metals or total-nutrient analysis (from the zip-locked bag(s)) are airdried. The entire sample is submitted to the lab (at least 2 cups), with site-ID and date are clearly labeled. In some cases, samples may be sent to the lab before they are dried, check with the lab and/or project-specific SAP for more information.

KCl filtrate samples are kept in the freezer until ready to ship. There are no specific holding times.

## **10.0 DATA AND RECORDS MANAGEMENT**

Project-specific data and records management requirements can be found in the project-specific SAP. To maintain the integrity of sample site IDs, sample bottles must be labeled properly and the information on the label must match the information on the Lab Sheet, or other sample

tracking or Chain-of-Custody form. Information on sample labels must be written in permanent ink.

Before leaving the field site, be sure that all required samples have been collected, labeled, and that all appropriate field sheets, field notes, and sample tracking forms have been filled out completely and accurately.

The data from the field forms is sent to the wetlands coordinator at the same time as the other field data collected for that day (ideally within 2 weeks from the date of the site visit).

See project-specific SAP for data forms and specific data management practices.

## 11.0 QUALITY ASSURANCE AND QUALITY CONTROL

Field replicates for sediment samples should be collected at a minimum rate of 1 replicate for every 10 regular samples, or as identified in a project-specific SAP. To collect the replicate sample, follow the steps outlined in this SOP under **Section 9.0**. This replicate sample should be collected immediately following the collection of the first sample. Note on the field sheet and in the field notebook that a replicate was collected. Refer to the project-specific SAP for more details and performance goals for replicate samples.

## **12.0 REFERENCES**

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## **13.0 FIGURES**

#### Figure 1. Example sample label for KCl extract samples

IW2019 Soil Filtrate	
MLID	
SITE NAME	
COLLECTION DATE	

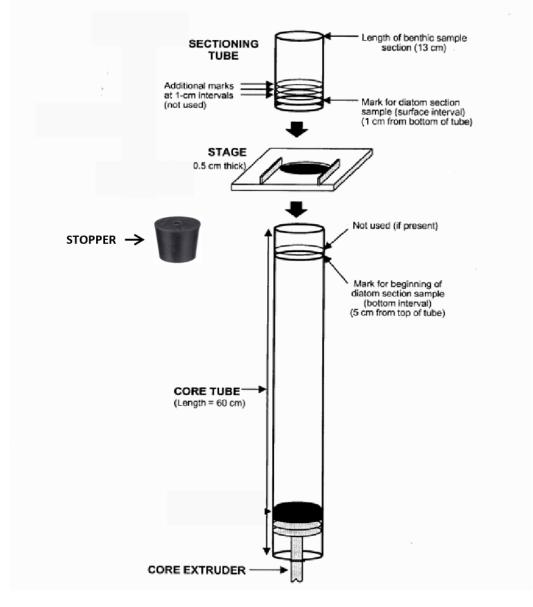
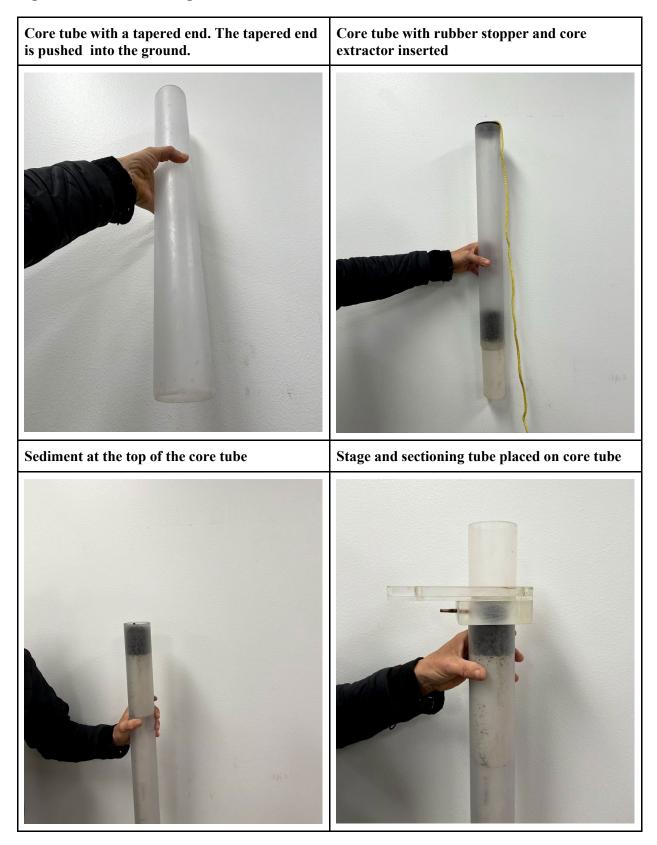


Figure 2. Illustration of the modified KB corer and sectioning apparatus (EPA, 2007).



*Figure 3.* Procedure using the core tube

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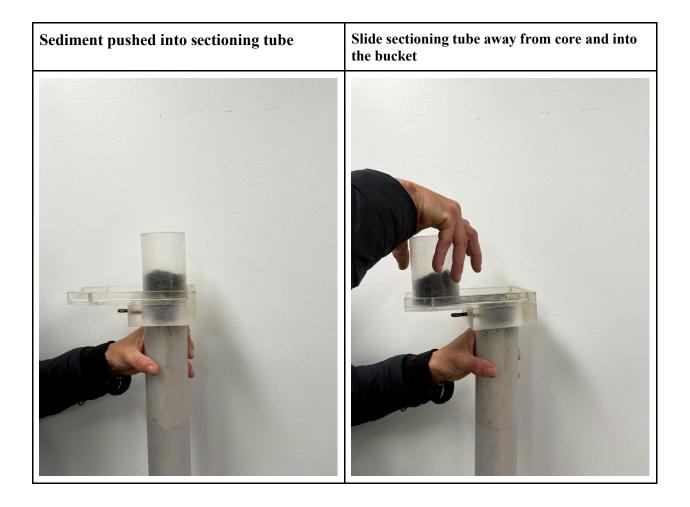


Figure 4. KCl extract gravity option



Figure 5. KCl extract Geopump option



## 14.0 APPENDICES APPENDIX 1 - SOP ACKNOWLEDGMENT AND TRAINING FORM

## **SOP Acknowledgement Form**

This SOP must be read and acknowledged annually, and this form must be kept on file at DWQ.

Document Title:	
Document Revision Number:	
Document Revision Date:	

#### Please sign below in accordance with the following statement:

"I have read and understood the above referenced document. I agree to perform the procedures described in this SOP in accordance with the document until such time that it is superseded by a more recent approved revision."

Date	Printed Name	Signature

## **SOP Training Form**

Training is required for all new samplers and Refreshers are required annually.

**Trainee:** Sign below to acknowledge that training on this SOP was received, understood, and all questions/concerns were addressed by the trainer.

**Trainer:** Sign below to acknowledge that training on this SOP was completed for the individual listed and that trainee is competent to perform the procedures described within.

Date	Trainee Printed Name	Trainee Signature	Trainer Printed Name	Trainer Signature		
Management Approval						
Printed Name:						
Signat	ure:					
Date:	Date:					

#### Appendix 2

To Make 2 Molar KCl Solution:

- 1. Multiply the molar weight by 2
- 2. Multiply above by the number of liters of solution needed

i.e. Molar weight \*2\*6 = salt for 6 liters of solution

- 3. Add salt to 6 liters of DI water
- 4. Add 100 mL of solution to the specimen cup and record initial weight on the Sample Weight Tracking Form (**Appendix 3**).

Example:

74.55 (g KCl/mol) \* 2 (moles/L) \* 4 (L of solution) = 596.4 g KCl salt + 4 Liters of DI Water

## Appendix 3

## KCl Sample Weight Tracking Form

Cup Name	MLID	Weight	Weight with Soil	Date Gathered	Date Filtered
					+

2019 IW SAV Sheet						
GPS coords of transect st	art					
Lat:						
Long:				Sampler(s)	:	
Wetland-Scale Cover Es	timates ~2	00 m of sa	ampling loo	cation		
%Algal mat		1	%SAV			7
%Floating Aquatic Veg		1	%Bare mu	ud/substrate		1
%Emergent Veg			%Benthic	mat		]
		-				
Quadrat:	1	2	3	4	5	Average
Plot location along transect (m)						
Water depth (cm)						
Height of SAV (cm)						
SAV cover (%)						
<sup>1</sup> SAV condition Cover - Spp1:						<u> </u>
Cover - Spp1: Cover - Spp2:						<u> </u>
Cover - Spp2: Cover - Spp3:		<u> </u>			<u> </u>	<u> </u>
		<b> </b>				+
<sup>2</sup> Filamentous algae cover (%) [Surface]						
Epiphytic Alg. cover (%)						
Duckweed cover (%)						
<sup>1</sup> SAV condition: 0 = absent 1 = Decomposing/senescing, 2 = Intact, but stressed, 3 = Healthy, F = Flowers/Fruits <sup>2</sup> Filamentous algae: Extent of algae on SAV and/or surface of pond in %; (x) = Veg Sample Collected						
Plant Vouchers or Comments:						
Fish Observed:						
Depth loose muck:						

Appendix 4 - Submersed Aquatic Vegetation Field Form