AECOM
1350 Deming Way
Suite 100
Middleton, WI 53562

T: +1-608-836-9800
aecom.com

March 20, 2023

Mr. John Sager
Wisconsin Department of Natural Resources
1701 North 4th Street
Superior, Wisconsin 54880

PFAS Site Investigation Work Plan – Supplement #1
Enbridge Energy Superior Terminal
2800 East 21st Street
Superior, Wisconsin
BRRTS No. 02-16-589282, FID# 816010580
AECOM Project No. 60683192

Dear Mr. Sager:

On the behalf of Enbridge Energy, Limited Partnership (Enbridge), AECOM Technical Services, Inc. (AECOM) is providing the Wisconsin Department of Natural Resources (WDNR) this first supplement to the previously submitted site investigation work plan for investigating Per- and Polyfluoroalkyl Substances (PFAS) at the above referenced facility. The original work plan was submitted on April 28, 2022 and approved by the WDNR on June 2, 2022. The original work plan was prepared in response to the WDNR's "Responsible Party (RP)" letter dated February 28, 2022, and in general accordance with Chapter NR 716, Wisconsin Administrative Code (WAC), requirements.

The results of the original site investigation were provided in a report submitted to the WDNR on November 4, 2022. Following review of the site investigation report, the WDNR provided comments in a letter dated January 20, 2023, which requested additional investigation. Therefore, Enbridge proposes to continue conducting the PFAS site investigation at the Superior Terminal (Terminal) in a phased approach. The purpose of this work plan supplement is the following:

- to evaluate the vertical extent of PFAS soil impacts at selected locations based on the results from the PFAS surface soil investigation, and
- to determine potential PFAS impacts to surface water from the storage and historic use of aqueous film-forming foam (AFFF) for fire suppression at the Terminal.

Based on the results of this investigation, subsequent investigation phases may be added to further evaluate the impacts of PFAS at the Terminal. This work plan supplement describes the scope and methods of the proposed additional investigation and does not repeat information regarding the background and site characteristics from the original work plan.

Involved Parties

Responsible Party
Enbridge Energy, Limited Partnership (Enbridge)
11 East Superior Street, Suite 125
Duluth, Minnesota 55802
Contact: Karl Beaster, PG, Senior Environmental Advisor
Karl.beaster@enbridge.com
(715) 718-1040

Environmental Consultant
AECOM Technical Services, Inc. (AECOM)
Leo Linnemanstons, PG, Senior Project Manager
1350 Deming Way, Suite 100
Middleton, Wisconsin 53562
leo.linnemanstons@aecom.com
(608) 828-8208

Proposed Drilling Subcontractor (as needed)
Dakota Technologies, Inc
5001 Boone Avenue North
New Hope, Minnesota 55428
(763) 424-4803

Buried Utilities Clearance Subcontractor
Northwestern Surveying & Engineering
603 Chestnut Street
Cloquet, Minnesota 55720
(218) 444-9394

Laboratory Subcontractor
PACE Analytical Laboratory
1700 Elm Street SE
Minneapolis, Minnesota 55414
(612) 607-1700
WDNR Certification: 999407970

Site Description

The Terminal is located at 2800 East 21st Street, Superior, Wisconsin in Douglas County (see Figure 1) and is approximately 560 acres in size, which occupies portions of Sections 30 and 31, Township 49N, Range 13W and Section 36, Township 49N and Range 14W. Figure 2 shows site features and the layout of the Terminal.

Previous Site Investigation

The purpose of the previous investigation was to evaluate the potential of PFAS in soil associated with the storage and historic use of AFFF for fire suppression at the Terminal. Site Investigation activities at the Terminal occurred in June, July, and August 2022. To evaluate the presence of PFAS in soil related to the use of AFFF at the Terminal, the site investigation included the following components:

- Surface Soil Samples (31 locations): one surface soil sample was collected from within the secondary containments of aboveground storage tanks (Tanks) numbered 1-29. These sample locations were

preferentially selected within the secondary containments to be from the lowest elevations following the apparent surface water drainage patterns. In addition, surface soil samples were collected at each of the two buildings in which AFFF and associated AFFF dispersal equipment were stored.

- Hydraulic Probe Boring (1 location): a direct push soil probe boring was conducted through the backfill of the former excavation at the 2012 AFFF discharge location. A soil sample was collected from the native soils encountered at base of the former excavation and second sample was collected approximately 2 ft below the first sample. The hydraulic probe boring was completed to 20 ft below ground surface.

Based on previous site investigations and the results of the surface soil investigation activities, the following conclusions were made:

- PFAS were detected in all surface soil samples at the site, but concentrations were less than NR720 generic residual contaminant levels (RCLs).
- In surface soil samples, PFAS was generally detected at low concentrations that have patterns and/or correlations that suggest historic releases in containment areas that may have been the result of different training practices and/or AFFF products. The following general observations were made:

  PFOA was detected in 30 of 31 samples, but only low-level concentrations <1 ug/kg.

  PFOS was detected in 28 of 31 samples, but with only three isolated tank locations (TK11, TK24, and TK25) having concentrations >10 ug/kg and six tank locations (TK02, TK13, TK15, TK20, TK23, and TK26) having concentrations >1 ug/kg.

  6:2 FTS was detected in 14 of 31 samples, but with only three tank locations (TK11, TK15, and TK25) having concentrations >1 ug/kg and five tank locations (TK02, TK13, TK19, TK20 and TK26) having concentrations >0.1 ug/kg.
- No PFAS was detected in the soil probe samples collected from the 2012 AFFF deployment excavation suggesting that remedial excavation had successfully removed impacted materials, and none had migrated vertically into the underlying red clay layer.
- Surface soil conditions in the containment areas at the site generally consist of a red, lean clay. Based on previous soil borings at the site the red clay layer extends to a depth of approximately 100 to 140 feet with bedrock encountered at depths greater than 260 ft below ground surface. The red clay layer has a very low permeability ($1x10^{-8}$ centimeters per second), which limits infiltration of surface water and migration of surface impacts into the subsurface and to groundwater.

Because the Terminal no longer uses AFFF that contain PFAS, a continuing source of PFAS impacts to soil is not present.

Surface Water Management

The surface water management information for the Terminal provided below was summarized from the *Enbridge Superior Terminal Water Management Plan* (May 2021), previously provided to and approved by the WDNR.

At this time, stormwater is directed through onsite conveyances to the Terminal's pond systems. Figure 3 shows the surface water flow directions and locations for the ponds and the permitted outfalls. The Terminal has three pond systems (004 West Ponds, 005 South Ponds and 006 East Ponds) for the management of stormwater and the approved non-storm discharges. Each of the pond systems has holding capacity and control structures at their terminus. Each system discharges via dry run ditches to the Nemadji River, two (Outfalls 005 and 006) flow through wetlands prior to joining the river.

Field Investigation

To evaluate the presence of PFAS in soil and surface water related to the former use of AFFF at the Terminal, this phase of the site investigation will include the following components:

- Vertical Profile Soil Samples (4 locations, see Figure 2): Vertical soil profile samples are planned to be collected from within the containment areas for Tanks 11, 15, 24, and 25. These sample locations were selected based on the four highest PFAS concentrations detected during the Surface Soil Investigation.

- Surface Water Samples (2 locations, see Figure 3): Surface water samples will be collected at the two permitted outfall structures (Outfalls 004 and 005) for the Terminal that discharge stormwater that is collected from the tank containment areas (Tanks 1 to 29), where PFAS were identified in the surface soil samples.

  Additional ASTs, identified as Tanks 30 to 45, are located in a separate area to the east of Grand Avenue and do not have a history of fire training where AFFF would have been discharged. Therefore, these AST locations and the associated outfall structure (Outfall 006) are not included as part of this investigation.

Pre-Field Planning

Prior to the start of sampling activities, AECOM will conduct a site reconnaissance to evaluate field conditions and determine locations for the proposed samples. Based on actual site conditions, field sampling methods may need to be adjusted, but they are still expected to be consistent with the methods described in this work plan.

Health and Safety Plan

The site-specific Health and Safety Plan (HASP) will be updated for the new field work to meet both AECOM and Enbridge Health and Safety requirements, processes, and procedures. The HASP will contain a summary of known site contaminants and other site hazards, emergency resources available, personnel protection equipment, decontamination procedures, and emergency procedures recommended for this project.

Utility Locating

Public utility locate calls will be made and utility locate tickets will be obtained from Digger's Hotline prior to ground disturbance. Because the Terminal is an active crude oil facility with significant buried infrastructure, Enbridge Ground Disturbance Standards will be followed. This includes conducting 4-way sweeps around borings that extend greater than 12 inches below ground surface. Where necessary, borings will be located away from marked infrastructure in areas that have been swept for subsurface utilities.

A subcontractor may be needed to perform required hydrovacuum excavations to confirm buried utility locations at or in the vicinity of planned soil sample locations. Spoils from hydrovacuum excavations will be managed in the Terminal's soil management area.

General PFAS Sampling Considerations

There are potential cross contamination issues associated with PFAS sampling due to the presence of these compounds in many commercial products. Therefore, AECOM PFAS-certified sampling teams will conduct the PFAS monitoring events. AECOM certification requires attending an internal PFAS sampling training course and reviewing the AECOM PFAS Sampling Guidance document designed to make AECOM samplers aware of the products known to have tested positive for PFAS compounds, as well as identifying PFAS-free products that are appropriate to use in the sampling environment.

AECOM will verify that the subcontractor is following PFAS-free drilling protocols and providing PFAS-free materials for the project, including drilling/decon water.

Vertical Soil Profiling

AECOM will direct a subcontractor to conduct shallow soil borings near selected locations where the four highest PFAS concentrations were found during the surface soil investigation. Prior to the start of the borings, these locations will be cleared for buried utilities to a depth approved by Enbridge Operations. Once the borehole locations are cleared, vertical soil profile samples will be collected at these borings locations to a maximum depth of 4 feet below ground surface (bgs). The vertical profile samples will be collected at the following intervals:

Interval ID	1	2	3	4	5
Depth Interval	0.0-0.5 ft	0.5-1.0 ft	1.5-2.0 ft	2.5-3.0 ft	3.5-4.0 ft

Because the first interval (0.0-0.5 ft) was collected and analyzed during the initial Surface Soil Investigation, that sample interval will be omitted from laboratory analysis for this investigation. Using the survey coordinates for the previous sample locations, AECOM plans that the vertical profile sampling will be conducted within 10 feet of the corresponding surface soil sample.

Depending on Terminal requirements for subsurface excavation, vertical soil profile samples will be collected using either hand auger methods or a hydraulic probe rig. The hand auger method will be performed using a decontaminated stainless steel bucket auger to collect soil samples in approximately 6-inch intervals. Soil grab samples will be carefully removed from the bucket auger to avoid soil slough in the bottom of the borehole or from extracting the auger from the borehole. The hydraulic probe method will directly push a macro-core sample tube to collect a continuous soil sample from the ground surface to the boring terminus. If required and conditions allow, the macro-core sample tube may be advanced by hand-driving to the required depth.

AECOM will classify soil from the boring according to the Unified Soil Classification System (USCS). Because of the presence of red clay and nearby surface waters, perched groundwater may infiltrate these boreholes. If present, the depth to water will be noted relative to the ground surface, and the boring will proceed to its planned depth. The soil sample location will be photographed, and its geographic location recorded using a hand-held GPS unit.

For laboratory analysis, a grab soil sample will be collected at each of the identified depth intervals. Soil samples for laboratory analysis will be transferred, with a decontaminated stainless-steel spoon or nitrile gloves, to appropriate laboratory-supplied containers, labeled, and maintained on ice in insulated coolers. Soil samples will be submitted to the laboratory for sequential analysis progressing from shallow to deep intervals. If PFAS is detected in the sample interval submitted, the next deeper sample will be submitted for analysis. If PFAS is not detected in a sample interval, then remaining deeper soil samples will not be analyzed.

Once soil sampling is completed, the soil boring will be abandoned by backfilling with bentonite in accordance with Chapter NR141, WAC.

Surface Water Sampling

Because the WDNR does not have surface water sampling guidance for PFAS, surface water samples will be collected from the selected locations following methods and procedures described in the Michigan Department of Environmental Quality's (MDEQ's) Surface Water PFAS Sampling Guidance (Attachment A). Because the sample locations are anticipated to be at ponds, AECOM is anticipating that a sufficient water depth will be available to use a direct submersion method to collect surface water samples. If the water depth is not sufficient for a direct submersion method, AECOM will use dedicated tubing and a peristaltic pump for sample collection.

AECOM will use laboratory provided PFAS-free sample containers. For the direct submersion method, the sealed sample bottle will be submersed into the water column, the cover will be removed to allow the container to fill, and then the cover will be replaced before removing the container from the water column. For the peristaltic pump method, the dedicated sample tube will be submerged into the water column, water will be pumped and allowed to discharge from the tubing, and then the sample container will be filled via the tubing.

Water samples will be collected upstream of the sampler, and caution will be exercised to minimize turbidity in the surface water body. Surface water samples will be centrifuged at the laboratory to consolidate suspended solids in the water sample container.

Water samples for laboratory analysis will be collected in appropriate laboratory-supplied containers, labeled, and maintained on ice in insulated coolers.

Laboratory Analyses

Samples will be shipped overnight in the cooler, on ice, under chain-of-custody protocol to the WDNR PFAS certified laboratory for analyses. Samples will be analyzed for the Wisconsin list of 33 PFAS using EPA modified Method 537.1 isotope dilution. The samples will be analyzed on a standard (15-day) turn-around-time (TAT). Level/Tier IV quality control reporting will be provided by the lab. Due to the use of the isotope dilution method, Matrix Spike/Spike Duplication (MS/MSD) analysis will not be requested.

Quality Assurance

Standard sampling protocols for PFAS compounds include the use of field and equipment blanks due to the possible ubiquitous nature of these compounds including the potential presence of these compounds in sampling equipment and supplies, and to assess the possibility of cross-contamination during sampling, transport, and sample storage. Due to the use of the isotope dilution method, Matrix Spike/Spike Duplicate (MS/MSD) analysis will not be requested. As such, the following Quality Assurance samples will be collected for this project:

Field Blank: One field blank will be collected during the sample collection activities. The field blank will be collected by pouring laboratory-certified PFAS-free water into a laboratory-provided sampling container.

Equipment Blank: One equipment blank will be obtained by pouring laboratory certified PFAS free water over the decontaminated sampling equipment and collecting the water in a laboratory provided bottle.

Water Blank: One sample of the water used by the driller will be collected and tested as a water blank. The water blank sample will be collected to characterize the wash water used during the decontamination process.

These blank samples will be shipped to the laboratory with the field samples. AECOM will provide laboratory data validation review using procedures described in the National Functional Guidelines for High Resolution Superfund Method Data Review (EPA, April 2016), as appropriate.

Equipment Decontamination

Decontamination of the stainless-steel soil sampling equipment will be performed between the collection of each soil sample and will consist of removing solids from the equipment, washing with Alconox, and then triple rinsed with PFAS-free water. Decontamination of the drilling rig and down-hole equipment will also include high-pressure cleaning before and after conducting the direct push soil probe borings.

Investigation Derived Waste

Excess soil generated during the advancement of the soil borings will be contained and stored in the Superior Terminal Soil Management Area. A composite waste characterization sample will be collected and analyzed. Pending receipt of the waste characterization laboratory results, the contaminated material will be transported off the property and properly disposed offsite at a licensed disposal facility.

Sample Notification and Site Investigation Report

AECOM will notify the WDNR (via WDNR Form 4400-249) within ten business days after receiving the sampling results and completing the data validation review. The report will generally follow NR 716.15 requirements, which includes, project contacts, site and background information, investigation methods, sampling and analysis requirements, field and analysis results, and conclusions and recommendations. Tables summarizing laboratory results and figures that include sample locations will be included. The required soil boring forms and well/borehole abandonment forms will be completed and attached to the report.

Project Schedule

Scheduling for initial field tasks is anticipated to commence upon receipt of concurrence of the proposed scope of work from the WDNR. A Subsurface Investigation report in general conformance to NR 716 will be submitted to the WDNR within 60 days of receipt of the analytical data.

Project Phase	Date
Site Investigation Review Letter Received	January 20, 2023
Work Plan Submitted to WNDR for Review	March 20, 2023
Surface Water and Vertical Soil Sampling	June 2023
Analytical Receipt	July 2023
Draft Site Investigation Report	August 2023

Enbridge will notify the WDNR of significant changes to the site investigation schedule.

Concurrence

Enbridge requests that WDNR provide written comments and concurrence with the scope of work presented in this site investigation work plan. The Work Plan review fee and Technical Assistance Form 4400-237 will be submitted to the WDNR separately.

If you have any questions, please contact Leo Linnemanstons at (608)828-8208.

Sincerely,

AECOM Technical Services, Inc.

Leo B. Linnemanstons, PG
Project Manager / Hydrogeologist
AECOM
leo.linnemanstons@aecom.com

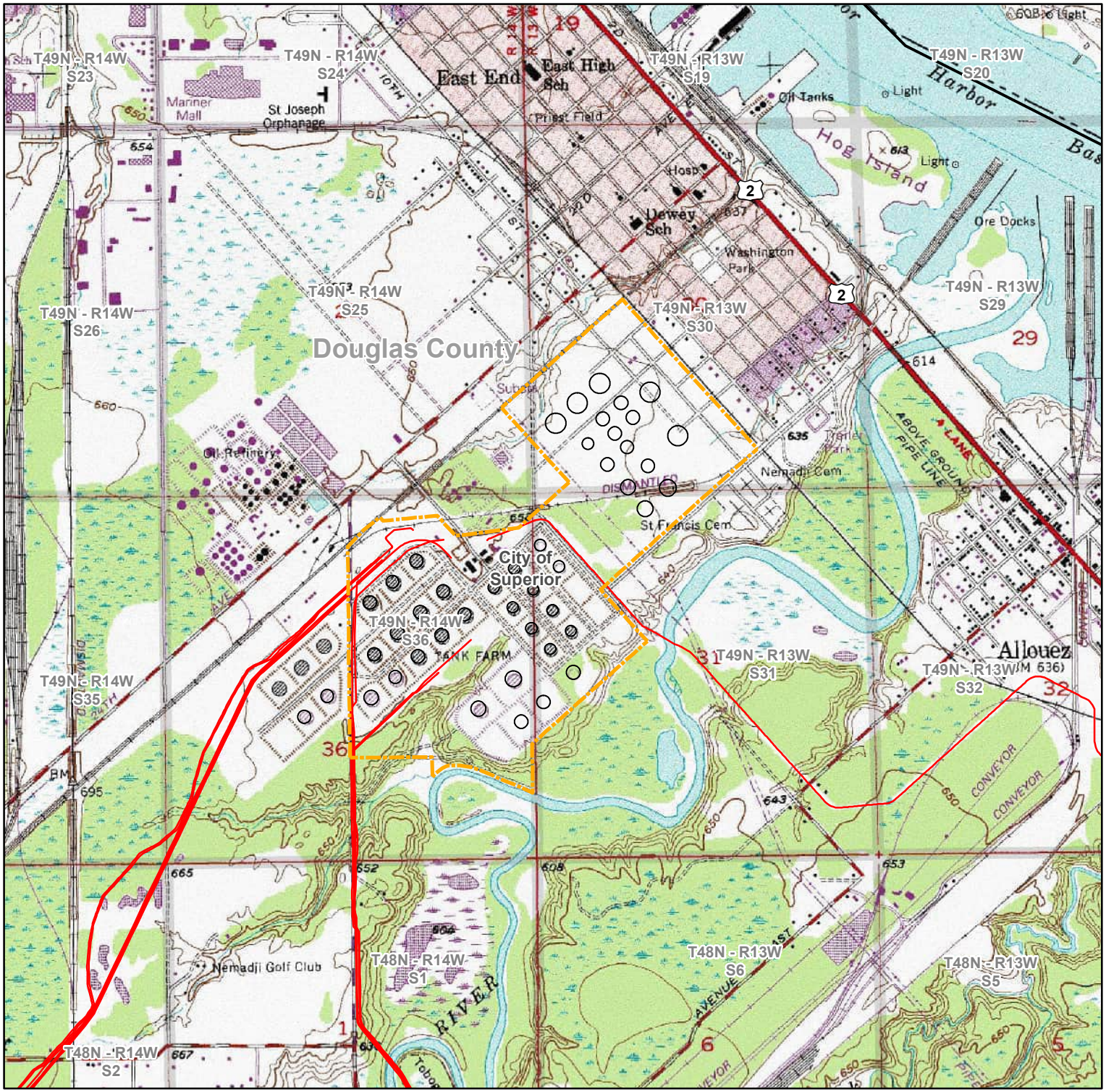
David Henderson, P.E.
Senior Project Engineer
AECOM
dave.henderson@aecom.com

Attachments:

Figure 1 Site Location Map
Figure 2 Vertical Soil Profile Sample Locations
Figure 3 Surface Water Sample Locations
MDEQ Surface Water PFAS Sampling Guidance

Cc: Karl Beaster, Senior Environmental Advisor, Enbridge
Bryan Stiemsma, Technical Manager, Enbridge
Nick Larabel, Environmental Advisor, Enbridge

Figures



Site Location

Enbridge Pipelines

Terminal Property Boundary

0 2,000 4,000

Feet

1 Inch = 2,000 Feet

Figure 1

SITE LOCATION

Superior Terminal

Enbridge Energy, L.P.

Superior, Wisconsin

ENBRIDGE

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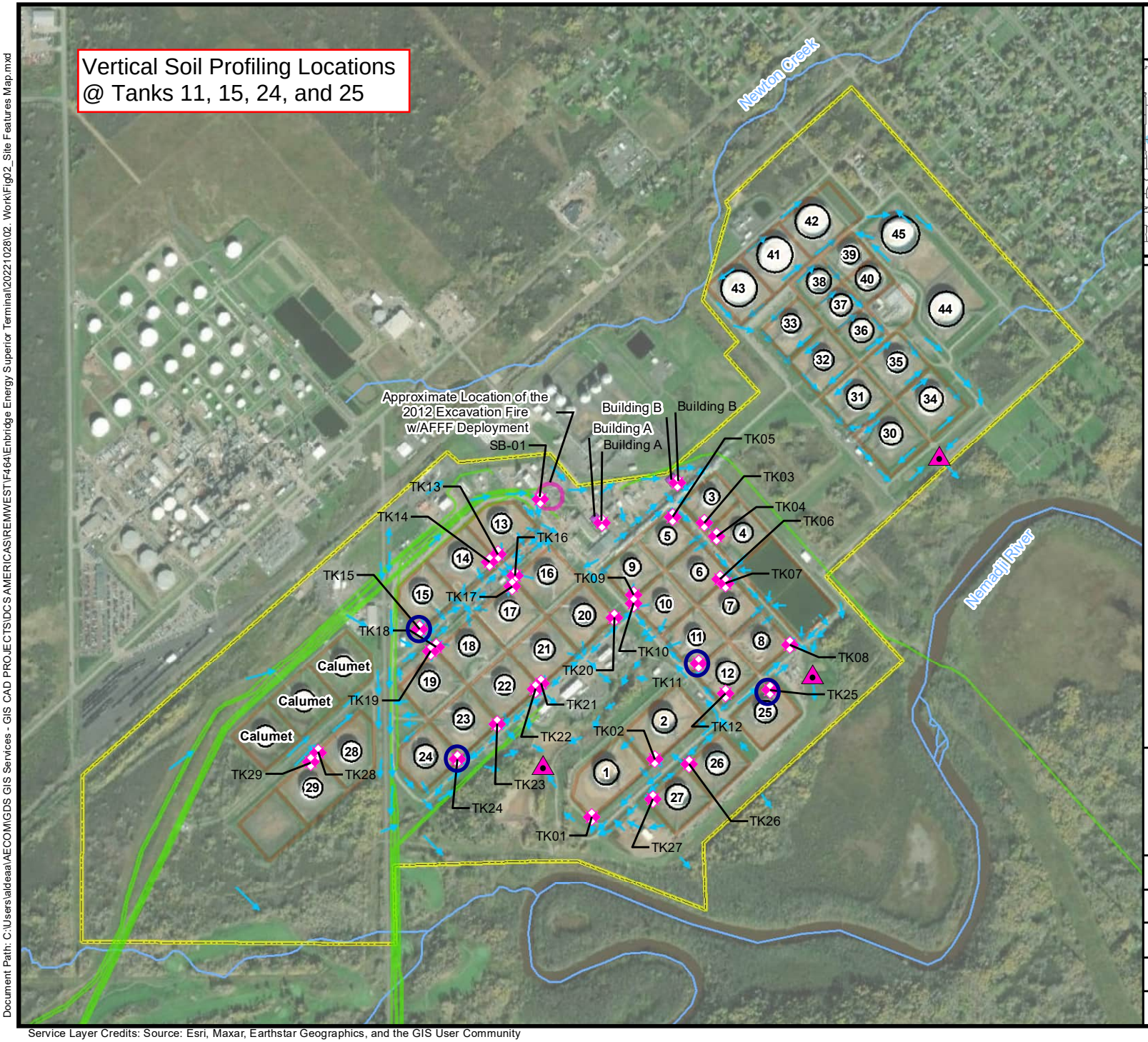

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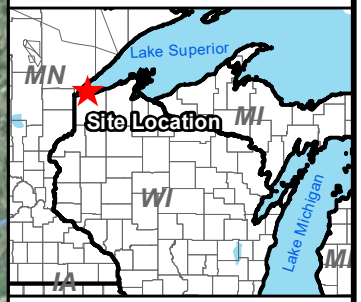

Vertical Soil Profile Samples

Enbridge Energy Superior Terminal
2800 East 21st Street
Superior, Wisconsin

Legend

- Sample location
- WPDES Outfalls
- Containment Berm
- Surface Flow Direction
- Storage Tank
- Enbridge Pipelines
- Terminal Property Boundary
- Rivers/Streams
- Planned Sample Locations

0 625 1,250 Feet

1 inch = 1,167 feet

N

Prepared by:

AECOM

200 Indiana Avenue
Stevens Point WI , USA
www.AECOM.com

Drawn:	AA	10/28/2022
Approved:	CS	10/28/2022
Scale:		1:14,000
Project No:		60683192
Figure No:		2

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Service Layer Credits: Source: Esri, Maxar, Earthstar Geographics, and the GIS User Community

Outfall Location

Surface Flow Direction

Superior Terminal Property Boundary

Planned Sample Locations

Drawn: 3/31/2020

Service Layer Credits: Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGRID, IGN, and the GIS User Community. City of Superior 2008, 1 ft. LIDAR DEM

WSP USA Inc.
302 W SUPERIOR ST
SUITE 70
DULUTH, MN 55802
TEL: +1 218 336 2285

SUPERIOR TERMINAL
SUPERIOR, WI

PREPARED ON BEHALF OF

ENBRIDGE ENERGY, LIMITED PARTNERSHIP

FIGURE 3

SURFACE WATER SAMPLING LOCATIONS

MDEQ Surface Water PFAS Sampling Guidance

SURFACE WATER PFAS SAMPLING

Guidance

Introduction

This sampling guidance contains the processes, decontamination procedures, and acceptable items and materials for sampling surface water for Per- and Polyfluoroalkyl Substances (PFAS). This guidance will be used to support the sampling objectives and procedures based on the Quality Assurance Project Plan (QAPP) developed prior to any field activities. This guidance assumes staff has basic familiarity with and/or understanding of basic surface water sampling procedures.

NOTE: Review the **General PFAS Sampling Guidance** prior to reviewing this guidance document.

The Michigan Department of Environmental Quality (MDEQ) intends to update the information contained within this Surface Water PFAS Sampling Guidance document as new information becomes available. The user of this Surface Water PFAS Sampling Guidance is encouraged to visit the Michigan PFAS Action Response Team webpage (www.michigan.gov/PFASresponse) to access the current version of this document.

PFAS has been detected in surface water in Michigan at concentrations of over 19,000 parts per trillion (ppt). Because PFAS compounds can be analyzed at concentrations in the parts per trillion (ppt) range, precautions must be taken to prevent cross-contamination. Therefore, there is a high possibility of false positives if decontamination procedures are not followed diligently. This sampling guidance covers both the collection of samples from shallow and deep surface water bodies.

This Surface Water PFAS Sampling Guidance discusses the collection of surface water samples and methods to prevent cross-contamination that can occur from:

- Field clothing and personal protective equipment (PPE)
- Personal care products (PCPs)
- Food packaging
- Sampling equipment
- Equipment decontamination
- Filtering of surface water
- Sample collection and handling
- Sample shipment

Contents

NOTE: Additional information about PFAS testing can be found on the Michigan PFAS Action Response Team (MPART) website: www.michigan.gov/PFASresponse

1. Potential Sources for PFAS Cross-Contamination

Potential sources for PFAS cross-contamination include items and materials used within the sampling environment, such as sampling equipment, field clothing, personal protective equipment (PPE), sun and biological protection products, personal hygiene, personal care products (PCPs), and food packaging. A detailed discussion about potential sources for PFAS cross-contamination is included in the **General PFAS Sampling Guidance**, which should be reviewed before reading this document. However, a high-level summary is presented in this guidance.

All of the items and materials discussed in each of the MDEQ's PFAS Sampling Guidance Documents are divided into three major groups:

- Prohibited (●) identifies items and materials that should not be used when sampling. It is well documented that they contain PFAS or that PFAS are used in their manufacture.

- Allowable (■) identifies items and materials that have been proven not to be sources of PFAS cross contamination and are considered acceptable for sampling.

- Needs Screening (▲) identifies items and materials that have the potential for PFAS cross-contamination due to a lack of scientific data or statements from manufacturers to prove otherwise. These items and materials are further sub-divided into two categories:

    - **Category 1:** Items and materials that will come in direct contact with the sample. These should not be used when sampling unless they are known to be PFAS-free, by collecting an equipment blank sample prior to use.

    - **Category 2:** Items and materials that will not come in direct contact with the sample. These should be avoided, if possible, unless they are known to be PFAS-free by collecting an equipment blank sample prior to use.

● - Prohibited ■ – Allowable ▲ - Needs Screening

Please note that at this time no published research is available that documents the use of various materials and effect on sample results. Therefore, a conservative approach is recommended, and the guidance is based on the collection of multiple environmental samples at various PFAS Sites. Sampling staff should take practical and appropriate precautions to avoid items that are likely to contain PFAS at the sampling site as well as avoid specific items during the sampling event.

A general overview of PFAS contamination sources during sampling can be found in **Section 4.2** of the **General PFAS Sampling Guidance**. Any items or materials utilized that are not identified in this guidance or not discussed in **Section 4.2** should be evaluated as described in **Section 4.2.1**.

Sampling staff should take practical and appropriate precautions to avoid items that are likely to contain PFAS at the sampling site as well as avoid specific items during the sampling event (see below).

1.1 Field Clothing and Personal Protection Equipment (PPE)

A general overview of field clothing and PPE can be found in **Section 4** of the **General PFAS Sampling Guidance**.

As with any field mobilization, it is the responsibility of all personnel to be aware of the physical, chemical and biological hazards associated with a particular site. Personal safety is paramount. The safety of staff should not be compromised by fear of PFAS-containing materials without any scientific basis. Any deviation from this guidance, including those necessary to ensure the health and safety of sampling personnel, should be recorded in field notes and discussed in the final report.

Depending on the project objectives and sampling plan, the collection of surface water samples could be as simple as a grab sample or as complex as a sample collected using a Van Dorn® sampler from a boat. Generally, for surface water sampling, approved field clothing (discussed in **Section 4** of the **General PFAS Sampling Guidance**) is required. Life jackets made of PFAS-free materials should be used. The coatings used on waders are of particular concern during surface water sampling. Ensure the waders are made from PFAS-free materials before use.

NOTE: Special attention should be given to clothing that has been advertised as having waterproof, water-repellant, or dirt and/or stain characteristics. They are likely to have PFAS in their manufacturing.

NOTE: Life jackets may have protective coatings that contain PFAS.

- ● Do not use waders made of Gore-Tex or other known PFAS containing materials.
- ■ Life jackets made of polyethylene foam and nylon shell fabric may be used.
- ■ Waders made of Neoprene or other PFAS-free materials may be used.

Any field clothing and/or PPE items that might be required for surface water sampling and not discussed in this guidance should be evaluated as described in **Section 4.2.2** of the **General PFAS Sampling Guidance**.

NOTE: Both field clothing and PPE should be kept dust and fiber free. During the sample collection, extra care should be taken so that no dust or fibers can fall into the sample bottle.

Powderless nitrile gloves should frequently be changed any time there is an opportunity for cross-contamination of the sampling including, but not limited to, the following activities:

- Each time sampling equipment is handled.
- Prior to sample collection.

● - Prohibited ■ – Allowable ▲ - Needs Screening

- After handling any sample, including QA/QC samples such as field reagent blanks or equipment rinsate blanks.
- After the handling of any non-dedicated sampling equipment, contact with non-decontaminated surfaces, or when judged necessary by field personnel.
- During and after decontamination of non-dedicated sampling equipment.

1.2 Personal Care Products (PCPs)

A number of sampling guidance documents recommend that personal hygiene and personal care products (PCPs) (e.g., cosmetics, shampoo, sunscreens, dental floss, etc.) not be used prior to and on the day(s) of sampling because the presence of PFAS in these products has been documented (OECD, 2002, Fujii, 2013, Borg and Ivarsson, 2017). However, if the MDEQ's sampling SOPs are followed, these items should not come into contact with the sampling equipment or the sample being collected. As of the date of this sampling guidance, cross-contamination of samples due to the use of PCPs has not been documented during the collection of thousands of samples. However, field personnel should be aware of the potential of cross-contamination if the sampling equipment or actual samples would come into contact with these products. The following precautions should be taken when dealing with personal hygiene or PCPs before sampling:

- ● Do not handle or apply PCPs in the sampling area.
- ● Do not handle or apply PCPs while wearing PPE that will be present during sampling.
- ■ Move to the staging area and remove PPE if applying personal care products becomes necessary.
- ■ Wash hands thoroughly after the handling or application of PCPs and, when finished, put on a fresh pair of powderless nitrile gloves.

1.3 Food Packaging

PFAS has been used by the paper industry as a special protective coating against grease, oil, and water for paper and paperboards, including food packaging since the late 1950s (Trier et al., 2018). PFAS application for food packaging includes paper products that come into contact with food such as paper plates, food containers, bags, and wraps (OECD, 2002). Pre-wrapped food or snacks (such as candy bars, microwave popcorn, etc.) must not be in the sampling and staging areas during sampling due to PFAS contamination of the packaging. When staff requires a break to eat or drink, they should remove their gloves, coveralls, and any other PPE, if worn, in the staging area and move to the designated area for food and beverage consumption. When finished, staff should wash their hands and put on a fresh pair of powderless nitrile gloves at the staging area, before returning to the sampling area.

- ● Do not handle, consume, or otherwise interact with pre-wrapped food or snacks, carry-out food, fast food, or other food items while on-site during sampling.
- ■ Move to the staging area and remove PPE prior to leaving the sampling and staging areas if consuming food on site becomes necessary.

2. Surface Water Sampling Equipment

Surface water sampling equipment that is also used for non-PFAS sampling such as dippers, Kemmerer®, or Van Dorn® samplers, should be decontaminated prior to collecting PFAS samples to avoid cross contamination. This non-dedicated equipment (equipment used for more than one water body or location) should be verified that it is PFAS free at least once prior to use. Surface water sampling equipment can fall into **Category 1** or **Category 2**:

● - Prohibited ■ – Allowable ▲ - Needs Screening

Category 1: Surface water sampling equipment that will come into contact with the surface water sample include sample bottles and various surface water samplers or tubing. Sample bottles should be provided by the laboratory and known to be PFAS free. Any surface water samplers, tubing, or materials that will come into contact with the surface water samples should be screened and known to be PFAS-free. The tubing should always be kept in the original cardboard or bag in which it was shipped. The tubing should always be stored in a clean location free of dust and fibers.

NOTE: As a precautionary action, an equipment rinsate blank should be collected even if the sampling materials are made of materials that are not expected to contain PFAS.

Category 2: Examples of field equipment that do **not** come into contact with the surface water samples include water quality meters, GPS receivers, notebooks, clipboards, and turbidity meters. The surface of some of these pieces of field equipment, or the storage boxes in which they are kept, might contain PFAS.

Do not use any equipment that contains any known fluoropolymers including, but not limited to:

- ● Do not use polytetrafluoroethylene (PTFE), that includes the trademark Teflon® and Hostaflon®, which can be found in many items, including but not limited to the lining of some hoses and tubing, some wiring, certain kinds of gears, and some objects that require the sliding action of parts.
- ● Do not use Polyvinylidene fluoride (PVDF), that includes the trademark Kynar®, which can be found in many items, including but not limited to tubing, films/coatings on aluminum, galvanized or aluminized steel, wire insulators, and lithium-ion batteries.
- ● Do not use Polychlorotrifluoroethylene (PCTFE), that includes the trademark Neoflon®, which can be found in many items, including but not limited to valves, seals, gaskets, and food packaging.
- ● Do not use Ethylene-tetrafluoro-ethylene (ETFE), that includes the trademark Tefzel®, which can be found in many items, including but not limited to wire and cable insulation and covers, films for roofing and siding, liners in pipes, and some cable tie wraps.
- ● Do not use Fluorinated ethylene propylene (FEP), that includes the trademarks Teflon® FEP and Hostaflon® FEP, and may also include Neoflon®, which can be found in many items, including but not limited to wire and cable insulation and covers, pipe linings, and some labware.

Note: Manufacturers can change the chemical composition of any product. As a result, all materials that will come into direct contact with the sample media should be tested to confirm they are "PFAS-free", i.e. will not contaminate samples at detectable levels. **There is no guarantee that materials in the 'Allowable' category will always be PFAS- free.**

- ● Do not use low density polyethylene (LDPE) for any items that will come into **direct contact** with the sample media. LDPE can be found in many items, including but not limited to containers and bottles, plastic bags, and tubing.
  - ▲ **However**, LDPE may be used if an equipment blank has confirmed it to be PFAS-free. LDPE does not contain PFAS in the raw material but may contain PFAS cross-contamination from the manufacturing process.
- ■ LDPE bags (e.g. Ziploc®) that **do not** come into direct contact with the sample media and do not introduce cross contamination with samples may be used.
- ■ Use materials that are either made of high density polyethylene (HDPE), polypropylene, silicone, or acetate.
- ■ Use only powderless nitrile gloves (which can be found at some hardware and major retail outlets).
- ■ Keep tubing in the original cardboard or bag in which it was shipped.

●- Prohibited ■ – Allowable ▲- Needs Screening

- ■ Store tubing in a clean location free of dust and fibers.
- ▲ Latex gloves should be screened before use.
- ▲ Post-It® Notes should be screened before use.

NOTE: Depending on the project objectives, boats might be required to be used during surface water sampling. Boats might have various parts that may contain PFAS, including protective water repellent coatings. When boats are used on rivers, samples should always be collected on the upgradient side of the boat.

Depending on the project data quality objectives, water samples can be collected as: a simple grab directly into the sample bottle; a grab sample at a selected depth using any of several collection bottles with subsequent transfer to the sample bottle(s); or as a depth integrated sample. A depth integrated sample can be collected using a simple weighted bottle constructed to allow gradual water inflow (e.g., chlorophyll sampler), or by using a Van Dorn® or Kemmerer® sampler and compositing grab samples from several depths. Composited samples are then transferred to the sample bottle.

Surface water sampling collection can be divided into two method categories as presented in the following Table 1.

Table 1. Surface Water Sampling Methods[1]

Depth to Surface Water Sample	Locations	Sampling Method
0-5 feet	Streams, rivers, creeks, tributaries, lakes, lagoons, ponds, and impoundments.	Direct method, swing, telescoping, and Van Dorn, depth integrating samplers.
Over 5 feet	Large streams, rivers, tributaries, lakes, lagoons, ponds, and impoundments.	Peristaltic pump, swing, telescoping, Van Dorn, Kemmerer, and depth integrating samplers.

[1]This table includes the most frequently used methods for surface water samples.

2.1 Container Immersion

Two types of immersion sampling equipment are available for surface water sampling: extension rods and submersible devices. Extension rods can be used to immerse the actual sample bottle, different types of beakers, or peristaltic pump tubing into the surface water. Submersible devices (i.e., Kemmerer Bottle, Van Dorn Sampler) are fully immersed into the surface water using a rope.

2.1.1 Extension Rods

The most common extension rods are telescoping or swing samplers. Both types of sampling equipment are very similar in design and concept, and both facilitate the immersion of either the sampling bottle or various beakers or scoops. Lists of various extension rod designs are provided below:

- Pendulum or angular beaker.
- Fixed scoop.
- Fix or rotatable head bottle holder.

A peristaltic pump can also be used with extension rods by attaching the tubing to the extension rods and immersing both the rods and the connected tubing to the desired depth in the surface water.

- ■ Use only sample collection equipment, tubing, beakers, and/or scoop materials that are known to be PFAS-free such as stainless steel, glass, HDPE, polyvinyl chloride (PVC), or silicone.
- ■ Extension rods made of materials such as aluminum that has been identified as being PFAS-free can be used.

A specialized extension rod that features a telescoping design for the handle could also be used as a subsurface grab sampler. The sample is collected using a cable from the handle, which has a ring that can be opened for the sample collection after the desired depth has been reached.

2.1.2 Submersible Devices

The most common submersible devices being used are Kemmerer Bottles or Van Dorn Samplers. These devices are primarily used when the samples are collected at depths greater than 5 feet from a boat and/or structure such as a bridge or pier. All submersible devices are submerged in the surface water using a rope.

NOTE: Careful evaluation of all submersible samplers' parts should be done. Any parts that might contain PFAS should be replaced with PFAS-free materials. Equipment rinsate blank samples should be collected to make certain the sampler is PFAS-free.

The Kemmerer Bottle sampler is typically constructed of a stainless-steel tube with polyurethane end seals that can collect a total sample volume of 1.2 liters. The Kemmerer Bottle is not ideal for the collection of samples close to the surface, as the tube is immersed vertically in the water.

The Van Dorn® bottle sampler is typically constructed of 1-liter transparent acrylic tube with two end stoppers. The sampler is suspended horizontally, which is ideal for the sample collection in shallow water bodies as well as sampling at depth.

When submersible samplers are used, the following recommendations should be followed:

- ● Do not use any sampling bottle with Teflon end seals.
- ■ Use a Kemmerer® Bottle made of stainless steel with polyurethane end seals.
- ■ Use a Van Dorn® bottle sampler that uses stoppers made of PFAS-free materials.
- ■ Use nylon line, stainless steel cable, or line or wires made of PFAS-free materials for sample collection.
- ■ Use tubing for the sampling ports made of HDPE, polypropylene, silicone, PVC, or other PFC-free materials.

2.2 Direct Sampling

For surface water samples collected near the shore (e.g., from streams, rivers, lakes, and other surface waters), the direct method can be used to collect the water samples directly into the sample container.

- ● Do not sample without powderless nitrile gloves.

- ● Never place the sample cap directly on the ground or boat deck during sampling.
- ■ Use powderless nitrile gloves
- ■ Hands should be well washed
- ■ Use HDPE sample bottles with Teflon®-free caps, provided by the laboratory.
- ■ If sample bottles that are known to be PFAS-free are not available, the sample container and lid should be rinsed with water that is known to be PFAS-free at least 3 times prior to collecting the sample.
- ■ If samples are collected while wading in the water body, the bottle should be immersed inverted and upstream of the collector.
- ■ If samples are collected from a boat, the bottles should be submerged upstream of the boat.

NOTE: Unless specifically required by the project objectives, surface water samples should ***not*** be taken at the top layer of the water body or of surface scums. PFAS are expected to accumulate at the surface water air interface or be present in the surface runoff, so samples taken at the surface are likely to result in high biased results that are not representative of the bulk surface water.

3. Equipment Decontamination

Field sampling equipment that is used at multiple sites or sampling locations (non-dedicated equipment) could become contaminated with PFAS.

The following should be considered when decontaminating any equipment that contacts the sampling media:

- ● Do not use Decon 90®.
- ■ Laboratory supplied PFAS-free deionized water is preferred for decontamination.
- ■ Alconox®, Liquinox®, and Citranox® can be used for equipment decontamination.
- ■ Sampling equipment can be scrubbed using a polyethylene or Polyvinyl chloride (PVC) brush to remove particulates.
- ■ Decontamination procedures should include triple rinsing with PFAS-free water.
- ■ Commercially available deionized water in an HDPE container may be used for decontamination if the water is verified to be PFAS-free.
- ▲ Municipal drinking water may be used for decontamination purposes if it is known to be PFAS-free.

4. Sample Collection and Handling

A preferred sampling sequence should be established prior to any sampling event to reduce the risk of cross contamination. In general, the sampling sequence should begin in areas expected or known to be least contaminated, proceeding to anticipated areas or identified to be most contaminated. If analytical results from past sampling events are available, the sampling sequence can be readily determined.

However, for many PFAS investigation sites, no PFAS sampling has been conducted. In these cases, all site information on possible PFAS uses and potential PFAS migration patterns (e.g., upgradient, downgradient) from PFAS sources at the site should be reviewed prior to the sampling event to help establish the sampling sequence.

If multiple samples (i.e., monitoring wells, surface water, residential) will be collected in an area where a PFAS release in the environment has been documented, samples that are known to be upgradient from the impacted area should be sampled first, followed by those that are furthest downgradient from the

suspected source. The remaining samples should be progressively sampled from the one most distant downgradient to those closer to the known PFAS source.

If no information is available about the site, samples are to be collected in the following order:

1. Drinking Water (e.g., residential wells)
2. Surface Water
3. Groundwater

When collecting and handling surface water samples:

- ● Do not insert or let tubing or any materials inside the sample bottle.
- ● Dust and fibers must be kept out of sample bottles.
- ● The sample cap should never be placed directly on the ground during sampling. If sampling staff must set the sample bottle cap down during sample collection and a second member of the sampling crew (wearing a fresh pair of powderless nitrile gloves) is not available, set the cap on a clean surface (cotton sheeting, HDPE sheeting, triple rinsed cooler lid, etc.).
- ● Regular/thick size markers (Sharpie® or otherwise) are to be avoided; as they may contain PFAS.
- ■ Fine or Ultra-Fine point Sharpies® may be used to label the empty sample bottle while in the staging area provided the lid is on the sample bottle and powderless nitrile gloves are changed following sample bottle labeling.
- ■ Ballpoint pens may be used when labeling sample containers. If ballpoint pens do not write on the sample container labels, preprinted labels from the laboratory may be used.
- ■ Hands should be well washed and gloved.
- ■ Use HDPE, or polypropylene sample bottles with Teflon®-free caps, provided by the laboratory.
- ■ Bottles should only be opened immediately prior to sampling.
- ■ Bottles should be capped immediately after collecting the sample.
- ■ Samples should be double bagged using resealable low density polyethylene (LDPE) bags (e.g., Ziploc®).
- ■ Follow any guidance or requirements in the PFAS analytical reference method that will be used for testing samples, for sample collection, storage, preservation, and holding times.

If a published testing method is not used, and in the absence of formal United States Environmental Protection Agency guidance for PFAS sample storage, the documentation in USEPA Method 537 Rev. 1.1 should be used as a guide for thermal preservation (holding temperature), and holding times for surface water or other samples. Samples must be chilled during storage and shipment, and must not exceed 50°F (10º C) during the first 48 hours after collection.

NOTE: USEPA Method 537 Rev. 1.1 was developed for the analysis of finished drinking water samples only. It was not designed for testing surface water or other matrices that could cause significant interferences to the method.

Surface water samples should be extracted as soon as possible but must be extracted within 14 days. Extracts must be stored at room temperature and analyzed within 28 days after extraction (EPA Method 537 Rev. 1.1).

5. Filtering of Surface Water

Since PFAS can adsorb to particulate matter, unfiltered samples may result in high-biased results. PFAS are known to absorb to various filters. As a result, filtering of surface water samples prior to delivery to the lab should be avoided unless called for in the project data quality objectives. To reduce the need for filtering, samples should be collected with as minimal disturbance to sediments as possible.

NOTE: It is recommended that filtering of the samples should **only be performed in the laboratory** in order to reduce the possibility of cross contamination.

If it is known beforehand that samples will need to be filtered the procedure should be discussed with the laboratory and sample handling methods and responsibilities should be described in the sampling workplan and QAPP.

The following recommendations should be used when considering filtering of the samples:

- ● **Field filtration of the sample is generally not advised.**
  - ▲ If filtering is absolutely necessary, if specifically requested by a client or for other reasons:
- ● Do not use any filters that contain any PFAS, such as PTFE filters
- ● Do not use nylon filters.
- ■ Glass filters are recommended to be used.
- ■ Consider use of a centrifuge in the laboratory to reduce the need for sample filtering.

6. Sample Shipment

When prepping samples for shipping:

- ■ Check the cooler periodically to ensure samples are well iced and at the proper temperature.
- ■ Refresh with regular ice, if needed, double bagged in LDPE resealable storage bags if needed.
- ■ Regular ice should be used to cool and maintain the sample at or below the proper temperature.
  - ▲ Chemical or blue ice may be used if it is known to be PFAS-free and it is absolutely certain that the sample is cooled and maintained at or below the proper temperature during collection and through transit to the laboratory.
- ■ Chain of Custody and other forms should be double bagged in LDPE (Ziploc®) storage bags and taped to the inside of the cooler lid.
- ■ The cooler should be taped closed with a custody seal and, if shipping, shipped by overnight courier.
- ■ Samples should be shipped as soon as possible (e.g. overnight) to ensure the samples arrive within the analytical holding time specified by the lab.

Michigan Department of Environmental Quality

MDEQ PFAS Sampling Quick Reference Field Guide[1]

All Items Used During Sampling Event

● Prohibited

• Items or materials that contain fluoropolymers such as
  ○ Polytetrafluoroethylene (PTFE), that includes the trademarks Teflon® and Hostaflon®
  ○ Polyvinylidene fluoride (PVDF), that includes the trademark Kynar®
  ○ Polycholotrifluoroethylene (PCTFE), that includes the trademark Neoflon ®
  ○ Ethylene-tetrafluoro-ethylene (ETFE), that includes the trademark Tefzel®
  ○ Fluorinated ethylene propylene (FEP), that includes the trademarks Teflon® FEP and Hostaflon® FEP
• Items or materials that contain any other fluoropolymer

Pumps, Tubing, and Sampling Equipment

● Prohibited	■ Allowable	▲Needs Screening[2]

• Items or materials containing any fluoropolymer (potential items include tubing, valves, or pipe thread seal tape)	• High-density polyethylene (HDPE)
	• Low-density polyethylene (LDPE) tubing
	• Polypropylene
	• Silicone
	• Stainless-steel
	• Any items used to secure sampling bottles made from:
	  ○ Natural rubber
	  ○ Nylon (cable ties)
	  ○ Uncoated metal springs
	  ○ Polyethylene
		• Any items or materials that will come into direct contact with the sample that have **not** been verified to be PFAS-free
		  ○ Do not assume that any sampling items or materials are PFAS-free based on composition alone

Sample Storage and Preservation

● Prohibited	■ Allowable	▲Needs Screening[2]
• Polytetrafluoroethylene (PTFE): Teflon® lined bottles or caps	• Glass jars[4] • Laboratory-provided PFAS-Free bottles: ○ HDPE or polypropylene • Regular wet ice • Thin HDPE sheeting • LDPE resealable storage bags (i.e. Ziploc®) that will not contact the sample media[6]	• Aluminium foil[4] • Chemical or blue ice[5] • Plastic storage bags other than those listed as ■ Allowable • Low-density polyethylene (LDPE) bottles

Field Documentation

● Prohibited	■ Allowable	▲Needs Screening[2]
• Clipboards coated with PFAS • Notebooks made with PFAS treated paper • PFAS treated loose paper • PFAS treated adhesive paper products	• Loose paper (non-waterproof, non-recycled) • Rite in the Rain® notebooks • Aluminium, polypropylene, or Masonite field clipboards • Ballpoint pens, pencils, and Fine or Ultra-Fine Point Sharpie® markers	• Plastic clipboards, binders, or spiral hard cover notebooks • All markers not listed as ■ Allowable • Post-It® Notes or other adhesive paper products • Waterproof field books

Decontamination

● Prohibited	■ Allowable	▲Needs Screening[2]
• Decon 90® • PFAS treated paper towel	• Alconox®, Liquinox®, or Citranox® • Triple rinse with PFAS-free deionized water • Cotton cloth or untreated paper towel	• Municipal water • Recycled paper towels or chemically treated paper towels

Clothing, Boots, Rain Gear, and PPE

● Prohibited	■ Allowable	▲Needs Screening[2]

• New or unwashed clothing
• Anything made of or with:
  ○ Gore-Tex™ or other water-resistant synthetics
• Anything applied with or recently washed with:
  ○ Fabric softeners
  ○ Fabric protectors, including UV protection
  ○ Insect resistant chemicals
  ○ Water, dirt, and/or stain resistant chemicals
	• Powderless nitrile gloves
	• Well-laundered synthetic or 100% cotton clothing, with most recent launderings not using fabric softeners
	• Made of or with:
	  ○ Polyurethane
	  ○ Polyvinyl chloride (PVC)
	  ○ Wax coated fabrics
	  ○ Rubber / Neoprene
	  ○ Uncoated Tyvek®
		• Latex gloves
		• Water and/or dirt resistant leather gloves
		• Any special gloves required by a HASP
		• Tyvek® suits, clothing that contains Tyvek®, or coated Tyvek®

Food and Beverages

● Prohibited	■ Allowable

• No food should be consumed in the staging or sampling areas, including pre-packaged food or snacks.
  ■ If consuming food on-site becomes necessary, move to the staging area and remove PPE. After eating, wash hands thoroughly and put on new PPE.
	• Brought and consumed only outside the vicinity of the sampling area:
	  ○ Bottled water
	  ○ Hydration drinks (i.e. Gatorade®, Powerade®)

Personal Care Products (PCPs) - for day of sample collection[6]

● Prohibited	■ Allowable	▲Needs Screening[2]
• Any PCPs[6], sunscreen, and insect repellent applied in the sampling area.	PCPs[6], sunscreens, and insect repellents applied in the staging area, away from sampling bottles and equipment followed by thoroughly washing hands: **PCPs[6]:** • Cosmetics, deodorants/antiperspirants, moisturizers, hand creams, and other PCPs[6] **Sunscreens:** • Banana Boat® for Men Triple Defense Continuous Spray Sunscreen SPF 30 • Banana Boat® Sport Performance Coolzone Broad Spectrum SPF 30 • Banana Boat® Sport Performance Sunscreen Lotion Broad Spectrum SPF 30 • Banana Boat® Sport Performance Sunscreen Stick SPF 50 • Coppertone® Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50 • Coppertone® Sport High Performance AccuSpray Sunscreen SPF 30 • Coppertone® Sunscreen Stick Kids SPF 55 • L'Oréal® Silky Sheer Face Lotion 50 • Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 50 • Meijer® Sunscreen Continuous Spray Broad Spectrum SPF 30 • Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50 • Meijer® Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70 • Neutrogena® Beach Defense Water+Sun Barrier Lotion SPF 70 • Neutrogena® Beach Defense Water+Sun Barrier Spray Broad Spectrum SPF 30 • Neutrogena® Pure & Free Baby Sunscreen Broad Spectrum SPF 60+ • Neutrogena® UltraSheer Dry-Touch Sunscreen Broad Spectrum SPF 30 **Insect Repellents:** • OFF® Deep Woods • Sawyer® Permethrin	• Products other than those listed as ■Allowable

[1] This table is not considered to be a complete listing of prohibited or allowable materials. All materials should be evaluated prior to use during sampling. The manufacturers of various products should be contacted in order to determine if PFAS was used in the production of any particular product.

[2] Equipment blank samples should be taken to verify these products are PFAS-free prior to use during sampling.

[3] **For surface water foam samples:** LDPE storage bags may be used in the sampling of foam on surface waters. In this instance, it is allowable for the LDPE bag to come into direct contact with the sample media.

[4] **For fish and other wildlife samples:** Depending on the project objectives, glass jars and aluminum foil might be used for PFAS sampling. PFAS has been found to bind to glass and if the sample is stored in a glass jar, a rinse of the jar is required during the sample analysis. PFAS are sometimes used as a protective layer for some aluminum foils. An equipment blank sample should be collected prior to any aluminum foil use.

[5] Regular ice is recommended as there are concerns that chemical and blue ice may not cool and maintain the sample at or below 42.8°F (6°C) (as determined by EPA 40 CFR 136 – NPDES) during collection and through transit to the laboratory.

[6] Based on evidence, avoidance of PCPs is considered to be precautionary because none have been documented as having cross-contaminated samples due to their use. However, if used, application of PCPs must be done at the staging area and away from sampling bottles and equipment, and hands must be thoroughly washed after the use of any PCPs prior to sampling.

● - Prohibited ■ - Allowable ▲ - Needs Screening