Sediment Sampling Plan
for
Coon Creek and
West Fork Kickapoo Watershed in
La Crosse, Monroe, and Vernon Counties, Wisconsin

Prepared By

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Prepared For

USDA NRCS
Natural Resources Conservation Service

USDA, Natural Resources Conservation Service

October 2021

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LIST OF ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
CoC	chain of custody
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethylene
DDT	dichloro-diphenyl-trichloroethane
EIS	Environmental Impact Statement
EPA	U.S. Environmental Protection Agency
ERP	Environmental Repair
GPS	global positioning system
HDPE	high density polyethylene
ID	identification
LUST	Leaking Underground Storage Tank
mg/kg	milligrams per kilogram
NRCS	Natural Resources Conservation Service
PCBs	polychlorinated biphenyls
PFAS	per- and polyfluoroalkyl substances
PFOA	perfluorooctanoic acid
PFOS	perfluorooctane sulfonate
PLAN	NRCS Watershed Project Plan
PVC	polyvinyl chloride
QA	quality assurance
QC	quality control
RCRA	Resource Conservation and Recovery Act
SOP	Standard Operating Procedure
Sundance-EA JV	Sundance-EA Partners, LLC
USGS	U.S. Geological Survey
WAC	Wisconsin Administrative Code
WDNR	Wisconsin Department of Natural Resources
WSLH	Wisonsin State Laboratory of Hygiene

1 Introduction

Sundance-EA Partners, LLC (Sundance-EA JV), is pleased to submit this Sediment Sampling Plan for the U.S. Department of Agriculture Natural Resources Conservation Service (NRCS) at Coon Creek and West Fork Kickapoo Watershed, in Lacrosse, Vernon, and Monroe Counties, Wisconsin. This additionally scoped Sediment Sampling Plan is provided under the NRCS Watershed Project Plan (PLAN) and Environmental Impact Statement (EIS) Contract Number 12SPEC18D0016 and Task Order 12FPC320F0134.

Coon Creek Watershed has an area of 90,601 acres (141.6 square miles) to the confluence with the Mississippi River. The focused planning area for the PLAN-EIS is 68,762 acres (107.4 square miles) and includes the village of Chaseburg, as shown on Figure 1. West Fork Kickapoo Watershed has an area of 75,387 acres (117.8 square miles) to the confluence with the Kickapoo River (a tributary of the Wisconsin River). The focused planning area for the PLAN-EIS is 63,761 acres (99.6 square miles) and includes the village of Liberty, as shown on Figure 2.

2 Background on Suite of Analytes

The initial suite of analytes was determined in 2001 based on two historical reports provided by NRCS for sediment testing in Plum Creek Watershed (NRCS, 2001a) and White Mound Lake Plain Honey Creek Watershed (NRCS, 2001b). This initial suite of analytes was selected based on other agricultural watersheds participating in the Pilot Dam Rehabilitation Program and the County Agents specializing in pesticides, County Conservationists, Wisconsin Department of Agriculture (Agricultural Chemical Spill Unit), and scientists at the U.S. Geological Survey (USGS) Upper Midwest Environmental Sciences Center.

The 2001 initial suite of analytes included dichloro-diphenyl-trichloroethane (DDT) and its derivatives, atrazine, and its metabolites, alachlor (Lasso), metolachlor (Dual), cyanazine (Bladex), dichlorodiphenyldichloroethylene (DDE), dichlorodiphenyldichloroethane (DDD), phosphorus, nitrogen (NO3 + NO2), and inorganic arsenic contamination in sediment. The 2001 suite of analytes commonly used in agricultural settings were DDT, its derivatives, and nitrogen- and phosphorus-based fertilizers. Arsenic was also analyzed because it was a commonly used in lead/arsenic pesticide for apple orchards.

In addition to the 2001 initial suite of analytes in sediment, NRCS is now proposing to determine the presence/absence of perfluorooctane sulfonate (PFOS) and/or perfluorooctanoic acid (PFOA), two chemicals that are part of a diverse group of human-made chemicals referred to as per- and polyfluoroalkyl substances (PFAS), the full suite of Resource Conservation and Recovery Act (RCRA) metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium), polychlorinated biphenyls (PCBs), gasoline, and diesel. The addition of PFOS and/or PFOA, RCRA metals, PCBs, gasoline, and diesel analysis is to determine the presence/absence PFOS and/or PFOA (an emerging contaminant) and potential impacts from Leaking Underground Storage Tank (LUST) sites and three Environmental Repair (ERP) sites with an open status within and just outside of the watersheds (WDNR, 2021).

3 Sediment Sampling Activities

The objective of this Sediment Sampling Plan is to determine the presence/absence DDT, its derivatives, atrazine, and its metabolites, alachlor (Lasso), metolachlor (Dual), cyanazine (Bladex), DDE, DDD, phosphorus, nitrogen (NO3 + NO2), of PFOS and/or PFOA, RCRA metals, PCBs, gasoline and diesel contamination in sediment at two distinct depths behind the dams within Coon Creek and West Fork Kickapoo Watershed. Table 1 details the proposed sediment sample locations, number of sediment samples, depth of sediment samples, and sample identification prefix to be collected from each watershed.

Table 1. Summary of Sediment Sample Locations

Sampling Locations	Number of Sediment Samples	Depths of Sediment Sample	Sample Identification Prefix
Coon Creek Watershed	2	0 to 0.5 feet and 5 to 6 feet	CC
West Fork Kickapoo Watershed	2	0 to 0.5 feet and 5 to 6 feet	WFK

Sediment sample locations will be selected from within each watershed behind dams and/or structure. The sediment samples will be collected from two depth intervals (0 to 0.5 feet and 5 to 6 feet) to assess the presence/absence and potential stratification of suite of analytes. Number and type of quality control samples will be determined by the NRCS sediment sampling crew.

The NRCS sediment sampling crew will follow the guidelines presented within the *USGS National Field Manual for the Collection of Water Quality Data* (USGS, 2018), *Guidance for Applying the Sediment Sampling and Analysis Requirements of Chapter NR 347, Wisconsin Administrative Code (WAC)* (WAC, 2003), and Sundance-EA JV's Standard Operating Procedure (SOP) for sediment sampling, which is provided as Attachment 1.

At each sediment sampling location, two co-located sediment samples will be collected, a surficial sediment sample from approximately the top 0.5 foot and a subsurface sediment sample from approximately 5 feet to 6 feet below surface. Co-located sediment sample locations will be surveyed using global positioning system (GPS).

A Tenite™ (clear plastic) tube, Ponar® sampler, or Ekman® sampler will be used to collect surficial sediments. For shallow areas reachable on foot, the samples will be collected using cores such as Tenite™ tubes. The coring device will be placed to the appropriate depth, withdrawn, and sampled onshore. A Ponar® or Ekman® sampler will be used to collect sediment samples from locations accessed by boat. The NRCS sediment sampling crew will determine and document the appropriate sampling tool for each specific location based upon site conditions.

A new, dedicated Tenite™ tube will be used at each sample location. Waders will be decontaminated (see details below) before entering a new sample location. Ponar® or Ekman® samplers will be decontaminated prior to use, between sample locations, and after final use to ensure no cross-contamination occurs.

Surface sediment samples for PFAS analyses should be collected using a clean, stainless-steel tool (e.g., a trowel or spoon or Ponar® grab sampler). For subsurface sediment samples from cores, single-use polyvinyl chloride (PVC), high density polyethylene (HDPE), or acetate liners shall be used. Samples for PFAS analysis should be collected from the cores directly or using a stainless-steel tool.

4 Sample Handling, Custody, and Disposal

To ensure sample authenticity and data defensibility, a proper sample handing system will be followed from the time of sample collection to final sample disposal. Activities include sample labeling; chain of custody (CoC) record completion; packing and shipping coordination; sample receipt, inspection, and log-in; sample custody and storage; and sample disposal.

5 Sample Designations

5.1 Sediment Sample Designation

The sample identifications (IDs) for co-located sediment samples in each watershed will include: two-digit year, three-character project ID, two or three-character watershed designation, location number, sample type, and sample depth. The sediment sample designations are as follows:

- Two-Digit Year: 21
- Three-Character Project ID: EIS (Environmental Impact Statement)
- Watershed: Sample Identification Prefix indicated in Table 1
- Location Number: sequential location number within watershed starting with 01
- Sample Type: SD (sediment)
- Sample Depth: 0 feet (surface) or 5 feet (subsurface)

For example, the first subsurface sediment sample collected from a depth of 5 feet to 6 feet at West Fork Kickapoo Watershed in 2021 for the EIS would be written as: 21EIS-WFK01-SD-5.

5.2 Quality Control Designation

Sample blanks will be denoted by year collected, three-character project ID, quality control (QC) designation, time of day sample was collected (where applicable), and month/date collected. Number and type of quality control samples will be determined by the NRCS sediment sampling crew.

The QC extensions are as follows:

- FB – Field Blank
- EB – Equipment Blank
- 99 – Duplicate Sample

For example, the sample ID for an equipment blank collected in the morning on 31 October 2021 during sediment sampling would be written as: 21EIS-EBAM-1031. The sample ID for an equipment blank collected on 31 October 2021 would be written as: 21EIS-EB-1031. An example of a parent sediment sample collected from the surface at 0 to 0.5 feet at West Fork Kickapoo Watershed in 2021 would be written as 21EIS-WFK01-SD-0 and would have the duplicate sample ID written as 21EIS-WFK99-SD-0.

6 Decontamination Procedures

To the degree possible, dedicated and/or disposable sampling equipment will be used for sediment sampling. Equipment blank samples will be collected off reusable sample tooling to verify that no residual contamination remains on the sampler and proper decontamination procedures have been implemented. One equipment blank will be collected per 20 samples during sediment sampling if reusable sampling equipment is used.

The decontamination process for reusable sampling equipment, the boat, and waders will follow the most current methods for PFAS decontamination procedures and will include at a minimum the use of PFAS-free detergent, PFAS-free water, and a final rinse with laboratory supplied PFAS-free deionized rinse water. Water for decontamination must be verified PFAS-free and will be supplied by the contracted laboratory.

7 Laboratory Analysis

Wisconsin State Lab of Hygiene will perform the analysis of sediment samples using the analytical methods summarized in Table 2. Sediment samples will be analyzed in accordance with this Sediment Sampling Plan, with standard turnaround times (15 working days).

Table 2. Sample Containers, Preservation, and Holding Times

Analyte	EPA Analytical Method	Units
Atrazine and metabolites	8141A[1]	mg/kg
Alachlor (Lasso)	8141A[1]	mg/kg
Metolachlor (Dual)	8141A[1]	mg/kg
Cyanazine (Bladex)	8141A[1]	mg/kg
DDT	8081B[2]	mg/kg
DDE	8081B[2]	mg/kg
DDD	8081B[2]	mg/kg
Phosphorus	365.4 [3]	mg/kg
Nitrogen (NO3 + NO2)	353.2 [4]/350.1 [5]	mg/kg
Nitrogen (ammonia)	353.2 [4]/350.1 [5]	mg/kg
RCRA Metals	6010B[6]	mg/kg
PFOS/PFOA	WSLH PFAS [7]	ng/g
PCBs	8082A[8]	mg/kg
Gasoline and Diesel	8015C[9]	mg/kg

Notes:
[1] SW-846 Method 8141A Organophosphorus Pesticides by Gas Chromatography: Capillary Column Technique
[2] SW-846 Method 8081B Organochlorine Pesticides by Gas Chromatography
[3] 365.4 Phosphorus, Total – Colormetric/Automated/Block Digester AA II
[4] 353.2 Nitrogen, Nitrate-Nitrite – Colormetric/Cadmium or Nitrate-Nitrate by Automated Colorimetry
[5] 350.1 Nitrogen, Ammonia – Colorimetric or Nitrogen, Ammonia – Semi Automated Colorimetry
[6] 6010B Inductively Coupled Plasma-Atomic Emission Spectrometry
[7] Wisconsin State Laboratory of Hygiene Perfluoroalkyl Substances (PFASs)
[8] SW-846 Method 8082A by gas chromatography
[9] SW-846 Method 8015C by gas chromatography
Details of these tests can be found at www.epa.gov/epaoswer/hazwaste/test/under.html
Acronyms and Abbreviations:
°C - degrees Celsius
mg/kg - milligrams per kilogram

8 Quality Assurance and Quality Control

Quality Assurance (QA)/QC samples will be collected to monitor accuracy, precision, and the presence of field contamination. The number and type of QA/QC samples will be determined by the NRCS sediment sampling crew. In general:

- One equipment blank will be collected for every 20 sediment samples collected and will be analyzed for PFAS, when non-disposable equipment is used.
- One field blank will be collected every day that sediment samples are collected.
- One field duplicate sample will be collected for every ten primary sediment samples collected, with at least one duplicate per day per matrix.
- Additional volume for parent samples for preparation of matrix spike and matrix spike duplicate sample analysis will be collected at a frequency of at least one per delivery group.

The NRCS project manager will designate a NRCS field lead to review field notes. Identified quality issues will be addressed the following day before work begins by both the NRCS project manager and field team lead.

9 References

U.S. Department of Agriculture Natural Resources Conservation Service (NRCS), 2001a. *Sediment Testing in the Plum Creek Watershed Pierce County, Wisconsin*. Dated July 2001.

NRCS, 2001b. *Sediment Testing at White Mound Lake Plain Honey Creek Watershed Structure #3, Sauk County, Wisconsin*. Dated October 2001.

U.S. Geological Survey (USGS), 2018. *National Field Manual for the Collection of Water Quality Data Version 1.1,* USGS, June 2018.

Wisconsin Administrative Code (WAC), 2003. *Guidance for Applying the Sediment Sampling and Analysis Requirements of Chapter NR 347, WAC.* Dated December 2003.

Wisconsin Department of Natural Resources (WDNR), 2021. *Petroleum Contamination and Leaking Underground Storage Tanks.*

Website accessed at

https://dnr.wisconsin.gov/topic/Brownfields/Petro.html

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Figures

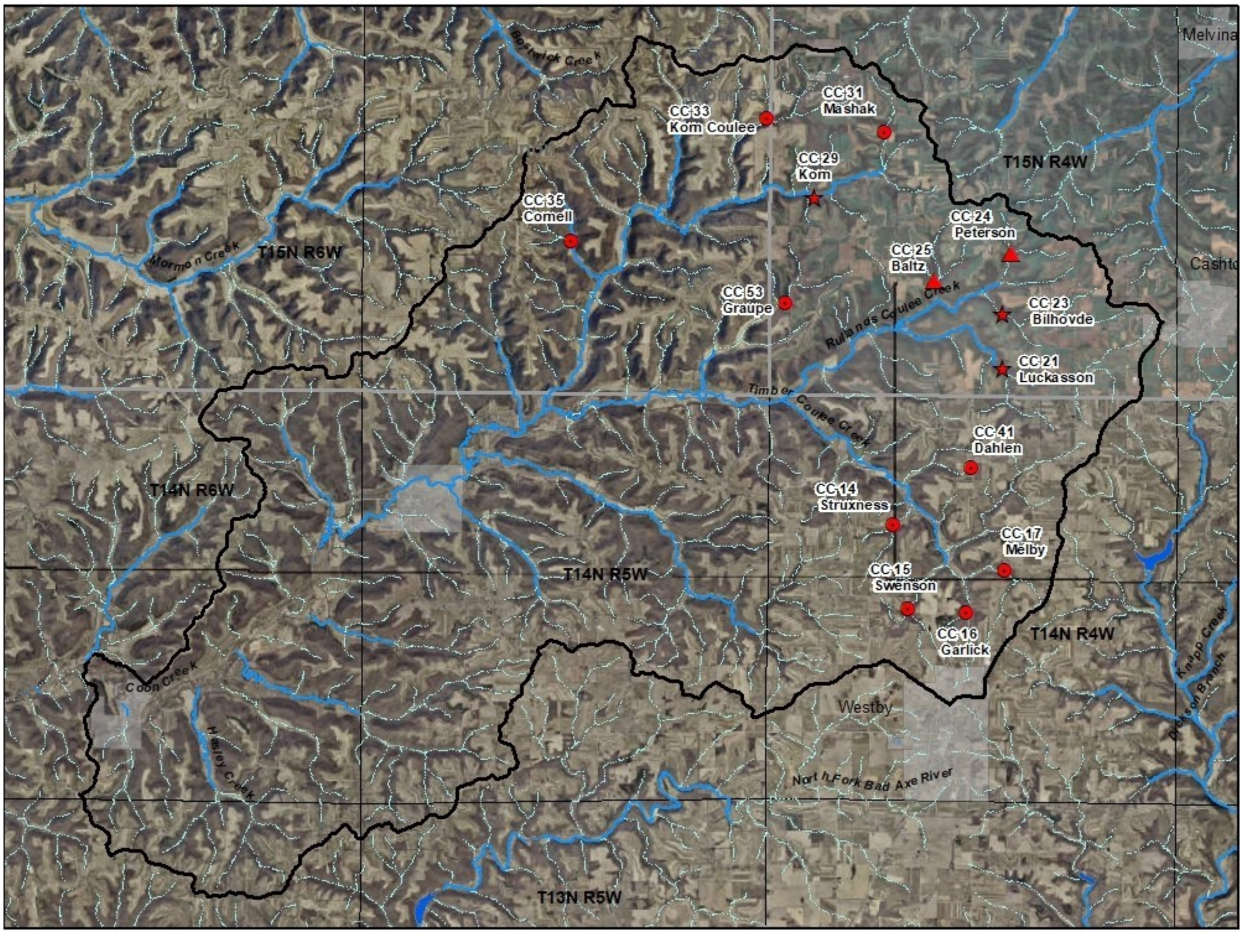

Figure 1. Coon Creek Watershed with Structure Locations.

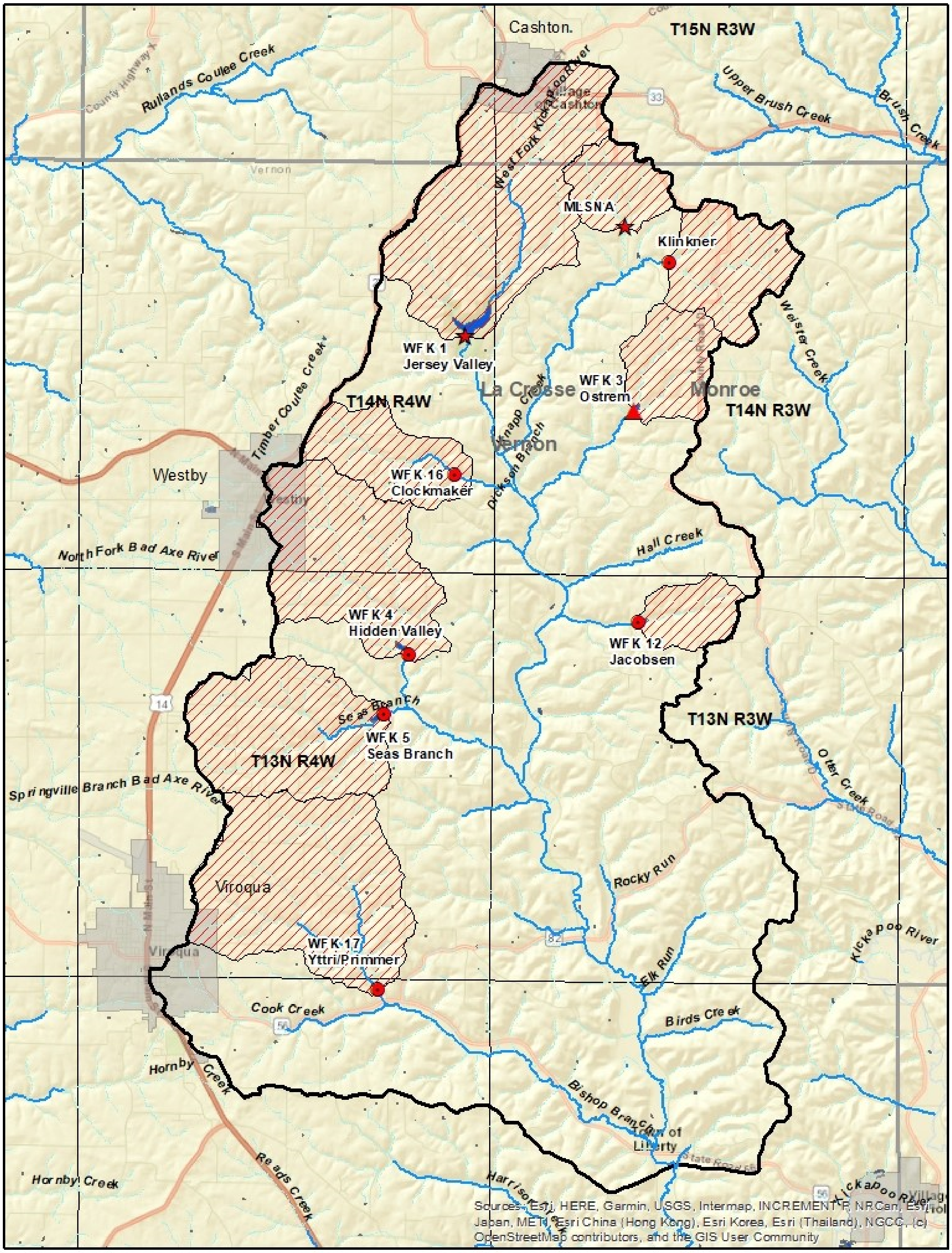

Figure 2. West Fork Kickapoo Watershed with Structure Locations.

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Attachments

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1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) delineates protocols for sampling sediments from streams, rivers, ditches, lakes, ponds, lagoons, and marine and estuarine systems.

Sundance-EA JV recognizes that other protocols have been developed that meet the criteria of quality and reproducibility. Clients may have their own sediment sampling protocols which may contain methodologies and procedures that address unique or unusual site-specific conditions or may be in response to local regulatory agency requirements.

2. PROCEDURES

The water content of sediment varies. Sediments range from soft to dense and fine to rocky. A variety of equipment may be necessary to obtain representative samples, even at a single site. Factors to consider in selecting the appropriate sampling equipment include sample location (edge or middle of the waterbody), depth of water and sediment, grain size, water velocity, and analytes of interest.

3. GENERAL PROCEDURES

1. Surface water and sediment samples are to be collected at the same location (if both are required in the project-specific Sampling and Analysis Plan).
2. Collect the surface water sample first. Sediment sampling usually results in disturbance of the sediments, which may influence the analytical results of the surface water samples.
3. Wear gloves when collecting samples. Comply with the Health and Safety Plan specifications for proper personal protective equipment.
4. If sampling from a boat or near waterbodies with depths of 4 feet or more, the sampling team will wear life jackets.
5. Wading into a waterbody disturbs the sediment. Move slowly and cautiously, approach the sample location from downstream. If flow is not strong enough to move entrained particles away from the sample location, wait for the sediment to resettle before sampling.
6. Collect samples first from areas suspected of being the least contaminated, thus minimizing the risk of cross-contamination.
7. Collecting samples directly into sample containers is not recommended. Sediment samples should be placed in Teflon-free, high density polyethylene (HDPE), stainless steel, or glass trays, pans, or bowls for sample preparation.
8. Use the proper equipment and material construction for the analytes of interest. For example, for perfluoroalkyl substances (PFAS) analysis, the sampling material in direct contact with the sediment or surface water must consist of HDPE or stainless steel.
9. Use proper PFAS decontamination methods before and after sampling and between samples.
10. Collect samples for volatile organic compound analysis first. Do not mix such samples before placing them in the sample containers. For composite volatile organic compound samples, place equal aliquots of each subsample in the sample container.

11. Sediment that will be analyzed for other than volatile organic compounds should be prepared as follows:
    - Place the sediment in a mixing container.
    - Divide the sediment into quarters.
    - Mix each quarter separately and thoroughly.
    - Combine the quarters and mix thoroughly.
    - For composite samples, mix each subsample as described above. Place equal aliquots of each subsample in a mixing container and follow the procedure described above.
12. Mark the sampling location on a site map. Record sampling location coordinates with a Global Positioning System unit, photograph (optional, recommended) and describe each location, and place a numbered stake above the visible high-water mark on the bank closest to the sampling location. The photographs and description must be adequate to allow the sampling station to be relocated at a future date.
13. Dispose of investigation-derived wastes according to applicable rules and regulations.

4. CORERS

A corer provides a vertical profile of the sediment, which may be useful in tracing historical contaminant trends. Because displacement is minimal, a corer is particularly useful when sampling for trace metals and organics. Corers can be constructed out of a variety of materials.

For example, a 2-inch diameter polyvinyl chloride pipe with a Teflon-free or HDPE liner can be lowered into the sediment; a 2-inch diameter well cap can be used to form an airtight seal and negative pressure as the pipe is withdrawn.

- Ensure that the corer and (optional) liner are properly cleaned.
- Stand downstream of the sample location.
- Force the corer into the sediment with a smooth continuous motion. Rotate (not rock) the corer if necessary to penetrate the sediment.
- Twist the corer to detach the sample; then withdraw the corer in a single smooth motion. If the corer does not have a nosepiece, place a cap on the bottom to keep the sediment in place.
- Remove the top of the corer and decant the water (into appropriate sample containers for surface water analysis, if required).
- Remove the nosepiece or cap and deposit the sample into a stainless steel, HDPE, or glass tray.
- Transfer the sample into sample containers using a stainless-steel spoon (or equivalent device).

5. SCOOPS AND SPOONS

When sampling at the margins of a waterbody or in shallow water, scoops and spoons may be the most appropriate sampling equipment. For collecting samples several feet from shore or in deeper water, the scoop or spoon may be attached to a pole or conduit.

- Stand downstream of the sample location.
- Collect the sample slowly and gradually to minimize disturbing the fine particles.
- Decant the water slowly to minimize loss of fine particles.
- Transfer the sediment to sample containers or mixing trays, as appropriate.

6. DREDGES

Three types of dredges are most frequently used: Peterson, Ponar, and Eckman. Many other dredge types are available; their applicability will depend upon site-specific factors.

6.1 PETERSON AND PONAR DREDGES

These dredges are suitable for hard, rocky substrates, deep waterbodies, and streams with fast currents. Ponars have top screens and side plates to prevent sample loss during retrieval.

- Open the jaws and place the cross bar into the proper notch.
- Lower the dredge to the bottom, making sure it settles flat.
- When tension is removed from the line, the cross bar will drop, enabling the dredge to close as the line is pulled upward during retrieval.
- Pull the dredge to the surface. Make sure the jaws are closed and that no sample was lost during retrieval.
- Open the jaws and transfer the sediment to sample containers or to a mixing tray.

6.2 ECKMAN DREDGE

The Eckman dredge works best in soft substrates in waterbodies with slow or no flow.

- Open the spring-loaded jaws and attach the chains to the pegs at the top of the sampler.
- Lower the dredge to the bottom, making sure it settles flat.
- Holding the line taut, send down the message to close the jaws.
- Pull the dredge to the surface. Make sure the jaws are closed and that no sample was lost during retrieval.
- Open the jaws and transfer the sediment to sample containers or a mixing tray.

7. REFERENCE

U.S. Geological Survey (USGS), 2018. *National Field Manual for the Collection of Water Quality Data* Version 1.1, USGS, June 2018.