



## Site Investigation Report Update - Groundwater

*Superior Refinery April 26, 2018 Incident*

*BRRTs Number: 02-16-581317*



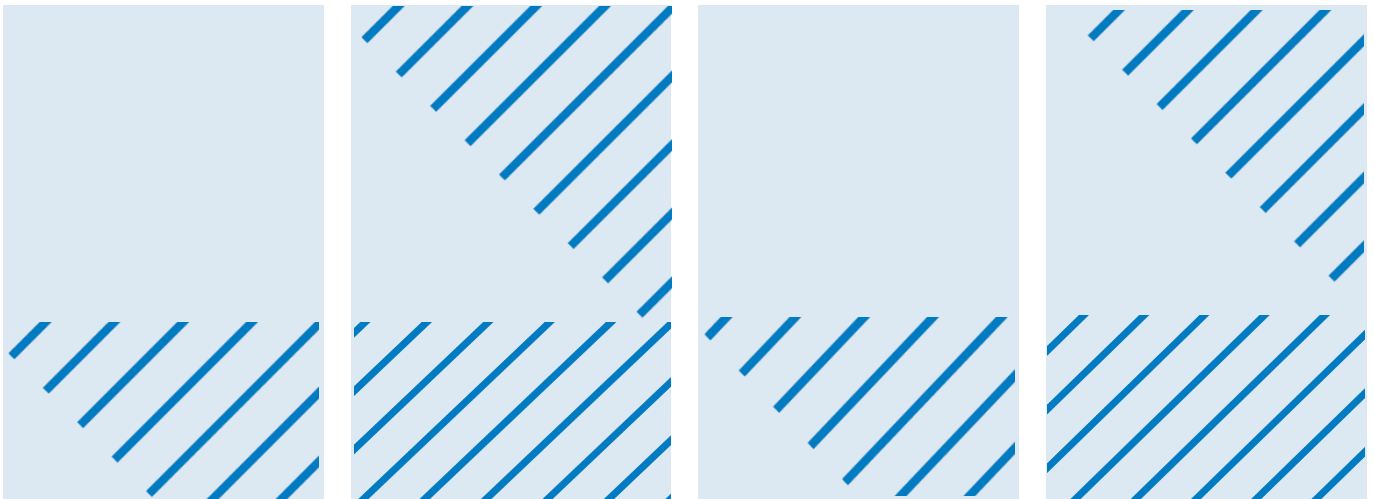
Prepared for  
Superior Refining Company LLC

Prepared by  
Barr Engineering Co.

August 2024

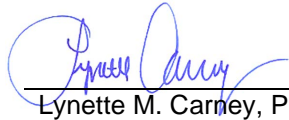
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## Certification

"I, Lynette M. Carney, hereby certify that I am a hydrogeologist as that term is defined in s. NR 712.03(1), Wis. Adm. Code, am registered in accordance with the requirements of ch. GHSS 2, Wis. Adm. Code or licensed in accordance with the requirements of ch. GHSS 3, Wis. Adm. Code; and that, to the best of my knowledge, all of the information contained in this document is correct, and the document was prepared in compliance with all applicable requirements in Chapters NR 700 to 726, Wis. Adm. Code."

A blue ink signature of Lynette M. Carney, written in a cursive style.

Lynette M. Carney, PG  
PG #: 1138

August 23, 2024  
Date



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August 2024

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## Abbreviations

AFFF	Aqueous Film Forming Foam
AIX	anion exchange
ATF	Asphalt Tank Farm
bgs	below ground surface
BRRTS	Bureau of Remediation and Redevelopment Tracking system
CSM	Conceptual Site Model
DC-RCL	Direct Contact Residual Contaminant Level
EPA	U.S. Environmental Protection Agency
ES	Enforcement Standards
FCCU	Fluidized Catalytic Cracking Unit
ft/yr	feet per year
GAC	granular activated carbon
GW-RCL	Groundwater Residual Contaminant Level
IGIWP	Initial Groundwater Investigation Work Plan
IW-1	Incident Well 1
J	qualified
LOD	limit of detection
LOQ	limit of quantitation
MDL	method detection limit
NEtFOSA	n-Ethylperfluorooctanesulfonamide
NEtFOSAA	n-Ethyl perfluorooctanesulfonamidoacetic acid
NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol
ng/l	nanograms per liter
O.D.	outside diameter
PAH	polynuclear aromatic hydrocarbon
PAL	Preventative Action Limits
PFOA	Perfluorooctanoic acid
PFAS	polyfluoroalkyl substances
PFOS	Perfluorooctanesulfonic acid
PFOSA	Perfluorooctanesulfonamide
PFHxS	Perfluorohexanesulfonic acid
PID	photoionization detector
ppm	parts per million
PVOC	petroleum volatile organic compound
QC	quality control
RAP	response action plan
RL	reporting limit
RPD	Relative Percent Difference
SI/RAP	Site Investigation/Remedial Action Plan
SOP	standard operation procedures
SRC	Superior Refining Company LLC
TPT	Twin Ports Testing
USGS	U.S. Geological Survey
VOC	volatile organic compound
WDNR	Wisconsin Department of Natural Resources



WWTP      wastewater treatment plant

## Executive Summary

This report summarizes the results of the hydrocarbon and per- and polyfluoroalkyl substances (PFAS) groundwater investigation completed by Barr Engineering Co. (Barr) on behalf of Superior Refining Company LLC (SRC). The investigation was initiated following a release of asphalt, Therminol®, and #6 fuel oil containing hydrocarbons during the April 26, 2018 explosion and fire (Incident) at the Superior Refinery (Site), which resulted in the use of Aqueous Film Forming Foam (AFFF) containing PFAS for fire suppression. The purpose of this investigation phase was to evaluate the PFAS and hydrocarbon groundwater conditions in the Incident (release source) area.

As outlined in *Appendix G of the Site Investigation Report Update* (IGIWP; Barr, 2021b; Appendix A), a single groundwater monitoring well was installed in the Incident area. As outlined in the IGIWP (Barr, 2021b), the well was installed in what is considered the “worst-case scenario” location for the Incident area (e.g., an area with the highest PFAS concentrations in near surface soil samples). This location was selected as most representative for evaluating the potential for PFAS groundwater impacts related to the Incident.

Groundwater samples were collected from the monitoring well for laboratory analysis of PFAS, petroleum volatile organic compounds (PVOCs), and polynuclear aromatic hydrocarbons (PAHs).

Results from two rounds of sampling identified concentrations of certain PFAS compounds above the Recommended Wisconsin Preventative Action Limits (PALs), but concentrations of all PFAS compounds were below the Recommended Wisconsin Enforcement Standards (ES). Results showed PVOc and PAH compound concentrations in the groundwater were below laboratory detection limits.

Based on the lack of soil-to-groundwater pathway exceedances in soil samples collected from the “worst case scenario” Incident area, and since PFAS detections in groundwater from the Incident (release source) area were below Recommended WI ES, there does not appear to be risk to the groundwater pathway from this release. In addition, due to the low permeability clay layer underlying the entire facility, groundwater flow is largely negligible across the facility and therefore the migration of PFAS and/or hydrocarbons from the facility to any potential off-site or downgradient receptors through groundwater is not considered a primary pathway of concern. Regardless, SRC will begin the next phase of the NR 700 process and prepare a response action plan (RAP) that will move the current stormwater treatment system from an interim action to a final remedy. Accordingly, SRC believes the Incident-related soil and groundwater investigation is complete.

# 1 Project Objectives

This *Site Investigation Report Update – Groundwater* builds upon the work completed and data collected in the initial and supplemental hydrocarbon and PFAS soil investigations at the Site, which indicated that PFOS, PFOA, and PFBS were not detected at the Site above the Wisconsin Default Soil to Groundwater Residual Contaminant Level (GW-RCL).

Specifically, this phase of the groundwater investigation was conducted at the request of Wisconsin Department of Natural Resources (WDNR) as set forth in WDNR's April 28, 2023 response to the *Site Investigation Report Update – Supplement A* (Barr, 2023). WDNR requested the Site determine whether there may be a residual impact to groundwater associated with PFAS compounds in the Incident area soils, even though PFOS, PFOA, or PFBS were not detected above the GW-RCL during the soil investigation.

SRC obtained groundwater samples for all PFAS compounds for which Wisconsin has set Recommended Wisconsin Enforcement Standards (ES) (Table 1). All results from this phase of the investigation were below these recommended standards.

As outlined in *Appendix G of the Site Investigation Report Update*; (Barr, 2021b; Appendix A) this groundwater investigation included the following objectives:

- Assess the condition of groundwater that may be in contact with hydrocarbon and PFAS-impacted soil beneath pervious surfaces within Incident impacted areas;
- Determine the need for additional investigation, interim action(s) and/or remedial action(s); and
- Collect/assess any information necessary to select an interim and/or recommended remedial action.

This report summarizes the groundwater investigation activities, results, and conclusions, provides relevant updates to the ongoing interim actions, and recommendations for next steps.

## 2 General Information

### Figures:

- Figure 1 provides a location map showing the Superior Refinery and the surrounding area using the U.S. Geological Survey (USGS) 7.5-minute topographic map (NR 716.09(2) (c) 5).
- Figure 2A provides an aerial image of the facility and the restricted access (fence line) boundaries in relation to the surrounding features along with area private water supply wells located within 1,200 feet of the facility boundary (required by NR 716.15 (2)(c) 6.).
- Figure 2B provides an aerial image of SRC owned property in the vicinity of the refinery.
- Figure 3A and Figure 3B provide Site features and refining operational process area details (required by NR 716.15(2) (c)6.).
- Figure 4 provides the existing monitoring well network and estimated groundwater flow established for the facility wide Bureau of Remediation and Redevelopment Tracking system (BRRTS) site 16-16-559511.

### Site Information:

BRRTs Number: 02-16-581317  
Facility Identification Number: 816009590  
Superior Refining Company LLC  
2407 Stinson Avenue  
Superior, Wisconsin  
Douglas County, Wisconsin  
NW ¼, NW ¼ of Section 36, T49N, R14W  
Latitude / Longitude: 46.690927 / 92.07179 (Site Center)  
WTM91 Coordinates: X: 361511, Y: 692726 (Site Center)

### Responsible Party:

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## 3 Background

A description of the Incident, along with the immediate and interim actions and previous investigation phases was provided in the *PFAS Soil Site Investigation Work Plan* (Barr, 2020b) and the *Interim Action Report – Hydrocarbon and AFFF* (Barr, 2021a). A summary of the Incident and immediate and interim actions is provided below.

### 3.1 Summary of April 2018 Incident

On April 26, 2018, an Incident occurred at the Superior Refinery while conducting a process shut down for a refinery-wide maintenance turnaround. Debris from the initial Incident punctured asphalt storage Tank 101, resulting in a release of asphalt that later ignited. During the Incident, fire also caused damage to process piping that contained Therminol® and #6 fuel oil in the Asphalt Tank Farm (ATF). The fire was later extinguished on the same day using a combination of water and AFFF which contained PFAS compounds as part of the manufacturer's formulation. The estimated extent of the release to pervious surfaces has been identified as the "Incident impacted area" outlined in red on Figure 5. Some of the water used for firefighting efforts ponded and pooled in the Tank 86 and 87 containment area (which is the target for the source well installation) and flowed overland to the north ditch along Stinson Avenue, carrying with it dilute amounts of hydrocarbons and firefighting foam chemicals.

### 3.2 Summary of Immediate and Interim Actions

In response to the Incident, immediate and interim actions were initiated. Immediate actions occurred during and immediately after the firefighting efforts to minimize the impacts of releases to the environment. The immediate and interim actions were implemented as soon as areas and infrastructure were deemed to be safe, accessible and authorization was granted by governmental oversight entities. Immediate actions included: extinguishing of the fire, securing the facility, controlling off-site migration of firewater, environmental monitoring and free product removal (Barr, 2021a).

Recovery of the asphalt and treatment of PFAS impacted firefighting and surface water was initiated shortly after the Incident. Recovery of liquid hydrocarbons was addressed with the immediate actions described above, except for what was mixed or embedded with the asphalt. The removal of accessible asphalt on the ground surface within the asphalt tank farm and process units, along with asphalt-coated equipment and materials began in May 2018 and was completed in March 2019. Stormwater from the Incident-impacted areas at the facility continues to be routed to the on-site WWTP and treated by a granular activated carbon (GAC) and anion exchange (AIX) resin treatment system for PFAS removal.

In addition, several large excavations within the Incident impacted area were completed to remove potentially contaminated soil and accommodate the removal and replacement of damaged infrastructure. These areas included large portions of the Fluidized Catalytic Cracking Unit (FCCU), Crude Unit and the ATF areas as shown in the *Site Investigation Report Update – Hydrocarbon and PFAS* (Barr, 2021b). The Incident clean-up and subsequent facility rebuild efforts have also included significant removal and disposal of refining equipment and associated insulation, electrical infrastructure, concrete cover, and equipment foundations from both inside the Incident-damaged process units and in the ATF.

### 3.3 Summary of Previous Investigation Phases

In the September 18, 2018 Responsible Party letter to SRC, the WDNR requested the completion of a site investigation in accordance with NR 700 (WDNR, 2018). Following receipt of this request from the

WDNR, SRC prepared a *Site Investigation Work Plan* (Barr, 2019) and conducted an initial phase of investigation focusing on hydrocarbons in soil in the fall of 2019.

Following completion of the initial hydrocarbon soil investigation, SRC prepared a *Site Investigation Report* (Barr, 2020a) followed by an initial *PFAS Soil Site Investigation Work Plan* (Barr, 2020b). The PFAS soil site investigation was conducted in summer and fall of 2020. SRC prepared a *Site Investigation Report Update – Hydrocarbon and PFAS* in February 2021 (Barr, 2021b). The WDNR requested additional PFAS soil investigation in their letter response to the *Site Investigation Report Update* dated April 22, 2021 (WDNR, 2021a). The *PFAS Site Investigation Work Plan – Supplement A* (Barr, 2021c) scope of work was approved by WDNR in their letter dated October 18, 2021 (WDNR, 2021b).

The investigation focused on characterizing soil conditions in pervious surface areas located within the release area boundaries, as shown on Figure 5. The estimated extent of the petroleum hydrocarbon release to pervious surfaces included portions of the ATF, refinery process areas, facility access roadways and the northern Stinson Avenue ditch (Figure 5). Low-level concentrations of petroleum compounds were detected in soil samples from three locations in the affected area with concentrations greater than the WDNR GW-RCLs. There were no detections of petroleum compounds greater than the Wisconsin Industrial Direct Contact Residual Contaminant Levels (DC-RCLs). Concentrations of PFAS compounds were detected in shallow soil samples at each location sampled but were all below the DC-RCLs and GW-RCLs.

The results were summarized in the *Site Investigation Report Update – Supplement A* (Barr, 2023). The WDNR responded to SRC with comments related to the *Site Investigation Report Update – Supplement A* in a letter dated April 28, 2023 (WDNR, 2023). As indicated above, WDNR agreed that for direct contact in an industrial setting, PFAS contamination was defined (WDNR, 2023). However, WDNR indicated that “PFAS contamination remaining in soil may be a concern for groundwater quality. The DNR will make a determination on definition of soil impacts that may result in the exceedance of the Wisconsin Department of Health’s recommended groundwater standards following the groundwater investigation.” Based on these comments, the groundwater investigation in the Incident area was initiated, and the analytical results thereof are presented in this report.



## 4 Investigation Methods

In February 2021, SRC submitted the *Initial Groundwater Investigation Work Plan* (IGIWP) as *Appendix G of the Site Investigation Report Update* (Barr, 2021a) to WDNR, which included rationale for well placement, a sampling and analysis plan, standard operation procedures (SOPs), and additional investigation details. The IGIWP was approved by the WDNR on April 22, 2021 (WDNR, 2021a). Implementation of the work was delayed until 2023 due to refinery re-build efforts in the investigation area.

### 4.1 Well Installation

One monitoring well (Incident Well 1 (IW-1)) was installed on November 10, 2023 in the “worst-case scenario” Incident (release source) area to evaluate groundwater for potential hydrocarbon and PFAS contamination. The well was installed within the containment berm for Tanks 86 and 87 (Figure 5 and Figure 6), an area with the highest concentrations of hydrocarbons and PFAS in the near surface soil samples (Barr, 2020a and Barr, 2021b).

IW-1 was installed by Twin Ports Testing (TPT) in native material (clay) to a depth of 14.5 feet below ground surface (bgs) using hollow stem auger. The well was constructed using 2-inch outside diameter (O.D.) schedule 40-PVC well screen and riser. The filter pack, annular space seal, and the surface completion were constructed in accordance with WDNR NR 141. A well construction log is provided in Appendix B.

Drilling equipment and well materials were decontaminated, and non- PFAS containing materials (e.g., Teflon™ containing materials) were used. Two rinsate blanks were collected from the drilling decontamination water and well construction materials prior to well installation and analyzed for PFAS compounds, some PFAS compounds were detected but below the Wisconsin ES and PAL.

The borehole was logged and documented by a Barr field geologist on the WDNR *Soil Boring Log Form* 4400-122 and well construction details are documented on the WDNR *Monitoring Well Construction Form* 4400-113A (Appendix B). During drilling, soil samples were collected for volatile organic compound (VOC) headspace screening and classification using dual tube sampling methods.

### 4.2 Well Development

On March 12, 2024, IW-1 was developed by surging the well screen interval with a bailer and pumping to remove accumulated fine sediment from within the filter pack and to establish hydraulic connection with the surrounding groundwater. This field activity was performed in conformance with Barr's SOP as detailed in the IGIWP and the WDNR *Groundwater Sampling Desk Reference and Field Manual* (WDNR, 1996a and WDNR, 1996b) and documented on the WDNR *Well Development Form* (Form 4400-113B; Appendix B).

### 4.3 Groundwater Sample Collection and Analysis

Following IW-1 development on March 12, 2024, the well was purged on March 19, 2024 (7 days later). After the purging, due to the anticipated slow recovery, the well was allowed to recover for 8 more days prior to collecting a groundwater sample on March 27, 2024 for analysis of PVOC by Method EPA 8260D, PAH by Method EPA 8270E SIM, and PFAS by Method WI SPE (LCMSMS per WDNR Document EA-19-0001). For verification purposes, a second purge and sample event (2 weekly purge events followed by

sample collection two weeks later) began on April 9, 2024. The second verification sample was collected on April 24, 2024. This approach resulted in the collection of a total of two groundwater samples approximately 30 days apart. Sample collection, special measures for collection of PFAS samples, chain-of custody documentation, and transport of samples followed applicable Barr SOPs (provided in the IGIWP).

Appropriate sample handling and documentation procedures, as described in Barr's SOP (provided in the IGIWP) and the *WDNR Groundwater Sampling Desk Reference and Field Manual* (WDNR, 1996a and WDNR 1996b), were followed.

## 4.4 Hydrocarbon Soil Samples

A photoionization detector (PID) with a 10.6 eV lamp was used to perform soil headspace screening. The PID was calibrated or checked against a known concentration of a calibration gas standard prior to collection of field measurements. Field screening notations and measurements were recorded in field logs.

Consistent with previous well installations at the Site, if potential hydrocarbon impacts (PID reading, staining, odor and/or sheen) are observed in boring soils, a soil sample is collected for laboratory analysis. Residual (semi-solid) asphalt was present at 1.5 feet bgs and black stained soil was observed below the asphalt from 1.5 to 2 feet bgs in the IW-1 boring. Soil samples were collected for analysis of PVOCs by Method EPA 8260D and PAHs by Method EPA 8270E SIM. Laboratory analyses were performed by Pace Analytical in Minneapolis, Minnesota (Pace). Appropriate sample handling, documentation, and quality control procedures, as described in the *Site Investigation Work Plan* (Barr, 2019) were followed.

## 4.5 Deviations

Deviations from the IGIWP were discussed and resolved with SRC personnel. Adjustments to the original scope consisted of the items listed below per NR716.15 (2) I.

- During well installation, the 2-3 foot excavation in which the well was to be installed brought concerns about the potential for surface water collecting in the excavation backfill, coupled with an already limited surface seal due to the shallow water table, causing a risk of surface water infiltration into the well. It was decided to abandon the surface excavation approach and drill the well through the existing surface material.
- Initial water levels were measured in IW-1 in January 2024 following a significant and rare late December 2023 rainfall event (a short period after installation). The monitoring well was purged and recovery monitored several times to evaluate the aquifer response and to determine if the water in the well may have been the result of potential surface water infiltration. After several rounds of well purging and aquifer testing, it was determined that the recharge rate was consistent and repeatable and therefore representative of groundwater inflow. The time required for complete well recovery was estimated to be 15 to 30 days, consistent with other shallow wells at the Site.

## 5 Results

Investigation activities for this phase occurred between November 10, 2023 and April 24, 2024. A soil boring log, well installation log, and well development form is provided in Appendix B. Representative photographs of the well installation, development, and sampling are included as Appendix C.

### 5.1 Geology and Hydrogeology

Stratigraphy at the IW-1 location consisted of 1.5 feet of silty sand with gravel and gravelly clay (fill) overlaying native fat clay. The native clay was of high plasticity, firm consistency, moist, reddish brown in color and of glaciolacustrine origin. Soft asphalt chunks were identified at 1.5 feet bgs and native clay with black staining was identified directly below from 1.5 to 2.0 feet bgs. The depth to groundwater ranges from 1 to 5 feet bgs at the site, with an average depth to groundwater of approximately 3 feet bgs. However, because of the shallow depth of the low permeability clay formation, groundwater did not readily enter the borehole during drilling.

### 5.2 PFAS Groundwater Analytical Summary

Groundwater samples were collected from IW-1 during two separate sampling events 30 days apart. PFAS sample results were compared to the Recommended Wisconsin ES, Recommended Wisconsin PAL. Analytical results of PFAS in the groundwater are summarized in Table 1. Copies of groundwater sample laboratory analytical reports are provided in Appendix D.

#### 5.2.1 Sampling Event 1

PFAS detections in groundwater from the first sampling event did not exceed the Recommended Wisconsin ES. Concentrations of PFHxS exceeded the Recommended Wisconsin PAL of 4 nanograms per liter (ng/l) with a concentration of 4.2 ng/l. Concentrations of the combined criteria for the sum of PFOS, PFOA, PFOSA, NEtFOSE, NEtFOSA, and NEtFOSAA exceeded the Recommended Wisconsin PAL of 2 ng/l with a summed concentration of 4.3 ng/l.

#### 5.2.2 Sampling Event 2

PFAS detections in groundwater from the second sampling event did not exceed the Recommended Wisconsin ES. Concentrations of PHFxs exceeded the Recommended Wisconsin PAL of 4 ng/l with a concentration of 4.8 ng/l. Concentrations of the combined criteria of the sum of PFOS, PFOA, FOSA, NEtFOSE, NEtFOSA, and NEtFOSAA exceeded the Recommended Wisconsin PAL of 2 ng/l with a summed concentration of 4.2 ng/l.

### 5.3 Hydrocarbon Groundwater Analytical Summary

Groundwater samples were collected from IW-1 during two sampling events approximately 30 days apart. Hydrocarbon sample results were compared to Wisconsin NR 140 ES and PAL criteria. Analytical results of hydrocarbons in the groundwater are summarized in Table 2. Copies of laboratory analytical reports are provided in Appendix D. No PVOC or PAH analytes were detected above laboratory reporting limits during either sampling event.

## 5.4 Hydrocarbon Soil Field Screening

During well installation, soil was continuously screened for signs of contamination (odor, discoloration, sheen, and PID headspace measurements). Observations and field headspace screening results are summarized on the boring log in Appendix B.

Residual (semi-solid) asphalt from the Incident appeared to be present at 1.5 feet bgs and black stained clay was observed below the asphalt from 1.5 to 2 feet bgs. No odor or sheen was present and the PID headspace was 0.0 parts per million (ppm) in this interval. The highest headspace reading from the boring was 1.5 ppm from 14 to 15 feet bgs which was likely due to moisture interference.

## 5.5 Hydrocarbon Soil Analytical Summary

Consistent with prior phases of work, the hydrocarbon soil sampling was limited to the intervals where suspected contamination was observed. This resulted in one sample from the IW-1 boring location from 1.5 to 2 feet bgs as described above. There were low-level detections of PAH compounds from this interval, but no concentrations exceeded WDNR DC-RCLs or WDNR GW-RCLs. There were no detections of PVOC compounds above laboratory detection limits. Table 3 summarizes the hydrocarbon soil sampling results compared to criteria. Copies of laboratory analytical reports for the hydrocarbon soil sample collection are included in Appendix D.

## 6 Quality Control (QC) and Investigation Procedures

Barr completed a quality control (QC) review of data collected as part of this investigation using QC procedures described in the IGIWP (Barr, 2021b). The review was conducted to assess the validity of the analytical results from Pace Analytical (Pace) reports 10676081, 10687707, and 10690769 and Merit reports S60291 and S61354. The review was performed in accordance with Barr's SOPs for data evaluation. Both field sampling and laboratory analytical procedures were examined in the review. Barr-defined qualifiers, based on EPA-defined qualifiers, were assigned in the data summary tables for this project during the evaluation process. The sample data were reported to the limit of detection (LOD)/method detection limit (MDL) and detections between the LOD/MDL and limit of quantitation (LOQ)/reporting limit (RL) were qualified (J) as estimated results.

### 6.1 Field Sampling Procedures

Three PVOC trip blanks, two PFAS field blanks, and six PFAS rinsate blanks were collected as part of the sampling events. The PVOC trip blank was analyzed to determine the extent of potential PVOC contamination introduced during sample transport and handling. No PVOC concentrations were detected above the LOD/MDL in the trip blank samples. Rinsate blank samples were collected to monitor potential PFAS contamination from equipment materials, decontamination procedures, sampling activities, sample transport, and sample storage. Field blank samples were collected to monitor potential PFAS contamination from sampling activities, sample transport, and sample storage. No PFAS target compounds were detected above the LOD/MDL in the rinsate or field blank samples or had significant detections that would impact final data use (Table 4).

Two of the rinsate blanks were samples of analyte-free water that were collected from the rinsing of sampling equipment prior to use at the project site to evaluate whether the material would introduce PFAS to the samples being collected. Four rinsate blanks were collected from the water used for decontamination of sampling equipment (Decontamination Water) and the two different liners used for soil sampling (Liner and CAB Liner). Some PFAS were detected between the LOD/MDL and LOQ/RL; however, no data were qualified for the rinsate blank detections.

No PVOC concentrations were detected above the LOD/MDL in the trip blanks. The field blanks were collected to monitor potential contamination from sampling activities, sample transport, and storage. Target compounds were not detected above the LOD/MDL or did not impact sample data except for an ADONA detection between the LOD/MDL and LOQ/RL in the field blank collected on April 24, 2024. Since the ADONA concentration for the associated IW-1 sample was less than the LOD/MDL, there was no data impact. The ADONA concentration in the associated field duplicate (source sample IW-1) was equal to the field blank concentration and was considered a potential false positive. The sample concentration was presented as non-detect and qualified "UB" in the data summary tables. Sample concentrations greater than five times the blank detection were not qualified.

Field (masked) duplicate sample results measure the reproducibility of measurements under a given set of conditions and were evaluated by calculating the Relative Percent Difference (RPD) values. Duplicate sample results were evaluated by calculating the RPD for compounds where both the native and field duplicate sample concentrations were greater than five times the reporting limit. The RPD formula is as follows:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where:

RPD	=	relative percent difference
S	=	native sample result
D	=	duplicate sample result

Sample IW-1 collected on March 27, 2024 and April 24, 2024 served as the field duplicate samples. Field duplicate sample results were evaluated by calculating the RPD for compounds where both the native and field duplicate sample concentrations were greater than five times the reporting limit. The RPDs for the field duplicate samples were within the acceptance criteria defined in the IGIWP.

## 6.2 Laboratory Quality Control

Laboratory procedures were evaluated utilizing technical holding times, preservation, method blank samples, accuracy data, precision data, and data package completeness. The technical holding times and preservation were within recommendations for the analyses. The method blank, accuracy data, and precision data met the applicable laboratory acceptance criteria, were not specific to this project and were not addressed or had no significant deviations that would impact final data use, with the following exceptions. Where an internal standard noted a matrix interference, the laboratory qualified the associated sample result 'I' and where the qualifier ion ratio was outside of control limits, the laboratory qualified the associated sample result 'q'. In these cases, the results were already qualified 'J' as being between the LOD/MDL and LOQ/RL and considered estimated so no additional qualifier was applied.

Data completeness was evaluated by comparing the analyses requested with the data packages as received. The samples were reported as specified on the chains of custody.

## 6.3 Data QC Conclusions

The data are deemed acceptable for the purposes of this project with the qualifications assigned during the data evaluation process.

## 6.4 Investigation Derived Waste

Waste generated by this investigation was disposed of in accordance with federal, state, and local regulations and Barr's SOP: *Investigative Derived Waste (provided in the IGIWP)*. Soil cuttings were placed in the on-site contaminated soil containment building (3-Sided Building) prior to proper disposal off-site. Waste water generated during development, purging, and sampling was containerized and disposed of at the wash slab at the facility. Water from the wash slab goes through the facility's water treatment plant where it is treated for PFAS before offsite discharge. Investigation derived waste disposal documentation will be provided with the final site investigation report.

## 7 Discussion and CSM Update

Data collected in the groundwater investigation have been evaluated to develop an understanding of Site conditions after completion of the immediate and interim actions. Hydrocarbons were not detected above criteria in groundwater. PFAS compounds associated with the use of AFFF during the Incident are present in the groundwater above the WI Recommended PAL but below the Recommended WI ES. Additional information regarding the extent of the release and changes to the CSM are provided below.

### 7.1 Groundwater Analytical Results

PFAS and hydrocarbon compounds were not detected in groundwater within the release area above the Recommended Wisconsin ES.

Since IW-1 is located in an area that represents the “worst-case scenario” for potential PFAS and hydrocarbon impacts to groundwater in the Incident (release source) area, this well is considered representative of the “worst case” groundwater conditions at the Site. In addition, given that previous phases of investigation resulted in no exceedances of PFAS in soil above the Wisconsin Default GW-RCLs and the fact that PFAS and hydrocarbon detections in groundwater at IW-1 are below the Wisconsin Recommended ES criteria, the extent of PFAS and hydrocarbon impacts to groundwater from this Incident (release source) appear to be sufficiently delineated for the purposes of risk evaluation.

### 7.2 Conceptual Site Model Summary and Update

A preliminary Conceptual Site Model (CSM) was developed as part of the *PFAS Soil Site Investigation Work Plan* (Barr, 2020b) as a guide to help focus investigation activities in the area affected by the Incident and to ensure efficient and effective data collection in support of the scientific and engineering basis for investigation decision-making.

The Site is situated in a relatively flat area and is surrounded by both developed and undeveloped land. Surficial geology consists of a glacio-lacustrine lean-to-fat clay layer that extends to at least 100 feet bgs. The Incident area includes the glacio-lacustrine clay layer with ground surface cover consisting of vegetation, gravel, asphalt pavement, and concrete. Groundwater in the vicinity of the facility is present at an average depth of approximately 3 ft with an estimated velocity of approximately 0.013 feet per year (ft/yr) (Gannett Fleming, 2014). Groundwater elevation contours developed from the existing monitoring well network is shown on Figure 5. Stormwater from Incident-impacted areas and process area wastewater is currently treated through the Refinery wastewater treatment plant (WWTP) and GAC/AIX systems prior to discharge off site. The treated water was previously discharged to Newton Creek but as of July 2021 is sent to the City of Superior POTW.

During the Incident, hydrocarbons and firefighting water containing AFFF were released to areas with pervious surfaces. The firefighting water acted as a transport mechanism for hydrocarbons and PFAS compounds as firefighting water followed the existing stormwater drainage features at the facility. Surface water samples collected following the Incident show the presence of hydrocarbons and PFAS in the on-site water retention ponds.

The results of this groundwater investigation support the preliminary CSM as it relates to soil and groundwater conditions, and no new model features, transport mechanisms, or exposure pathways have been identified or incorporated as a result of this groundwater investigation. A graphic CSM model is provided as Figure 7.



### 7.2.1 Migration Pathways

The potential migration pathways for hydrocarbon and PFAS compounds in soil and groundwater are determined by the properties of the released compound and the characteristics of the transport media. Because of the relatively thick and impermeable surficial clay soils at the refinery, releases tend to migrate horizontally along the ground surface. As stated in the *Site Investigation Work Plan* (Barr, 2020b) and outlined in the Facility-wide *Site Investigation/Remedial Action Plan* (SI/RAP) for this facility (Gannett Fleming, 2014) some vertical migration is possible in the surficial air-filled desiccation fractures within the clay. However, once a compound reaches the saturated conditions at the shallow groundwater table, it is not expected to penetrate the unfractured clay because of the high entry pressure (Bradbury et al., 1985). As a result, lateral subsurface migration of released compounds is not considered a significant transport pathway.

The low permeability of the clay at the Site significantly impedes the potential hydrocarbon vapor migration of compounds in the unsaturated zone. Additionally, the refinery has internal controls in place that further minimize potential direct contact exposure to impacted soil and groundwater. The refinery is surrounded by a 24-hour per day, 7-day per week security system that includes controlled access perimeter fencing, video surveillance system, and security guards. These safeguards prevent the public from accessing the facility. The refinery also has an internal safe work permit program that requires any employees or contractors to obtain a work permit prior to working in any refinery area. This permit system includes a separate work instruction for soil excavation projects and defines the minimum project requirements, safe work practices, and control measures that are to be utilized for all trenching, excavation, or other soil disturbance activities at the refinery.

According to information summarized in the Facility-wide SI/RAP, the hydrocarbon soil vapor exposure pathway has not been evaluated at any of the previously closed or currently active petroleum release locations. This decision was approved by the WDNR since these releases are located within, or adjacent to process areas, equipment or other infrastructure that are not designed for human occupancy. No structures designed for human occupancy are present within 30 feet of known areas of petroleum-contaminated soil or groundwater (Gannett Fleming, 2014).

### 7.2.2 Receptor Risk Evaluation

Potential direct contact risk receptors within the refinery are limited to workers. Human exposure risk through direct or indirect contact with soil, groundwater, or vapor is low as concentrations of hydrocarbon and PFAS compounds in surficial soil samples do not exceed the direct contact screening criteria.

As outlined in the *Site Investigation Report Update – Supplement A* (Barr, 2023) PFAS concentrations in soil were not detected above the Wisconsin Default GW-RCLs, which would suggest PFAS is not likely to leach from soil to groundwater. Based on the lack of soil-to-groundwater pathway exceedances in soil samples collected from the Incident area, and since PFAS detections in groundwater from the Incident (release source) area were below Recommended WI ES, there does not appear to be risk to the groundwater pathway from this release. In addition, due to the low permeability clay layer underlying the entire facility, groundwater flow is largely negligible across the facility and therefore the migration of PFAS and/or hydrocarbons from the facility to any potential off-site or downgradient receptors through groundwater is not considered a primary pathway of concern.



## 8 Conclusions and Recommendations

The following conclusions were developed based on the results and discussion presented in this investigation report:

### 8.1 Soil and Groundwater Investigation

- No hydrocarbon or PFAS groundwater detections exceeded the Recommended Wisconsin ES. Since the Incident (release source) well is located in an area that represents the “worst-case scenario” for potential impacts to groundwater, and due to the lack of hydrocarbon and PFAS detections in groundwater above recommended criteria, coupled with the presence of a low permeability clay layer underlying the entire facility, no further groundwater investigation is recommended.
- The WDNR previously agreed the extent of PFAS contamination greater than the Wisconsin direct contact RCLs has been defined (WDNR, 2023), pending confirmation that a soil-to-groundwater pathway for PFAS was not present, which has been confirmed by this phase of the investigation. Therefore, no additional soil investigation is recommended.

### 8.2 Status of Ongoing Activities

- The hydrocarbon-contaminated areas of soil identified south of the Crude Unit and west of Tank 104 are considered parts of separate historical releases. The need for additional investigation in these areas, and identification of a pathway to site closure, are being evaluated separately from this post-Incident investigation.
- The removal of shallow hydrocarbon (asphalt) surface impacts near Tank 87 began in summer 2023 and are on-going. Documentation of remediation activities will be provided to the WDNR when complete.

### 8.3 Recommendations

Since the soil and groundwater investigation is considered complete, recommendations for the next steps of the NR 700 Incident response include:

- Continuation of the hydrocarbon (asphalt) surface clean up near Tank 87 and preparation of a cleanup documentation report upon completion.
- While soil and groundwater results were below applicable RCLs and Enforcement Standards, SRC will begin the next phase of the NR 700 process and prepare a response action plan (RAP) that will move the current stormwater treatment system from an interim action to a final remedy.

## 9 References

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- WDNR, 2021a. Site Investigation Report Update, Superior Refining Company LLC, BRRTS Number: 02-16-581317. April 22, 2021.
- WDNR, 2021b. PFAS Soil Investigation Work Plan – Supplement A approval, Superior Refining Company LLC, BRRTS Number: 02-16-581317. October 18, 2021.
- WDNR, 2023. Site Investigation Report Update – Supplement A, Superior Refining Company LLC, BRRTS Number: 02-16-581317. April 28, 2023.



## Tables

Table 1  
Groundwater Analytical Results Summary - PFAS  
SRC Post-Incident Site Investigation  
Superior, WI

Location Date Sample Type Data Status			IW-1 3/27/2024		IW-1 4/24/2024	
			N	FD	N	FD
			Validated	Validated	Validated	Validated
Parameter	Recommended WI Enforcement Standard	Recommended WI Preventative Action Limit				
Last Updated	2020	2020				
Exceedance Key	No Exceedances	Bold				
Per- and Polyfluoroalkyl Substances (ng/l)						
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)			< 0.71 U	< 0.70 U	< 0.69 U	< 0.69 U
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS)			< 0.65 U	< 0.64 U	< 0.63 U	< 0.63 U
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS)			0.81 J	0.74 J	0.96 J	0.86 J
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2 FTS)			19	18	31	32
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (N-EtFOSE)	PFAS	PFAS	< 0.71 U	< 0.70 U	< 0.69 U	< 0.69 U
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (N-MeFOSE)			< 0.65 U	< 0.64 U	< 0.63 U	< 0.63 U
4,8-dioxa-3H-perfluorononanoic acid (ADONA)			< 0.33 U	< 0.33 U	< 0.32 U	< 0.34 UB
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)			< 0.39 U	< 0.39 U	< 0.38 U	< 0.38 U
Hexafluoropropylene oxide dimer acid (HFPO-DA)	300	30	< 0.29 U	< 0.29 U	< 0.28 U	< 0.28 U
Methylperfluoro-1-octanesulfonamide (N-MEFOSA)			< 0.55 U	< 0.54 U	< 0.53 U	< 0.54 U
n-Ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	PFAS	PFAS	< 0.59 U	< 0.59 U	< 0.57 U	< 0.58 U
n-Ethylperfluorooctansulfonamide (N-EtFOSA)	PFAS	PFAS	< 0.55 U	< 0.54 U	< 0.53 U	< 0.54 U
n-Methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)			< 0.30 U	< 0.30 U	< 0.29 U	< 0.30 U
Perfluorobutanesulfonic acid (PFBS)	450000	90000	3.8	3.9	3.7	4.1
Perfluorobutanoic acid (PFBA)	10000	2000	32	32	35	35
Perfluorodecanesulfonic acid (PFDS)			< 0.55 U	< 0.54 U	< 0.53 U	< 0.54 U
Perfluorodecanoic acid (PFDA)	300	60	< 0.49 U	< 0.49 U	< 0.48 U	< 0.48 U
Perfluorododecanesulfonic acid (PFDOS)			< 0.55 U	< 0.54 U	< 0.53 U	< 0.54 U
Perfluorododecanoic acid (PFDoA)	500	100	< 0.91 U	< 0.90 U	< 0.88 U	< 0.89 U
Perfluoroheptanesulfonic acid (PFHpS)			< 0.46 U	< 0.46 U	< 0.45 U	< 0.45 U
Perfluoroheptanoic acid (PFHpA)			14	14	15	15
Perfluorohexanesulfonic acid (PFHxS)	40	4	4.2	4.2	4.8	5.2
Perfluorohexanesulfonic acid (PFHxS) - Branched			1.8 J	1.7 J	2.0	2.1
Perfluorohexanesulfonic acid (PFHxS) - Linear			2.7	2.7	3.0	3.4
Perfluorohexanoic acid (PFHxA)	150000	30000	74	74	84	86
Perfluorononanesulfonic acid (PFNS)			< 0.49 U	< 0.49 U	< 0.48 U	< 0.48 U
Perfluorononanoic acid (PFNA)	30	3	0.53 J	0.59 J	0.51 J	0.52 J
Perfluorooctanesulfonamide (PFOSA / FOSA)	PFAS	PFAS	< 0.52 U	< 0.51 U	< 0.50 U	< 0.51 U
Perfluorooctanesulfonic acid (PFOS)	PFAS	PFAS	0.50 J	0.50 J	0.66 J	0.55 J
Perfluorooctanesulfonic acid (PFOS) - Branched			< 0.33 U	< 0.33 U	0.38 J	0.38 J
Perfluorooctanesulfonic acid (PFOS) - Linear			< 0.33 U	< 0.33 U	0.35 J	< 0.32 U
Perfluorooctanoic acid (PFOA)	PFAS	PFAS	3.8	3.8	3.5	3.5
Perfluoropentanesulfonic acid (PFPeS)			1.8	2.0	1.9	2.0
Perfluoropentanoic acid (PFPeA)			200	190	230	230
Perfluorotetradecanoic acid (PFTA)	10000	2000	< 0.73 U	< 0.73 U	< 0.71 U	< 0.72 U
Perfluorotridecanoic acid (PFTrDA)			< 0.62 U	< 0.61 U	< 0.60 U	< 0.61 U
Perfluoroundecanoic acid (PFUnA)	3000	600	< 0.42 U	< 0.41 U	< 0.41 U	< 0.41 U
Sum of PFOS, PFOA, FOSA, NEtFOSE, NEtFOSA, and NEtFOSAA	20	2	4.3	4.3	4.2	4.1

Notes:

N: Normal Sample

FD: Field Duplicate Sample

PFAS: Analyte is included in the criteria sum.

J: Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

U: The analyte was analyzed for, but was not detected.

UB: The analyte was detected in one of the associated laboratory, equipment, field or trip blank samples and is considered non-detect at the concentration reported by the laboratory.

Table 2  
Groundwater Analytical Results Summary - PVOCs and PAHs  
SRC Post-Incident Site Investigation  
Superior, WI

				Location	IW-1		IW-1	
				Date	3/27/2024		4/24/2024	
				Sample Type	N	FD	N	FD
				Data Status	Validated	Validated	Validated	Validated
Parameter	Units	Wisconsin Groundwater Public Health Enforcement Standards	Wisconsin Public Health Preventative Action Limits					
<b>Last Updated</b>		07/01/2023	07/01/2023					
<b>Exceedance Key</b>		No Exceedances	No Exceedances					
Semivolatile Organic Compounds								
1-Methylnaphthalene	ug/l				< 0.0053 U	< 0.0053 U	< 0.0053 U	< 0.0052 U
2-Methylnaphthalene	ug/l				< 0.0072 U	< 0.0072 U	< 0.0072 U	< 0.0071 U
Acenaphthene	ug/l				< 0.0045 U	< 0.0045 U	< 0.0045 U	< 0.0044 U
Acenaphthylene	ug/l				< 0.0041 U	< 0.0041 U	< 0.0041 U	< 0.0040 U
Anthracene	ug/l	3000			< 0.0072 U	< 0.0072 U	< 0.0071 U	< 0.0071 U
Benz(a)anthracene	ug/l				< 0.0045 U	< 0.0045 U	< 0.0045 U	< 0.0044 U
Benzo(a)pyrene	ug/l	0.2	0.02		< 0.0048 U	< 0.0048 U	< 0.0048 U	< 0.0047 U
Benzo(b)fluoranthene	ug/l	0.2	0.02		< 0.0073 U	< 0.0073 U	< 0.0072 U	< 0.0071 U
Benzo(g,h,i)perylene	ug/l				< 0.0098 U	< 0.0098 U	< 0.0097 U	< 0.0096 U
Benzo(k)fluoranthene	ug/l				< 0.0080 U	< 0.0080 U	< 0.0079 U	< 0.0078 U
Chrysene	ug/l	0.2	0.02		< 0.0078 U	< 0.0078 U	< 0.0077 U	< 0.0077 U
Dibenz(a,h)anthracene	ug/l				< 0.010 U	< 0.010 U	< 0.010 U	< 0.010 U
Fluoranthene	ug/l	400	80		< 0.014 U	< 0.014 U	< 0.014 U	< 0.013 U
Fluorene	ug/l	400	80		< 0.0052 U	< 0.0052 U	< 0.0052 U	< 0.0051 U
Indeno(1,2,3-cd)pyrene	ug/l				< 0.0097 U	< 0.0097 U	< 0.0096 U	< 0.0095 U
Naphthalene	ug/l	100	10		< 0.015 U	< 0.015 U	< 0.015 U	< 0.015 U
Phenanthrene	ug/l				< 0.013 U	< 0.013 U	< 0.013 U	< 0.013 U
Pyrene	ug/l	250			< 0.012 U	< 0.012 U	< 0.012 U	< 0.012 U
Volatile Organic Compounds								
1,2,4-Trimethylbenzene	ug/l	480 c	96 c		< 0.13 U	< 0.13 U	< 0.13 U	0.20 J
1,3,5-Trimethylbenzene	ug/l	480 c			< 0.11 U	< 0.11 U	< 0.11 U	< 0.11 U
Benzene	ug/l	5	0.5		< 0.21 U	< 0.21 U	< 0.21 U	< 0.21 U
Ethyl benzene	ug/l	700			< 0.11 U	< 0.11 U	< 0.11 U	0.12 J
Methyl tertiary butyl ether (MTBE)	ug/l	60			< 0.13 U	< 0.13 U	< 0.13 U	< 0.13 U
Toluene	ug/l	800			< 0.21 U	< 0.21 U	< 0.21 U	< 0.21 U
Xylene, total	ug/l	2000 (4)			< 0.42 U	< 0.42 U	< 0.42 U	< 0.42 U

Notes:

N: Normal Sample

FD: Field Duplicate Sample

J: Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

U: The analyte was analyzed for, but was not detected.

c: Value represents the criteria for Trimethylbenzenes (1,2,4- and 1,3,5- combined).

(4): Xylene includes meta-, ortho-, and para-xylene combined.

**Table 3**  
**Soil Analytical Results Summary - PVOCs and PAHs**  
**SRC Post-Incident Site Investigation**  
**Superior, WI**

					Location
					Date
					Depth
					Data Status
					IW-1
					11/10/2023
					1.5 - 2 ft
					Validated
Parameter	Units	Wisconsin Groundwater RCLs, DF=2	Wisconsin Not to Exceed Direct Contact Non-Industrial RCLs	Wisconsin Not to Exceed Direct Contact Industrial RCLs	
<b>Last Updated</b>		12/01/2018	12/01/2018	12/01/2018	
<b>Exceedance Key</b>		No Exceedances	No Exceedances	No Exceedances	
General Parameters					
Moisture	%				24.7
Semivolatile Organic Compounds					
1-Methylnaphthalene	mg/kg		17.6	72.7	0.0126 J
2-Methylnaphthalene	mg/kg		239	3010	0.0164
Acenaphthene	mg/kg		3590	45200	< 0.0027 U
Acenaphthylene	mg/kg				0.0023 J
Anthracene	mg/kg	196.9492	17900	100000	< 0.0012 U
Benz(a)anthracene	mg/kg		1.14	20.8	0.0092 J
Benzo(a)pyrene	mg/kg	0.47	0.115	2.11	0.0095 J
Benzo(b)fluoranthene	mg/kg	0.4781	1.15	21.1	0.0148
Benzo(g,h,i)perylene	mg/kg				0.0122 J
Benzo(k)fluoranthene	mg/kg		11.5	211	0.0054 J
Chrysene	mg/kg	0.1442	115	2110	0.0175
Dibenz(a,h)anthracene	mg/kg		0.115	2.11	< 0.0016 U
Fluoranthene	mg/kg	88.8778	2390	30100	0.0214
Fluorene	mg/kg	14.8299	2390	30100	0.0058 J
Indeno(1,2,3-cd)pyrene	mg/kg		1.15	21.1	0.0086 J
Naphthalene	mg/kg	0.6582	5.52	24.1	0.0119 J
Phenanthrene	mg/kg				0.0217
Pyrene	mg/kg	54.5455	1790	22600	0.0166
Volatile Organic Compounds					
1,2,4-Trimethylbenzene	mg/kg	1.3787 (1)	219	219	< 0.0226 U
1,3,5-Trimethylbenzene	mg/kg	1.3787 (1)	182	182	< 0.0218 U
Benzene	mg/kg	0.0051	1.6	7.07	< 0.0105 U
Ethyl benzene	mg/kg	1.57	8.02	35.4	< 0.0261 U
Methyl tertiary butyl ether (MTBE)	mg/kg	0.027	63.8	282	< 0.0227 U
Toluene	mg/kg	1.1072	818	818	< 0.0181 U
Xylene, total	mg/kg	3.96	260	260	< 0.0442 U

Notes:

N: Normal Sample

FD: Field Duplicate Sample

J: Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

U: The analyte was analyzed for, but was not detected.

(1): Representing the criteria for combined Trimethylbenzenes.

Table 4  
QC Analytical Results Summary - PFAS  
SRC Post-Incident Site Investigation  
Superior, WI

Location Date Sample Type Data Status			RB-DECON 8/01/2023 Rinse Blank Validated	RB-PVC 8/01/2023 Rinse Blank Validated	FB-01 3/27/2024 Field Blank Validated	RB-01 3/27/2024 Rinse Blank Validated	RB-02 3/27/2024 Rinse Blank Validated	FB-01 4/24/2024 Field Blank Validated	RB-01 4/24/2024 Rinse Blank Validated	RB-02 4/24/2024 Rinse Blank Validated
Parameter	Recommended WI Enforcement Standard	Recommended WI Preventative Action Limit								
Last Updated	2020	2020								
Exceedance Key	No Exceedances	No Exceedances								
Per- and Polyfluoroalkyl Substances (ng/l)										
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)			< 0.72 U	< 0.71 U	< 0.69 U	< 0.72 U	< 0.71 U	< 0.69 U	< 0.71 U	< 0.66 U
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS)			< 0.66 U	< 0.65 U	< 0.63 U	< 0.66 U	< 0.65 U	< 0.63 U	< 0.65 U	< 0.61 U
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS)			< 0.34 U	< 0.33 U	< 0.32 U	< 0.34 U	< 0.33 U	< 0.32 U	< 0.33 U	< 0.31 U
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2 FTS)			< 0.50 U	< 0.49 U	< 0.48 U	< 0.50 U	< 0.49 U	< 0.48 U	< 0.49 U	< 0.46 U
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (N-EtFOSE)	PFAS	PFAS	< 0.72 U	< 0.71 U	< 0.69 U	< 0.72 U	< 0.71 U	< 0.69 U	< 0.71 U	< 0.66 U
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (N-MeFOSE)			< 0.66 U	0.69 J	< 0.63 U	< 0.66 U	< 0.65 U	< 0.63 U	< 0.65 U	< 0.61 U
4,8-dioxa-3H-perfluorononanoic acid (ADONA)			0.36 J	0.35 J	0.33 J	< 0.34 U	< 0.33 U	0.34 J	0.35 J	< 0.31 U
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)			< 0.40 U	< 0.39 U	< 0.38 U	< 0.40 U	< 0.39 U	< 0.38 U	< 0.39 U	< 0.36 U
Hexafluoropropylene oxide dimer acid (HFPO-DA)	300	30	< 0.29 U	< 0.29 U	< 0.28 U	< 0.29 U	< 0.29 U	< 0.28 U	< 0.29 U	< 0.27 U
Methylperfluoro-1-octanesulfonamide (N-MEFOSA)			< 0.56 U	< 0.55 U	< 0.54 U	< 0.56 U	< 0.55 U	< 0.54 U	< 0.55 U	< 0.51 U
n-Ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	PFAS	PFAS	< 0.60 U	0.71 J	< 0.58 U	< 0.60 U	< 0.59 U	< 0.58 U	< 0.59 U	< 0.55 U
n-Ethylperfluoroctansulfonamide (N-EtFOSA)	PFAS	PFAS	< 0.56 U	< 0.55 U	< 0.54 U	< 0.56 U	< 0.55 U	< 0.54 U	< 0.55 U	< 0.51 U
n-Methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)			< 0.31 U	< 0.30 U	0.30 J	< 0.31 U	< 0.30 U	< 0.30 U	< 0.30 U	0.39 J
Perfluorobutanesulfonic acid (PFBS)	450000	90000	0.38 J	0.36 J	< 0.17 U	< 0.18 U	< 0.17 U	< 0.17 U	< 0.17 U	0.30 J
Perfluorobutanoic acid (PFBA)	10000	2000	0.75 J	0.73 J	0.36 J	0.39 J	0.39 J	< 0.25 U	< 0.26 U	< 0.24 U
Perfluorodecanesulfonic acid (PFDS)			< 0.56 U	< 0.55 U	< 0.54 U	< 0.56 U	< 0.55 U	< 0.54 U	< 0.55 U	< 0.51 U
Perfluorodecanoic acid (PFDA)	300	60	< 0.50 U	< 0.49 U	< 0.48 U	< 0.50 U	< 0.49 U	< 0.48 U	< 0.49 U	< 0.46 U
Perfluorododecanesulfonic acid (PFDOS)			< 0.56 U	< 0.55 U	< 0.54 U	< 0.56 U	< 0.55 U	< 0.54 U	< 0.55 U	< 0.51 U
Perfluorododecanoic acid (PFDoA)	500	100	< 0.93 U	< 0.91 U	< 0.89 U	< 0.93 U	< 0.91 U	< 0.89 U	< 0.91 U	< 0.85 U
Perfluoroheptanesulfonic acid (PFHpS)			< 0.47 U	< 0.46 U	< 0.45 U	< 0.47 U	< 0.46 U	< 0.45 U	< 0.46 U	< 0.43 U
Perfluoroheptanoic acid (PFHpA)			< 0.41 U	< 0.40 U	< 0.39 U	< 0.41 U	< 0.40 U	< 0.39 U	< 0.40 U	< 0.38 U
Perfluorohexanesulfonic acid (PFHxS)	40	4	< 0.57 U	< 0.56 U	< 0.55 U	< 0.57 U	< 0.56 U	< 0.55 U	< 0.56 U	< 0.53 U
Perfluorohexanesulfonic acid (PFHxS) - Branched			< 0.57 U	< 0.56 U	< 0.55 U	< 0.57 U	< 0.56 U	< 0.55 U	< 0.56 U	< 0.53 U
Perfluorohexanesulfonic acid (PFHxS) - Linear			< 0.57 U	< 0.56 U	< 0.55 U	< 0.57 U	< 0.56 U	< 0.55 U	< 0.56 U	< 0.53 U
Perfluorohexanoic acid (PFHxA)	150000	30000	< 0.34 UB	< 0.36 UB	< 0.23 U	< 0.24 U	< 0.23 U	< 0.23 U	< 0.23 U	0.24 J
Perfluorononanesulfonic acid (PFNS)			< 0.50 U	< 0.49 U	< 0.48 U	< 0.50 U	< 0.49 U	< 0.48 U	< 0.49 U	< 0.46 U
Perfluorononanoic acid (PFNA)	30	3	< 0.38 U	< 0.37 U	< 0.37 U	< 0.38 U	< 0.37 U	< 0.37 U	< 0.37 U	< 0.35 U
Perfluorooctanesulfonamide (PFOSA / FOSA)	PFAS	PFAS	< 0.53 U	< 0.52 U	< 0.51 U	< 0.53 U	< 0.52 U	< 0.51 U	< 0.52 U	< 0.49 U
Perfluorooctanesulfonic acid (PFOS)	PFAS	PFAS	0.54 J	0.36 J	< 0.32 U	< 0.34 U	< 0.33 U	< 0.32 U	< 0.33 U	< 0.31 U
Perfluorooctanesulfonic acid (PFOS) - Branched			< 0.34 U	< 0.33 U	< 0.32 U	< 0.34 U	< 0.33 U	< 0.32 U	< 0.33 U	< 0.31 U
Perfluorooctanesulfonic acid (PFOS) - Linear			< 0.34 U	< 0.33 U	< 0.32 U	< 0.34 U	< 0.33 U	< 0.32 U	< 0.33 U	< 0.31 U
Perfluorooctanoic acid (PFOA)	PFAS	PFAS	< 0.38 U	< 0.37 U	< 0.37 U	< 0.38 U	< 0.37 U	< 0.37 U	< 0.37 U	< 0.35 U
Perfluoropentanesulfonic acid (PFPeS)			< 0.22 U	< 0.22 U	< 0.21 U	< 0.22 U	< 0.22 U	< 0.21 U	< 0.22 U	< 0.20 U
Perfluoropentanoic acid (PFPeA)			0.46 J	< 0.20 U	< 0.20 U	< 0.21 U	< 0.20 U	0.33 J	< 0.20 U	< 0.19 U
Perfluorotetradecanoic acid (PFTA)	10000	2000	< 0.75 U	< 0.73 U	< 0.72 U	< 0.75 U	< 0.73 U	< 0.72 U	< 0.73 U	< 0.69 U
Perfluorotridecanoic acid (PFTrDA)			< 0.63 U	< 0.62 U	< 0.61 U	< 0.63 U	< 0.62 U	< 0.61 U	< 0.62 U	< 0.58 U
Perfluoroundecanoic acid (PFUnA)	3000	600	< 0.43 U	< 0.42 U	< 0.41 U	< 0.43 U	< 0.42 U	< 0.41 U	< 0.42 U	< 0.39 U
Sum of PFOS, PFOA, FOSA, NEtFOSE, NEtFOSA, and NEtFOSAA	20	2	0.54	1.1	ND	ND	ND	ND	ND	ND

Notes:

PFAS: Analyte is included in the criteria sum.

J: Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

U: The analyte was analyzed for, but was not detected.

UB: The analyte was detected in one of the associated laboratory, equipment, field or trip blank samples and is considered non-detect at the concentration reported by the laboratory.

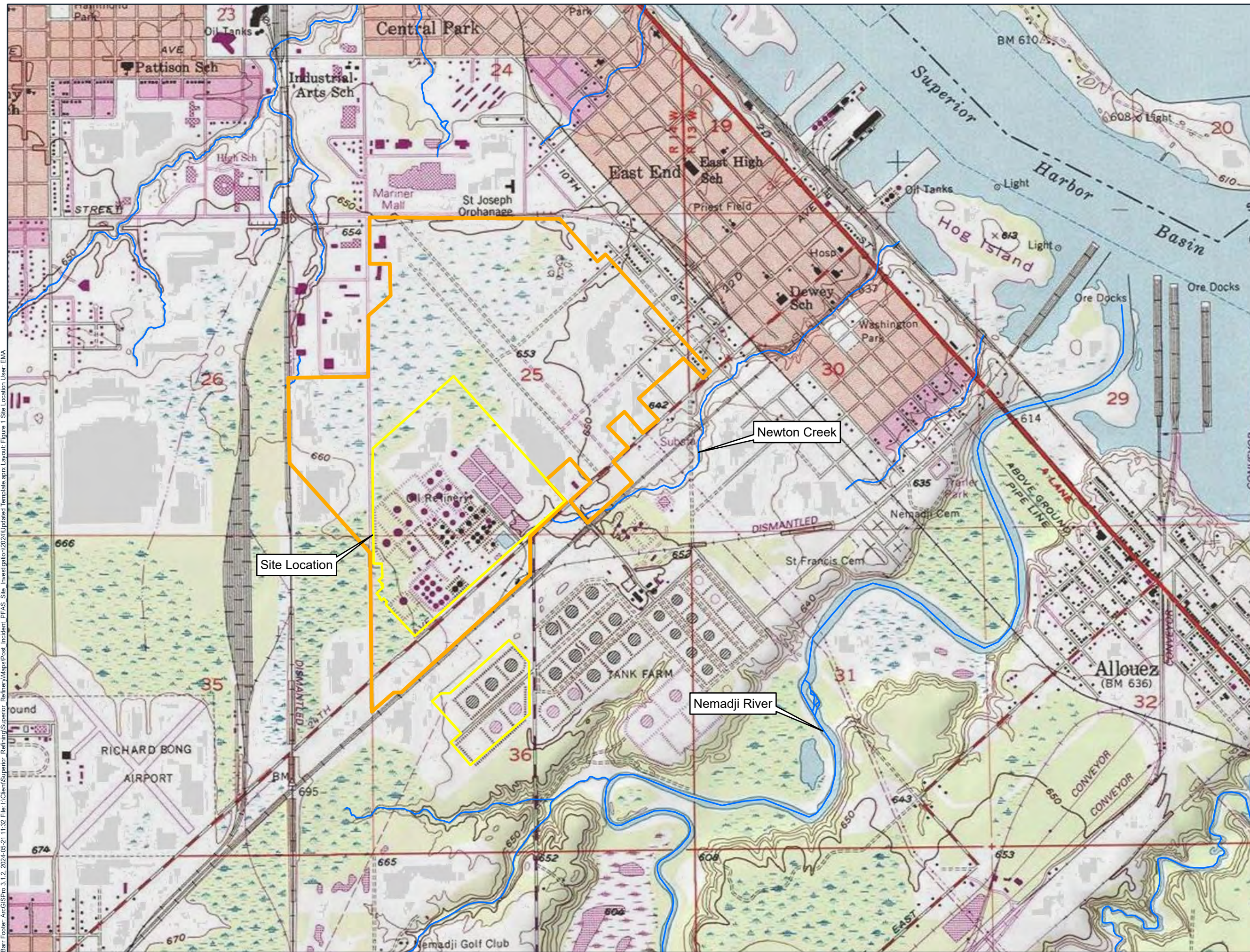
ND: Not detected.



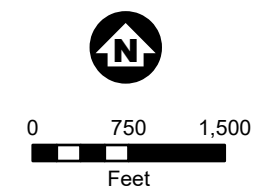
## Figures



Barr Footer ArcGISPro 3.1.2, 2024-05-21 11:32 File: I:\Client\Superior Refining\Superior Refining\Maps\Post Incident PFAS Site Investigation 2024\Updated Template.aprx Layout: Figure 1 Site Location User: EMA



- Approximate SRC Property Boundaries for Contiguous Operations
- Approximate Fenceline Boundaries for Refining-Related Activities



**Site Location**  
Superior Refining  
Company LLC (SRC)  
Superior, WI

FIGURE 1

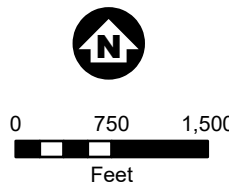




Barr Footer ArcGISPro 3.1.2, 2024-05-21 11:44 File: I:\Client\Superior Refining\Superior Refining\Maps\Post Incident PFAS Site Investigation 2024\Updated Template.aprx Layout: Figure 2A SRC Owned Property User: EWA



- Approximate SRC Property
- Boundaries for Contiguous Operations
- Surficial Water



**SRC Owned Property**  
Superior Refining  
Company LLC (SRC)  
Superior, WI

FIGURE 2A



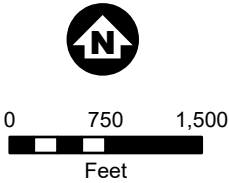


Barr Footer ArcGISPro 3.1.2, 2024-05-21 13:11 File: I:\Client\Superior Refining\Superior Refining\Maps\Post Incident PFAS Site Investigation\2024\Updated Template.aprx Layout: Figure 2B Facility Boundary User: EMA



- Approximate Fenceline  
Boundaries for Refining-  
Related Activities
- Surficial Water
- Private Water Supply  
Well (Historical)
- Private Water Supply  
Well (Post-1989)

Note: Private water supply well  
locations were obtained from  
WDNR (post-1989) and the  
Wisconsin Geological Survey  
database (pre-1989).  
Creek/River data from USGS.



**Facility Boundary**  
Superior Refining  
Company LLC (SRC)  
Superior, WI

FIGURE 2B







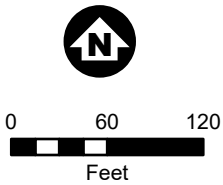


Barr Footer ArcGISPro 3.1.2, 2024-05-21 14:21 File: I:\Client\Superior Refining\Superior Refining\Maps\Post Incident PFAS Site Investigation\2024\Updated Template.aprx Layout: Figure 3B Site Features - Process Area Detail User: EWA



Process Unit Battery Limits

Nearmap Imagery: 5/7/2022



**Site Features**  
**Process Area Detail**  
Superior Refining  
Company LLC (SRC)  
Superior, WI

FIGURE 3B

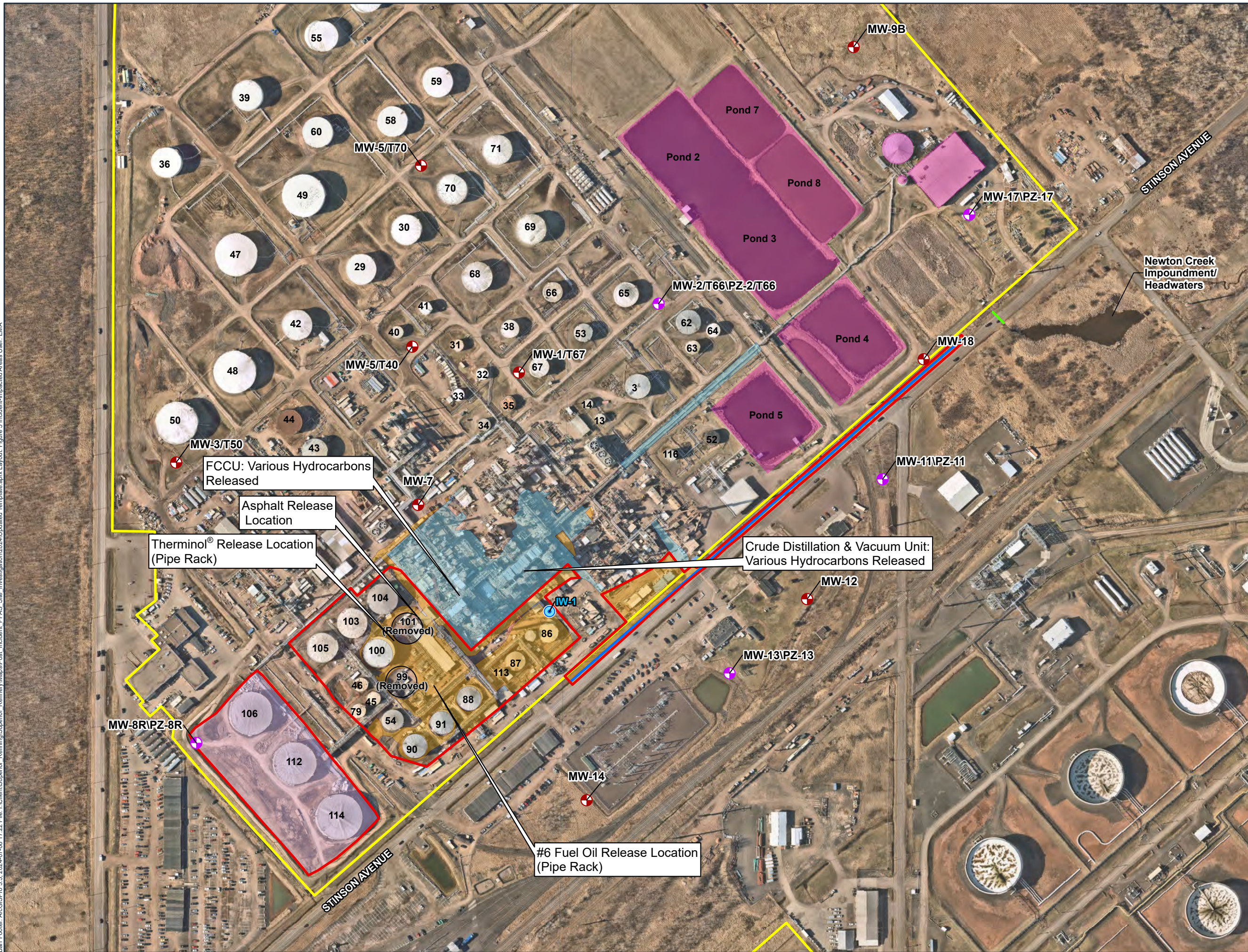








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- Existing Monitoring Well
- Existing Monitoring Well & Piezometer Pair
- Incident Well IW-1
- Stinson Ave Ditch
- Culvert

Date of Incident/Releases-  
April 26, 2018

- Potential Incident Impacted Pervious Surface
- PFAS Impacted Surface
- Water Storage and Treatment System
- Approximate Fenceline Boundaries for Refining-Related Activities
- Incident Impacted Unpaved (Pervious) Surface-Firefighting Water Storage
- Incident Impacted Paved (Impervious) Surface
- Incident Impacted Unpaved (Pervious) Surface

Note: Release extent based on information provided to Barr by SRC.

Nearmap Imagery: 5/7/2022



0 150 300  
Feet

**Incident-Impacted Areas**  
Superior Refining  
Company LLC (SRC)  
Superior, WI

FIGURE 5

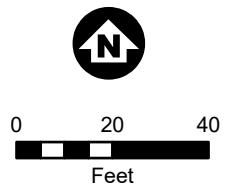




- Incident Well IW-1
- Potential Incident Impacted Pervious Surface

Incident occurred April 26, 2018

Nearmap Imagery: 5/16/2023

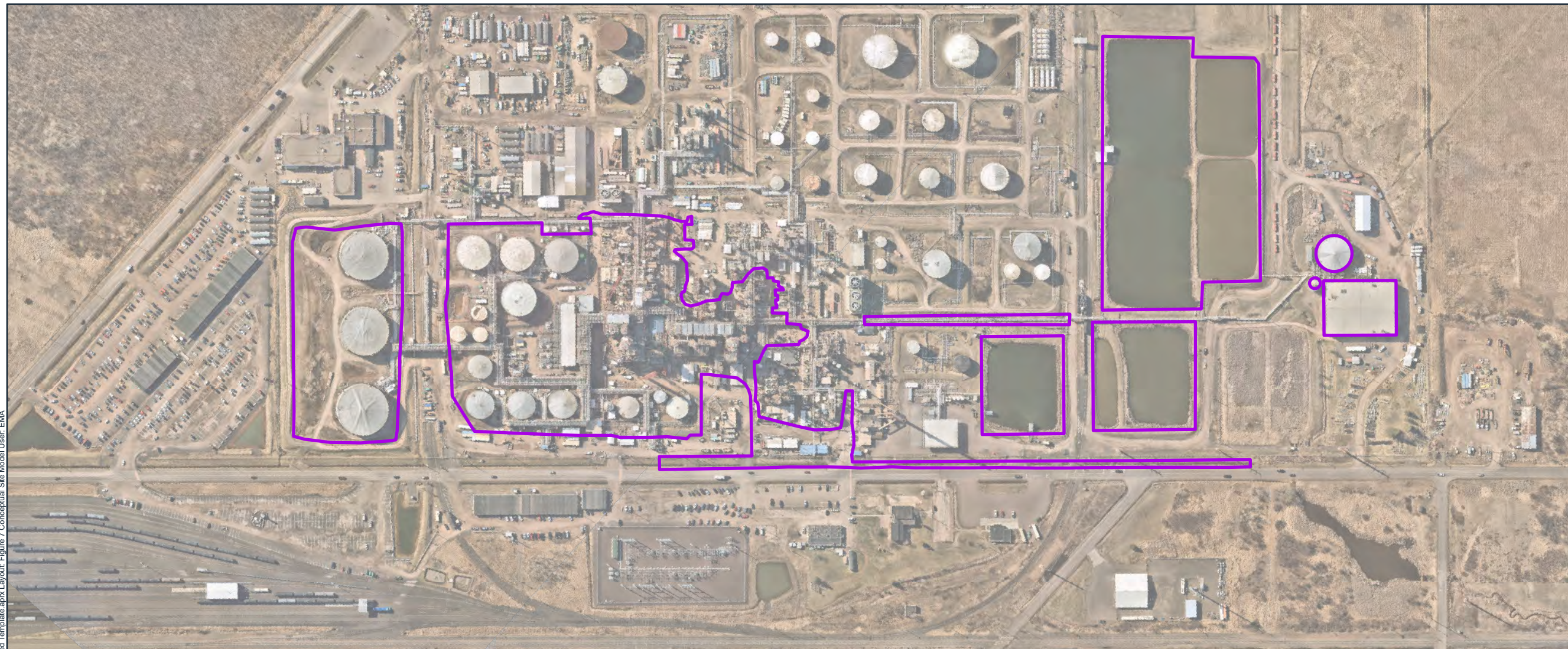


**Incident Well Location**  
Superior Refining  
Company LLC (SRC)  
Superior, WI

FIGURE 6

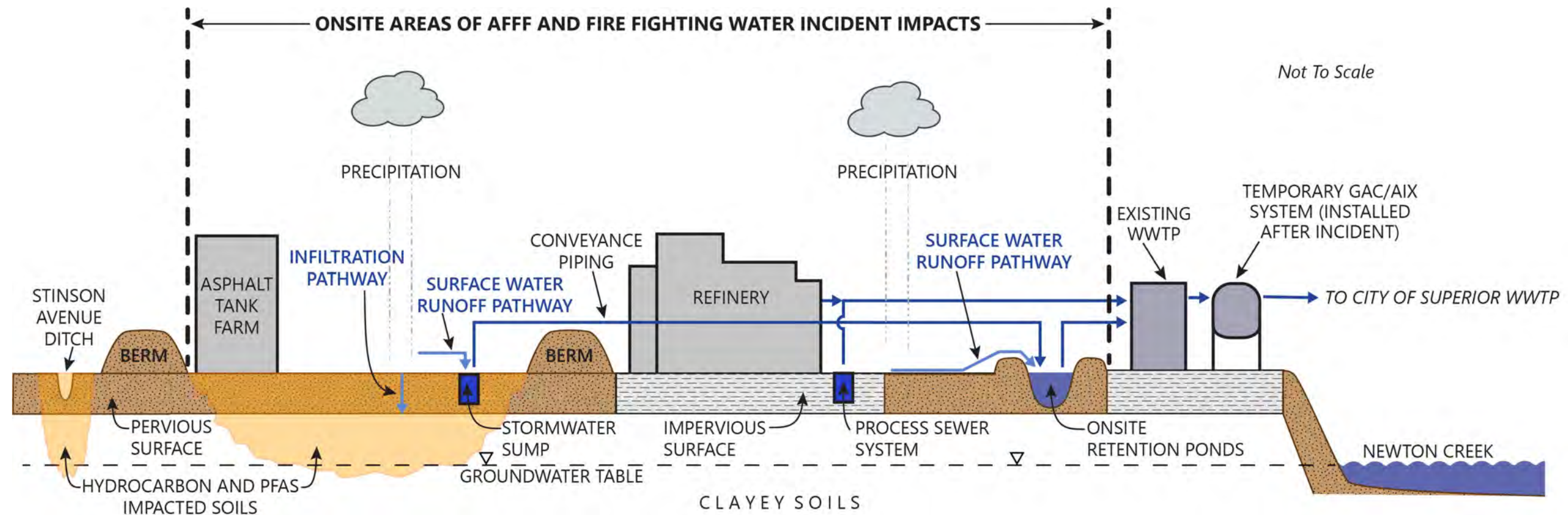


Barr Footer ArcGISPro 3.3, 2024-07-08 17:19 File: I:\Client\Superior\_Refining\Superior\_Refinery\MapaPost\_Incident\_PFA\_Site\_Investigation\2024\Updated\_Template.aprx Layout: Figure 7 Conceptual Site Model User: EMA



Area in contact with AFFF and/or firefighting water

Incident occurred April 26, 2018



Conceptual Site Model  
Superior Refining  
Company LLC (SRC)  
Superior, WI

FIGURE 7

**BARR**





## Appendices



# **Appendix A**

## **Initial Groundwater Investigation Work Plan**

## Appendix G: Initial Groundwater Investigation Work Plan

Superior Refinery April 26, 2018 Incident  
BRRTS Number: 02-16-581317  
February 2021

### 1.0 Work Plan Objective

The purpose of this work plan is to outline the approach to an initial investigation of potential hydrocarbon and per and polyfluoroalkyl substances (PFAS) impacts to groundwater as a result of the April 26, 2018 explosion and resulting fires (Incident) at the Superior Refinery (Site). Superior Refining Company LLC (SRC) has prepared this work plan in response to the Wisconsin Department of Natural Resources (WDNR) request in their letter dated September 18, 2018 (WDNR, 2018).

This initial groundwater site investigation work plan (SIWP) follows hydrocarbon and PFAS soil investigations associated with a release of hydrocarbons (asphalt, Therminol<sup>®</sup>, #6 fuel oil in the Asphalt Tank Farm and a range of asphalt to liquefied petroleum gases (LPGs) materials in the damaged process units) and Aqueous Film Forming Foam (AFFF) containing per- and polyfluoroalkyl substances (PFAS) during the Incident (Barr, 2021a and Barr, 2021b). As recommended in the *Site Investigation Report Update* (SIRU) (Barr, 2021b), this work plan has been developed to assess potential impacts to groundwater based on hydrocarbon and PFAS soil concentrations previously identified in the shallow soils of the areas impacted by the Incident. As requested by the WDNR, this site investigation work plan has been developed following the requirements of NR 716 and, in particular, the site investigation scoping requirements in NR 716.07 and *Site Investigation Work Plan Checklist* (WDNR, 2019).<sup>1</sup>

This groundwater SIWP includes the following activities:

- Assess the condition of groundwater that may be in contact with hydrocarbon and PFAS-impacted soil beneath pervious surfaces within the Incident impacted areas;
- Determine the need for additional investigation, interim action(s) and/or remedial action(s); and
- Collect/assess any additional information necessary to select an interim and/or recommended remedial action.

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<sup>1</sup> This site investigation demonstrates SRC's willingness to work with the WDNR in the investigation and remediation of AFFF released during the Incident, in direct response to WDNR's position that SRC must take such actions under current legal obligations enforced by WDNR with respect to PFAS. SRC is taking these actions without waiving but expressly preserving its right to object to, challenge, or dispute WDNR's position in any regard under any existing or future asserted legal obligation as to any PFAS compounds.

## 2.0 Background Information

This groundwater SIWP was developed as an appendix to the SIRU (Barr, 2021b). Therefore, pertinent information regarding the Incident, Site description, facility specific information, physical setting, most current conceptual site model (CSM), professional certification and additional background information required as part of a SIWP under NR 716.07 is provided in the SIRU (Barr, 2021b).

## 3.0 Groundwater Regulatory Guidance

The WDNR regulatory framework around hydrocarbon compounds in groundwater has been well established and published regulatory criteria have been developed; however, as emerging chemicals of interest, the same is not true for PFAS compounds. The primary purpose of this section is to outline the characteristics and nature of potential PFAS compounds that could migrate to groundwater and to establish the methods and site characterization principles that apply to the proposed groundwater investigation work.

### 3.1 AFFF and PFAS

AFFF is typically manufactured by combining hydrocarbon foaming agents with fluorinated surfactants (ITRC, 2020a). The composition of AFFF containing PFAS has varied over the last decade depending on the manufacturer and has transitioned from the historical formulation (perfluorooctanoic acid (PFOA) and perfluorooctanesulfonate (PFOS)-based) to a fluorotelomer based product and then to a shorter carbon chained (perfluorohexane sulfonate [PFHxS]-based) formulation.

Based on the analytical results from surface water samples collected from the Incident impacted areas showing detections of various PFAS compounds, multiple AFFF formulations may have been utilized during the Incident (Barr, 2021a). This information affects both the parameters to be tested for during this investigation and the understanding of the ways these PFAS compounds could interact in the environment.

### 3.2 PFAS Regulatory Guidance

As discussed in the previous PFAS soil investigation work plan (Barr, 2020a), this family of emerging contaminants continues to be studied and the sampling methods and threshold risk screening comparison criteria are not fully developed or defined by applicable regulations. Therefore, it is important to identify the framework and guidelines under which the site groundwater investigation work will be completed. Despite the lack of a regulatory framework for PFAS, the groundwater investigation was designed to be consistent with NR 716. In particular, the site investigation incorporated the scoping requirements in NR 716.07 and the *Site Investigation Work Plan Checklist* (WDNR, 2019) along with a reliance on the existing body of knowledge related to PFAS properties and their fate and transport in soils (ITRC, 2020d, 2020e).

The WDNR has not developed sampling guidance for PFAS sample collection (soil or groundwater) and has not approved specific laboratory analytical methods or certified specific laboratories for PFAS analysis.

The Wisconsin Department of Health Services (DHS) has recently published recommended groundwater standards for 18 PFAS compounds and although these recommended criteria are still in the rule making process, they will be used for comparison purposes in this investigation.

## **4.0 Sampling and Analysis Plan**

As described above, the objectives of this investigation are to investigate potential hydrocarbon and PFAS impacts to groundwater in the Incident impacted areas, to incorporate the results into the CSM to evaluate potential migration pathways and to determine if additional investigation, interim action measures and/or remedial action(s) are needed. To accomplish these objectives, a phased investigation strategy will be implemented to collect the necessary data to characterize and evaluate the impacts of hydrocarbon and PFAS compounds related to the Incident. Once this initial groundwater investigation phase has been completed, the CSM will be updated and, if necessary, additional investigation phases may be evaluated, designed, and implemented.

### **4.1 Incident Impacted Release Area Assessment**

The Incident and subsequent firefighting efforts resulted in the release of hydrocarbons and AFFF containing PFAS to pervious ground surfaces (Barr, 2021a). These compounds became comingled during the Incident response and were largely contained onsite in containment dikes, stormwater, and fire water retention ponds and/or stormwater drainage features.

Hydrocarbon and PFAS soil investigations have been performed in the area impacted by the Incident (Barr, 2020 and Barr, 2020b). Results of these investigations are summarized in the SIRU (Barr, 2021b).

This proposed scope of the groundwater investigation will focus on the characterization of groundwater quality within the Incident impacted Asphalt Tank Farm area. To assess the Incident related hydrocarbon and PFAS impacts to groundwater, one monitoring well will be installed within the impacted (source) area near the locations of soil samples with some of the highest concentrations of these compounds. The monitoring well will be installed at a time and location that ensures that the well will not be damaged or compromised by ongoing refinery rebuild efforts. It is estimated that the well will be installed during the second or third quarter of 2021 when rebuild activities in the source well target area are largely complete. The proposed source well target area is shown on Figure G-1.

### **4.2 Laboratory Analysis**

To determine the potential Incident related hydrocarbon and PFAS impact to groundwater in the source well area, samples will be analyzed for PFAS, petroleum volatile organic compounds (PVOCs), and polycyclic aromatic hydrocarbons (PAHs) by the laboratories and methods listed in Table G-1. The WDNR is in the process of certifying laboratories for PFAS analysis, but as of this report there are no laboratories accredited for PFAS by the State of Wisconsin. If available and timely for this phase of the PFAS investigation, the chosen PFAS analytical laboratory will be certified in the state of Wisconsin, but if not, we will proceed with the laboratory currently analyzing the onsite ponds, WWTP and offsite surface water receptors (Barr, 2020a). Information from this laboratory is provided in Table G-1.

### 4.3 Data Evaluation

Analytical data from groundwater hydrocarbon samples will be compared to the preventive action limit (PAL) and enforcement standard (ES) from the WDNR Chapter 140 Public Health Groundwater Quality Standards. Analytical data from the groundwater PFAS samples will be compared to the Wisconsin DHS recommended PFAS criteria. This DHS criteria list includes 18 PFAS compounds including five additional compounds not analyzed in previous interim action and investigation phases for surface water and soil. Two of the additional five PFAS compounds (HFPO-DA and ADONA) are not applicable to this event as they are more recently manufactured replacement compounds that were used to manufacture high performance fluoropolymers and coatings. Of the remaining three additional compounds, the laboratory currently has the ability to analyze one, and two are in method development with the laboratory. These two remaining compounds will be included if the laboratory has demonstrated they can produce quality analytical data for these two compounds in time for this investigation.

### 4.4 Methods

Field activities discussed in this section have been designed to provide the necessary data for completion of the investigation objectives defined above. Detailed descriptions of the planned investigation activities are presented below. This section has been developed in accordance with the requirements of NR 716.09 (2) (f).

#### 4.4.1 Project Health and Safety Plan

A project health and safety plan (PHASP) will be prepared for the investigation.

#### 4.4.2 Standard Operating Procedures

Attachment A provides the primary standard operating procedures (SOPs) that will be followed during this field investigation including Barr's SOP with specific PFAS information, *Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples*. PFAS compounds are present in many everyday items and have been widely used to produce products that are water resistant, stain resistant, heat resistant and/or oil resistant. Field personnel will be required to use specific sampling techniques, decontamination procedures, PFAS-free equipment and avoid wearing lotion, deodorant, cosmetics, sunscreen, waterproof clothing, stain-resistant clothing, and clothing washed in fabric softener when completing PFAS sampling field work. Updates to this work plan and associated SOPs will be prepared as needed for each subsequent phase of investigation work.

#### 4.4.3 Soil Classification and Field Screening

Soil recovered during well construction will be described in the field in accordance with the Universal Soil Classification System. Soils encountered will be described in accordance with ASTM-2488, *Standard Soil Practice for Description and Identification of Soils (Visual/Manual Method)* and will be screened in the field for volatile organic vapors with a photoionization detector (PID). Additionally, soil will be inspected for other evidence of contamination such as staining, odors, discoloration, and/or sheen, and the observations documented on a soil boring log for each location. Depth to water will be recorded, where encountered.

The field screening techniques for soils are as follows: visual examination, distinguishable odor, headspace organic vapor screening (> 10 ppm), and oil staining and/or sheen. The results of these four screening procedures will be used to screen soil samples for possible hydrocarbon contamination. A PID with a 10.6 eV lamp will be used to complete soil headspace screening for each sample interval in accordance with the applicable Barr SOP (Appendix B). The PID will be calibrated or checked against a known concentration of a calibration gas standard prior to collection of field measurements. Field representatives will document the field screening activities and measurements in a project-dedicated field logbook or on field log data sheets.

#### **4.4.4 Well Installation**

One monitoring well will be installed in the area impacted by the Incident to evaluate groundwater for potential hydrocarbon and PFAS contamination. The final well location will be determined once refinery rebuild activities have been largely completed in the source well target area and a location can be identified that is accessible, out of the way of operations and maintenance activities, and is unlikely to accumulate standing water during rain or snow melt events. The proposed source well target location is within the containment berm for Tanks 86 and 87 (Figure G-1) as some of the highest concentrations of hydrocarbons and PFAS were detected in the near surface soil samples collected from borings in this area (Barr, 2020 and Barr, 2021b).

Groundwater at the Site is estimated to be at an average depth of 3 feet below ground surface (bgs). The source well will be installed in the native clay to an approximate depth of 13 feet bgs and will be constructed in a manner that minimizes the potential for cross-contamination by residual impacted soils and/or surface water overlying the screened interval. First, two to three feet of surficial soil will be excavated to remove shallow residual PFAS impacted soil above the water table. Once the overburden is removed, the source well will be completed in the native clay using a hollow stem auger and constructed using 2-inch outside diameter (O.D.) schedule 40-PVC well screen. The filter pack, annular space seal and surface completion will be constructed in accordance with WDNR NR 141.

The selected well driller will provide drilling equipment and well materials that have been decontaminated and are free of any PFAS containing materials (e.g. Teflon™ containing materials) or contamination. Up to four rinsate blanks will be collected from the drilling equipment and/or well construction materials prior to the field event and analyzed for PFAS compounds. During well installation, soil samples will be collected for VOC headspace screening and classification using a stainless-steel split spoon sampler. Soil samples will not be collected for laboratory analysis.

The borehole will be logged and well construction details documented by a Barr field geologist and presented on the WDNR *Soil Boring Log Form* 4400-122 and WDNR *Monitoring Well Construction Form* 4400-113A. The new source well will be designated as Incident Well IW-1.



#### **4.4.5 Well Development**

Once installed, the source well will be developed by surging and bailing water to remove accumulated fine sediment from within the filter pack and to establish hydraulic connection with the surrounding aquifer. This field activity will be performed in conformance with Barr's SOP (Attachment A) and the WDNR Groundwater Sampling Desk Reference *and Field Manual* (WDNR, 1996a and WDNR, 1996b) and documented on the WDNR *Well Development Form* (Form 4400-113B).

#### **4.4.6 Groundwater Sample Collection and Analysis**

Once the well has been developed, it will be purged for a second time a minimum of seven days later. After the second purging, the well will be allowed to recover for a minimum of seven days, then will be gauged and a groundwater sample will be collected for PVOC, PAH and PFAS analysis. For verification purposes, a second purge and sample event (2 weekly purge events followed by sample collection 1 week later) will begin one week after the first sample is collected. This approach will result in the collection of a total of 2 groundwater samples approximately 30 days apart. The top of riser elevation relative to mean sea level (msl) will be surveyed.

A summary of analytes, laboratory methods, method detection limits (MDL), reporting limits (RL), and criteria is presented in Table G-1. A summary of the proposed sampling event is presented in Table G-2. A summary of potential laboratory QA/QC samples are presented in Table G-3. The proposed Quality Assurance/Quality Control (QA/QC) field samples are presented in Table G-4. Sample collection, special measures for collection of PFAS samples, chain-of custody documentation, and transport of samples will follow applicable Barr SOPs (Attachment A).

Appropriate sample handling and documentation procedures, as described in Barr's SOP (Attachment A) and the WDNR Groundwater Sampling Desk Reference *and Field Manual* (WDNR, 1996a and WDNR 1996b), will be followed.

#### **4.4.7 Sample Labeling and Numbering**

Sample nomenclature will be represented by abbreviated letter designators, followed by a unique location number. Samples will be labeled according to the location from which they are collected. Standard designators are as follows: MW = monitoring well; FB = field blank, RB = rinse blank and FD = field duplicate.

#### **4.4.8 Field Records**

All field activities and data will be recorded daily in a dedicated field notebook or on dedicated field data collection forms. The Barr field technician will record work times and dates, field data (boring logs, field screening results, field analytical data, sample depths, water levels, etc.), project health and safety information, internal Barr communications, client communications, decision-making processes and rationale, documentation of changes to the investigation scope, and any other observations or activities relevant to the project. Field investigation information will also be recorded as appropriate on the field forms.

#### 4.4.9 Investigation Derived Waste

Plans for managing investigation-derived waste are being provided in accordance with NR 716.09 (2) (f) 7. Waste generated by this investigation will be disposed of in accordance with federal, state, and local regulations and Barr's SOP: *Investigative Derived Waste*. It is anticipated that soil cuttings from the well installation will be placed in the on-site soil disposal containment building (3-Sided Building) and groundwater generated from well development, purging and sampling will be collected and directed to the on-site oil water separator/WWTP/GAC & IX for treatment.

#### 4.4.10 Reporting

Investigation activities, analytical results and data evaluations will be summarized in an Investigation Report in accordance with NR 716.15. The report will summarize the data collected during the investigation phase and compare analytical results to current State of Wisconsin risk-screening criteria and/or the recommended DHS standards relevant to the media and facility setting. The report will include the following elements: introduction; property setting; investigation results; QA/QC procedures and results; a preliminary risk-screening evaluation; conclusions; and recommendations. A monitoring well construction log and a property map showing the well location will be developed. Laboratory reports will also be attached to the report. Recommendations for future investigation work or response action plan development will be based on the results presented in the report.

### 4.5 Quality Assurance / Quality Control

#### 4.5.1 Project Data Quality Objectives

The data and investigative information generated will be used to determine impacts to groundwater and the overall nature and extent of any potential risks to human health and environment at the Site. This section has been developed in accordance with the requirements of NR 716.09 (2) (f) 5 and 6. The data will satisfy the Property Data Quality Objectives (DQOs) presented below:

- Analytical results must accurately reflect the groundwater quality.
- Field collection of samples for risk-based evaluations will require a high level of data quality since the sampling will be used to determine the potential risks associated with the release.
- Laboratory results must be of sufficient quality for making a determination that the identified chemicals of interest (COIs) either do or do not present risks to human health or the environment when compared to criteria established by the appropriate regulatory agency and/or governmental organization. In most cases, for COIs with established criteria, the MDL (also referred to as limits of detection (LOD) in the State of Wisconsin) will be lower than the appropriate risk-based values and applicable State criteria (see Table G-1). In some cases, laboratory instrumentation limitations and sample matrix may result in final MDLs greater than the associated risk standard. Guidance on how to handle these situations will be evaluated on a case-by-case basis.

#### **4.5.2 Quality Assurance Objectives**

The laboratory analyses will be used for the determination of overall compliance with project objectives. The laboratory will ensure the production of quality analytical data by overall quality assurance systems that are supported by documented quality control checks. The particular types and frequencies of quality control checks analyzed with samples are defined in the laboratory's SOPs and Quality Assurance Manual (QAM), which are available for review upon request. Laboratory acceptance criteria is included with each analytical report.

Quality assurance objectives (QAOs) have been established to ensure precision, accuracy, representativeness, comparability, and sensitivity (PARCCS) of laboratory analytical data and to meet the quality control (QC) acceptance criteria of analytical protocols in support of project needs. Overall, QAO procedures for field sampling, chain-of-custody, laboratory analysis, and reporting will provide the level of data required for determining the concentration of potential contaminants.

#### **4.5.3 Precision**

Precision measures the reproducibility of measurements under a given set of conditions.

##### **4.5.3.1 Field Precision Objectives**

Precision of field sampling will be assessed by comparing the analytical results between field duplicate samples. A field duplicate sample is a second aliquot of a sample generated in the field that, when collected, processed, and analyzed by the same organization, provide precision information for the entire measurement system, including: sample acquisition, sample constituent heterogeneity, handling, shipping, storage, preparation, and analysis. Field duplicate samples are submitted to the laboratory as blind (masked) samples. The relative percent difference (RPD) will be calculated using the equation below for each pair of duplicate analysis where both results are greater than five times the reporting limit.

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where:

S = First sample value (original or matrix spike value)

D = Second sample value (duplicate or matrix spike duplicate value)

Table G-4 lists the frequency and criteria for field duplicate samples.

##### **4.5.3.2 Laboratory Precision Objectives**

Precision in the laboratory may be assessed through the calculation of RPDs for laboratory control samples/laboratory control sample duplicates (LCS/LCSD), matrix spike/matrix spike duplicates (MS/MSD) or laboratory duplicates and will be analyzed at the frequency presented in Table G-3. Laboratory precision criteria will be included in the laboratory's reports.

#### **4.5.4 Accuracy**

Accuracy is the degree of agreement between an observed value and an accepted reference value and measures bias in a measurement system.

##### **4.5.4.1 Field Accuracy Objectives**

Accuracy in the field is assessed through field equipment calibration and maintenance, use of field blank samples, and through the adherence to sample handling, preservation and holding time requirements. Field equipment is tested and maintained when needed using manufacturers' recommendations. Table G-4 lists the frequency, description, and criteria for blank samples.

##### **4.5.4.2 Laboratory Accuracy Objectives**

Accuracy of laboratory results may be assessed using the analytical results of LCS/LCSD, MS/MSD samples, surrogate standards, extracted internal standards (EIS), and/or method blanks. The percent recovery (%R) for LCS, MS, surrogates, and EIS will be calculated using the following equation: (for LCS, surrogates, and EIS, B is zero):

$$\%R = \frac{A - B}{C} \times 100$$

Where:

- A = The analyte concentration determined experimentally from the spiked sample
- B = The background level determined by a separate analysis of the unspiked sample
- C = The amount of the spike added

Table G-3 lists the frequency and criteria for the LCS, MS, surrogates, EIS, and method blank samples. Laboratory accuracy criteria will be included in the laboratory's reports.

#### **4.5.5 Representativeness**

Representativeness is defined as a measure of the degree to which data accurately and precisely represents a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program to provide samples representative of Site conditions and proper laboratory protocol. The representativeness criteria will be satisfied by following the associated work plan and by the use of proper sampling techniques and appropriate analytical procedures. Sample collection procedures (Attachment A) will describe proper purging and stabilization techniques for groundwater samples that will aid in ensuring a sample is representative of Site conditions.

#### **4.5.6 Comparability**

Comparability is defined as the confidence with which one set of data can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling methods, sample preparative procedures, analytical methods and holding times. Comparability

will be satisfied by ensuring that the sample plan is followed, and proper and consistent sampling techniques are used.

#### **4.5.7 Sensitivity**

Sensitivity expresses the methodology's and laboratory's ability to meet or exceed the applicable criteria. Sensitivity is dependent upon instrument sensitivity, sample matrix, and composition effects, and will be monitored by the laboratory. Laboratory sensitivity will be assessed by comparing the analytical MDLs to the applicable criteria. Actual MDLs achieved will depend on sample size available, sample matrix interferences, and dilutions. Laboratory MDLs are listed in Table G-1.

### **4.6 Data Reporting**

#### **4.6.1 Field Data Reporting**

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of the measurements made in the field. Field documentation of well logs, boring logs, sample identifications, etc. will be contained in the final field reports.

#### **4.6.2 Laboratory Data Reporting**

Laboratory analyses reports will be submitted to Barr upon completion. Results will be reported to the MDL. The results between the MDL and RL will be qualified ("J") indicating estimated concentrations. As part of their report, the laboratory may qualify (flag) their data for such items as concentration between the MDL and RL, estimated concentration due to poor spike recovery, or concentration of chemical also found in the laboratory method blank. The laboratory will perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the chain-of-custody, the report format shall consist of the following:

- Date of issuance
- Project name and number
- Condition of samples upon receipt at the laboratory
- Cross-referencing of laboratory sample to project sample identification numbers
- Sample collection and receipt date
- Laboratory analysis performed
- Reference method used for analysis
- Laboratory batch number
- Sample preparation and analysis dates
- Sample results

- Laboratory MDL/LOD and RL/LOQ for each analyte
- Quality control data and acceptance criteria (including method blank results, laboratory control sample recoveries, surrogate recoveries, and extracted internal standard recoveries)
- Discussion and/or qualification of any laboratory quality control checks which failed to meet acceptance criteria
- Discussion and/or qualification of any holding times that were not met
- Data qualifier definitions
- Discussion of technical problems or other observations which may have created analytical difficulties
- Any deviations from intended analytical strategy
- Signature of the laboratory project manager

#### 4.7 Data Review

Analytical and data review procedures will be performed on the data. Data quality evaluation procedures will use the QC acceptance limits specified in the laboratory reports. The specific requirements which will be checked during data evaluation (where applicable) are:

- Holding times
- Preservation
- Blank data
- Laboratory control sample data
- Matrix spike data
- Surrogate standard data
- Extracted internal standard data
- Duplicate sample data

The data reviewer will identify any out-of-control data points and data omissions and interact with the laboratory to correct data deficiencies. Upon completing data review, the data reviewer will provide any qualifiers and will indicate whether the data are usable as reported, usable as an estimated concentration, or unusable.

The electronic data deliverable (EDD) sample data will be verified against the laboratory hard copy report by a Barr data technician to verify that the results in the EDD and the hardcopy report accurately reflect

the data collected. The EDD will be entered into a Barr computer database and the data will be output in a spreadsheet format to be used in report data tables. Data tables are reviewed by the Barr project manager before the report is submitted to the WDNR.

## 5.0 Schedule

Depending on site rebuild efforts and weather conditions, the investigation activities outlined above will begin within six months of receiving WDNR approval of this work plan. It is anticipated the investigation work can commence in the second or third quarter of 2021. Two rounds of groundwater samples will be collected within approximately 45 days of completion of well development. Following the collection of samples, laboratory analysis will take approximately 2 weeks to complete. Within 90 days of receiving laboratory results from the second round of groundwater sampling, an investigation report update will be prepared to summarize the results of the hydrocarbon and PFAS groundwater investigation. If necessary, this report will make recommendations for additional investigation, interim action, or remedial action. Final schedules will be dependent on approval of this work plan by the WDNR, coordination with the contractors, weather conditions, facility accessibility during the refinery rebuild activities and receipt of analytical results.

### Tables

Table G-1	Groundwater Analytical Parameter MDL/LOD, RL/LOQ and Criteria
Table G-2	Sample Event Summary
Table G-3	Laboratory Quality Control Samples
Table G-4	Field Quality Control Samples

### Figures

Figure G-1	Source Well Location
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### Attachments

Attachment A	Barr Standard Operating Procedures
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## References

- Barr, 2019. *Site Investigation Work Plan*, Superior Refinery April 26, 2018 Fire, BRRTS Number 02-16-581317, Prepared for Superior Refining Company LLC. June 2019.
- Barr, 2020. *Site Investigation Report*, Superior Refinery April 26, 2018 Incident, BRRTS Number 02-16-581317, Prepared for Superior Refining Company LLC. February 2020.
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- ITRC, 2020c. Fact Sheet – Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS), Technical and Regulatory Guidance Document PFAS-1, Section 5 Fate and Transport, Interstate Technology & Regulatory Council, PFAS Team, Washington D.C., April 2020.
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- WDNR, 1996b. *Groundwater Sampling Field Manual*, Bureau of Drinking Water and Groundwater, Publ-DG-038 96, September 1996.
- WDNR, 2018. *BRRTS Site Responsibility Letter and Site Investigation Request*, Superior Refinery Fire SERTs ID 20180426N016-1 and BRRTS 02-16-581317. September 18, 2018.



WDNR, 2019. *Site Investigation Work Plan Preparation Checklist*, WDNR Remediation and Redevelopment Program NR 700 Process Document RR-096. April 2019.

WDNR, 2020. WDNR Letter with Site Investigation Report Review Comments, BRRTS ID 02-16-581317, Wisconsin Department of Natural Resources, June 25, 2020.

## Tables

**Table G-1**  
**Groundwater Analytical Parameters, MDL/LOD, RL/LOQ, and Criteria**  
**Initial Groundwater Site Investigation Work Plan**  
**SRC Post-Incident Site Investigation**  
**Superior, Wisconsin**

Parameter	MDL/LOD (ng/L)	RL/LOQ (ng/L)	WI DNR Recommended Preventive Action Limit (PAL) (ng/L)	WI DNR Recommended Enforcement Standard (ES) (ng/L)
<b>Per- and Polyfluorinated Alkyl Substances (PFAS) by ASTM D7979(M) w/Isotope Dilution - Merit Laboratories</b>				
Perfluorobutanoic acid (PFBA)	0.7	6	2	10
Perfluorobutanesulfonic acid (PFBS)	0.2	2	90	450
Perfluoropentanoic acid (PFPeA)	0.6	5	---	---
Perfluoropentanesulfonic acid (PFPeS)	0.5	2	---	---
Perfluorohexanoic acid (PFHxA)	0.8	2	30	150
Perfluorohexanesulfonic acid (PFHxS)	0.7	2	4	40
Perfluoroheptanoic acid (PFHpA)	0.8	2	---	---
Perfluoroheptane sulfonic acid (PFHpS)	1	2	---	---
Perfluorooctanoic acid (PFOA)	0.3	2	2(ii)	20(ii)
Perfluorooctanesulfonic acid (PFOS)	0.7	2	2(ii)	20(ii)
Perfluorononanoic acid (PFNA)	0.6	2	3	30
Perfluorononanesulfonic acid (PFNS)	0.7	2	---	---
Perfluorodecanoic acid (PFDA)	0.5	2	60	300
Perfluorodecane sulfonic acid (PFDS)	0.7	2	---	---
Perfluoroundecanoic acid (PFUnDA)	0.6	2	0.6	3
Perfluorododecanoic acid (PFDoDA)	0.6	2	100	500
Perfluorooctanesulfonamide (FOSA)	0.4	2	2(ii)	20(ii)
Perfluorotridecanoic acid (PFTTrDA)	0.5	2	---	---
Perfluorotetradecanoic acid (PFTeDA)	0.6	2	2	10
N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)	1	2	2(ii)	20(ii)
N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)	0.6	2	---	---
4:2 FTS	0.8	2	---	---
6:2 FTS	1	2	---	---
8:2 FTS	1	2	---	---
N-Ethyl perfluorooctanesulfonamidoethanol (NEtFOSE)	Pending		2(ii)	20(ii)
Perfluorooctadecanoic acid (PFODA)	Pending		80	400
N-Ethyl Perfluorooctane sulfonamide (NEtFOSA)	0.6	2	2(ii)	20(ii)
<b>Polycyclic Aromatic Hydrocarbons (PAH) - EPA 8270 SIM - Pace Analytical Services, LLC</b>				
1-Methylnaphthalene	0.00613	0.04	---	---
2-Methylnaphthalene	0.011	0.04	---	---
Acenaphthene	0.0081	0.04	---	---
Acenaphthylene	0.00643	0.04	---	---
Anthracene	0.00818	0.04	600	3000
Benzo(a)anthracene	0.0117	0.04	---	---
Benzo(a)pyrene	0.00879	0.04	0.02	0.2
Benzo(b)fluoranthene	0.00777	0.04	0.02	0.2
Benzo(g,h,i)perylene	0.00841	0.04	---	---
Benzo(k)fluoranthene	0.00846	0.04	---	---
Chrysene	0.0111	0.04	0.02	0.2
Dibenz(a,h)anthracene	0.0108	0.04	---	---
Fluoranthene	0.0106	0.04	80	400
Fluorene	0.00676	0.04	80	400
Indeno(1,2,3-cd)pyrene	0.0191	0.04	---	---
Naphthalene	0.011	0.04	10	100
Phenanthrene	0.0102	0.04	---	---
Pyrene	0.0152	0.04	50	250

**Table G-1**  
**Groundwater Analytical Parameters, MDL/LOD, RL/LOQ, and Criteria**  
**Initial Groundwater Site Investigation Work Plan**  
**SRC Post-Incident Site Investigation**  
**Superior, Wisconsin**

Parameter	MDL/LOD (ng/L)	RL/LOQ (ng/L)	WI DNR Recommended Preventive Action Limit (PAL) (ng/L)	WI DNR Recommended Enforcement Standard (ES) (ng/L)
<b>Petroleum Volatile Organic Compounds (PVOC) - EPA 8260 - Pace Analytical Services, LLC</b>				
1,2,4-Trimethylbenzene	0.172	1	Combined 96	Combined 480
1,3,5-Trimethylbenzene	0.124	1		
Benzene	0.120	1	0.5	5
Ethylbenzene	0.0747	1	140	700
Methyl-tert-butyl ether	0.116	1	12	60
Toluene	0.122	1	160	800
Xylene, Total (calculated)	0.287	3	400	2,000

Notes:

MDL/LOD - Method Detection Limit/Limit of Detection

RL/LOQ - Reporting Limit/Limit of Quantitation

MDL/LOD studies are performed annually or more often as needed per method requirements. RL/LOQ are estimated values, approximately 10/3 of MDL/LOD, laboratory report will include actual value. MDL/LOD and RL/LOQ values are subject to change and may vary based on initial volume, dilution factor, and possible matrix interferences.

ng/L = nanogram per liter

Merit typically reports PFHxS and PFOS as linear and branched also.

(ii) DHS recommends a combined enforcement standard of 20 ng/L and combined preventive action limit of 2 ng/L for FOSA, NtFOSE, NtFOSA, NtFOSAA, PFOS, and PFOA.

**Table G-2**  
**Sample Event Summary**  
**Initial Groundwater Site Investigation Work Plan**  
**SRC Post-Incident Site Investigation**  
**Superior, Wisconsin**

Sample Type	Laboratory Analytical Parameters	Laboratory Method	Estimated Maximum Number of Investigative Samples <sup>1</sup>	Grab Sample	Quality Assurance/Quality Control (QA/QC) Samples				
					Rinsate Blank (RB) <sup>2</sup>	Field Duplicate (FD)	Field Blank (FB)	Trip Blank (TB)	Total
Equipment (e.g., PVC, filter pack sand)	PFAS	ASTM D7979(M)	0	X	4	0	0	0	4
Groundwater*	PFAS	ASTM D7979(M)	1	X	0	1	1	0	3
	PVOCs	EPA 8260	1	X	0	1	1	1	4
	PAHs	EPA 8270 SIM	1	X	0	1	1	0	3

PFAS – Per- and Polyfluoroalkyl Substances  
PVOCs - Petroleum Volatile Organic Compounds  
PAHs - Polycyclic Aromatic Hydrocarbons

\* Number of samples is for one sampling event.

<sup>1</sup>Actual number of samples will be determined based on field observations and/or locations as described in the Work Plan.

<sup>2</sup>One blank collected per equipment type when the equipment is not known to be PFAS-free. Further information is provided in Table G-4.

Field screening parameters at each sampling location will include visual, distinguishable odor, and soil organic vapor headspace.

**Table G-3**  
**Laboratory Quality Control Samples**  
**Initial Groundwater Site Investigation Work Plan**  
**SRC Post-Incident Site Investigation**  
**Superior, Wisconsin**

Parameter	Frequency	Comments
Method Blank	1 per batch of 20 or fewer samples, with every analytical batch or as stated in the method, whichever is more frequent	Analyte-free media processed simultaneously with, and under the same conditions, as samples. Used to assess possible sources of laboratory contamination present at concentrations that may impact analytical results. Target analytes should not have a reportable concentration above the MDL.
Laboratory Control Sample (LCS) or Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	1 LCS or 1 LCS/LCSD per batch of 20 or fewer samples, with every analytical batch or as stated in the method, whichever is more frequent	Analyte-free media spiked with a known concentration of analyte processed with, and under the same conditions, as samples. Recovery is used to evaluate overall analytical method accuracy independent of sample matrix effects. If analyzed in duplicate, the calculated relative percent difference (RPD) is used to assess the overall analytical method precision.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	1 MS or 1 MS/MSD set per batch of 20 or fewer samples (may or may not be project samples)	A sample spiked with a known concentration of analyte processed with, and under the same conditions, in order to assess the accuracy of a method in a given sample matrix. If analyzed in duplicate, the calculated RPD is used to assess the precision of a method in a given sample matrix.
Laboratory Duplicate	1 per batch of 20 or fewer samples, where applicable	A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method. It may be a duplicate of a sample or a duplicate of a matrix spike.
Surrogates	Surrogates are added to each sample for organic analyses (blanks, spiked samples, project samples, QC samples) prior to sample extraction	Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Recovery is used to evaluate the analytical method efficiency.
Extracted Internal Standard (EIS)	Added to each sample (blanks, spiked samples, project samples, QC samples) prior to sample extraction	Isotopically labeled internal standard (exact match, if available) added prior to extraction, centrifuging, filtering, or phase separation that goes through the same sample extraction and analysis. It is used to calculate a target analyte concentration.

**Table G-4**  
**Field Quality Control Samples**  
**Initial Groundwater Site Investigation Work Plan**  
**SRC Post-Incident Site Investigation**  
**Superior, Wisconsin**

Parameter	Frequency	Comments
Rinsate Blank	Prior to equipment use unless equipment is known to be PFAS-free	A sample of analyte-free water that has been collected from the rinsing of sampling equipment. It is used to check that equipment being considered for use at a project site would not introduce the PFAS of concern to the samples being collected. Best practice is to evaluate prior to using the equipment at the project site. Target analytes should not have reportable concentrations above the MDL or at levels that would impact the project samples.
Field Blank	1 per sampling event <sup>1</sup>	A sample of analyte-free water exposed to environmental conditions at the sampling site by transferring from one sample container to another or by removing the lid and exposing a container filled with analyte-free water to the atmosphere for the time equivalent necessary to fill a container. Collected instead of an Equipment Blank if disposable/single use sampling equipment is used. Target analytes should not have a reportable concentration above half the reporting limit or 1/10 the sample concentration, whichever is higher.
Field Duplicate	1 per sampling event <sup>1</sup>	Sample collected in duplicate using the same collection methods to verify reproducibility. Analyzed at the laboratory. RPD $\leq$ 30% for analyte concentrations $>$ 5x the reporting limit. For analyte concentrations $\leq$ 5x, professional judgement used.

<sup>1</sup> Sampling event is equivalent to an investigation phase (multi-day or back-to-back field event).

## Figures





Existing Monitoring Well

Existing Monitoring Well & Piezometer Pair

PFAS Soil Boring Location (Sept 4-5, 2020)

PFAS Hand Auger Location (PFAS-HA-# Sept 1, 2020  
PFAS-HA-#B Oct 16, 2020)

Hydrocarbon Soil Boring Location (Sept and Nov 2019)

Hydrocarbon Hand Auger Location (Oct 2019)

Test Pit Location (Sept 2019)

Crude Unit (Insight, 5/5/2019 and Barr, 7/6/2020)

FCCU Unit (Insight, 6/13/2019)

Tank Farm Unit (Insight, 6/13/2019)

Tank Farm Unit (Insight, 8/1/2019)

FCCU Unit (Insight, 11/19/2019)

Incident occurred April 26, 2018

Note: Release extent based on information provided to Barr by SRC.

Bathtub excavation limits are based on information provided to Barr by SRC.

0

100

200

Feet

PROPOSED SOURCE WELL TARGET AREA

Superior Refining Company, LLC (SRC)

Superior, WI

FIGURE G-1

Key:

Potential Incident Impacted Pervious Surface

Approximate Location of Refinery Rebuild Bathtub Construction Excavation (7.5 to 8 ft bgs)

Proposed Area for Source Groundwater Monitoring Well

Approximate Fenceline Boundaries for Refining-Related Activities

Incident Impacted Unpaved (Pervious) Surface- Firefighting Water Storage

PFAS Impacted Surface Water Storage and Treatment System

Incident Impacted Paved (Impervious) Surface

Incident Impacted Unpaved (Pervious) Surface

Estimated Extent of Residual Hydrocarbon Contaminated Soil Above Groundwater RCL Criteria

Stinson Ave Ditch



## Attachments

**Attachment A**

**Barr Standard Operating Procedures**

## Attachment A

### Index of Standard Operating Procedures (SOP)

#### Site Investigation Work Plan Superior Refinery April 2018 Explosion and Fire Superior, Wisconsin

<b>Barr Engineering SOP Title</b>
Collection and Disposal of Investigative Derived Waste
Collection of Groundwater Samples from a Temporary or Permanent Monitoring Well (Includes Well Purging and Stabilization)
Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples
Collection of Quality Control Samples
Decontamination of Sampling Equipment
Documentation on a Chain-of-Custody (COC)
Domestic Transport of Samples to Laboratories within the USA - States and Territories
Field Screening Soil Samples
Monitoring Well Development Oversight
Routine Level SVOC, PAH, DRO, and TPH Data Evaluation
Routine Level VOC, GRO, and TPH Data Evaluation




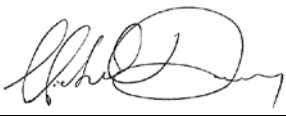
# Standard Operating Procedure

## Collection and Disposal of Investigative Derived Waste

Revision 7

October 6, 2020

Approved By:

<u>John Juntilla</u>		<u>10/06/20</u>
Print	Technical Reviewer    Signature	Date
<u>Michael Dupay</u>		<u>10/06/20</u>
Print	QA Manager    Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

# Collection and Disposal of Investigative Derived Waste

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to define the procedures for the collection and disposal of investigative derived waste (IDW) generated during field investigation activities. This procedure is applicable to sampling IDW which are materials containing pollutants derived during investigation activities including drill cuttings, drilling fluids, cleaning liquids, waste water, DNAPL, soil and rock samples, protective clothing and equipment, or any other items or materials which are exposed to, or may contain pollutants that must be characterized for off-site disposal.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- IDW can be contaminated with various hazardous substances, characterization may be necessary.

## 3.0 Responsibilities

The Barr Project Manager is responsible for determining whether any solid or liquid-phase product needs to be containerized and characterized for off-site disposal.

Experienced Field Technicians are responsible for the proper sample identification, collection and management of samples, documentation and sample transport to the laboratory.

The role of the Field Safety Representative is to oversee on-site safety activities.

Project staff are responsible for ordering sample containers prior to the sampling event.

## 4.0 Safety

Barr staff is responsible for conducting aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protection equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling material contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies

- Applicable sampling equipment
- Weatherproof container labels
- Plastic garbage bags
- Chemical resistant gloves (e.g., nitrile)
- IDW containers
- Permanent markers
- Plastic covering

## 6.0 Procedure

The Barr Project Manager is responsible for determining if IDW can be left on-site or if it must be disposed of off-site. Two general objectives that will be considered when managing IDW are the minimization of IDW generation and managing the IDW consistent with the final remedy for the site. The extent to which the objectives can be met is dependent on the site-specific circumstances.

Any IDW that is required to be containerized will be containerized separately by media until laboratory data are received to determine the appropriate disposition of the materials. Containerization and disposal of personal protective equipment and/or other materials, if necessary, will be determined on a project by project basis and discussed in the project Sampling and Analysis Plan (SAP).

### 6.1 Calibration

Calibration is not applicable to this SOP.

### 6.2 Sampling

Representative samples will be collected, and/or composited, preserved, and handled following Barr's matrix specific sampling SOP. Sampling equipment will be cleaned following Barr's 'Decontamination of Sampling Equipment' SOP.

The samples must be delivered to the laboratory via hand or overnight delivery courier in accordance with all Federal, State and Local transportation regulations and Barr's 'Domestic Transport of Samples to the Laboratory' SOP.

### 6.3 Data Reduction/Calculations

Data reduction or calculations are not applicable to this SOP.

### 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

## 7.1 QA/QC Samples

QA/QC samples are defined in Barr's SOP 'Collection of Quality Control Samples'. The sampling frequency should be performed as written in the project scope of work and/or documentation (e.g., Work Plan, SAP, or Quality Assurance Project Plan).

## 7.2 Measurement Criteria

Measurement criteria are not applicable to this SOP.

## 8.0 Records

The field technician will document the IDW sampling event on the field log data sheet and/or field notebook. They will also document the type and number of bottles on the chain-of-custody record, as appropriate. The analysis for each container and the laboratory used will be documented on the chain-of-custody record. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation is listed in the SOPs referenced in this procedure.

The field documents and COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: collection of samples, collection of QC samples, decontamination of sampling equipment, domestic transport of samples, and documentation on a COC.

## 9.0 References

Environmental Protection Agency, 9345.3-03FS. January 1992. *Guide to Management of Investigation-Derived Wastes*





# Standard Operating Procedure

## Collection of Groundwater Samples from a Temporary or Permanent Monitoring Well (Includes Well Purging and Stabilization)

Revision 2

March 14, 2019

Approved By:

<u>Kristen Jung</u>	<u></u>	<u>03/14/19</u>
Print	Technical Reviewer Signature	Date
<u>Terri Olson</u>	<u></u>	<u>03/14/19</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

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# Collection of Groundwater Samples from a Monitoring Well (Includes Well Purging and Stabilization)

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the methods used for monitoring well purging, stabilization, and sampling (excluding residential/water supply systems). The SOP also provides details regarding the calculation of purge volumes and measurement of groundwater stabilization criteria and identifies the common container, preservative, and holding times for typical groundwater sample analyses.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Sample collection methods can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance.
- Collection of groundwater samples from residential/water supply systems are not discussed within this SOP.
- Dedicated sampling equipment and/or decontamination of sampling equipment is required to prevent cross-contamination.
- Low-flow sampling methods are not discussed within this SOP.
- Sample collection using 'clean hands/dirty hands' methods is not discussed within this SOP.
- If sampling for per- and polyfluorinated alkyl substances (PFAS), special consideration must be taken to avoid accidental contamination of environmental samples - see Barr's SOP 'Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples'.

## 3.0 Responsibilities

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, SAP, etc.).

Experienced Field Technician(s) are responsible for the measurement of well pumping rates, calculation of well purge volume, field screening procedures, field equipment and calibration, proper sample identification, collection of samples, quality control procedures, and documentation.

Equipment Technicians are responsible for maintaining equipment in working order and aiding in troubleshooting equipment issues.

The role of the Field Safety Representative is to oversee on-site safety activities.

Project staff are responsible for ordering sample containers prior to the sampling event.

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling waters contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies\*

- Water quality meter (e.g., YSI, or equivalent)
- Polyethylene bailer and rope
- Sample tubing and fittings
- Turbidimeter (optional)
- Coolers
- Ice
- Chemical resistant gloves (e.g., nitrile)
- Calculator
- Locks/keys
- Pump (peristaltic or submersible), power source, and appropriate drive tubing
- Cord reel (optional)
- Graduated measuring container
- Plastic bags
- Waterproof ink pen or pencil
- Clock or stopwatch
- Sample containers (method specific)
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

\* See Barr's PFAS SOP for a list of prohibited and acceptable items.

## 6.0 Procedure

This section describes the procedure(s) for calibrating field equipment, measuring pumping rates, calculating purge volumes, well purging, measuring well stabilization, and the sampling, handling, and delivery of groundwater samples. Best practices include setting up the purging, stabilization, and sampling equipment in an upwind direction from any potential source of contamination.

This SOP describes the groundwater collection from a bore hole, temporary well, or permanent monitoring well. Typically, a direct-push (Geoprobe® or equivalent) will be used to create the bore hole or temporary well by advancing the direct-push sampler to the desired sampling interval (sampling depth). When the sampling depth is reached, small diameter extension rods are inserted through the steel probe rods to hold the groundwater sampler screen in place while the rods and screen sheath are retracted, exposing the screen. The groundwater sampler screen can typically be exposed up to 41 inches, but can be exposed a shorter length depending on project requirements. Alternately, a small diameter PVC well screen and riser pipe may be installed in the bore hole for use as a temporary well. Polyethylene (or project specified) tubing is placed into the bore hole or temporary well, and a peristaltic pump (or equivalent) or project specified pump is used to draw water samples to the surface for collection. Well

stabilization is not always necessary for temporary wells but if required by the project, see Section 6.2.6 of this SOP.

After each borehole or temporary well is constructed, the probe rods are decontaminated by the drilling contractor in accordance with project requirements. The polyethylene (or project specified) tubing is discarded after each sample is collected and new tubing is used for the collection of the next sample. The borehole and temporary well locations will be permanently sealed following applicable state and local regulations.

## 6.1 Calibration

The water quality meter and turbidimeter will be calibrated as per the applicable Barr SOP. The meters will undergo calibration checks, at a minimum, before and after sampling. The calibration check will be documented on a calibration form (as appropriate) and/or in the field notebook. Any significant issues found during the calibration check will be noted in the field notebook and the Equipment Technicians will be notified.

## 6.2 Purging/Well Stabilization/Sampling

Prior to sampling, purging of the monitoring well is performed to remove stagnant water from within the well and to stabilize the well to allow for representative groundwater sample collection. The term 'purge volume' refers to the amount of water removed from a well before groundwater sample collection occurs.

Purging well volumes and stabilizing to remove stagnant water from a temporary well may not be necessary due to the short time frame between well installation and sampling. Purging and well stabilization procedure for temporary wells may vary by project or by well. Recommended practice is to purge a temporary well until the water clears, if possible, prior to sampling; however, purging prior to sampling may not be possible at all if water is limited (as it might be in a perched water zone), or water recharge is slow (as it would be in a clayey or silty water bearing zone).

### 6.2.1 Purge Volume

The volume of standing water in the well is calculated to determine the purge volume that needs to be removed from the well. The water level must be measured in order to determine the volume (see applicable Barr SOP). Calculation of the purge volume is addressed in Section 6.3, Data Reduction/Calculation of this SOP and Table 1. If a well is pumped dry, this constitutes an adequate purge and the well can be sampled following recovery. Refer to project documentation for volumes required to be purged.

### 6.2.2 Bailer Purging

A bailer can be used for slowly recovering wells with minimal water volume and a depth to groundwater greater than 25 feet. A new disposable polyethylene bailer with a check valve can be attached to a cord reel or a downrigger and support assembly. Polyethylene bailers can be hauled using stainless steel wire or new nylon line (rope).

- Put on gloves for skin protection and to prevent sample contamination.

- Secure the bailer and lower slowly into the water column until the bailer is submerged. Avoid rapid movements of the bailer to minimize turbidity. A cord reel can be used to aid in the lowering of the bailer.
- Raise the bailer and empty the water collected from the bailer into a graduated measuring container.
- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

### 6.2.3 Peristaltic Pump Purging

A peristaltic pump is used when the water level is within suction lift (e.g., within about 25 feet of the ground surface but may be less at higher altitudes). It usually is a low-volume suction pump with low pumping rates suitable for sampling shallow, small-diameter wells.

- Put on gloves for skin protection and to prevent sample contamination.
- Lower tubing into the well water to the desired depth (typically near the middle of the water column within the well screen interval) and cut to the desired length.
- Connect the well tubing to the drive tubing entering the pump.
- Connect the drive tubing exiting the pump to the short section of tubing entering the flow-through cell or graduated measuring container.
- Turn on pump and set the speed at the desired rate of flow.
- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

### 6.2.4 Submersible Pump Purging

A submersible pump is used when the water level is greater than the suction lift associated with a peristaltic pump. It is commonly used in conjunction with a control box to achieve the desired pumping rate (low to high). Variable rate submersible pumps are available to fit inside 2 inch or larger wells.

#### 6.2.4.1 1.5-inch Submersible Pump

This is a type of submersible pump that can be used in 2-inch or larger diameter wells. It can purge water from depths down to 200 feet or greater, depending on pump model and manufacturer.

- Put on gloves for skin protection and to prevent sample contamination.
- Attach appropriate diameter tubing to pump intake, secure the tubing to the pump using a hose clamp or zip tie, lower pump, and secure at desired depth (typically near the middle of the water column within the well screen interval).
- Cut off tubing, allowing additional tubing length for discharge.
- Plug the pump into the controller. Pump will begin pumping using the variable speed controller. There are varieties of speed controllers available, typically designed for a specific pump.
- Attach the controller to the power supply (e.g., car battery, generator).
- Attach the tubing to the flow-through cell for the water quality meter.

*Note: If water is considerably turbid after initial pump start-up, the flow-through cell may be connected after purge water has cleared visually.*

Turn on the controller and dial the speed control to the desired flow rate. The controller can slow the purge rate down to the optimum rate.

*Note: If the submersible pump is not running, turn off the pump and then disconnect from the power supply. Check connections and try again.*

- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

#### 6.2.4.2 3 or 4-inch Submersible Pump

This pump may be used to purge water samples from any depth.

- Put on gloves for skin protection and to prevent sample contamination.
- Attach purging hose to the pipe connected on the top of the submersible pump.
- Lower the submersible pump slowly into the well until it is completely submersed into the water and secure at desired depth (typically near the middle of the water column within the well screen interval).
- Connect the pump to a sufficiently sized generator with an extension cord.
- Attach the flow-through cell for the water quality meter.

*Note: If water is considerably turbid after initial pump start-up, the flow-through cell may be connected after purge water has cleared visually.*

- Turn on pump and if it does not start, check connections to generator.
- Adjust flow rate to desired rate with the valve and measure the flow rate with the graduated measuring container.
- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

#### 6.2.5 Well Purging with In-place Plumbing

In-place plumbing consists of dedicated, submersible pumps that are permanently installed in a well.

- Put on gloves for skin protection and to prevent sample contamination.
- Turn switch to start the generator, put choke on, pull recoil rope, and let generator idle until it is running smooth.
- Connect the pump to the generator with an extension cord.
- Connect the pipe, elbow, and valve to the discharge pipe of the submersible pump (located at the top of the well) and turn on the generator.

*Note: If the pump does not start, check the connection from the generator to the pump.*

- When water flows from discharge of the pump, adjust the flow according to desired flow rate and measure the flow rate with the graduated measuring container.
- Attach the flow-through cell for the water quality meter.

*Note: If water is considerably turbid after initial pump start-up, the flow-through cell may be connected after purge water has cleared visually.*

- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

*Note: Each dedicated pump has its own pipe, elbow, and valve. These pieces are left at each well.*

### 6.2.6 Well Stabilization

Well stabilization is typically conducted to help verify that the groundwater sample is representative of aquifer conditions. A well is considered 'stabilized' after the well purge volume has been met and the groundwater (or well) stabilization parameter measurements are within acceptable limits for three consecutive readings. Well stabilization parameters may vary by project or regulatory agency but at a minimum typically include pH, temperature, and specific conductance (temperature corrected electrical conductivity). Dissolved oxygen (DO) and oxidation-reduction potential (ORP) may also be used as stabilization parameters.

The procedure to stabilize a well includes recording well stabilization parameter measurements collected with the water quality meter at the beginning of the well purging process and after subsequently purged well volumes. A well volume is measured as the volume of water present inside a well screen and/or casing (i.e., from the base of the well to the water level measurement) and is defined in the footnotes of Table 1. Groundwater aliquots used for stabilization parameter measurements are typically collected by either directing the purge water discharge line through a flow-through cell or by pouring groundwater from a bailer into a container holding the water quality meter probe (depending on the purging method used).

Documentation of the well stabilization process typically includes recording pertinent information such as the pump type, pumping rate, volume pumped, and well stabilization measurements on the field log data sheets or field notebook. If only the minimum parameters are used for stabilization, the DO and ORP should still be measured and recorded as they may be needed to interpret other chemical parameter results. Turbidity is measured with a standalone turbidimeter but is typically not used as a stabilization parameter. A qualitative determination of turbidity may also be noted (e.g. clear, cloudy, very cloudy, etc.).

The well may be sampled after three consecutive measurements (typically one well volume per measurement), collected at the intervals described above, are within specific project criteria or the criteria presented in Section 7.2, Measurement Criteria of this SOP.

If field parameters do not stabilize after five well volumes have been purged, then the field technician will verify that the probes and related equipment are functioning properly and that operator error is not an issue. They will also re-evaluate whether or not water is being withdrawn from the appropriate depth to effectively evacuate the well. If the checks produce no new insight, a decision will need to be made by the project team on whether to collect samples for laboratory analysis. When samples are collected, it will be clearly documented that stabilization was not achieved; at a minimum, this fact will be reported on the field log data sheets and in the Field Sampling Report.

If the well was purged dry, it shall be allowed to recharge and the samples should then be collected. If there is insufficient sample volume for the analyses being sampled, the project team will need to decide if sampling should be carried out or if a reduced prioritized list of analyses should be collected.

### 6.2.7 Sampling

The project team will determine the order for sampling the wells but general guidelines are below:

- Where water quality data are available, the least contaminated wells would be sampled first, proceeding to increasingly contaminated wells.

- Where the distribution of contaminants is not known, wells considered to be up gradient from likely sources of contamination would be sampled first and downgradient wells closest to the suspected contamination would be last.
- Make certain to keep records of the order in which wells were sampled.

Similar to purging, sampling requires the use of pumps or bailers. It may be appropriate to use a different device to sample than that which was used to purge. The most common example of this is the use of a pump to purge and a bailer to sample. There are several factors to take into consideration when choosing a sampling device. The experience of the project team will be used to determine which is appropriate and care should be taken when reviewing the advantages or disadvantages of any one device.

To reduce potential contamination, samples for PFAS should be collected first. See Barr's SOP 'Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples'. To prevent the possible loss of some volatile organic compounds (VOCs), samples for volatile parameters should be collected second with as little agitation and disturbance as possible, then proceed in order towards the least volatile parameter as listed in Barr's 'Water Sampling Guidelines' form. The 40 mL vials used to collect the VOC samples should be checked for air bubbles. Air bubbles may be caused by insufficient meniscus when sealing the vial, degassing after sample collection or during sample shipment, or reaction between the sample and preservative (HCl). If air bubbles > 6 mm (pea-sized) are observed during sampling, discard the vial and recollect the sample using a new vial. If air bubbles are believed to be due to the sample reacting with the preservative, the sample should be collected in an unpreserved vial if possible.

Put on new sampling gloves at each sampling site to reduce the risk of sample cross-contamination and exposure to skin. Never reuse gloves.

Prepare sampling containers by filling out the label, using an indelible permanent pen, with the following information at a minimum:

- Sample ID
- Date and time of sample collection
- Preservative
- Sample analysis (if required by the lab)

When filling the containers, do not insert the tubing into the containers and do not overfill preserved containers. When samples are containerized, place the filled sample containers in a sampling cooler with ice, turn off any equipment, disassemble the sampling apparatus, dispose of one-time use (disposable) equipment, and decontaminate reusable equipment per Barr's SOP 'Decontamination of Sampling Equipment'.

#### 6.2.7.1 Bailer Sampling

After the well has been purged and stabilized, secure the bailer and slowly lower into the top of the water column making certain not to stir up the water with the bailer, which could result in volatilizing the samples. Keep the bailer in the top portion of the water column when collecting the sample.

When the bailer is filled, slowly raise the bailer out of the well. A clean tarp may be used to cover the ground to minimize the contact of the rope with the ground. Fill containers in the order listed in Barr's 'Water Sampling Guidelines' form.



### 6.2.7.2 Peristaltic / Submersible Pump Sampling

After the well has been purged and stabilized, disconnect the tubing exiting the pump from the flow-through cell, if used and fill containers as listed in Barr's 'Water Sampling Guidelines' form.

### 6.2.7.3 Check Valve Sampling

Sampling temporary wells through tubing with a check valve may be conducted following a drilling subcontractor's procedure.

### 6.2.8 Preservation

Container volume, type, and preservative are important considerations in sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat analyses. The container type varies with the analysis required. Typically, the analytical laboratory will preserve the container before shipment. Preservation and shelf life vary; contact the laboratory to determine if an on-hand container is still useful. Barr's 'Water Sampling Guidelines' form lists the parameter, container type, container volume, and preservative for many of the most common parameters collected.

### 6.2.9 Handling

The samples will be bubble wrapped or bagged after collection, stored in a sample cooler, and packed on double bagged wet ice. Samples will be kept cold ( $\leq 6^{\circ}\text{C}$ , but not frozen), until receipt at the laboratory (where applicable).

*Note: Samples may need to be stored indoors in winter to prevent freezing.*

### 6.2.10 Shipment/Delivery

Once the cooler is packed to prevent breaking of bottles, the proper chain-of-custody (COC) documentation is signed and placed inside a plastic bag then added to the cooler.

Samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured.

Custody seals may be present, but at a minimum, the coolers must be taped shut to prevent the lid from opening during shipment.

The coolers must be delivered to the laboratory via hand or overnight delivery courier, if possible, in accordance with Federal, State and Local transportation regulations and Barr's SOP 'Domestic Transport of Samples to the Laboratory'.

## 6.3 Data Reduction/Calculations

Table 1 provides the volume of water (per foot or meter of depth) based on the diameter of the casing or hole. The following are two examples of calculations used in Table 1:

Volume of Standing Water (V), cubic feet

$$V = (\pi)(r^2)(h)$$

Where:  $\pi$  = 3.1416

r = Well radius (ft)

$h$  = Total well depth (ft) – depth to static water (ft) = Water column height (ft)

*Note: For the table calculations, 'h' is equal to one foot.*

#### Well Volume (WV), gallons

$$WV = (V)(7.48)$$

Where:  $V$  = Volume of standing water, cubic feet

7.48 = Cubic foot to US Gallons conversion factor

Calculate the volume of water to be purged using the equation below:

$$VP = (WV)(NMV)$$

Where:  $VP$  = Volume of water to be purged

$WV$  = Well volume in gallons

$NMV$  = Number of well volumes to be purged per project requirements

## 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

### 7.1 QA/QC Samples

QA/QC samples are defined in Barr's SOP 'Collection of Quality Control Samples'. The sampling frequency should be performed at the frequency noted in the project scope of work and/or documentation (e.g., Work Plan, SAP, or QAPP).

### 7.2 Well Stabilization Criteria

Well stabilization criteria to be used if there are no project specific criteria:

- pH  $\pm$  0.1 standard units
- Temperature  $\pm$  0.5 °C
- Specific conductance  $\pm$  5%
- Optional Criteria:
  - ORP  $\pm$  10 mV
  - Dissolved oxygen  $\pm$  10% (> 0.5 mg/L)
 

*Note: Three consecutive readings  $\leq$  0.5 mg/L can be considered stabilized.*
  - Turbidity  $\pm$  10% (> 5 Nephelometric Turbidity Units (NTU))
 

*Note: Three consecutive readings  $\leq$  5 NTU can be considered stabilized.*

## 8.0 Records

The field technician will document the pumping flow rate, well volume, time purged, volume purged, water level, total well depth and stabilization test measurements on the field log data sheet and/or field notebook. They will also document the type and number of bottles on the chain-of-custody record, as appropriate. The analysis for each container and the laboratory used will be documented on the chain-of-custody record. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Chain-of-custody (COC)
- Sample label
- Custody seal (if applicable)
- Water Level Data Sheet
- Field Log Data Sheet
- Field Log Cover Sheet
- Field Sampling Report
- Water Sampling Guidelines (includes sampling order, container, preservation, and holding time)

The field documents and COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: water level measurement, water quality meter, turbidimeter, collection of QC samples, collection of PFAS samples, decontamination of sampling equipment, investigative derived waster, domestic transport of samples, and documentation on a COC.

## 9.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3.*

Environmental Protection Agency, EPA/540/P-91/007. 1999. *Compendium of ERT Groundwater Sampling Procedures.*

Minnesota Pollution Control Agency, Water Quality Division. 2006. *Sampling Procedures for Groundwater Monitoring Wells.*

**Table 1**  
**Volume of Water in Casing or Hole**

<b>Diameter of Casing or Hole (In)</b>	<b>Gallons per Foot of Depth (WV)</b>	<b>Cubic Feet per Foot of Depth (V)</b>	<b>Liters per Meter of Depth</b>	<b>Cubic Meters per Meter of Depth</b>
1	0.041	0.0055	0.509	$0.509 \times 10^{-3}$
1½	0.092	0.0123	1.142	$1.142 \times 10^{-3}$
2	0.163	0.0218	2.024	$2.024 \times 10^{-3}$
2½	0.255	0.0341	3.167	$3.167 \times 10^{-3}$
3	0.367	0.0491	4.558	$4.558 \times 10^{-3}$
3½	0.500	0.0668	6.209	$6.209 \times 10^{-3}$
4	0.653	0.0873	8.110	$8.110 \times 10^{-3}$
4½	0.826	0.1104	10.26	$10.26 \times 10^{-3}$
5	1.020	0.1364	12.67	$12.67 \times 10^{-3}$
5½	1.234	0.1650	15.33	$15.33 \times 10^{-3}$
6	1.469	0.1963	18.24	$18.24 \times 10^{-3}$
7	2.000	0.2673	24.84	$24.84 \times 10^{-3}$
8	2.611	0.3491	32.43	$32.43 \times 10^{-3}$
9	3.305	0.4418	41.04	$42.04 \times 10^{-3}$
10	4.080	0.5454	50.67	$50.67 \times 10^{-3}$
11	4.937	0.6600	61.31	$61.31 \times 10^{-3}$
12	5.875	0.7854	72.96	$72.96 \times 10^{-3}$
14	8.000	1.069	99.35	$99.35 \times 10^{-3}$
16	10.44	1.396	129.65	$129.65 \times 10^{-3}$
18	13.22	1.767	164.18	$164.18 \times 10^{-3}$
20	16.32	2.182	202.68	$202.68 \times 10^{-3}$
22	19.75	2.640	245.28	$245.28 \times 10^{-3}$
24	23.50	3.142	291.85	$291.85 \times 10^{-3}$
26	27.58	3.687	342.52	$342.52 \times 10^{-3}$
28	32.00	4.276	397.41	$397.41 \times 10^{-3}$
30	36.72	4.909	456.02	$456.02 \times 10^{-3}$
32	41.78	5.585	518.87	$518.87 \times 10^{-3}$
34	47.16	6.305	585.68	$585.68 \times 10^{-3}$
36	52.88	7.069	656.72	$656.72 \times 10^{-3}$

1 gallon = 3.7854 liters

1 liter = 0.26417 gallons

1 meter = 3.281 feet

1 gallon water weighs 8.33 lbs. = 3.785 kilograms

1 liter water weighs 1 kilogram = 2.205 lbs.

1 gallon per foot of depth = 12.419 liters per foot of depth

1 gallon per meter of depth =  $12.419 \times 10^{-3}$  cubic meters per meter of depth





# Standard Operating Procedure

## Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples

Revision 0

March 12, 2019

Approved By:

Kevin McGilp		03/12/19
Print	Technical Reviewer Signature	Date
Terri Olson		03/12/19
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the methods used when collecting liquid (e.g., drinking water, groundwater, surface water, wastewater) and solid (e.g., soil, sediment, wipe) samples for per- and polyfluorinated alkyl substances (PFAS) analysis.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Sample collection methods can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance.
- PFAS samples are susceptible to contamination from many sources. Special consideration must be taken to avoid accidental contamination of environmental samples due to the presence of fluoropolymers, such as polytetrafluoroethylene (PTFE, e.g., Teflon®), in many consumer products and sampling materials.
- Dedicated or disposable sampling equipment and/or decontamination of sampling equipment should be used to prevent cross-contamination, where applicable.
- Since there are many individual PFAS, the substances of concern can vary by project. If a PFAS project list is not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance to develop an appropriate PFAS project list.

## 3.0 Responsibilities

Equipment Technicians are responsible for maintaining equipment in working order and aiding in troubleshooting equipment issues.

The role of the Field Safety Representative is to oversee on-site safety activities.

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, SAP, etc.).

Experienced Field Technician(s) are responsible for the proper sample identification, collection of samples, quality control procedures, and documentation.

Project staff are responsible for ordering sample bottles prior to the sampling event.

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected

contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling waters contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies

A summary of prohibited and acceptable materials is provided in Table 1. The list represents best practices when sampling but is subject to change as new information becomes available. Equipment and/or materials listed in other referenced SOPs may be used if known to be PFAS-free. If presence is unknown, it is highly recommended that rinsate blanks, or the materials themselves, be collected and submitted to the laboratory prior to use for analysis of the PFAS project list.

## 6.0 Procedure

This section describes the procedure(s) for the sampling, handling, and delivery of liquid and solid PFAS samples.

### 6.1 Calibration

Please refer to the individual field equipment SOP to be used during sampling.

### 6.2 Sampling

PFAS are man-made fluorinated and environmentally persistent compounds that do not occur naturally in the environment. Due to the presence of PFAS in common consumer products, the environment, and in equipment typically used to collect samples, care must be taken during sampling operations to minimize exposure of the sample to human, atmospheric, and other potential sources of contamination. A conservative approach is to exclude materials known to contain PFAS. When PFAS-containing equipment or supplies cannot be eliminated (e.g., fire retardant clothing at a refinery), consider collecting a sample of the material or a rinsate blank sample to show the extent of possible PFAS contamination. Use appropriate SOPs for sampling according to the matrix being collected.

#### 6.2.1 Source/Import Materials

Since PFAS is commonly found in many products, including equipment typically used to collect samples, materials being brought onto a project site should be screened for the project list of PFAS prior to use. Source/import materials may include, but are not limited to:

- Water used for drilling and decontamination
- Pumps, and drilling equipment that contacts the soil or water being sampled (e.g., drill augers, drill rods, direct-push sample liners, and well casing and screens)
- Personal protective equipment (PPE), including Tyvek®, leather gloves and boots (treated or not)
- Food wrappers and containers
- Additional items listed in Table 1

Depending on the item, sample a portion of the material or collect a rinsate blank by rinsing the material with PFAS-free water (typically supplied by the selected laboratory) and send to the laboratory for PFAS analysis. Best practice for a project is to define what is considered PFAS-free prior to beginning sampling operations. The material is considered acceptable for use if the PFAS results reported as nondetections, or less than the reporting limit, meet project requirements. As the current trends regarding acceptable and prohibited materials is evolving with respect to this emerging contaminant, it is recommended that the project team is consulted prior to sampling to determine if any changes have been made to the acceptable substances list.

### 6.2.2 Water and Soil Samples

Put on new sampling gloves at each sampling site to reduce the risk of sample cross-contamination and exposure to skin. Never reuse gloves. Use the sampling SOP that is appropriate for the type of sample being collected. **Collect PFAS samples first at each sampling location to minimize contact with other types of sample containers that may contain PFAS.** Avoid contact with the prohibited materials listed in Table 1 if possible.

Field blanks are typically collected with PFAS samples. Due to the possible areas of contamination, as well as the demand for increasingly lower reporting limits, the water used for the field blank is typically supplied by the lab. When collecting the field blank, pour the field blank water into the sample bottle and label this bottle as the field blank. Trip blanks, if required by the project, are supplied by the laboratory. They should accompany each cooler of PFAS samples and field blanks collected. Document the field and trip blank samples on the chain-of-custody (COC).

Turn off any equipment, disassemble the sampling apparatus, dispose of one-time use (disposable) equipment, and decontaminate reusable equipment per Barr's SOP 'Decontamination of Sampling Equipment'. Whenever possible, materials used for decontamination will need to be PFAS-free.

### 6.2.3 Preservation

Sample container size, type, and preservative are important considerations in sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat analyses. The container type varies with the matrix and analysis required. If preservation is required, the analytical laboratory will preserve the container before shipment. Barr's 'Water Sampling Guidelines' and 'Soil Sampling Guidelines' forms list the container type, container size, and preservative.

### 6.2.4 Handling

Prepare sample bottles/jars by filling out the label, using an indelible marker (e.g., fine point Sharpie®) with the following information at a minimum.

- Sample ID
- Date and time of sample collection
- Preservative
- Sample analysis (if required by the lab)

If placed into a bag, samples can be labeled directly on the bag, minimizing potential for contaminating sample. The bagged samples and blanks will be stored in a separate sample cooler (other sampling



containers may contain PFAS) and packed on bagged wet ice (not chemical ice packs – see Table 1). Samples will be kept cold ( $\leq 6^{\circ}\text{C}$ , but not frozen), until receipt at the laboratory.

*Note: Samples may need to be stored indoors in winter to prevent freezing.*

#### 6.2.5 Shipment/Delivery

Once the cooler is packed to reduce bottle shifting during transport, the proper COC documentation is signed and placed inside a plastic bag then added to the cooler.

Samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured.

Custody seals may be present, but at a minimum, the coolers must be taped shut to prevent the lid from opening during shipment.

The coolers must be delivered to the laboratory via hand or overnight delivery courier, if possible, in accordance with Federal, State and Local transportation regulations and Barr's SOP 'Domestic Transport of Samples to the Laboratory'.

#### 6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

#### 6.4 Disposal

Project-specific protocols for disposal of PFAS-contaminated investigation derived waste (IDW) should be established before sampling begins. Project IDW disposal plans should be adhered to in order to ensure that materials are stored and disposed of properly. Waste generated by this process will be disposed of in accordance with the project requirements, Federal, State and Local regulations, and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

### 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities allow the self-verification of the quality and consistency of the work.

#### 7.1 QA/QC Samples

QA/QC samples are defined in Barr's SOP 'Collection of Quality Control Samples'. The sampling frequency should be performed at the frequency noted in the project scope of work and/or documentation (e.g., Work Plan, SAP, or QAPP). To demonstrate that sample contamination has not occurred during field sampling, one field blank should be processed per day or per project requirements. If a trip blank was provided, it should be included with each PFAS cooler or per project requirements. The PFAS concentrations in the field and trip blank samples should not be detected at the level required for the project.

### 8.0 Records

The field technician will document the order in which the wells were sampled, any potential sources of contamination (e.g., changes in weather, wind direction, activity in the area), and any field test measurements on the field log data sheet and/or field notebook. They will also document the type and

number of bottles on the chain-of-custody record, as appropriate. The analysis for each bottle and the laboratory used will be documented on the chain-of-custody record. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation is specific to the sampling SOP being used.

The field documents and COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: collection of various matrices (e.g., groundwater, surface water, soil), low-flow sampling, field equipment, collection of QC samples, decontamination of sampling equipment, investigative derived waste, domestic transport of samples, and documentation on a COC.

## 9.0 References

Interstate Technology and Regulatory Council. 2018. *Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS)*.

Michigan Department of Environmental Quality. 2018. *General PFAS Sampling Guidance*.

Michigan Department of Environmental Quality. 2018. *MDEQ PFAS Sampling Quick reference Field Guide*.

New Hampshire Department of Environmental Services. 2016. *Perfluorinated Compound (PFC) Sample Collection Guidance*.

USEPA. 2018. *Method 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)*.

**Table 1**  
**Prohibited and Acceptable Items for PFAS Sampling**

Prohibited Items	Acceptable Items
<b>Field Equipment</b>	
Teflon® containing materials	High-density polyethylene (HDPE)
Storage of samples in containers made of LDPE materials	Acetate liners
Teflon® tubing	Silicon tubing
Waterproof field books	Loose paper (non-waterproof)
Plastic clipboards, binders, or spiral hard cover notebooks	Aluminum field clipboards or with Masonite
Post-It Notes	Sharpies®, pens
Chemical (blue or black) ice packs	Regular ice
<b>Field Clothing and Personal Protective Equipment (PPE)</b>	
New clothing or water resistant, waterproof, or stain- treated clothing, clothing containing Gore-Tex®. Avoid any sort of synthetic “performance” fabrics	Well-laundered clothing, defined as clothing that has been washed 6 or more times after purchase, made of synthetic or natural fibers (preferable cotton)
Clothing laundered using fabric softener	No fabric softener
Boots containing Gore-Tex® Leather boots and gloves may require pre-screening	Boots made with polyurethane and polyvinyl chloride (PVC) Disposable PFAS-free over-boots PFAS-free leather boots and gloves
Tyvek® (coated variety)	Cotton Clothing Plain, uncoated Tyvek® (must verify prior to use)
No cosmetics, moisturizers, hand cream, or other related products as part of personal cleaning/showering routine on the morning of sampling	<p align="center"><b>Sunscreens</b></p> <p>Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, Baby sunscreens that are “free” or “natural”</p> <p align="center"><b>Insect Repellents</b></p> <p>Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellent, Herbal Armor, California Baby Natural Bug Spray, BabyGanics</p> <p align="center"><b>Sunscreen and insect repellent</b></p> <p>Avon Skin So Soft Bug Guard Plus – SPF 30 Lotion</p>
<b>Sample Containers</b>	
LDPE or glass containers	HDPE or polypropylene
Teflon® -lined caps	Lined or unlined HDPE or polypropylene caps
<b>Rain Events</b>	
Waterproof or water resistant rain gear	Gazebo tent that is only touched or moved prior to and following sampling activities
<b>Equipment Decontamination</b>	
Decon 90	Alconox® and/or Liquinox®
Water from an on-site well	PFAS-free water
<b>Food Considerations</b>	
All food and drink, with exceptions as noted for acceptable items	Bottled water and hydration drinks (i.e. Gatorade® and Powerade®) to be brought and consumed only in the staging area
<b>General</b>	
<i>Prohibited includes materials or equipment containing:</i>	
Teflon®, polytetrafluoroethylene (PTFE) Food containers with waterproof coatings Anything with fluoro in the name Fluorinated ethylene propylene (FEP) Ethylene tetrafluoroethylene (ETFE) Low density polyethylene (LDPE) Polyvinylidene fluoride (PVDF)	Anything including the trademarks Teflon® and Hostaflon® Anything including the trademark Kynar® Anything including Polychlorotrifluoroethylene (PCTFE), that includes the trademark Neoflon® Anything including the trademark Tefzel® Anything including the trademarks Teflon® FEP and Hostaflon® FEP




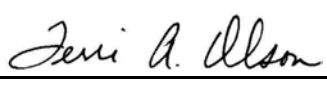
# Standard Operating Procedure

## Collection of Quality Control Samples

Revision 7

May 8, 2019

Approved By:

Andrea Nord		05/08/19
Print	Technical Reviewer Signature	Date
Terri Olson		05/08/19
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

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# Collection of Quality Control Samples

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures used in the collection and handling of field quality control (QC) samples: field blanks, equipment (rinsate) blanks, trip blanks, field (masked) duplicate samples, matrix spikes and matrix spike duplicate samples.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- The type and frequency of quality control samples can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance.
- Laboratory analysis specific QC samples (e.g., method blanks, laboratory control samples) are not discussed within this SOP.

## 3.0 Responsibilities

Experienced Field Technicians are responsible for the accurate collection of QC samples and the laboratory is responsible for the accurate set-up and analysis of QC samples.

Project staff are responsible for ordering sample containers prior to the sampling event.

The role of the Field Safety Representative is to oversee on-site safety activities.

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, Sampling Analysis Plan (SAP), etc.).

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies\*

- Laboratory-certified containers appropriate for the required analysis
- Sample containers/media (method specific)
- Chemical resistant gloves (e.g., nitrile)
- Sample labels
- Matrix specific sampling devices and equipment
- Analyte-free water
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

\* See Barr's PFAS SOP for a list of prohibited and acceptable items.

## 6.0 Procedure

This section provides the definitions and sampling procedure(s) for field derived QC samples.

### 6.1 Calibration

Calibration is not applicable to this SOP.

### 6.2 Sampling

General considerations to be taken into account when planning and conducting sampling operations are the required sample amount, sample holding times, sample handling, and special precautions for trace contaminant sampling. Matrix specific sampling SOPs should be followed for the collection and preservation of samples. The QC samples will be handled in the same manner as the sample group for which they are intended (i.e. stored and transported with the sample group).

#### 6.2.1 Blank Samples

Blank samples are used to monitor for potential contamination at a sampling site and may consist of field, equipment, rinsate, and trip blank samples. Each of these measure different potential sources of contamination. When collecting a blank for dissolved parameters, the blank water sample should be filtered before adding it to the sample container.

##### 6.2.1.1 Field Blank

A field blank (FB) is prepared on-site and is a sample of analyte-free water exposed to environmental conditions at the sampling site by either 1) transferring the water from one container to another or 2) by removing the lid and exposing a container filled with analyte-free water to the atmosphere for the time necessary to fill the container(s). It measures the potential for sample cross contamination due to site conditions.

##### 6.2.1.2 Equipment Blank

An equipment blank (EB) is prepared on-site and is a sample of analyte-free water that has been collected after field decontamination of sampling equipment (e.g., bailer or pump, hand-trowel and bowl) and prior to sampling the next location. It measures the potential for sample cross contamination due to insufficient decontamination. An equipment blank is not collected from disposable or dedicated equipment.

*Note: Prior to May 2019, the terms 'Equipment Blank' and 'Rinsate Blank' were used interchangeably and carried the same definition. To help better define the blank being collected, the term 'Rinsate Blank' is defined as listed below.*

#### 6.2.1.3 Rinsate Blank

A rinsate (or rinse blank, RB) is a sample of analyte-free water that has been collected from the rinsing of sampling equipment. It is used to check that equipment being considered for use at a project site would not introduce the target analyte of concern to the samples being collected. Best practice is to evaluate prior to using the equipment at the project site.

#### 6.2.1.4 Trip Blank

A trip blank (TB) is a sample of analyte-free water prepared or provided by the laboratory along with the sampling containers. Trip blank sample containers are not to be opened in the field and accompany the samples during collection, storage, and transport to the analytical laboratory. It measures the potential for sample cross contamination due to sample transport and handling.

A trip blank sample is used when sampling volatile parameters (e.g., volatile organic compounds (VOC)/gasoline range organic (GRO)/ total petroleum hydrocarbon (TPH)). Analyte-free water is used for an aqueous trip blank and methanol (or other applicable sample preservative) is used for a soil trip blank. A trip blank should be included for each sample cooler containing VOC samples and documented on the chain-of-custody (COC) form along with the samples and the required analysis. Trip blanks may also be used for per- and polyfluorinated alkyl substances (PFAS).

#### 6.2.2 Material Check

A material check (MC) is a sample of material (e.g. bentonite, sand) that has been collected to verify that the material being considered for use at a project site will not introduce the target analyte of concern to the samples being collected.

#### 6.2.3 Field (Masked) Duplicate

A field (masked) duplicate is a sample collected at the same time as an original/source sample using the same procedures, equipment, and types of containers. It measures the precision associated with sample homogeneity, collection, preservation, and storage, as well as laboratory procedures.

The field duplicate is collected in a separate container and assigned a different sample identification (e.g., M-1 or FD) than the original/source sample. The date sampled must be included on the sample container label and COC for holding time determination but not the time sampled so that the original/source sample will be blind to the laboratory. Containers designated for a particular analysis (e.g., semi-volatile organic compounds) must be filled sequentially before jars designated for another analysis are filled (e.g., metals). The field duplicate sample is analyzed using the same method as the original/source sample.

#### 6.2.4 Matrix Spike and Matrix Spike Duplicate

Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) samples are two aliquots of a sample to which known quantities of analytes are added (spiked) in the laboratory. The MS and MSD are prepared and analyzed exactly like their original/source sample aliquot. For some analyses, it is required that three separate sample aliquots are collected in the field for each analysis. One aliquot is analyzed to determine the concentrations in the original/source sample, a second sample aliquot serves as the MS, and the third sample aliquot serves as the MSD. The purpose of the MS and MSD is to quantify the bias and precision caused by the sample matrix.



## 6.3 Data Reduction/Calculations

### 6.3.1 Field Duplicate

Field duplicate sample results are evaluated by calculating the Relative Percent Difference (RPD) value. The RPD formula is as follows:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where: RPD = relative percent difference  
S = native sample result  
D = duplicate sample result

Note: The RPD equation may also be used to calculate the precision between the MS and MSD.

### 6.3.2 MS/MSD

MS/MSD recoveries are calculated using the following equation:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where: %R = % recovery  
SSR = spiked sample result  
SR = native/source sample result  
SA = spike added to native/source sample

## 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's 'Investigative Derived Waste' SOP. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

### 7.1 QA/QC Samples

The frequency of QC samples is generally one field blank or equipment blank/field duplicate/MS/MSD per twenty samples; however, specific project requirements may require alternative sampling frequencies.

### 7.2 Measurement Criteria

Criteria are defined in project specific documentation or in Barr's data evaluation SOPs.

## 8.0 Records

The field technician will document the type and number of QC samples collected during each sampling event on a COC and in a project dedicated field logbook or on field log data sheets.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Field Log Data Sheet
- COC form
- Sample label
- Custody seal (if applicable)

Field documentation and COC are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: sample collection, investigative derived waste, decontamination of sampling equipment, and documentation on a COC.

## 9.0 References

EPA QA/G-5. 2002. Guidance for Quality Assurance Project Plans.

EPA SW-846. 2014. *Chapter One: Project Quality Assurance and Quality Control*.

## Attachment A

### Index of Standard Operating Procedures (SOP)

#### Site Investigation Work Plan Superior Refinery April 2018 Explosion and Fire Superior, Wisconsin

<b>Barr Engineering SOP Title</b>
Collection and Disposal of Investigative Derived Waste
Collection of Groundwater Samples from a Temporary or Permanent Monitoring Well (Includes Well Purging and Stabilization)
Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples
Collection of Quality Control Samples
Decontamination of Sampling Equipment
Documentation on a Chain-of-Custody (COC)
Domestic Transport of Samples to Laboratories within the USA - States and Territories
Field Screening Soil Samples
Monitoring Well Development Oversight
Routine Level SVOC, PAH, DRO, and TPH Data Evaluation
Routine Level VOC, GRO, and TPH Data Evaluation




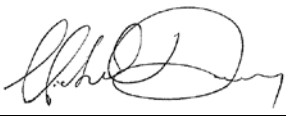
# Standard Operating Procedure

## Collection and Disposal of Investigative Derived Waste

Revision 7

October 6, 2020

Approved By:

<u>John Juntilla</u>		<u>10/06/20</u>
Print	Technical Reviewer    Signature	Date
<u>Michael Dupay</u>		<u>10/06/20</u>
Print	QA Manager    Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

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# Collection and Disposal of Investigative Derived Waste

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to define the procedures for the collection and disposal of investigative derived waste (IDW) generated during field investigation activities. This procedure is applicable to sampling IDW which are materials containing pollutants derived during investigation activities including drill cuttings, drilling fluids, cleaning liquids, waste water, DNAPL, soil and rock samples, protective clothing and equipment, or any other items or materials which are exposed to, or may contain pollutants that must be characterized for off-site disposal.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- IDW can be contaminated with various hazardous substances, characterization may be necessary.

## 3.0 Responsibilities

The Barr Project Manager is responsible for determining whether any solid or liquid-phase product needs to be containerized and characterized for off-site disposal.

Experienced Field Technicians are responsible for the proper sample identification, collection and management of samples, documentation and sample transport to the laboratory.

The role of the Field Safety Representative is to oversee on-site safety activities.

Project staff are responsible for ordering sample containers prior to the sampling event.

## 4.0 Safety

Barr staff is responsible for conducting aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protection equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling material contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies

- Applicable sampling equipment
- Weatherproof container labels
- Plastic garbage bags
- Chemical resistant gloves (e.g., nitrile)
- IDW containers
- Permanent markers
- Plastic covering

## 6.0 Procedure

The Barr Project Manager is responsible for determining if IDW can be left on-site or if it must be disposed of off-site. Two general objectives that will be considered when managing IDW are the minimization of IDW generation and managing the IDW consistent with the final remedy for the site. The extent to which the objectives can be met is dependent on the site-specific circumstances.

Any IDW that is required to be containerized will be containerized separately by media until laboratory data are received to determine the appropriate disposition of the materials. Containerization and disposal of personal protective equipment and/or other materials, if necessary, will be determined on a project by project basis and discussed in the project Sampling and Analysis Plan (SAP).

### 6.1 Calibration

Calibration is not applicable to this SOP.

### 6.2 Sampling

Representative samples will be collected, and/or composited, preserved, and handled following Barr's matrix specific sampling SOP. Sampling equipment will be cleaned following Barr's 'Decontamination of Sampling Equipment' SOP.

The samples must be delivered to the laboratory via hand or overnight delivery courier in accordance with all Federal, State and Local transportation regulations and Barr's 'Domestic Transport of Samples to the Laboratory' SOP.

### 6.3 Data Reduction/Calculations

Data reduction or calculations are not applicable to this SOP.

### 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

## 7.1 QA/QC Samples

QA/QC samples are defined in Barr's SOP 'Collection of Quality Control Samples'. The sampling frequency should be performed as written in the project scope of work and/or documentation (e.g., Work Plan, SAP, or Quality Assurance Project Plan).

## 7.2 Measurement Criteria

Measurement criteria are not applicable to this SOP.

## 8.0 Records

The field technician will document the IDW sampling event on the field log data sheet and/or field notebook. They will also document the type and number of bottles on the chain-of-custody record, as appropriate. The analysis for each container and the laboratory used will be documented on the chain-of-custody record. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation is listed in the SOPs referenced in this procedure.

The field documents and COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: collection of samples, collection of QC samples, decontamination of sampling equipment, domestic transport of samples, and documentation on a COC.

## 9.0 References

Environmental Protection Agency, 9345.3-03FS. January 1992. *Guide to Management of Investigation-Derived Wastes*





# Standard Operating Procedure

## Collection of Groundwater Samples from a Temporary or Permanent Monitoring Well (Includes Well Purging and Stabilization)

Revision 2

March 14, 2019

Approved By:

<u>Kristen Jung</u>	<u></u>	<u>03/14/19</u>
Print	Technical Reviewer    Signature	Date
<u>Terri Olson</u>	<u></u>	<u>03/14/19</u>
Print	QA Manager    Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

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# Collection of Groundwater Samples from a Monitoring Well (Includes Well Purging and Stabilization)

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the methods used for monitoring well purging, stabilization, and sampling (excluding residential/water supply systems). The SOP also provides details regarding the calculation of purge volumes and measurement of groundwater stabilization criteria and identifies the common container, preservative, and holding times for typical groundwater sample analyses.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Sample collection methods can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance.
- Collection of groundwater samples from residential/water supply systems are not discussed within this SOP.
- Dedicated sampling equipment and/or decontamination of sampling equipment is required to prevent cross-contamination.
- Low-flow sampling methods are not discussed within this SOP.
- Sample collection using 'clean hands/dirty hands' methods is not discussed within this SOP.
- If sampling for per- and polyfluorinated alkyl substances (PFAS), special consideration must be taken to avoid accidental contamination of environmental samples - see Barr's SOP 'Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples'.

## 3.0 Responsibilities

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, SAP, etc.).

Experienced Field Technician(s) are responsible for the measurement of well pumping rates, calculation of well purge volume, field screening procedures, field equipment and calibration, proper sample identification, collection of samples, quality control procedures, and documentation.

Equipment Technicians are responsible for maintaining equipment in working order and aiding in troubleshooting equipment issues.

The role of the Field Safety Representative is to oversee on-site safety activities.

Project staff are responsible for ordering sample containers prior to the sampling event.

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling waters contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies\*

- Water quality meter (e.g., YSI, or equivalent)
- Polyethylene bailer and rope
- Sample tubing and fittings
- Turbidimeter (optional)
- Coolers
- Ice
- Chemical resistant gloves (e.g., nitrile)
- Calculator
- Locks/keys
- Pump (peristaltic or submersible), power source, and appropriate drive tubing
- Cord reel (optional)
- Graduated measuring container
- Plastic bags
- Waterproof ink pen or pencil
- Clock or stopwatch
- Sample containers (method specific)
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

\* See Barr's PFAS SOP for a list of prohibited and acceptable items.

## 6.0 Procedure

This section describes the procedure(s) for calibrating field equipment, measuring pumping rates, calculating purge volumes, well purging, measuring well stabilization, and the sampling, handling, and delivery of groundwater samples. Best practices include setting up the purging, stabilization, and sampling equipment in an upwind direction from any potential source of contamination.

This SOP describes the groundwater collection from a bore hole, temporary well, or permanent monitoring well. Typically, a direct-push (Geoprobe® or equivalent) will be used to create the bore hole or temporary well by advancing the direct-push sampler to the desired sampling interval (sampling depth). When the sampling depth is reached, small diameter extension rods are inserted through the steel probe rods to hold the groundwater sampler screen in place while the rods and screen sheath are retracted, exposing the screen. The groundwater sampler screen can typically be exposed up to 41 inches, but can be exposed a shorter length depending on project requirements. Alternately, a small diameter PVC well screen and riser pipe may be installed in the bore hole for use as a temporary well. Polyethylene (or project specified) tubing is placed into the bore hole or temporary well, and a peristaltic pump (or equivalent) or project specified pump is used to draw water samples to the surface for collection. Well

stabilization is not always necessary for temporary wells but if required by the project, see Section 6.2.6 of this SOP.

After each borehole or temporary well is constructed, the probe rods are decontaminated by the drilling contractor in accordance with project requirements. The polyethylene (or project specified) tubing is discarded after each sample is collected and new tubing is used for the collection of the next sample. The borehole and temporary well locations will be permanently sealed following applicable state and local regulations.

## 6.1 Calibration

The water quality meter and turbidimeter will be calibrated as per the applicable Barr SOP. The meters will undergo calibration checks, at a minimum, before and after sampling. The calibration check will be documented on a calibration form (as appropriate) and/or in the field notebook. Any significant issues found during the calibration check will be noted in the field notebook and the Equipment Technicians will be notified.

## 6.2 Purging/Well Stabilization/Sampling

Prior to sampling, purging of the monitoring well is performed to remove stagnant water from within the well and to stabilize the well to allow for representative groundwater sample collection. The term 'purge volume' refers to the amount of water removed from a well before groundwater sample collection occurs.

Purging well volumes and stabilizing to remove stagnant water from a temporary well may not be necessary due to the short time frame between well installation and sampling. Purging and well stabilization procedure for temporary wells may vary by project or by well. Recommended practice is to purge a temporary well until the water clears, if possible, prior to sampling; however, purging prior to sampling may not be possible at all if water is limited (as it might be in a perched water zone), or water recharge is slow (as it would be in a clayey or silty water bearing zone).

### 6.2.1 Purge Volume

The volume of standing water in the well is calculated to determine the purge volume that needs to be removed from the well. The water level must be measured in order to determine the volume (see applicable Barr SOP). Calculation of the purge volume is addressed in Section 6.3, Data Reduction/Calculation of this SOP and Table 1. If a well is pumped dry, this constitutes an adequate purge and the well can be sampled following recovery. Refer to project documentation for volumes required to be purged.

### 6.2.2 Bailer Purging

A bailer can be used for slowly recovering wells with minimal water volume and a depth to groundwater greater than 25 feet. A new disposable polyethylene bailer with a check valve can be attached to a cord reel or a downrigger and support assembly. Polyethylene bailers can be hauled using stainless steel wire or new nylon line (rope).

- Put on gloves for skin protection and to prevent sample contamination.

- Secure the bailer and lower slowly into the water column until the bailer is submerged. Avoid rapid movements of the bailer to minimize turbidity. A cord reel can be used to aid in the lowering of the bailer.
- Raise the bailer and empty the water collected from the bailer into a graduated measuring container.
- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

### 6.2.3 Peristaltic Pump Purging

A peristaltic pump is used when the water level is within suction lift (e.g., within about 25 feet of the ground surface but may be less at higher altitudes). It usually is a low-volume suction pump with low pumping rates suitable for sampling shallow, small-diameter wells.

- Put on gloves for skin protection and to prevent sample contamination.
- Lower tubing into the well water to the desired depth (typically near the middle of the water column within the well screen interval) and cut to the desired length.
- Connect the well tubing to the drive tubing entering the pump.
- Connect the drive tubing exiting the pump to the short section of tubing entering the flow-through cell or graduated measuring container.
- Turn on pump and set the speed at the desired rate of flow.
- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

### 6.2.4 Submersible Pump Purging

A submersible pump is used when the water level is greater than the suction lift associated with a peristaltic pump. It is commonly used in conjunction with a control box to achieve the desired pumping rate (low to high). Variable rate submersible pumps are available to fit inside 2 inch or larger wells.

#### 6.2.4.1 1.5-inch Submersible Pump

This is a type of submersible pump that can be used in 2-inch or larger diameter wells. It can purge water from depths down to 200 feet or greater, depending on pump model and manufacturer.

- Put on gloves for skin protection and to prevent sample contamination.
- Attach appropriate diameter tubing to pump intake, secure the tubing to the pump using a hose clamp or zip tie, lower pump, and secure at desired depth (typically near the middle of the water column within the well screen interval).
- Cut off tubing, allowing additional tubing length for discharge.
- Plug the pump into the controller. Pump will begin pumping using the variable speed controller. There are varieties of speed controllers available, typically designed for a specific pump.
- Attach the controller to the power supply (e.g., car battery, generator).
- Attach the tubing to the flow-through cell for the water quality meter.

*Note: If water is considerably turbid after initial pump start-up, the flow-through cell may be connected after purge water has cleared visually.*

Turn on the controller and dial the speed control to the desired flow rate. The controller can slow the purge rate down to the optimum rate.

*Note: If the submersible pump is not running, turn off the pump and then disconnect from the power supply. Check connections and try again.*

- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

#### 6.2.4.2 3 or 4-inch Submersible Pump

This pump may be used to purge water samples from any depth.

- Put on gloves for skin protection and to prevent sample contamination.
- Attach purging hose to the pipe connected on the top of the submersible pump.
- Lower the submersible pump slowly into the well until it is completely submersed into the water and secure at desired depth (typically near the middle of the water column within the well screen interval).
- Connect the pump to a sufficiently sized generator with an extension cord.
- Attach the flow-through cell for the water quality meter.

*Note: If water is considerably turbid after initial pump start-up, the flow-through cell may be connected after purge water has cleared visually.*

- Turn on pump and if it does not start, check connections to generator.
- Adjust flow rate to desired rate with the valve and measure the flow rate with the graduated measuring container.
- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

#### 6.2.5 Well Purging with In-place Plumbing

In-place plumbing consists of dedicated, submersible pumps that are permanently installed in a well.

- Put on gloves for skin protection and to prevent sample contamination.
- Turn switch to start the generator, put choke on, pull recoil rope, and let generator idle until it is running smooth.
- Connect the pump to the generator with an extension cord.
- Connect the pipe, elbow, and valve to the discharge pipe of the submersible pump (located at the top of the well) and turn on the generator.

*Note: If the pump does not start, check the connection from the generator to the pump.*

- When water flows from discharge of the pump, adjust the flow according to desired flow rate and measure the flow rate with the graduated measuring container.
- Attach the flow-through cell for the water quality meter.

*Note: If water is considerably turbid after initial pump start-up, the flow-through cell may be connected after purge water has cleared visually.*

- Sampling may begin once desired volume is purged and the well has stabilized (see Section 6.2.6, Well Stabilization of this SOP).

*Note: Each dedicated pump has its own pipe, elbow, and valve. These pieces are left at each well.*

### 6.2.6 Well Stabilization

Well stabilization is typically conducted to help verify that the groundwater sample is representative of aquifer conditions. A well is considered 'stabilized' after the well purge volume has been met and the groundwater (or well) stabilization parameter measurements are within acceptable limits for three consecutive readings. Well stabilization parameters may vary by project or regulatory agency but at a minimum typically include pH, temperature, and specific conductance (temperature corrected electrical conductivity). Dissolved oxygen (DO) and oxidation-reduction potential (ORP) may also be used as stabilization parameters.

The procedure to stabilize a well includes recording well stabilization parameter measurements collected with the water quality meter at the beginning of the well purging process and after subsequently purged well volumes. A well volume is measured as the volume of water present inside a well screen and/or casing (i.e., from the base of the well to the water level measurement) and is defined in the footnotes of Table 1. Groundwater aliquots used for stabilization parameter measurements are typically collected by either directing the purge water discharge line through a flow-through cell or by pouring groundwater from a bailer into a container holding the water quality meter probe (depending on the purging method used).

Documentation of the well stabilization process typically includes recording pertinent information such as the pump type, pumping rate, volume pumped, and well stabilization measurements on the field log data sheets or field notebook. If only the minimum parameters are used for stabilization, the DO and ORP should still be measured and recorded as they may be needed to interpret other chemical parameter results. Turbidity is measured with a standalone turbidimeter but is typically not used as a stabilization parameter. A qualitative determination of turbidity may also be noted (e.g. clear, cloudy, very cloudy, etc.).

The well may be sampled after three consecutive measurements (typically one well volume per measurement), collected at the intervals described above, are within specific project criteria or the criteria presented in Section 7.2, Measurement Criteria of this SOP.

If field parameters do not stabilize after five well volumes have been purged, then the field technician will verify that the probes and related equipment are functioning properly and that operator error is not an issue. They will also re-evaluate whether or not water is being withdrawn from the appropriate depth to effectively evacuate the well. If the checks produce no new insight, a decision will need to be made by the project team on whether to collect samples for laboratory analysis. When samples are collected, it will be clearly documented that stabilization was not achieved; at a minimum, this fact will be reported on the field log data sheets and in the Field Sampling Report.

If the well was purged dry, it shall be allowed to recharge and the samples should then be collected. If there is insufficient sample volume for the analyses being sampled, the project team will need to decide if sampling should be carried out or if a reduced prioritized list of analyses should be collected.

### 6.2.7 Sampling

The project team will determine the order for sampling the wells but general guidelines are below:

- Where water quality data are available, the least contaminated wells would be sampled first, proceeding to increasingly contaminated wells.



- Where the distribution of contaminants is not known, wells considered to be up gradient from likely sources of contamination would be sampled first and downgradient wells closest to the suspected contamination would be last.
- Make certain to keep records of the order in which wells were sampled.

Similar to purging, sampling requires the use of pumps or bailers. It may be appropriate to use a different device to sample than that which was used to purge. The most common example of this is the use of a pump to purge and a bailer to sample. There are several factors to take into consideration when choosing a sampling device. The experience of the project team will be used to determine which is appropriate and care should be taken when reviewing the advantages or disadvantages of any one device.

To reduce potential contamination, samples for PFAS should be collected first. See Barr's SOP 'Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples'. To prevent the possible loss of some volatile organic compounds (VOCs), samples for volatile parameters should be collected second with as little agitation and disturbance as possible, then proceed in order towards the least volatile parameter as listed in Barr's 'Water Sampling Guidelines' form. The 40 mL vials used to collect the VOC samples should be checked for air bubbles. Air bubbles may be caused by insufficient meniscus when sealing the vial, degassing after sample collection or during sample shipment, or reaction between the sample and preservative (HCl). If air bubbles > 6 mm (pea-sized) are observed during sampling, discard the vial and recollect the sample using a new vial. If air bubbles are believed to be due to the sample reacting with the preservative, the sample should be collected in an unpreserved vial if possible.

Put on new sampling gloves at each sampling site to reduce the risk of sample cross-contamination and exposure to skin. Never reuse gloves.

Prepare sampling containers by filling out the label, using an indelible permanent pen, with the following information at a minimum:

- Sample ID
- Date and time of sample collection
- Preservative
- Sample analysis (if required by the lab)

When filling the containers, do not insert the tubing into the containers and do not overfill preserved containers. When samples are containerized, place the filled sample containers in a sampling cooler with ice, turn off any equipment, disassemble the sampling apparatus, dispose of one-time use (disposable) equipment, and decontaminate reusable equipment per Barr's SOP 'Decontamination of Sampling Equipment'.

#### 6.2.7.1 Bailer Sampling

After the well has been purged and stabilized, secure the bailer and slowly lower into the top of the water column making certain not to stir up the water with the bailer, which could result in volatilizing the samples. Keep the bailer in the top portion of the water column when collecting the sample.

When the bailer is filled, slowly raise the bailer out of the well. A clean tarp may be used to cover the ground to minimize the contact of the rope with the ground. Fill containers in the order listed in Barr's 'Water Sampling Guidelines' form.

### 6.2.7.2 Peristaltic / Submersible Pump Sampling

After the well has been purged and stabilized, disconnect the tubing exiting the pump from the flow-through cell, if used and fill containers as listed in Barr's 'Water Sampling Guidelines' form.

### 6.2.7.3 Check Valve Sampling

Sampling temporary wells through tubing with a check valve may be conducted following a drilling subcontractor's procedure.

### 6.2.8 Preservation

Container volume, type, and preservative are important considerations in sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat analyses. The container type varies with the analysis required. Typically, the analytical laboratory will preserve the container before shipment. Preservation and shelf life vary; contact the laboratory to determine if an on-hand container is still useful. Barr's 'Water Sampling Guidelines' form lists the parameter, container type, container volume, and preservative for many of the most common parameters collected.

### 6.2.9 Handling

The samples will be bubble wrapped or bagged after collection, stored in a sample cooler, and packed on double bagged wet ice. Samples will be kept cold ( $\leq 6^{\circ}\text{C}$ , but not frozen), until receipt at the laboratory (where applicable).

*Note: Samples may need to be stored indoors in winter to prevent freezing.*

### 6.2.10 Shipment/Delivery

Once the cooler is packed to prevent breaking of bottles, the proper chain-of-custody (COC) documentation is signed and placed inside a plastic bag then added to the cooler.

Samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured.

Custody seals may be present, but at a minimum, the coolers must be taped shut to prevent the lid from opening during shipment.

The coolers must be delivered to the laboratory via hand or overnight delivery courier, if possible, in accordance with Federal, State and Local transportation regulations and Barr's SOP 'Domestic Transport of Samples to the Laboratory'.

## 6.3 Data Reduction/Calculations

Table 1 provides the volume of water (per foot or meter of depth) based on the diameter of the casing or hole. The following are two examples of calculations used in Table 1:

Volume of Standing Water (V), cubic feet

$$V = (\pi)(r^2)(h)$$

Where:  $\pi$  = 3.1416

$r$  = Well radius (ft)

$h$  = Total well depth (ft) – depth to static water (ft) = Water column height (ft)

*Note: For the table calculations, 'h' is equal to one foot.*

#### Well Volume (WV), gallons

$$WV = (V)(7.48)$$

Where:  $V$  = Volume of standing water, cubic feet

7.48 = Cubic foot to US Gallons conversion factor

Calculate the volume of water to be purged using the equation below:

$$VP = (WV)(NMV)$$

Where:  $VP$  = Volume of water to be purged

$WV$  = Well volume in gallons

$NMV$  = Number of well volumes to be purged per project requirements

## 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

### 7.1 QA/QC Samples

QA/QC samples are defined in Barr's SOP 'Collection of Quality Control Samples'. The sampling frequency should be performed at the frequency noted in the project scope of work and/or documentation (e.g., Work Plan, SAP, or QAPP).

### 7.2 Well Stabilization Criteria

Well stabilization criteria to be used if there are no project specific criteria:

- pH  $\pm$  0.1 standard units
- Temperature  $\pm$  0.5 °C
- Specific conductance  $\pm$  5%
- Optional Criteria:
  - ORP  $\pm$  10 mV
  - Dissolved oxygen  $\pm$  10% (> 0.5 mg/L)
 

*Note: Three consecutive readings  $\leq$  0.5 mg/L can be considered stabilized.*
  - Turbidity  $\pm$  10% (> 5 Nephelometric Turbidity Units (NTU))
 

*Note: Three consecutive readings  $\leq$  5 NTU can be considered stabilized.*

## 8.0 Records

The field technician will document the pumping flow rate, well volume, time purged, volume purged, water level, total well depth and stabilization test measurements on the field log data sheet and/or field notebook. They will also document the type and number of bottles on the chain-of-custody record, as appropriate. The analysis for each container and the laboratory used will be documented on the chain-of-custody record. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Chain-of-custody (COC)
- Sample label
- Custody seal (if applicable)
- Water Level Data Sheet
- Field Log Data Sheet
- Field Log Cover Sheet
- Field Sampling Report
- Water Sampling Guidelines (includes sampling order, container, preservation, and holding time)

The field documents and COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: water level measurement, water quality meter, turbidimeter, collection of QC samples, collection of PFAS samples, decontamination of sampling equipment, investigative derived waster, domestic transport of samples, and documentation on a COC.

## 9.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3.*

Environmental Protection Agency, EPA/540/P-91/007. 1999. *Compendium of ERT Groundwater Sampling Procedures.*

Minnesota Pollution Control Agency, Water Quality Division. 2006. *Sampling Procedures for Groundwater Monitoring Wells.*

**Table 1**  
**Volume of Water in Casing or Hole**

<b>Diameter of Casing or Hole (In)</b>	<b>Gallons per Foot of Depth (WV)</b>	<b>Cubic Feet per Foot of Depth (V)</b>	<b>Liters per Meter of Depth</b>	<b>Cubic Meters per Meter of Depth</b>
1	0.041	0.0055	0.509	$0.509 \times 10^{-3}$
1½	0.092	0.0123	1.142	$1.142 \times 10^{-3}$
2	0.163	0.0218	2.024	$2.024 \times 10^{-3}$
2½	0.255	0.0341	3.167	$3.167 \times 10^{-3}$
3	0.367	0.0491	4.558	$4.558 \times 10^{-3}$
3½	0.500	0.0668	6.209	$6.209 \times 10^{-3}$
4	0.653	0.0873	8.110	$8.110 \times 10^{-3}$
4½	0.826	0.1104	10.26	$10.26 \times 10^{-3}$
5	1.020	0.1364	12.67	$12.67 \times 10^{-3}$
5½	1.234	0.1650	15.33	$15.33 \times 10^{-3}$
6	1.469	0.1963	18.24	$18.24 \times 10^{-3}$
7	2.000	0.2673	24.84	$24.84 \times 10^{-3}$
8	2.611	0.3491	32.43	$32.43 \times 10^{-3}$
9	3.305	0.4418	41.04	$42.04 \times 10^{-3}$
10	4.080	0.5454	50.67	$50.67 \times 10^{-3}$
11	4.937	0.6600	61.31	$61.31 \times 10^{-3}$
12	5.875	0.7854	72.96	$72.96 \times 10^{-3}$
14	8.000	1.069	99.35	$99.35 \times 10^{-3}$
16	10.44	1.396	129.65	$129.65 \times 10^{-3}$
18	13.22	1.767	164.18	$164.18 \times 10^{-3}$
20	16.32	2.182	202.68	$202.68 \times 10^{-3}$
22	19.75	2.640	245.28	$245.28 \times 10^{-3}$
24	23.50	3.142	291.85	$291.85 \times 10^{-3}$
26	27.58	3.687	342.52	$342.52 \times 10^{-3}$
28	32.00	4.276	397.41	$397.41 \times 10^{-3}$
30	36.72	4.909	456.02	$456.02 \times 10^{-3}$
32	41.78	5.585	518.87	$518.87 \times 10^{-3}$
34	47.16	6.305	585.68	$585.68 \times 10^{-3}$
36	52.88	7.069	656.72	$656.72 \times 10^{-3}$

1 gallon = 3.7854 liters

1 liter = 0.26417 gallons

1 meter = 3.281 feet

1 gallon water weighs 8.33 lbs. = 3.785 kilograms

1 liter water weighs 1 kilogram = 2.205 lbs.

1 gallon per foot of depth = 12.419 liters per foot of depth

1 gallon per meter of depth =  $12.419 \times 10^{-3}$  cubic meters per meter of depth





# Standard Operating Procedure

## Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples

Revision 0

March 12, 2019

Approved By:

Kevin McGilp		03/12/19
Print	Technical Reviewer Signature	Date
Terri Olson		03/12/19
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the methods used when collecting liquid (e.g., drinking water, groundwater, surface water, wastewater) and solid (e.g., soil, sediment, wipe) samples for per- and polyfluorinated alkyl substances (PFAS) analysis.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Sample collection methods can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance.
- PFAS samples are susceptible to contamination from many sources. Special consideration must be taken to avoid accidental contamination of environmental samples due to the presence of fluoropolymers, such as polytetrafluoroethylene (PTFE, e.g., Teflon®), in many consumer products and sampling materials.
- Dedicated or disposable sampling equipment and/or decontamination of sampling equipment should be used to prevent cross-contamination, where applicable.
- Since there are many individual PFAS, the substances of concern can vary by project. If a PFAS project list is not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance to develop an appropriate PFAS project list.

## 3.0 Responsibilities

Equipment Technicians are responsible for maintaining equipment in working order and aiding in troubleshooting equipment issues.

The role of the Field Safety Representative is to oversee on-site safety activities.

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, SAP, etc.).

Experienced Field Technician(s) are responsible for the proper sample identification, collection of samples, quality control procedures, and documentation.

Project staff are responsible for ordering sample bottles prior to the sampling event.

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected



contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling waters contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies

A summary of prohibited and acceptable materials is provided in Table 1. The list represents best practices when sampling but is subject to change as new information becomes available. Equipment and/or materials listed in other referenced SOPs may be used if known to be PFAS-free. If presence is unknown, it is highly recommended that rinsate blanks, or the materials themselves, be collected and submitted to the laboratory prior to use for analysis of the PFAS project list.

## 6.0 Procedure

This section describes the procedure(s) for the sampling, handling, and delivery of liquid and solid PFAS samples.

### 6.1 Calibration

Please refer to the individual field equipment SOP to be used during sampling.

### 6.2 Sampling

PFAS are man-made fluorinated and environmentally persistent compounds that do not occur naturally in the environment. Due to the presence of PFAS in common consumer products, the environment, and in equipment typically used to collect samples, care must be taken during sampling operations to minimize exposure of the sample to human, atmospheric, and other potential sources of contamination. A conservative approach is to exclude materials known to contain PFAS. When PFAS-containing equipment or supplies cannot be eliminated (e.g., fire retardant clothing at a refinery), consider collecting a sample of the material or a rinsate blank sample to show the extent of possible PFAS contamination. Use appropriate SOPs for sampling according to the matrix being collected.

#### 6.2.1 Source/Import Materials

Since PFAS is commonly found in many products, including equipment typically used to collect samples, materials being brought onto a project site should be screened for the project list of PFAS prior to use. Source/import materials may include, but are not limited to:

- Water used for drilling and decontamination
- Pumps, and drilling equipment that contacts the soil or water being sampled (e.g., drill augers, drill rods, direct-push sample liners, and well casing and screens)
- Personal protective equipment (PPE), including Tyvek®, leather gloves and boots (treated or not)
- Food wrappers and containers
- Additional items listed in Table 1

Depending on the item, sample a portion of the material or collect a rinsate blank by rinsing the material with PFAS-free water (typically supplied by the selected laboratory) and send to the laboratory for PFAS analysis. Best practice for a project is to define what is considered PFAS-free prior to beginning sampling operations. The material is considered acceptable for use if the PFAS results reported as nondetections, or less than the reporting limit, meet project requirements. As the current trends regarding acceptable and prohibited materials is evolving with respect to this emerging contaminant, it is recommended that the project team is consulted prior to sampling to determine if any changes have been made to the acceptable substances list.

### 6.2.2 Water and Soil Samples

Put on new sampling gloves at each sampling site to reduce the risk of sample cross-contamination and exposure to skin. Never reuse gloves. Use the sampling SOP that is appropriate for the type of sample being collected. **Collect PFAS samples first at each sampling location to minimize contact with other types of sample containers that may contain PFAS.** Avoid contact with the prohibited materials listed in Table 1 if possible.

Field blanks are typically collected with PFAS samples. Due to the possible areas of contamination, as well as the demand for increasingly lower reporting limits, the water used for the field blank is typically supplied by the lab. When collecting the field blank, pour the field blank water into the sample bottle and label this bottle as the field blank. Trip blanks, if required by the project, are supplied by the laboratory. They should accompany each cooler of PFAS samples and field blanks collected. Document the field and trip blank samples on the chain-of-custody (COC).

Turn off any equipment, disassemble the sampling apparatus, dispose of one-time use (disposable) equipment, and decontaminate reusable equipment per Barr's SOP 'Decontamination of Sampling Equipment'. Whenever possible, materials used for decontamination will need to be PFAS-free.

### 6.2.3 Preservation

Sample container size, type, and preservative are important considerations in sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat analyses. The container type varies with the matrix and analysis required. If preservation is required, the analytical laboratory will preserve the container before shipment. Barr's 'Water Sampling Guidelines' and 'Soil Sampling Guidelines' forms list the container type, container size, and preservative.

### 6.2.4 Handling

Prepare sample bottles/jars by filling out the label, using an indelible marker (e.g., fine point Sharpie®) with the following information at a minimum.

- Sample ID
- Date and time of sample collection
- Preservative
- Sample analysis (if required by the lab)

If placed into a bag, samples can be labeled directly on the bag, minimizing potential for contaminating sample. The bagged samples and blanks will be stored in a separate sample cooler (other sampling

containers may contain PFAS) and packed on bagged wet ice (not chemical ice packs – see Table 1). Samples will be kept cold ( $\leq 6^{\circ}\text{C}$ , but not frozen), until receipt at the laboratory.

*Note: Samples may need to be stored indoors in winter to prevent freezing.*

#### 6.2.5 Shipment/Delivery

Once the cooler is packed to reduce bottle shifting during transport, the proper COC documentation is signed and placed inside a plastic bag then added to the cooler.

Samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured.

Custody seals may be present, but at a minimum, the coolers must be taped shut to prevent the lid from opening during shipment.

The coolers must be delivered to the laboratory via hand or overnight delivery courier, if possible, in accordance with Federal, State and Local transportation regulations and Barr's SOP 'Domestic Transport of Samples to the Laboratory'.

### 6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

### 6.4 Disposal

Project-specific protocols for disposal of PFAS-contaminated investigation derived waste (IDW) should be established before sampling begins. Project IDW disposal plans should be adhered to in order to ensure that materials are stored and disposed of properly. Waste generated by this process will be disposed of in accordance with the project requirements, Federal, State and Local regulations, and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities allow the self-verification of the quality and consistency of the work.

### 7.1 QA/QC Samples

QA/QC samples are defined in Barr's SOP 'Collection of Quality Control Samples'. The sampling frequency should be performed at the frequency noted in the project scope of work and/or documentation (e.g., Work Plan, SAP, or QAPP). To demonstrate that sample contamination has not occurred during field sampling, one field blank should be processed per day or per project requirements. If a trip blank was provided, it should be included with each PFAS cooler or per project requirements. The PFAS concentrations in the field and trip blank samples should not be detected at the level required for the project.

## 8.0 Records

The field technician will document the order in which the wells were sampled, any potential sources of contamination (e.g., changes in weather, wind direction, activity in the area), and any field test measurements on the field log data sheet and/or field notebook. They will also document the type and

number of bottles on the chain-of-custody record, as appropriate. The analysis for each bottle and the laboratory used will be documented on the chain-of-custody record. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation is specific to the sampling SOP being used.

The field documents and COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: collection of various matrices (e.g., groundwater, surface water, soil), low-flow sampling, field equipment, collection of QC samples, decontamination of sampling equipment, investigative derived waste, domestic transport of samples, and documentation on a COC.

## 9.0 References

Interstate Technology and Regulatory Council. 2018. *Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS)*.

Michigan Department of Environmental Quality. 2018. *General PFAS Sampling Guidance*.

Michigan Department of Environmental Quality. 2018. *MDEQ PFAS Sampling Quick reference Field Guide*.

New Hampshire Department of Environmental Services. 2016. *Perfluorinated Compound (PFC) Sample Collection Guidance*.

USEPA. 2018. *Method 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)*.

**Table 1**  
**Prohibited and Acceptable Items for PFAS Sampling**

Prohibited Items	Acceptable Items
<b>Field Equipment</b>	
Teflon® containing materials	High-density polyethylene (HDPE)
Storage of samples in containers made of LDPE materials	Acetate liners
Teflon® tubing	Silicon tubing
Waterproof field books	Loose paper (non-waterproof)
Plastic clipboards, binders, or spiral hard cover notebooks	Aluminum field clipboards or with Masonite
Post-It Notes	Sharpies®, pens
Chemical (blue or black) ice packs	Regular ice
<b>Field Clothing and Personal Protective Equipment (PPE)</b>	
New clothing or water resistant, waterproof, or stain- treated clothing, clothing containing Gore-Tex®. Avoid any sort of synthetic “performance” fabrics	Well-laundered clothing, defined as clothing that has been washed 6 or more times after purchase, made of synthetic or natural fibers (preferable cotton)
Clothing laundered using fabric softener	No fabric softener
Boots containing Gore-Tex® Leather boots and gloves may require pre-screening	Boots made with polyurethane and polyvinyl chloride (PVC) Disposable PFAS-free over-boots PFAS-free leather boots and gloves
Tyvek® (coated variety)	Cotton Clothing Plain, uncoated Tyvek® (must verify prior to use)
No cosmetics, moisturizers, hand cream, or other related products as part of personal cleaning/showering routine on the morning of sampling	<p align="center"><b>Sunscreens</b></p> <p>Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, Baby sunscreens that are “free” or “natural”</p> <p align="center"><b>Insect Repellents</b></p> <p>Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellent, Herbal Armor, California Baby Natural Bug Spray, BabyGanics</p> <p align="center"><b>Sunscreen and insect repellent</b></p> <p>Avon Skin So Soft Bug Guard Plus – SPF 30 Lotion</p>
<b>Sample Containers</b>	
LDPE or glass containers	HDPE or polypropylene
Teflon® -lined caps	Lined or unlined HDPE or polypropylene caps
<b>Rain Events</b>	
Waterproof or water resistant rain gear	Gazebo tent that is only touched or moved prior to and following sampling activities
<b>Equipment Decontamination</b>	
Decon 90	Alconox® and/or Liquinox®
Water from an on-site well	PFAS-free water
<b>Food Considerations</b>	
All food and drink, with exceptions as noted for acceptable items	Bottled water and hydration drinks (i.e. Gatorade® and Powerade®) to be brought and consumed only in the staging area
<b>General</b>	
<i>Prohibited includes materials or equipment containing:</i>	
Teflon®, polytetrafluoroethylene (PTFE) Food containers with waterproof coatings Anything with fluoro in the name Fluorinated ethylene propylene (FEP) Ethylene tetrafluoroethylene (ETFE) Low density polyethylene (LDPE) Polyvinylidene fluoride (PVDF)	Anything including the trademarks Teflon® and Hostaflon® Anything including the trademark Kynar® Anything including Polychlorotrifluoroethylene (PCTFE), that includes the trademark Neoflon® Anything including the trademark Tefzel® Anything including the trademarks Teflon® FEP and Hostaflon® FEP





# Standard Operating Procedure

## Collection of Quality Control Samples

Revision 7

May 8, 2019

Approved By:

Andrea Nord		05/08/19
Print	Technical Reviewer Signature	Date
Terri Olson		05/08/19
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: \_\_\_\_\_

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Initials: \_\_\_\_\_

Date: \_\_\_\_\_

# Collection of Quality Control Samples

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures used in the collection and handling of field quality control (QC) samples: field blanks, equipment (rinsate) blanks, trip blanks, field (masked) duplicate samples, matrix spikes and matrix spike duplicate samples.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- The type and frequency of quality control samples can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance.
- Laboratory analysis specific QC samples (e.g., method blanks, laboratory control samples) are not discussed within this SOP.

## 3.0 Responsibilities

Experienced Field Technicians are responsible for the accurate collection of QC samples and the laboratory is responsible for the accurate set-up and analysis of QC samples.

Project staff are responsible for ordering sample containers prior to the sampling event.

The role of the Field Safety Representative is to oversee on-site safety activities.

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, Sampling Analysis Plan (SAP), etc.).

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies\*

- Laboratory-certified containers appropriate for the required analysis
- Sample containers/media (method specific)
- Chemical resistant gloves (e.g., nitrile)
- Sample labels
- Matrix specific sampling devices and equipment
- Analyte-free water
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

\* See Barr's PFAS SOP for a list of prohibited and acceptable items.

## 6.0 Procedure

This section provides the definitions and sampling procedure(s) for field derived QC samples.

### 6.1 Calibration

Calibration is not applicable to this SOP.

### 6.2 Sampling

General considerations to be taken into account when planning and conducting sampling operations are the required sample amount, sample holding times, sample handling, and special precautions for trace contaminant sampling. Matrix specific sampling SOPs should be followed for the collection and preservation of samples. The QC samples will be handled in the same manner as the sample group for which they are intended (i.e. stored and transported with the sample group).

#### 6.2.1 Blank Samples

Blank samples are used to monitor for potential contamination at a sampling site and may consist of field, equipment, rinsate, and trip blank samples. Each of these measure different potential sources of contamination. When collecting a blank for dissolved parameters, the blank water sample should be filtered before adding it to the sample container.

##### 6.2.1.1 Field Blank

A field blank (FB) is prepared on-site and is a sample of analyte-free water exposed to environmental conditions at the sampling site by either 1) transferring the water from one container to another or 2) by removing the lid and exposing a container filled with analyte-free water to the atmosphere for the time necessary to fill the container(s). It measures the potential for sample cross contamination due to site conditions.

##### 6.2.1.2 Equipment Blank

An equipment blank (EB) is prepared on-site and is a sample of analyte-free water that has been collected after field decontamination of sampling equipment (e.g., bailer or pump, hand-trowel and bowl) and prior to sampling the next location. It measures the potential for sample cross contamination due to insufficient decontamination. An equipment blank is not collected from disposable or dedicated equipment.

*Note: Prior to May 2019, the terms 'Equipment Blank' and 'Rinsate Blank' were used interchangeably and carried the same definition. To help better define the blank being collected, the term 'Rinsate Blank' is defined as listed below.*



#### 6.2.1.3 Rinsate Blank

A rinsate (or rinse blank, RB) is a sample of analyte-free water that has been collected from the rinsing of sampling equipment. It is used to check that equipment being considered for use at a project site would not introduce the target analyte of concern to the samples being collected. Best practice is to evaluate prior to using the equipment at the project site.

#### 6.2.1.4 Trip Blank

A trip blank (TB) is a sample of analyte-free water prepared or provided by the laboratory along with the sampling containers. Trip blank sample containers are not to be opened in the field and accompany the samples during collection, storage, and transport to the analytical laboratory. It measures the potential for sample cross contamination due to sample transport and handling.

A trip blank sample is used when sampling volatile parameters (e.g., volatile organic compounds (VOC)/gasoline range organic (GRO)/ total petroleum hydrocarbon (TPH)). Analyte-free water is used for an aqueous trip blank and methanol (or other applicable sample preservative) is used for a soil trip blank. A trip blank should be included for each sample cooler containing VOC samples and documented on the chain-of-custody (COC) form along with the samples and the required analysis. Trip blanks may also be used for per- and polyfluorinated alkyl substances (PFAS).

#### 6.2.2 Material Check

A material check (MC) is a sample of material (e.g. bentonite, sand) that has been collected to verify that the material being considered for use at a project site will not introduce the target analyte of concern to the samples being collected.

#### 6.2.3 Field (Masked) Duplicate

A field (masked) duplicate is a sample collected at the same time as an original/source sample using the same procedures, equipment, and types of containers. It measures the precision associated with sample homogeneity, collection, preservation, and storage, as well as laboratory procedures.

The field duplicate is collected in a separate container and assigned a different sample identification (e.g., M-1 or FD) than the original/source sample. The date sampled must be included on the sample container label and COC for holding time determination but not the time sampled so that the original/source sample will be blind to the laboratory. Containers designated for a particular analysis (e.g., semi-volatile organic compounds) must be filled sequentially before jars designated for another analysis are filled (e.g., metals). The field duplicate sample is analyzed using the same method as the original/source sample.

#### 6.2.4 Matrix Spike and Matrix Spike Duplicate

Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) samples are two aliquots of a sample to which known quantities of analytes are added (spiked) in the laboratory. The MS and MSD are prepared and analyzed exactly like their original/source sample aliquot. For some analyses, it is required that three separate sample aliquots are collected in the field for each analysis. One aliquot is analyzed to determine the concentrations in the original/source sample, a second sample aliquot serves as the MS, and the third sample aliquot serves as the MSD. The purpose of the MS and MSD is to quantify the bias and precision caused by the sample matrix.

## 6.3 Data Reduction/Calculations

### 6.3.1 Field Duplicate

Field duplicate sample results are evaluated by calculating the Relative Percent Difference (RPD) value. The RPD formula is as follows:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where: RPD = relative percent difference  
S = native sample result  
D = duplicate sample result

Note: The RPD equation may also be used to calculate the precision between the MS and MSD.

### 6.3.2 MS/MSD

MS/MSD recoveries are calculated using the following equation:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where: %R = % recovery  
SSR = spiked sample result  
SR = native/source sample result  
SA = spike added to native/source sample

## 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's 'Investigative Derived Waste' SOP. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

### 7.1 QA/QC Samples

The frequency of QC samples is generally one field blank or equipment blank/field duplicate/MS/MSD per twenty samples; however, specific project requirements may require alternative sampling frequencies.

### 7.2 Measurement Criteria

Criteria are defined in project specific documentation or in Barr's data evaluation SOPs.

## 8.0 Records

The field technician will document the type and number of QC samples collected during each sampling event on a COC and in a project dedicated field logbook or on field log data sheets.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Field Log Data Sheet
- COC form
- Sample label
- Custody seal (if applicable)

Field documentation and COC are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: sample collection, investigative derived waste, decontamination of sampling equipment, and documentation on a COC.

## 9.0 References

EPA QA/G-5. 2002. Guidance for Quality Assurance Project Plans.

EPA SW-846. 2014. *Chapter One: Project Quality Assurance and Quality Control*.





# Standard Operating Procedure

## Decontamination of Sampling Equipment

Revision 2

October 6, 2020

Approved By:

<u>John W. Juntilla</u>		<u>10/06/20</u>
Print	Technical Reviewer    Signature	Date
<u>Michael Dupay</u>		<u>10/06/20</u>
Print	QA Manager    Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Decontamination of Sampling Equipment

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to define the process used for decontaminating environmental sampling-related equipment including pumps, meters, and materials coming into contact with actual sampling equipment or with sampling personnel. This procedure is applicable to all personnel who are collecting samples and/or decontaminating sampling and field equipment.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Equipment used once and discarded such as bailers, protective gear, and filtration devices are not part of this SOP.

## 3.0 Responsibilities

The equipment technician is responsible for ensuring field equipment has been thoroughly decontaminated and prepared for use out in the field. The field technician(s) are responsible for decontamination in the field at each individual sampling point and for ensuring adherence to any investigative derived waste (IDW) project-specific requirements set forth in a QAPP or SAP (if applicable).

The role of the Field Safety Representative is to oversee on-site safety activities.

## 4.0 Safety

Barr staff is responsible for implementing aspects of the job safely. Where available, refer to the appropriate Project Health and Safety Plan (PHASP) to determine the proper personal protection equipment (PPE) required when using this SOP. Barr staff is responsible for conducting all aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protection equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies

- Non-phosphorus detergent (e.g., Liquinox™)
- Scrub brush made of inert materials
- Oven
- Bucket
- Tap water
- Analyte-free water (e.g., distilled or deionized (DI) water, or equivalent)
- Kimwipes®, or equivalent
- Chemical resistant gloves (e.g., nitrile)
- Spray bottle
- Organic solvent (e.g. methanol)

## 6.0 Procedure

This section describes the procedure(s) for the decontamination of equipment used to sample water, soil, or air.

### 6.1 Calibration

Calibration is not applicable to this SOP.

### 6.2 Operation

Decontamination of sampling equipment will be performed before sampling and after working at each sampling point, if applicable.

#### 6.2.1 Water Sampling Equipment

Equipment that does not contact sample water or the inside of the well should be rinsed with analyte-free water and inspected for remaining particles or surface film. If these are noted, repeat cleaning and rinse procedures.

Equipment that contacts sample water or the inside of the well should be cleaned (inside and outside where possible) with a non-phosphorus detergent solution applied with a spray bottle and/or scrub brush (if needed). Rinse with analyte-free water and containerize with other IDW if required by the SAP or QAPP and inspect for remaining particles or surface film. If these are noted, repeat cleaning and rinse procedures. Shake off remaining water and allow to air dry.

The internal surfaces of pumps and tubing that cannot be adequately cleaned by the above methods alone will also be cleaned by first circulating a non-phosphorus detergent solution through them followed by circulating analyte-free water. Special care will be exercised to ensure that the "rinse" fluids will be circulated in sufficient quantities to completely flush out contaminants and detergents.

When transporting or storing equipment after cleaning, the equipment will be stored in a manner that minimizes the potential for contamination.

#### 6.2.2 Soil/Sediment Sampling Equipment

A variety of samplers (split-barrel, split-barrel with brass liners, piston sampler, backhoe, hand-auger, or shovel) may be used to retrieve soil from sampling locations. The soil sample will either be sealed within the sampler (e.g., collecting volatile samples) or the soil sample will be transferred to laboratory-supplied containers depending on the analysis to be conducted on the soil sample. The equipment required to transfer the soil from the sampler to the laboratory-supplied sample containers includes: stainless-steel

spoons or scoops and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the PHASP.

All soil sampling equipment, including split-barrels, stainless-steel spoons and scoops, will be carefully cleaned before and during sampling with a tap water and non-phosphorus detergent solution, using a brush if necessary to remove particulate matter and films. The equipment is then rinsed three times with tap water and/or three times with analyte-free water. Inspect equipment and repeat procedure if any residual soil or visible contaminants are present. Dry sampler with a Kimwipes®. Organic solvents (e.g., methanol) may be used to aid with desorbing organic material but should be kept to a minimum and must be collected and containerized if used.

At the completion of the work day, the samplers should be decontaminated following the procedure above and stored in a manner that minimizes the potential for contamination.

### 6.2.3 Air Sampling Equipment

For non-laboratory manifold equipment, methanol soak manifold components for a minimum of two hours. Remove from the methanol bath and place in an oven pre-heated to 90 °C and continue to heat manifold components for at least 3 hours or until interior and exterior surface inspections of the manifold components indicate that they are free of liquid methanol.

### 6.2.4 Handling

All equipment will be handled in a manner that minimizes cross-contamination between points. After cleaning, the equipment will be visibly inspected to detect any residues or other substances that may exist after normal cleaning. If inspection reveals that decontamination was insufficient, the decontamination procedures will be repeated.

## 6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

## 6.4 Disposal

IDW generated by this process will be disposed of in accordance with Federal, State and Local regulations and/or as required by project-specific SAP or Work Plan. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

### 7.1 QA/QC Samples

Decontamination procedures may be monitored through the use of an equipment blank which consists of analyte-free water processed through non-disposable or non-dedicated aqueous or solid sampling equipment after equipment decontamination and before field sample collection. The equipment blank is analyzed for the same parameters as the samples at a project specific frequency (e.g., one per twenty samples).

## 7.2 Measurement Criteria

Equipment blank results should be below the laboratory's method detection limit or reporting limit (depending on the data quality objectives).

## 8.0 Records

When required, the field technician(s) will document the field equipment decontamination procedures in a project dedicated field logbook or on field log data sheets.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation is listed in the applicable sample collection SOP.

Field documentation and COC are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual."

Other Barr SOP subjects referenced within this SOP: collection of samples and investigative derived waste.

## 9.0 References

ASTM. 2015. Standard Practice for Decontamination of Field Equipment Used at Waste Sites.







# Standard Operating Procedure Documentation on a Chain-of-Custody (COC)

Revision 6

February 26, 2020

Approved By:

Andrea Nord		02/26/20
Print	Technical Reviewer Signature	Date
Terri Olson		02/26/20
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Documentation on a Chain-of-Custody (COC)

## 1.0 Scope and Applicability

The purpose of this procedure is to describe how to properly document information on a Chain-of-Custody (COC). A COC is a legally binding document that identifies sample identification, analyses required, and shows traceable possession of samples from the time they are obtained until they are introduced as evidence in legal proceedings. A Field Technician completes the information on the COC at the time he/she collects samples and the COC accompanies the samples during transport to a storage facility or to the laboratory for analysis.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- The SOP does not apply to sample aliquots that are only collected for field screening purposes.
- The SOP does not apply to samples remaining on-site.

## 3.0 Responsibilities

Experienced Field Technicians are responsible for the proper sample identification and for accurate and complete documentation on the COC.

## 4.0 Procedure

The COC is the most important sampling document; it must be filled out accurately and completely every time a sample is collected. The instructions below are specific to Barr's COC for air canisters and Barr's COC typically used for solid and liquid samples. The COC for air canisters is typically used when collecting soil gas, soil vapor, emissions, or indoor and outdoor air samples in an evacuated canister. The COC for solid and liquid samples is typically used when collecting matrices such as groundwater, surface water, drinking water, waste water, storm water, soil, sediment, oil, paint chips, bulk materials, etc. Information common to both COCs and specific to each COC are detailed below.

Some of the information on a COC may be filled out ahead of time (e.g., report and invoice recipient details, project number, project name, project manager, purchase order number, etc.) while other information should be completed during sampling. Complete one COC or more, as needed, for each set of project samples. The COC should be completed prior to leaving the sampling location.

Laboratory supplied COCs may be used but may differ in the information captured. The use of a Barr COC is recommended as it allows for more efficient data processing within Barr's systems. If there are any questions, please contact a member of Barr's Data Quality team.

The laboratory receiving the samples will sign the COC, record the date and time of sample receipt, assign a laboratory work order number, document sample condition, and document whether custody seals were used and if they were intact.

#### 4.1 Common Chain-of-Custody Information

- Barr office location managing the work.
- Two digit identification for the state or province the samples originated from/sampled in.
- COC numbered pages (e.g., 1 of 1).
- Report and invoice recipient information.
- Purchase order number (if applicable).
- Barr project name and number.
- Sample location.
- Sample collection date and time.
- Sample matrix abbreviation (see "Matrix Code" on COC).
- Analysis requested.
- Field Technician (i.e. sampler) name.
- Barr Project Manager and project Data Quality (DQ) Manager names.
- Laboratory name and location in which samples are to be relinquished.
- Requested due date.
- Signature of Field Technician (i.e. sampler) under the first 'relinquished by'.
- Signature of sample transferee.
- Date and time of sample transfers.
- Method of transport (ground courier, air carrier, sampler, etc.).
- Air Bill number (if applicable).

#### 4.2 Completing a Chain-of-Custody for Air Canisters

Lab deliverable contents (based on project needs).

- Canister serial # and size.
- Flow controller serial #.
- Initial and final vacuum measurement (in inches of mercury).
- Start and stop times that the canister was drawing sample.
- Total time calculated from the start and stop times.
- Matrix code.
- PID reading (indicate if ppm or ppb).
- Sample comments (if any).
- Identify the report deliverable contents and electronic data deliverable contents requested.

### 4.3 Completing a Chain-of Custody for Solid and Liquid Samples

- Sample start and stop depth (if applicable) and unit of measurement (meter, feet, inches, etc.).
- Information regarding whether to perform sample Matrix Spike (MS) and MS duplicate (MSD).
- Container preservative type (see "Preservative Code" on COC).
- Information regarding whether the sample was field filtered.
- Number of each container type and the total number of containers for the sample.
- Presence or absence of ice.

### 4.4 Distribution of the COC Pages

Page one (white copy) accompanies the sample shipment to the laboratory and page two (yellow copy) is the Field Document copy. The Field Technician must scan and email a copy to the Barr Data Management Administrator for filing on Barr's internal network project files. Alternatively, the yellow hardcopy may be routed to the Barr Data Management Administrator for electronic filing. This read-only electronic copy will be distributed to and available for use by the project team via Barr's internal network project file access.

## 5.0 Quality Control and Quality Assurance (QA/QC)

The Field Technician should review the COC for accurate and complete documentation.

## 6.0 Records

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Chain-of-Custody for Air Canisters
- Chain-of-Custody

A copy of the COC is provided to a Barr Data Management Administrator for storage on the internal Barr network files.

Additional records information can be found in Barr's "Records Management System Manual".

## 7.0 References

United States Environmental Protection Agency. 2002. *Guidance for Quality Assurance Project Plans*. EPA QA/G-5.



# Standard Operating Procedure Domestic Transport of Samples to Laboratories within the United States of America – States and Territories

Revision 3

February 27, 2020

Approved By:

Andrea Nord

Print

QA Manager

A handwritten signature in black ink, appearing to read "Andrea Nord", is written over a horizontal line.

Signature

02/27/20

Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

Initials: \_\_\_\_\_

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Date: \_\_\_\_\_

# Standard Operating Procedures for the Domestic Transport of Samples to the Laboratories within the United States of America – States and Territories

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures necessary for personal delivery or shipment of samples from locations within the United States of America (USA) and its territories to analytical laboratories located within the USA and its territories. This procedure applies to the transportation of ground and surface water, soil, wipe, sediment, paint chip, debris, air samples and their corresponding quality control samples to the appropriate laboratory. This SOP applies to samples that are classified as non-regulated, non-hazardous, or “Dangerous Goods in Excepted Quantities” samples prior to shipment.

Soil samples that are preserved with flammable chemicals (methanol) and unused sample vials containing flammable or corrosive chemical preservatives are examples of materials that are classified as “Dangerous Goods in Excepted Quantities”. Materials classified as Dangerous Goods in Excepted Quantities have limitations on the volume/weight of the material allowed in each shipment, and have additional packaging, labeling, and shipping requirements than non-regulated and non-hazardous samples and sampling media.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Maintaining proper sample temperatures (<6°C or ambient air temperature in accordance with the analytical method requirements) and delivering samples to the laboratory within 24 to 48 hours from collection are primary concerns.
- This procedure does not apply to the transportation of samples to laboratories outside of the USA and its territories.
- This procedure does not apply to samples that are classified as “hazardous” according to USDOT, PHMSA, and/or RCRA and must be packaged, labeled, and/or transported in accordance with USDOT’s hazardous materials regulations (49 CFR Parts 100-180).
- This procedure does not apply to samples that are classified as “dangerous goods” and must follow the International Air Transportation Association’s (IATA) dangerous goods regulations (DGR) for packaging, labeling, and/or air transport.

### 3.0 Responsibilities

The field technician(s) shall ensure the security, temperature, and packaging of environmental samples during transport and shipment.

### 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When samples may be contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of chemical preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

### 5.0 Equipment, Reagents, and Supplies

- Rigid cooler
- Heavy bag for containing ice and preventing leakage of melted water
- Ice
- Packing tape
- Dangerous Goods in Excepted Quantities Label with the number "8" added indicating the hazard class. This label must be used for coolers containing unused sample containers with corrosive preservative.
- Absorbent padding
- Bubble-wrap/bubble bags (inner packing material)
- Ziploc® baggies
- Shipping Airbill – if shipping via overnight commercial courier service
- Dangerous Goods in Excepted Quantities Label with the number "3" added indicating the hazard class. This label must be used for coolers containing methanol preservative
- Items listed in Section 8.0 Records

### 6.0 Procedure

#### 6.1 Packaging of non-regulated or non-hazardous samples requiring ambient air temperature per the analytical method of analysis

Sample matrices that do not require thermal preservation (ice) typically include wipe, paint chip, debris, and air samples. These samples may or may not require chemical preservatives depending upon the analytical method of analysis. The classification of "non-regulated" or "non-hazardous" in this context is based upon the nature of the sample prior to chemical preservation/fixation.

For samples that are stored at ambient air temperature, the samples will be placed in a jar, baggie or shipping carton (i.e. cooler, cardboard box, envelope) and accompanied with the proper COC.

Place the samples in a shipping carton in a manner that will avoid breakage. Fill out the chain-of-custody (COC) completely and include required copies with the samples. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Once the shipping carton is packed to prevent samples breaking, the COC is signed off and placed in the cooler or box. Adhere two to three strips of packaging tape from top to bottom on the cooler or box.

Custody seals must be adhered over the shipping carton lid or enclosure if project quality assurance plan or sampling and analysis plan require them. The custody seal must be adhered to the crack of the lid on two opposing sides of the cooler or over the flap(s) of the box or envelope to ensure the carton remained shut and the contents have not been tampered with during transit.

## **6.2 Packaging of non-regulated or non-hazardous samples requiring thermal preservation per the analytical method of analysis**

Samples matrices that require thermal preservation (ice) typically include water, soil and sediment samples. Glass containers should be packed in bubble wrap or other cushioning material to avoid breakage.

*Note: Bubble-wrap is the preferred packing material.*

Line a rigid plastic cooler (i.e. shipping container) with a strong plastic bag. This bag will serve as an outer liner and contain the wet ice, absorbent materials and sample containers.

Place samples and cushioning absorbent material inside the plastic bag and add enough absorbent padding to absorb the sample liquid within the package. Package ice in double-lined Ziploc® bags to ensure sample labels will not be compromised, and the cooler(s) will not leak melt water. Add enough ice to the cooler to maintain a constant temperature at  $\leq 6^{\circ}\text{C}$ , (but not frozen) until the samples arrive at the laboratory. Zip tie the plastic bag shut.

Before sealing cooler, fill out the COC completely and include required copies with the samples. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Adhere two to three strips of packaging tape on the cooler from top to bottom, and adhere an additional strip of tape covering the gap between the lid and sides of cooler to seal the cooler to avoid leakage.

Custody seals must be adhered on the cooler if project quality assurance plan or sampling and analysis plan require them. The custody seal must be adhered to the crack of the lid on two opposing sides of the cooler to ensure the contents have not been tampered with during transit.

Follow the labelling instructions in Section 6.4 of this SOP.



## 6.3 Packaging of samples classified as “Dangerous Goods in Excepted Quantities”

### 6.3.1 Soil Samples Preserved with Methanol (Flammable) – Hazard Class 3

Soil samples that are preserved with flammable chemicals (methanol) are an example of materials that are classified as hazard class “3” “Dangerous Goods in Excepted Quantities”.

Follow the packaging instructions listed in Section 6.2 of this SOP with the following addition: *Methanol sample containers must be placed in a Ziploc® Baggie to meet shipping requirements for preventing leakage.*

Each cooler shall not exceed 500 mL of methanol (50 vials, 10 mL of methanol per vial) and each vial shall not have more than 10 mL of methanol to meet the requirements of a Dangerous Goods in Excepted Quantities. A label with the hazard class number “3” indicates the cooler contains flammable (or reactive/oxidizer) materials (in this case a flammable methanol sample preservative). Additional labeling instructions are found in Section 6.4.2 of this SOP.

### 6.3.2 Unused Sample Jars – Hazard Class 3 (Flammable) and Hazard Class 8 (Corrosive) Chemicals

Unused sample vials containing flammable or corrosive chemical preservatives are examples of materials that are classified as “Dangerous Goods in Excepted Quantities”.

Follow the packaging instructions listed in Section 6.2 of this SOP with the following additions:

Each chemical, may have a limitation as to the volume or weight of the chemical and the number of inner containers (sample containers) allowed within each outer shipping container (cooler) to meet the requirements of a Dangerous Goods in Excepted Quantities. A label with the hazard class number “3” indicates the cooler contains flammable (or reactive/oxidizer) materials (in this case a flammable methanol sample preservative). A label with the hazard class number “8” indicates the cooler contains a corrosive material (in this case an acid or base sample preservative). Additional labeling instructions are found in Section 6.4.2 of this SOP.

## 6.4 Labeling of Outer Shipping Container or Carton

### 6.4.1 Shipping Label

Attach the shipping address label to the top of the cooler or to the cooler handle tag. Attach a second label with the same information should also be attached with packaging tape to the cooler in event that the original label is damaged or destroyed during sample shipment.

Directional arrow labels (Figure 1) must also be attached to the outside of the cooler according to the hazardous materials shipping regulations. Directional arrow labels indicate the upright position during sample shipment.



Figure 1 - Directional Arrows Label

#### 6.4.2 Dangerous Goods in Excepted Quantities Label

When shipping materials classified as Dangerous Goods in Excepted Quantities, the cooler must have a Dangerous Goods in Excepted Quantities Label (Figure 2). This label is placed on two opposing sides of the cooler. The label indicates the hazard class number and the name and address of the shipper or consignee. In cases where the package contents have more than one hazard class assigned, the primary (most hazardous) hazard class is listed on the label. Table 1 includes a Summary of United Nations Hazard Classes.



Figure 2 - Dangerous Goods in Excepted Quantities Label

Footnotes:

- (1) The "\*" must be replaced by the primary hazard class, or when assigned, the division of each of the hazardous materials contained in the package.
- (2) The "\*\*" must be replaced by the name of the shipper or consignee if not shown elsewhere on the package.

Table 1 – Summary of United Nations Hazard Classes

<b>Class 1</b>	Explosives
<b>Class 2</b>	Gases
<b>Class 3</b>	Flammable Liquids
<b>Class 4</b>	Flammable Solids; Substances Liable to Spontaneous Combustion; Substances Which, in Contact with Water, Emit Flammable Gases (e.g., soil sample contaminated with high concentrations of gasoline released from an underground storage tank)
<b>Class 5</b>	Oxidizing Substances and Organic Peroxide
<b>Class 6</b>	Toxic and Infectious Substances (e.g., samples of refuse collected from a solid waste landfill)
<b>Class 7</b>	Radioactive Material
<b>Class 8</b>	Corrosives (e.g., nitric acid used for preservation of some groundwater samples) (see Note)
<b>Class 9</b>	Miscellaneous Dangerous Goods

#### 6.4.2.1 Dangerous Goods Air Waybill Statement and Shippers Declaration

A shipping paper (i.e. bill of lading) is not required when offering the cooler for air transport via a commercial courier service (e.g. Federal Express or United Parcel Service).

A document such as an air waybill accompanies a shipment that is transported by aircraft. The air waybill must include the statement “Dangerous Goods in Excepted Quantities” and indicate the number of packages associated with each air waybill. This phrase is typically written behind the Barr project number in the PO or comments section on the air waybill.

A shipper’s declaration for dangerous goods is also required. Some air waybills also have a box you must also check off that says “Dangerous Goods no Shipper’s Declaration Required”.

### 6.5 Transport/Delivery Options

Account for the samples before shipping and compare to the COC. Refer to Barr’s SOP ‘Documentation on a Chain-of-Custody (COC) for further information. Ship samples during times when the laboratory will be able to accept and quickly analyze them. Whenever possible, select mode of transport/delivery to ensure delivery to the laboratory will occur with ample holding time remaining for the specified analytical methods required for the samples. Avoid sending samples during holidays and weekends. All Federal, State and Local shipping regulations must be met.

**Personal Delivery.** The samples are delivered to the laboratory by the field technician(s). The COC is signed and dated by the laboratory representative.

**Ground Transport.** The same procedures are followed as above; i.e., the COC is signed and dated and the top copy is sent with the samples. The cooler or box is then secured with packaging tape and a courier form is filled out for the designated laboratory. The cooler or box is then left in the services area for pickup via ground transport and delivery.

**Air Transport.** Follow the procedures above, replacing the courier form with the overnight courier air bill via Federal Express or United Parcel Service, for example. Include the date, project number, type of

delivery service desired, parcel weight, number of coolers or boxes on the air bill. Also include the phrase "Dangerous Goods in Excepted Quantities", when applicable.

## 7.0 Quality Control and Quality Assurance (QA/QC)

Not Applicable.

## 8.0 Records

Examples of common field documentation are available in Barr's "Compendium of Field Documentation".

Field documentation specific to this SOP are listed below:

- Chain-of-custody (COC)
- Custody seal (if applicable)
- Dangerous Goods in Excepted Quantities Label
- Directional Arrow Label

COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: documentation on a COC.

## 9.0 References

49 CFR Part 173.4a – Excepted Quantities October 1, 2011 Online

<https://www.govinfo.gov/app/details/CFR-2011-title49-vol2/CFR-2011-title49-vol2-sec173-4>

*ASTM International. 2015. ASTM Method D6911 – 15 Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis<sup>1</sup>. ASTM January 15, 2015.*





# Standard Operating Procedure

## Field Screening Soil Samples

Revision 8

April 9, 2019

Approved By:

<u>John W. Juntilla</u>		<u>04/09/19</u>
Print	Technical Reviewer Signature	Date
<u>Terri A. Olson</u>		<u>04/09/19</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Field Screening of Soil Samples

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedure for properly screening soil or sediment samples in the field. This procedure applies to field technicians responsible for field screening soil or sediment samples.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Screening techniques can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance, if applicable.
- Interferences on the test can be caused by any contaminant that can cause an oil sheen on water. The samples will be carefully observed for characteristic appearance or odors which may indicate a possible contaminant other than coal tar or petroleum substances.
- Sunlight and low temperatures may interfere with headspace development.
- Water and soil particles may interfere with PID and FID measurements.
- Decontamination of screening equipment is required to prevent cross-contamination.
- Contact the local one call system prior to digging to have public utilities identified at sampling locations. Privately owned underground utilities, if present, typically will not be identified by the one call system and contracting with a private utility locator may be necessary.

## 3.0 Responsibilities

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, SAP, etc.).

Experienced Field Technicians are responsible for the proper sample identification, field screening procedures, field equipment and calibration, quality control procedures, and documentation.

Equipment Technicians are responsible for maintaining equipment in working order and aiding in troubleshooting equipment issues.

The role of the Field Safety Representative is to oversee on-site safety activities.

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent

sample contact with the skin and eyes. When screening soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies

- Photoionization detector (PID)
- Flame ionization detector (FID)
- Squirt bottle with tap water
- Waterproof ink pen or pencil
- Polyethylene bags
- Chemical resistant gloves (e.g., nitrile)
- Stainless steel spoon
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

## 6.0 Procedure

The field screening techniques for soils are as follows: visual examination, odor, headspace organic vapor screening, and oil sheen. The results of these four screening procedures may be used to screen soil samples for possible contamination.

### 6.1 Calibration

The PID or FID shall be calibrated or checked against a known concentration of a calibration gas standard prior to collection of field measurements. Calibration of the PID or FID shall follow the recommended procedures as described in the manufacturer's operation manual or as per the applicable Barr SOP.

Regular calibration checks (bump tests) are expected to be performed by the field technician a minimum of once per day of use in the field. It is recommended that bump tests be conducted around mid-day and at the end of the day. More frequent bump testing may be completed if warranted by field conditions. The bump testing results should be recorded in the field log book or field log data sheets.

If problems occur during calibration, during bump tests, or if the unit will not stay calibrated, the field technician should document the issue in the field notes then contact the equipment technician or project manager for assistance.

### 6.2 Screening Techniques

The field screening techniques for soils are as follows: visual examination, odor, headspace organic vapor screening, and oil sheen. The results of these four screening procedures may be used to screen soil samples for possible contamination. To prevent sample cross-contamination, the screening equipment is carefully cleaned before and after working with each sample per Barr's SOP 'Decontamination of Sampling Equipment'.

#### 6.2.1 Visual Examination

A visual examination of the soil sample will include noting any discoloration of the soil or visible oiliness or tar.

#### 6.2.2 Odor

The field technician will note odor only if noticed incidentally while handling the soil sample. Field technicians will not unduly expose themselves to sample odors. Odor will be described as trace, light, moderate, or strong, and appropriate description of the type of odor, if evident.

### 6.2.3 Headspace Organic Vapor Screening

The polyethylene bag headspace method recommended by the Minnesota Pollution Control Agency will be used in the field to screen soils suspected to contain volatile organic compounds. The screening method is intended to be used in conjunction with other “real time” observations.

The following equipment is required to conduct headspace organic vapor screening: PID or FID, polyethylene bag, log book or record sheet, and appropriate PPE. Soil samples collected from a split-barrel sampler or a direct-push (i.e., Geoprobe) sample liner will be collected immediately after opening the barrel or liner. If the sample is collected from an excavation wall, soil pile, or backhoe bucket, it will be collected from a freshly exposed surface.

- Half-fill the bag with the sample to be analyzed using a stainless-steel spoon or a gloved hand and immediately seal it. Agitate the bag for 15 seconds and manually break up any soil clumps within the bag.
- Allow headspace development for approximately 10 minutes. The sample should be kept in a shaded area out of direct sunlight. Ambient temperatures during headspace development should be recorded. When ambient temperatures are below 50°F, headspace development should be conducted inside a heated vehicle or building. After completing the headspace development, agitate the bag for an additional 15 seconds.
- Quickly puncture the bag with the sampling probe of the PID or FID at a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particles.
- Record the highest PID or FID meter response as the headspace concentration. The maximum response will likely occur between 0 to 5 seconds.
- When using a FID, it may be necessary to correct for methane. In this case, take a reading first with the carbon filter, then without. This will require two duplicate bag samples. The second reading less the first is the headspace adjusted for methane. Adjusted readings less than zero are considered zero. Methane correction is not necessary if a PID is used.

### 6.2.4 Oil Sheen Test

The oil sheen or hydrocarbon test is a method used to immediately determine the approximate magnitude of coal tar or petroleum contamination in soil by observation of the sample in the field. The test is useful in soils which do not have a high binding capacity with petroleum compounds or polycyclic aromatic hydrocarbons (PAHs) (i.e., petroleum compounds or PAHs are free on the surface of the soil particles and can be released by a stream of water).

The equipment required to conduct the oil sheen test includes: a stainless-steel spoon, a squirt bottle filled with tap water, a log book or field log data sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan.

The procedure for conducting the oil sheen test consists of obtaining approximately 50 grams (about 30 cc) of representative soil with the spoon and then directing a stream of water onto the soil in the spoon with the squirt bottle until the soil is saturated and water begins to collect around the soil. The amount of oil sheen present on the water is determined by observation and the results of the test are reported as a magnitude of oil sheen observed: none, trace, light, moderate, heavy or rainbow. The test results, sample location, and observations of the sample's appearance and odor are recorded in the log book or field log data sheet.



The specific soil types at the area of investigation should be accounted for when performing the oil sheen test. The best results are obtained in silts, sands, and/or gravels with low organic content. The results obtained from clay soils may appear deceptively low. Typical descriptions of each test result are provided in the table below.

<b>Oil Sheen Test Result</b>	<b>Description</b>
None	No sheen detected.
Trace	Possible or faint oil sheen observed (may not continue to generate sheen as additional water is added).
Light	Obvious sheen that may not cover entire water surface
Moderate	Definite oil sheen that covers entire surface, but "rainbow colors" not distinguishable.
Heavy	Definite oil film or product that does not display rainbow colors.
Rainbow	Definite oil sheen, film or product that displays rainbow colors.

### 6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

### 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

Field background readings are measured for the headspace organic vapor screening. PID and FID readings should be duplicated every 20 field samples.

## 8.0 Records

The field technician(s) will document the field screening activities and measurements in a project dedicated field logbook or on field log data sheets.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Field Sampling Report
- Field Log Data Sheet

Field documentation are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual."

Other Barr SOP subjects referenced within this SOP: PID and FID equipment, decontamination of sampling equipment, and investigative derived waste.

## 9.0 References

PID and FID operation manuals.





# Standard Operating Procedure Monitoring Well Development Oversight

Revision 6

October 22, 2019

Approved By:

<u>John W. Juntilla</u>		<u>10/22/19</u>
Print	Technical Reviewer Signature	Date
<u>Terri A. Olson</u>		<u>10/22/19</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Monitoring Well Development Oversight

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe oversight provided on monitoring well development or redevelopment. These procedures are performed with the objective of obtaining representative groundwater information and water quality samples from aquifers.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Well development should be completed by an appropriately licensed or registered well contractor unless allowed by rules governing wells and borings.
- Best practice is to have a minimum of one week pass between monitoring well development and monitoring well sampling unless there are other project requirements.
- If well will be sampled for per- and polyfluorinated alkyl substances (PFAS), special consideration must be taken to avoid accidental contamination of the well during the development process - see Barr's SOP 'Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples'.

## 3.0 Responsibilities

Experienced Field Technicians are responsible for overseeing the well development, quality control procedures, and documentation.

The role of the Field Safety Representative is to oversee on-site safety activities.

The well drilling contractors are typically responsible for the development of monitoring wells at the time of installation and have the necessary tools, equipment, chemicals, applicable licenses or registrations that may be required to perform the development work. Successful development of a new well may be a requirement of the drilling specifications.

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When working with liquids contaminated with corrosive materials, emergency eye flushing facilities should be available.

## 5.0 Equipment, Reagents, and Supplies

- Pumps^ (e.g., submersible or peristaltic)
- Pump discharge hose/tubing
- Chemical resistant gloves (e.g., nitrile)
- Surge block (optional)
- Turbidimeter (optional)
- Water level indicator or interface probe
- Bailers
- Water quality meter (optional)
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

\* See Barr's PFAS SOP for a list of prohibited and acceptable items.

## 6.0 Procedure

These procedures are used to remove the fine-grained materials from a well or well bore as a result of boring or well construction. Monitoring wells must be developed to provide water free of suspended solids and to yield representative samples. Well development should result in a well that yields visibly clear groundwater.

### 6.1 Calibration

If used, the water quality meter and turbidimeter will be calibrated as per the applicable Barr SOP. The meters will undergo calibration checks, at a minimum, before and after sampling. The calibration check will be documented on a calibration form (as appropriate) and/or in the field notebook. Any significant issues found during the calibration check will be noted in the field notebook and the Equipment Technicians will be notified.

### 6.2 Development

Successful development methods include bailing, surging, pumping/over-pumping, and jetting with water. The basic principle behind each method is to create reversals of water flow into and out of the well screen (and/or bore hole) to break-down any potential mud cake or disturbed zones where fine-grained particles may be concentrated at the borehole-formation interface, and to draw the finer materials into the well or borehole for removal. This process also helps remove fine fraction formation materials in proximity to the borehole wall, leaving behind a "natural" pack of coarser-grained materials.

#### 6.2.1 Bailing

In relatively clean, permeable formations where water flows freely into the borehole, bailing is an effective development technique. Let the bailer fall down the well until it strikes the surface of the groundwater which produces an outward surge. Rapidly withdraw the bailer to create a drawdown and/or after the bailer hits the groundwater lower it to the bottom of the well and agitate it with rapid short strokes. Continue bailing with repeated up and down "surging motions" until water bailed from the well is free from suspended particles.

*Note: During this process, if the well goes dry, stop bailing and let the well recharge before continuing.*

#### 6.2.2 Surge Block

A surge block is a tool used to break up bridging of fine grained material by inducing agitation and inducing flow into and out of the well and aquifer formation. Bridging is the tendency for particles moving towards a well under unidirectional flow (pumping) to develop a blockage that restricts subsequent

particles to move into a well. Surge block is used alternately with either a pump or bailer. Let the surge block fall down the well until it strikes the groundwater surface. This creates a vigorous outward surge; rapidly retrieve the surge block. Lower the surge block to the top of the well intake and begin a pumping action with a typical stroke of approximately 3 feet and gradually work downward through the screened interval. Remove the surge block at regular intervals to discard the loosened suspended particles by either bailing or pumping. Continue the cycle of surging/bailing/pumping until satisfactory development has been attained.

### 6.2.3 Pumping/Over-pumping

In both pumping techniques, the groundwater flow is induced to flow into the well and the fine particulate material moves into the well and is discharged by the pump. In the case of over-pumping, the pump is operated at a capacity that substantially exceeds the ability of the formation to deliver water. Once pumping has begun, start the surging action by lowering and raising the hose/pumping apparatus through the screened interval. Bailing or bailing and surging may be combined with pumping for efficient well development. Continue pumping until such time as satisfactory development has been attained based on field observation of visibly clear water produced. If an analytical measure is needed, use turbidity meter readings to document initial turbidity and final turbidity readings. Well stabilization parameters may also be measured and documented pre- and post-development.

If pumping/over-pumping is completed by air lifting, the air compressor must be of an oil-less type or fitted with an oil trap capable of removing compressor oil from the air stream to avoid contaminating the well or boring.

*Note: The types of pumps used are described in Barr's SOPs 'Collection of Groundwater Samples from a Temporary or Permanent Monitoring Well (Includes Well Purging and Stabilization)' or 'Collection of Groundwater Samples using Low-Flow Purging and Sampling'.*

### 6.2.4 High Velocity Jetting

Development by high velocity jetting may be completed with either water or air. In practice, jetting with water is typically followed by or simultaneously occurring air-lift pumping/over pumping to remove the fine materials. The jetting procedure consists of operating a horizontal water jet(s) inside of the well screen so high velocity streams of water shoot through the screen openings into the sand pack/formation. The jetting tool is worked similar to a surge block. The jetting tool ideally will have four openings located 90 degrees apart and should be worked up and down the screened interval while being rotated. At a minimum, the amount of water introduced during jetting and, if feasible, an additional 10 well volumes of water should be purged from the well.

## 6.3 Data Reduction/Calculations

The calculations for well volume and volume of water to be purged are included in Barr's SOP 'Collection of Groundwater Samples from a Temporary or Permanent Monitoring Well (Includes Well Purging and Stabilization)'.

## 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

QA/QC objectives (e.g., turbidity, well recovery rate, water quality parameters) are specific to each project and/or well. Discuss QA/QC procedures with the project team prior to well development.

## 8.0 Records

The field technician(s) will document the method of development, any deviations from this SOP, volume of water purged, and any volume of water introduced to the well (e.g., high velocity jetting, flushing).

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Field Log Cover Sheet
- Field Log Data Sheet

The field documents are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: water quality meter, turbidimeter, well recovery rate testing, collection of PFAS samples, decontamination of sampling equipment, groundwater purging/sampling, low-flow purging/sampling, and investigative derived waste.

## 9.0 References

American Society for Testing and Materials (ASTM), D5521/D5521M-13. 2013. *Standard Guide for Development of Groundwater Monitoring Wells in Granular Aquifers*.

Environmental Protection Agency, Offices of Waste Programs Enforcement and Solid Waste and Emergency Response. 1986. *RCRA Ground-Water Monitoring Technical Enforcement Document*.

Johnson Filtration Systems. 1986. *Groundwater and Wells*.

National Water Well Association. *Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells*.

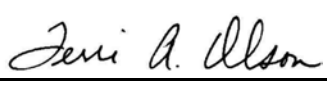


**Standard Operating Procedure**  
**Routine Level Semivolatile Organic Compounds (SVOC),**  
**Polycyclic Aromatic Hydrocarbons (PAH), Diesel Range**  
**Organics (DRO), and Total Petroleum Hydrocarbons (TPH)**  
**Data Evaluation**

Revision 7

January 2, 2020

Approved By:

<u>Marta Nelson</u>		<u>01/02/20</u>
Print	Technical Reviewer    Signature	Date
<u>Terri Olson</u>		<u>01/02/20</u>
Print	QA Manager    Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Routine Level Semivolatile Organic Compounds (SVOC), Polycyclic Aromatic Hydrocarbons (PAH), Diesel Range Organics (DRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

## 1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of semivolatile organic compounds data provided by laboratories to be used in Barr Engineering Co. (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine SVOC (including PAHs and phenols), TPH at various carbon ranges (e.g., TPH as fuel oil, TPH as motor oil, TPH as jet fuel), and DRO data evaluation for analyses by the following technologies:

- Gas Chromatography/Flame Ionization Detector (GC/FID)
  - Method examples: EPA 8015, EPA 8100, WI DRO
- Gas Chromatography/Mass Spectrometry (GC/MS)
  - Method example: EPA 625, EPA 8270
- Gas Chromatography/Mass Spectrometry-Selective Ion Monitoring (GC/MS-SIM)
  - Method example: EPA 8270
- High Performance Liquid Chromatography (HPLC)
  - Method example: EPA 610, EPA 8310
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

The letter indicator for the various EPA method revisions have been intentional omitted. Multiple versions of the approved methods would be applicable for review under this SOP. In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review. Laboratories may not provide all the review elements in this SOP, review only those that are provided.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.



### 3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

### 4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

#### 4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136, WI DRO method, and the Test Methods for Evaluating Solid Waste (SW-846) are used as guidance for the recommended holding time and preservation acceptance criteria listed in Table 1.

Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
SVOC/PAH/TPH	Aqueous	≤ 6° C	Ice	7 days extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6° C	Ice	14 days extraction/ addl. 40 days analysis
DRO	Aqueous	≤ 6° C	Ice, HCl < 2 pH; sodium azide for carbonate aquifer	7 days extraction/47 days collection to analysis 48 hours if not HCl or sodium azide preserved
	Sediment/Soil	≤ 6° C	Ice	10 days solvent addition/ 47 days collection to extraction and analysis
TCLP SVOC	Various	--	NA	14 days TCLP extraction/7 days extraction/addl. 40 days analysis

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an 'H' ("Recommended sample preservation, extraction or analysis holding time was exceeded."). Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subject to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

## 4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG). Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination	
Sample Result	Recommended Action for Associated Data
Non-detect	No action required
< 5x blank concentration	Qualify with 'UB'
≥ 5x blank concentration	Use professional judgment

UB = The analyte is detected in one of the associated laboratory, equipment, field or trip blank samples and is considered non-detect at the concentration reported by the laboratory.

*Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)*

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with 'J' ("Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.") or 'R' ("The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.").

### 4.3 Deuterated Monitoring Compounds (DMC) and Surrogates

DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are only used for the SVOC GC/MS analysis. Table 3 presents the recommended DMCs with their associated target compounds.

Table 3 – DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
<i>2,4-Dichlorophenol-d<sub>3</sub></i>	2,4-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol	2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol
<i>2-Chlorophenol-d<sub>4</sub></i>	2-Chlorophenol	
<i>2-Nitrophenol-d<sub>4</sub></i>	Isophorone	2-Nitrophenol
<i>4-6-Dinitro-2-methylphenol-d<sub>2</sub></i>	4,6-Dinitro-2-methylphenol	
<i>4-Chloroaniline-d<sub>4</sub></i>	4-Chloroaniline Hexachlorocyclopentadiene	3,3'-Dichlorobenzidine
<i>4-Methylphenol-d<sub>8</sub></i>	2-Methylphenol 4-Methylphenol	2,4-Dimethylphenol
<i>4-Nitrophenol-d<sub>4</sub></i>	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol	4-Nitrophenol 4-Nitroaniline
<i>Acenaphthylene-d<sub>8</sub></i>	Naphthalene 2-Methylnaphthalene 2-Chloronaphthalene	Acenaphthylene Acenaphthene
<i>Anthracene-d<sub>10</sub></i>	Hexachlorobenzene Atrazine	Phenanthrene Anthracene
<i>Benzo(a)pyrene-d<sub>12</sub></i>	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
<i>Bis-(2-chloroethyl) ether-d<sub>8</sub></i>	Bis-(2-chloroethyl) ether 2,2'-oxybis(1-chloropropane)*	bis(2-Chloroethoxy) methane
<i>Dimethylphthalate-d<sub>6</sub></i>	Caprolactum 1,1'-Biphenyl Dimethylphthalate Diethylphthalate	Di-n-butylphthalate Butylbenzylphthalate bis(2-ethylhexyl)phthalate Di-n-octylphthalate
<i>Fluorene-d<sub>10</sub></i>	Dibenzofuran Fluorene 4-Chlorophenyl-phenylether	4-Bromophenyl-phenylether Carbazole

(Table 3 continued on next page)

Table 3 – DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
<i>Nitrobenzene-d<sub>5</sub></i>	Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene	2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine
<i>Phenol-d<sub>5</sub></i>	Benzaldehyde	Phenol
<i>Pyrene-d<sub>10</sub></i>	Fluoranthrene Pyrene	Benzo(a)anthracene Chrysene
SIM DMC and Associated Target Compounds		
<i>Fluoranthene-d<sub>10</sub></i>	Fluoranthene Pyrene Benzo(a)anthracene Chrysene Benzo(b)fluoranthene	Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
<i>2-Methylnaphthalene-d<sub>10</sub></i>	Naphthalene 2-Methylnaphthalene Acenaphthylene Acenaphthene	Fluorene Pentachlorophenol Phenanthrene Anthracene

\* = Chemical name changed by Integrated Risk Information System (IRIS) on November 30, 2007 from Bis(2-chloroisopropyl)ether to Bis(2-chloro-1-methylethyl)ether (common name). 2,2'-oxybis(1-chloropropane) is CAS index name.

Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other DMC or surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. All samples (blanks, spiked samples, project samples, QC samples) should contain DMC or surrogates. If a sample does not contain DMC or surrogates or the method does not require surrogates (WI DRO), professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of DMC or surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each DMC or surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Surrogates are not required for some methods (e.g., WI DRO). If used for WI DRO, the method requires that the surrogates must not elute within the WI DRO window (C<sub>10</sub>-C<sub>28</sub>). If the laboratory report includes a surrogate spike recovery for WI DRO, use professional judgment to assess the data.

Table 4 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

<b>Table 4 – Guidelines for Single DMC or Surrogate</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
%R > Upper Limit	Qualify with 'J+'	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
%R within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.  
The analyte may or may not be present in the sample.

Table 5 includes guidance where multiple surrogates are analyzed per analytical fraction.

<b>Table 5 – Guidelines for Multiple DMC or Surrogates</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with 'J+'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.  
The analyte may or may not be present in the sample.

#### 4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (20 or less samples of the same matrix - WI DRO requires an additional LCSD analyzed at the end of 20 samples).
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision

(when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

<b>Table 6 – Guidelines for Laboratory Control Samples</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
%R > Upper Limit	Qualify with 'J+' or use professional judgment	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
RPD > Upper Limit	Qualify with 'J' or use professional judgment	
%R and RPD within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

#### 4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Ideally, blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The MS/MSD duplicate pairs may be substituted for laboratory duplicates. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with U, UB, <, or R. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

<b>Table 7 – Guidelines for Laboratory Duplicates</b>	
<b>% RPD</b>	<b>Recommended Action for Associated Data</b>
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with 'J'

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

#### 4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with U, UB, <, or R. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs  $\leq 30\%$  for aqueous samples and  $\leq 40\%$  for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times ( $>5x$ ) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times ( $>5x$ ) the RL, professional judgment should be used to determine if qualification is appropriate.

<b>Table 8 – Guidelines for Field Duplicates</b>	
<b>% RPD</b>	<b>Recommended Action for Associated Data</b>
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with 'J'

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

#### 4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to DRO in the WI method)

- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added ( $>4x$ ), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

<b>Table 9 – Guidelines for Matrix Spikes</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
%R > Upper Limit	Qualify with 'J+' or use professional judgment	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
RPD > Upper Limit	Qualify with 'J' or use professional judgment	
%R and RPD within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

#### 4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately



documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

## 5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data evaluation may include the completion of a Routine Level Quality Control Report. This may be a report produced via EQuIS DQM (Environmental Quality Information System Data Quality Module) or a hardcopy as found in Barr's "Compendium of Data Quality Assessment Documentation". Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented. If using EQuIS DQM, reason codes will also be applied. The reason codes are defined in the software. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

## 6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information. Documentation of the data evaluation may include but is not limited to an email to the project team, data evaluation summary report, technical memo, or section within a project report.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

## 7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3.*

Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review.*

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

## Attachment 1

### Revision History

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 10
		Attachments	Added Attachment 3
3.2	04/2011	Document Wide	Added analytical methods to applicability section.
		Attachments	Updated Attachment 1 and 2 to include current forms.
4.0	04/06/12	Document Wide	Major revision
5.0	06/17/13	Cover page	Added Calgary office
		I	Added waste rock and drill cores to examples of product sample
		III, IV, V, VI, VII	Added 'project specific requirements' as possible criteria source
		VI	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		VI	Clarified field duplicate criteria as < one value and not a range
		IX	Added statement regarding multiple qualifiers
6	01/19/16	Document Wide	SOP restructuring, new format
7	01/02/20	Document Wide	Updated for new qualifiers

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# Standard Operating Procedure

## Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

Revision 7

January 2, 2020

Approved By:

<u>Michael Dupay</u>		<u>01/02/20</u>
Print	Technical Reviewer Signature	Date
<u>Terri Olson</u>		<u>01/02/20</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

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# Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

## 1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of VOC, GRO, and TPH data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine VOC (including BTEX), GRO, and TPH (in the approximate gasoline carbon range, C<sub>6</sub>-C<sub>10</sub>) data evaluation for analyses by the following technologies:

- Gas Chromatography/Flame Ionization Detector (GC/FID)
  - Method examples: EPA 8015, WI GRO (GRO)
- Gas Chromatography/Photoionization Detector (GC/PID)
  - Method example: EPA 8021, WI GRO (PVOC)
- Gas Chromatography/Electrolytic Conductivity Detector (GC/ELCD)
  - Method example: EPA 8021
- Gas Chromatography/Mass Spectrometry (GC/MS)
  - Method example: EPA 624, EPA 8260
- Gas Chromatography/Mass Spectrometry-Selective Ion Monitoring (GC/MS-SIM)
  - Method example: EPA 8260
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

The letter indicator for the various EPA method revisions have been intentional omitted. Multiple versions of the approved methods would be applicable for review under this SOP. In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review. Laboratories may not provide all the review elements in this SOP, review only those that are provided.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

### 3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

### 4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report evaluation. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

#### 4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136, WI GRO method, and the Test Methods for Evaluating Solid Waste (SW-846) are used as guidance for the recommended holding time and preservation acceptance criteria listed in Table 1.

Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
VOC/PVOC	Aqueous	≤ 6 °C	HCl < 2 pH	14 days
	Aqueous	≤ 6 °C	Unpreserved	7 days
	Sediment/Soil	≤ 6 °C	1:1 soil:solvent (e.g., 10 g soil:10 mL MeOH in lab pre-weighed vial)	14 days
GRO (WI Method)	Aqueous	≤ 6 °C	HCl < 2 pH	14 days
	Sediment/Soil	≤ 6 °C	1:1 soil:solvent (e.g., 10 g soil:10 mL MeOH in lab pre-weighed vial)	21 days

(Table 1 continued on next page)

Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
TPH	Aqueous	≤ 6 °C	HCl or H <sub>2</sub> SO <sub>4</sub> < 2 pH	7 day extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6 °C	Zero headspace*	14 days extraction/ addl. 40 days analysis
TCLP	Various	≤ 6 °C	No preservative	14 days TCLP extraction/ addl. 14 days analysis

\* = Alternatively, samples may be collected as per the VOC analysis.

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an 'H' ("Recommended sample preservation, extraction or analysis holding time was exceeded."). Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subject to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

## 4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG) – laboratories should analyze a method blank at least once every 12 hours. Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Trip blanks should be placed in each transport cooler containing VOC sample containers prior to shipment into the field and remain with the associated VOC samples submitted to the laboratory for VOC analysis; including sample storage through analysis.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination	
Sample Result	Recommended Action for Associated Data
Non-detect	No action required
< 5x blank concentration	Qualify with 'UB'
≥ 5x blank concentration	Use professional judgment

UB = The analyte is detected in one of the associated laboratory, equipment, field or trip blank samples and is considered non-detect at the concentration reported by the laboratory.

*Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)*

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with 'J' ("Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.") or 'R' ("The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.").

#### 4.3 Deuterated Monitoring Compounds (DMC) and Surrogates

DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are only used for the VOC GC/MS analysis. Table 3 presents the recommended DMCs with their associated target compounds.

Table 3 –DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
1,1,2,2-Tetrachloroethane- <i>d</i> <sub>2</sub>	1,1,2,2-Tetrachloroethane	1,2-Dibromo-3-chloropropane
1,1-Dichloroethane- <i>d</i> <sub>2</sub>	trans-1,2-Dichloroethene 1,1-Dichloroethene	cis-1,2-Dichloroethene
1,2-Dichlorobenzene- <i>d</i> <sub>4</sub>	Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene	1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene
1,2-Dichloroethane- <i>d</i> <sub>4</sub>	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl-tert-butyl ether	1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
1,2-Dichloropropane- <i>d</i> <sub>6</sub>	Cyclohexane Methylcyclohexane	1,2-Dichloropropane Bromodichloromethane
1,4-Dioxane- <i>d</i> <sub>8</sub>	1,4-Dioxane	
2-Butanone- <i>d</i> <sub>5</sub>	Acetone	2-Butanone
2-Hexanone- <i>d</i> <sub>5</sub>	4-Methyl-2-pentanone	2-Hexanone
Benzene- <i>d</i> <sub>6</sub>	Benzene	
Chloroethane- <i>d</i> <sub>5</sub>	Dichlorodifluoromethane Chloromethane Bromomethane	Chloroethane Carbon disulfide
Chloroform- <i>d</i>	1,1-Dichloroethane Bromochloromethane Chloroform	Dibromochloromethane Bromoform
Toluene- <i>d</i> <sub>8</sub>	Trichloroethene Toluene Tetrachloroethene Ethylbenzene	o-Xylene m,p-Xylene Styrene Isopropylbenzene
trans-1,3-Dichloropropene- <i>d</i> <sub>4</sub>	cis-1,3-Dichloropropene trans-1,3-Dichloropropene	1,1,2-Trichloroethane
Vinyl Chloride- <i>d</i> <sub>3</sub>	Vinyl chloride	

Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other DMCs or surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. All samples (blanks, spiked samples, project samples, QC samples) should contain DMCs or surrogates. If a sample does not contain DMC or surrogates or the method does not require surrogates (e.g., WI GRO), professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of the DMC or surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each DMC or surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

While not required for WI GRO analyses, surrogates are required for PVOC. The method minimum surrogate recovery is 80%; there is no method maximum recovery. Use professional judgment when evaluating surrogates for WI GRO samples.

Table 4 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

<b>Table 4 – Guidelines for Single DMC or Surrogate</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
%R > Upper Limit	Qualify with 'J+'	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
%R within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.  
The analyte may or may not be present in the sample.

Table 5 includes guidance where multiple surrogates are analyzed per analytical fraction.

<b>Table 5 – Guidelines for Multiple DMC or Surrogates</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with 'J+'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.  
The analyte may or may not be present in the sample.



#### 4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (typically 20 or less samples of the same matrix - WI GRO requires an additional LCSD analyzed at the end of 20 samples)
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 6 – Guidelines for Laboratory Control Samples		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R > Upper Limit	Qualify with 'J+' or use professional judgment	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
RPD > Upper Limit	Qualify with 'J' or use professional judgment	
%R and RPD within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

#### 4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Ideally, blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The MS/MSD duplicate pairs may be substituted for laboratory duplicates. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with U, UB, <, or R. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times ( $>5x$ ) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times ( $>5x$ ) the RL, professional judgment should be used to determine if qualification is appropriate.

<b>Table 7 – Guidelines for Laboratory Duplicates</b>	
<b>% RPD</b>	<b>Recommended Action for Associated Data</b>
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with 'J'

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

#### 4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with U, UB, <, or R. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs  $\leq 30\%$  for aqueous samples and  $\leq 40\%$  for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times ( $>5x$ ) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times ( $>5x$ ) the RL, professional judgment should be used to determine if qualification is appropriate.

<b>Table 8 – Guidelines for Field Duplicates</b>	
<b>% RPD</b>	<b>Recommended Action for Associated Data</b>
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with 'J'

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

#### 4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to GRO in the WI method)
- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added ( $>4x$ ), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

Table 9 – Guidelines for Matrix Spikes		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R > Upper Limit	Qualify with 'J+' or use professional judgment	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
RPD > Upper Limit	Qualify with 'J' or use professional judgment	
%R and RPD within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

#### 4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

#### 5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data evaluation may include the completion of a Routine Level Quality Control Report. This may be a report produced via EQuIS DQM (Environmental Quality Information System Data Quality Module) or a hardcopy as found in Barr's "Compendium of Data Quality Assessment Documentation". Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented. If using EQuIS DQM, reason codes will also be applied. The reason codes are defined in the software. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

#### 6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information. Documentation of the data evaluation may include but is not limited to an email to the project team, data evaluation summary report, technical memo, or section within a project report.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

#### 7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3.*

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Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review*.

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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## Attachment 1

### Revision History

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 10
		Attachments	Added Attachment 3
3.2	04/2011	Document Wide	Added analytical methods to applicability section.
		Attachments	Updated Attachment 1 and 2 to include current forms.
4.0	04/06/12	Document Wide	Major revision
5.0	06/17/13	Cover page	Added Calgary office
		I	Added waste rock and drill cores to examples of product sample
		III, IV, V, VI, VII	Added 'project specific requirements' as possible criteria source
		VI	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		VI	Clarified field duplicate criteria as < one value and not a range
		IX	Added statement regarding multiple qualifiers
6	01/15/16	Document Wide	SOP restructuring, new format
7	01/02/20	Document Wide	Updated for new qualifiers




# Standard Operating Procedure

## Decontamination of Sampling Equipment

Revision 2

October 6, 2020

Approved By:

<u>John W. Juntilla</u>		<u>10/06/20</u>
Print	Technical Reviewer    Signature	Date
<u>Michael Dupay</u>		<u>10/06/20</u>
Print	QA Manager    Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Decontamination of Sampling Equipment

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to define the process used for decontaminating environmental sampling-related equipment including pumps, meters, and materials coming into contact with actual sampling equipment or with sampling personnel. This procedure is applicable to all personnel who are collecting samples and/or decontaminating sampling and field equipment.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Equipment used once and discarded such as bailers, protective gear, and filtration devices are not part of this SOP.

## 3.0 Responsibilities

The equipment technician is responsible for ensuring field equipment has been thoroughly decontaminated and prepared for use out in the field. The field technician(s) are responsible for decontamination in the field at each individual sampling point and for ensuring adherence to any investigative derived waste (IDW) project-specific requirements set forth in a QAPP or SAP (if applicable).

The role of the Field Safety Representative is to oversee on-site safety activities.

## 4.0 Safety

Barr staff is responsible for implementing aspects of the job safely. Where available, refer to the appropriate Project Health and Safety Plan (PHASP) to determine the proper personal protection equipment (PPE) required when using this SOP. Barr staff is responsible for conducting all aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protection equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When sampling soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.



## 5.0 Equipment, Reagents, and Supplies

- Non-phosphorus detergent (e.g., Liquinox™)
- Scrub brush made of inert materials
- Oven
- Bucket
- Tap water
- Analyte-free water (e.g., distilled or deionized (DI) water, or equivalent)
- Kimwipes®, or equivalent
- Chemical resistant gloves (e.g., nitrile)
- Spray bottle
- Organic solvent (e.g. methanol)

## 6.0 Procedure

This section describes the procedure(s) for the decontamination of equipment used to sample water, soil, or air.

### 6.1 Calibration

Calibration is not applicable to this SOP.

### 6.2 Operation

Decontamination of sampling equipment will be performed before sampling and after working at each sampling point, if applicable.

#### 6.2.1 Water Sampling Equipment

Equipment that does not contact sample water or the inside of the well should be rinsed with analyte-free water and inspected for remaining particles or surface film. If these are noted, repeat cleaning and rinse procedures.

Equipment that contacts sample water or the inside of the well should be cleaned (inside and outside where possible) with a non-phosphorus detergent solution applied with a spray bottle and/or scrub brush (if needed). Rinse with analyte-free water and containerize with other IDW if required by the SAP or QAPP and inspect for remaining particles or surface film. If these are noted, repeat cleaning and rinse procedures. Shake off remaining water and allow to air dry.

The internal surfaces of pumps and tubing that cannot be adequately cleaned by the above methods alone will also be cleaned by first circulating a non-phosphorus detergent solution through them followed by circulating analyte-free water. Special care will be exercised to ensure that the "rinse" fluids will be circulated in sufficient quantities to completely flush out contaminants and detergents.

When transporting or storing equipment after cleaning, the equipment will be stored in a manner that minimizes the potential for contamination.

#### 6.2.2 Soil/Sediment Sampling Equipment

A variety of samplers (split-barrel, split-barrel with brass liners, piston sampler, backhoe, hand-auger, or shovel) may be used to retrieve soil from sampling locations. The soil sample will either be sealed within the sampler (e.g., collecting volatile samples) or the soil sample will be transferred to laboratory-supplied containers depending on the analysis to be conducted on the soil sample. The equipment required to transfer the soil from the sampler to the laboratory-supplied sample containers includes: stainless-steel

spoons or scoops and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the PHASP.

All soil sampling equipment, including split-barrels, stainless-steel spoons and scoops, will be carefully cleaned before and during sampling with a tap water and non-phosphorus detergent solution, using a brush if necessary to remove particulate matter and films. The equipment is then rinsed three times with tap water and/or three times with analyte-free water. Inspect equipment and repeat procedure if any residual soil or visible contaminants are present. Dry sampler with a Kimwipes®. Organic solvents (e.g., methanol) may be used to aid with desorbing organic material but should be kept to a minimum and must be collected and containerized if used.

At the completion of the work day, the samplers should be decontaminated following the procedure above and stored in a manner that minimizes the potential for contamination.

### 6.2.3 Air Sampling Equipment

For non-laboratory manifold equipment, methanol soak manifold components for a minimum of two hours. Remove from the methanol bath and place in an oven pre-heated to 90 °C and continue to heat manifold components for at least 3 hours or until interior and exterior surface inspections of the manifold components indicate that they are free of liquid methanol.

### 6.2.4 Handling

All equipment will be handled in a manner that minimizes cross-contamination between points. After cleaning, the equipment will be visibly inspected to detect any residues or other substances that may exist after normal cleaning. If inspection reveals that decontamination was insufficient, the decontamination procedures will be repeated.

## 6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

## 6.4 Disposal

IDW generated by this process will be disposed of in accordance with Federal, State and Local regulations and/or as required by project-specific SAP or Work Plan. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

The QC activities described below allow the self-verification of the quality and consistency of the work.

### 7.1 QA/QC Samples

Decontamination procedures may be monitored through the use of an equipment blank which consists of analyte-free water processed through non-disposable or non-dedicated aqueous or solid sampling equipment after equipment decontamination and before field sample collection. The equipment blank is analyzed for the same parameters as the samples at a project specific frequency (e.g., one per twenty samples).

## 7.2 Measurement Criteria

Equipment blank results should be below the laboratory's method detection limit or reporting limit (depending on the data quality objectives).

## 8.0 Records

When required, the field technician(s) will document the field equipment decontamination procedures in a project dedicated field logbook or on field log data sheets.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation is listed in the applicable sample collection SOP.

Field documentation and COC are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual."

Other Barr SOP subjects referenced within this SOP: collection of samples and investigative derived waste.

## 9.0 References

ASTM. 2015. Standard Practice for Decontamination of Field Equipment Used at Waste Sites.





# Standard Operating Procedure Documentation on a Chain-of-Custody (COC)

Revision 6

February 26, 2020

Approved By:

Andrea Nord		02/26/20
Print	Technical Reviewer Signature	Date
Terri Olson		02/26/20
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
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Initials: _____	Date: _____

# Documentation on a Chain-of-Custody (COC)

## 1.0 Scope and Applicability

The purpose of this procedure is to describe how to properly document information on a Chain-of-Custody (COC). A COC is a legally binding document that identifies sample identification, analyses required, and shows traceable possession of samples from the time they are obtained until they are introduced as evidence in legal proceedings. A Field Technician completes the information on the COC at the time he/she collects samples and the COC accompanies the samples during transport to a storage facility or to the laboratory for analysis.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- The SOP does not apply to sample aliquots that are only collected for field screening purposes.
- The SOP does not apply to samples remaining on-site.

## 3.0 Responsibilities

Experienced Field Technicians are responsible for the proper sample identification and for accurate and complete documentation on the COC.

## 4.0 Procedure

The COC is the most important sampling document; it must be filled out accurately and completely every time a sample is collected. The instructions below are specific to Barr's COC for air canisters and Barr's COC typically used for solid and liquid samples. The COC for air canisters is typically used when collecting soil gas, soil vapor, emissions, or indoor and outdoor air samples in an evacuated canister. The COC for solid and liquid samples is typically used when collecting matrices such as groundwater, surface water, drinking water, waste water, storm water, soil, sediment, oil, paint chips, bulk materials, etc. Information common to both COCs and specific to each COC are detailed below.

Some of the information on a COC may be filled out ahead of time (e.g., report and invoice recipient details, project number, project name, project manager, purchase order number, etc.) while other information should be completed during sampling. Complete one COC or more, as needed, for each set of project samples. The COC should be completed prior to leaving the sampling location.

Laboratory supplied COCs may be used but may differ in the information captured. The use of a Barr COC is recommended as it allows for more efficient data processing within Barr's systems. If there are any questions, please contact a member of Barr's Data Quality team.

The laboratory receiving the samples will sign the COC, record the date and time of sample receipt, assign a laboratory work order number, document sample condition, and document whether custody seals were used and if they were intact.

#### 4.1 Common Chain-of-Custody Information

- Barr office location managing the work.
- Two digit identification for the state or province the samples originated from/sampled in.
- COC numbered pages (e.g., 1 of 1).
- Report and invoice recipient information.
- Purchase order number (if applicable).
- Barr project name and number.
- Sample location.
- Sample collection date and time.
- Sample matrix abbreviation (see "Matrix Code" on COC).
- Analysis requested.
- Field Technician (i.e. sampler) name.
- Barr Project Manager and project Data Quality (DQ) Manager names.
- Laboratory name and location in which samples are to be relinquished.
- Requested due date.
- Signature of Field Technician (i.e. sampler) under the first 'relinquished by'.
- Signature of sample transferee.
- Date and time of sample transfers.
- Method of transport (ground courier, air carrier, sampler, etc.).
- Air Bill number (if applicable).

#### 4.2 Completing a Chain-of-Custody for Air Canisters

Lab deliverable contents (based on project needs).

- Canister serial # and size.
- Flow controller serial #.
- Initial and final vacuum measurement (in inches of mercury).
- Start and stop times that the canister was drawing sample.
- Total time calculated from the start and stop times.
- Matrix code.
- PID reading (indicate if ppm or ppb).
- Sample comments (if any).
- Identify the report deliverable contents and electronic data deliverable contents requested.

### 4.3 Completing a Chain-of Custody for Solid and Liquid Samples

- Sample start and stop depth (if applicable) and unit of measurement (meter, feet, inches, etc.).
- Information regarding whether to perform sample Matrix Spike (MS) and MS duplicate (MSD).
- Container preservative type (see "Preservative Code" on COC).
- Information regarding whether the sample was field filtered.
- Number of each container type and the total number of containers for the sample.
- Presence or absence of ice.

### 4.4 Distribution of the COC Pages

Page one (white copy) accompanies the sample shipment to the laboratory and page two (yellow copy) is the Field Document copy. The Field Technician must scan and email a copy to the Barr Data Management Administrator for filing on Barr's internal network project files. Alternatively, the yellow hardcopy may be routed to the Barr Data Management Administrator for electronic filing. This read-only electronic copy will be distributed to and available for use by the project team via Barr's internal network project file access.

## 5.0 Quality Control and Quality Assurance (QA/QC)

The Field Technician should review the COC for accurate and complete documentation.

## 6.0 Records

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Chain-of-Custody for Air Canisters
- Chain-of-Custody

A copy of the COC is provided to a Barr Data Management Administrator for storage on the internal Barr network files.

Additional records information can be found in Barr's "Records Management System Manual".

## 7.0 References

United States Environmental Protection Agency. 2002. *Guidance for Quality Assurance Project Plans*. EPA QA/G-5.



# Standard Operating Procedure Domestic Transport of Samples to Laboratories within the United States of America – States and Territories

Revision 3

February 27, 2020

Approved By:

Andrea Nord

Print

QA Manager

A handwritten signature in black ink, appearing to read "Andrea Nord", is written over a horizontal line.

Signature

02/27/20

Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: \_\_\_\_\_

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# Standard Operating Procedures for the Domestic Transport of Samples to the Laboratories within the United States of America – States and Territories

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures necessary for personal delivery or shipment of samples from locations within the United States of America (USA) and its territories to analytical laboratories located within the USA and its territories. This procedure applies to the transportation of ground and surface water, soil, wipe, sediment, paint chip, debris, air samples and their corresponding quality control samples to the appropriate laboratory. This SOP applies to samples that are classified as non-regulated, non-hazardous, or “Dangerous Goods in Excepted Quantities” samples prior to shipment.

Soil samples that are preserved with flammable chemicals (methanol) and unused sample vials containing flammable or corrosive chemical preservatives are examples of materials that are classified as “Dangerous Goods in Excepted Quantities”. Materials classified as Dangerous Goods in Excepted Quantities have limitations on the volume/weight of the material allowed in each shipment, and have additional packaging, labeling, and shipping requirements than non-regulated and non-hazardous samples and sampling media.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Maintaining proper sample temperatures (<6°C or ambient air temperature in accordance with the analytical method requirements) and delivering samples to the laboratory within 24 to 48 hours from collection are primary concerns.
- This procedure does not apply to the transportation of samples to laboratories outside of the USA and its territories.
- This procedure does not apply to samples that are classified as “hazardous” according to USDOT, PHMSA, and/or RCRA and must be packaged, labeled, and/or transported in accordance with USDOT’s hazardous materials regulations (49 CFR Parts 100-180).
- This procedure does not apply to samples that are classified as “dangerous goods” and must follow the International Air Transportation Association’s (IATA) dangerous goods regulations (DGR) for packaging, labeling, and/or air transport.

### 3.0 Responsibilities

The field technician(s) shall ensure the security, temperature, and packaging of environmental samples during transport and shipment.

### 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When samples may be contaminated with corrosive materials, emergency eye flushing facilities should be available.

Some of the sample containers may require the use of chemical preservatives. Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

### 5.0 Equipment, Reagents, and Supplies

- Rigid cooler
- Heavy bag for containing ice and preventing leakage of melted water
- Ice
- Packing tape
- Dangerous Goods in Excepted Quantities Label with the number "8" added indicating the hazard class. This label must be used for coolers containing unused sample containers with corrosive preservative.
- Absorbent padding
- Bubble-wrap/bubble bags (inner packing material)
- Ziploc® baggies
- Shipping Airbill – if shipping via overnight commercial courier service
- Dangerous Goods in Excepted Quantities Label with the number "3" added indicating the hazard class. This label must be used for coolers containing methanol preservative
- Items listed in Section 8.0 Records

### 6.0 Procedure

#### 6.1 Packaging of non-regulated or non-hazardous samples requiring ambient air temperature per the analytical method of analysis

Sample matrices that do not require thermal preservation (ice) typically include wipe, paint chip, debris, and air samples. These samples may or may not require chemical preservatives depending upon the analytical method of analysis. The classification of "non-regulated" or "non-hazardous" in this context is based upon the nature of the sample prior to chemical preservation/fixation.

For samples that are stored at ambient air temperature, the samples will be placed in a jar, baggie or shipping carton (i.e. cooler, cardboard box, envelope) and accompanied with the proper COC.

Place the samples in a shipping carton in a manner that will avoid breakage. Fill out the chain-of-custody (COC) completely and include required copies with the samples. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Once the shipping carton is packed to prevent samples breaking, the COC is signed off and placed in the cooler or box. Adhere two to three strips of packaging tape from top to bottom on the cooler or box.

Custody seals must be adhered over the shipping carton lid or enclosure if project quality assurance plan or sampling and analysis plan require them. The custody seal must be adhered to the crack of the lid on two opposing sides of the cooler or over the flap(s) of the box or envelope to ensure the carton remained shut and the contents have not been tampered with during transit.

## **6.2 Packaging of non-regulated or non-hazardous samples requiring thermal preservation per the analytical method of analysis**

Samples matrices that require thermal preservation (ice) typically include water, soil and sediment samples. Glass containers should be packed in bubble wrap or other cushioning material to avoid breakage.

*Note: Bubble-wrap is the preferred packing material.*

Line a rigid plastic cooler (i.e. shipping container) with a strong plastic bag. This bag will serve as an outer liner and contain the wet ice, absorbent materials and sample containers.

Place samples and cushioning absorbent material inside the plastic bag and add enough absorbent padding to absorb the sample liquid within the package. Package ice in double-lined Ziploc® bags to ensure sample labels will not be compromised, and the cooler(s) will not leak melt water. Add enough ice to the cooler to maintain a constant temperature at  $\leq 6^{\circ}\text{C}$ , (but not frozen) until the samples arrive at the laboratory. Zip tie the plastic bag shut.

Before sealing cooler, fill out the COC completely and include required copies with the samples. Refer to Barr's SOP 'Documentation on a Chain-of-Custody (COC)' for further information.

Adhere two to three strips of packaging tape on the cooler from top to bottom, and adhere an additional strip of tape covering the gap between the lid and sides of cooler to seal the cooler to avoid leakage.

Custody seals must be adhered on the cooler if project quality assurance plan or sampling and analysis plan require them. The custody seal must be adhered to the crack of the lid on two opposing sides of the cooler to ensure the contents have not been tampered with during transit.

Follow the labelling instructions in Section 6.4 of this SOP.

## 6.3 Packaging of samples classified as “Dangerous Goods in Excepted Quantities”

### 6.3.1 Soil Samples Preserved with Methanol (Flammable) – Hazard Class 3

Soil samples that are preserved with flammable chemicals (methanol) are an example of materials that are classified as hazard class “3” “Dangerous Goods in Excepted Quantities”.

Follow the packaging instructions listed in Section 6.2 of this SOP with the following addition: *Methanol sample containers must be placed in a Ziploc® Baggie to meet shipping requirements for preventing leakage.*

Each cooler shall not exceed 500 mL of methanol (50 vials, 10 mL of methanol per vial) and each vial shall not have more than 10 mL of methanol to meet the requirements of a Dangerous Goods in Excepted Quantities. A label with the hazard class number “3” indicates the cooler contains flammable (or reactive/oxidizer) materials (in this case a flammable methanol sample preservative). Additional labeling instructions are found in Section 6.4.2 of this SOP.

### 6.3.2 Unused Sample Jars – Hazard Class 3 (Flammable) and Hazard Class 8 (Corrosive) Chemicals

Unused sample vials containing flammable or corrosive chemical preservatives are examples of materials that are classified as “Dangerous Goods in Excepted Quantities”.

Follow the packaging instructions listed in Section 6.2 of this SOP with the following additions:

Each chemical, may have a limitation as to the volume or weight of the chemical and the number of inner containers (sample containers) allowed within each outer shipping container (cooler) to meet the requirements of a Dangerous Goods in Excepted Quantities. A label with the hazard class number “3” indicates the cooler contains flammable (or reactive/oxidizer) materials (in this case a flammable methanol sample preservative). A label with the hazard class number “8” indicates the cooler contains a corrosive material (in this case an acid or base sample preservative). Additional labeling instructions are found in Section 6.4.2 of this SOP.

## 6.4 Labeling of Outer Shipping Container or Carton

### 6.4.1 Shipping Label

Attach the shipping address label to the top of the cooler or to the cooler handle tag. Attach a second label with the same information should also be attached with packaging tape to the cooler in event that the original label is damaged or destroyed during sample shipment.

Directional arrow labels (Figure 1) must also be attached to the outside of the cooler according to the hazardous materials shipping regulations. Directional arrow labels indicate the upright position during sample shipment.



Figure 1 - Directional Arrows Label

#### 6.4.2 Dangerous Goods in Excepted Quantities Label

When shipping materials classified as Dangerous Goods in Excepted Quantities, the cooler must have a Dangerous Goods in Excepted Quantities Label (Figure 2). This label is placed on two opposing sides of the cooler. The label indicates the hazard class number and the name and address of the shipper or consignee. In cases where the package contents have more than one hazard class assigned, the primary (most hazardous) hazard class is listed on the label. Table 1 includes a Summary of United Nations Hazard Classes.



Figure 2 - Dangerous Goods in Excepted Quantities Label

Footnotes:

- (1) The "\*" must be replaced by the primary hazard class, or when assigned, the division of each of the hazardous materials contained in the package.
- (2) The "\*\*" must be replaced by the name of the shipper or consignee if not shown elsewhere on the package.

Table 1 – Summary of United Nations Hazard Classes

<b>Class 1</b>	Explosives
<b>Class 2</b>	Gases
<b>Class 3</b>	Flammable Liquids
<b>Class 4</b>	Flammable Solids; Substances Liable to Spontaneous Combustion; Substances Which, in Contact with Water, Emit Flammable Gases (e.g., soil sample contaminated with high concentrations of gasoline released from an underground storage tank)
<b>Class 5</b>	Oxidizing Substances and Organic Peroxide
<b>Class 6</b>	Toxic and Infectious Substances (e.g., samples of refuse collected from a solid waste landfill)
<b>Class 7</b>	Radioactive Material
<b>Class 8</b>	Corrosives (e.g., nitric acid used for preservation of some groundwater samples) (see Note)
<b>Class 9</b>	Miscellaneous Dangerous Goods

#### 6.4.2.1 Dangerous Goods Air Waybill Statement and Shippers Declaration

A shipping paper (i.e. bill of lading) is not required when offering the cooler for air transport via a commercial courier service (e.g. Federal Express or United Parcel Service).

A document such as an air waybill accompanies a shipment that is transported by aircraft. The air waybill must include the statement “Dangerous Goods in Excepted Quantities” and indicate the number of packages associated with each air waybill. This phrase is typically written behind the Barr project number in the PO or comments section on the air waybill.

A shipper’s declaration for dangerous goods is also required. Some air waybills also have a box you must also check off that says “Dangerous Goods no Shipper’s Declaration Required”.

### 6.5 Transport/Delivery Options

Account for the samples before shipping and compare to the COC. Refer to Barr’s SOP ‘Documentation on a Chain-of-Custody (COC) for further information. Ship samples during times when the laboratory will be able to accept and quickly analyze them. Whenever possible, select mode of transport/delivery to ensure delivery to the laboratory will occur with ample holding time remaining for the specified analytical methods required for the samples. Avoid sending samples during holidays and weekends. All Federal, State and Local shipping regulations must be met.

**Personal Delivery.** The samples are delivered to the laboratory by the field technician(s). The COC is signed and dated by the laboratory representative.

**Ground Transport.** The same procedures are followed as above; i.e., the COC is signed and dated and the top copy is sent with the samples. The cooler or box is then secured with packaging tape and a courier form is filled out for the designated laboratory. The cooler or box is then left in the services area for pickup via ground transport and delivery.

**Air Transport.** Follow the procedures above, replacing the courier form with the overnight courier air bill via Federal Express or United Parcel Service, for example. Include the date, project number, type of

delivery service desired, parcel weight, number of coolers or boxes on the air bill. Also include the phrase "Dangerous Goods in Excepted Quantities", when applicable.

## 7.0 Quality Control and Quality Assurance (QA/QC)

Not Applicable.

## 8.0 Records

Examples of common field documentation are available in Barr's "Compendium of Field Documentation".

Field documentation specific to this SOP are listed below:

- Chain-of-custody (COC)
- Custody seal (if applicable)
- Dangerous Goods in Excepted Quantities Label
- Directional Arrow Label

COCs are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: documentation on a COC.

## 9.0 References

49 CFR Part 173.4a – Excepted Quantities October 1, 2011 Online

<https://www.govinfo.gov/app/details/CFR-2011-title49-vol2/CFR-2011-title49-vol2-sec173-4>

*ASTM International. 2015. ASTM Method D6911 – 15 Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis<sup>1</sup>. ASTM January 15, 2015.*





# Standard Operating Procedure

## Field Screening Soil Samples

Revision 8

April 9, 2019

Approved By:

<u>John W. Juntilla</u>		<u>04/09/19</u>
Print	Technical Reviewer Signature	Date
<u>Terri A. Olson</u>		<u>04/09/19</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
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# Field Screening of Soil Samples

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedure for properly screening soil or sediment samples in the field. This procedure applies to field technicians responsible for field screening soil or sediment samples.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Screening techniques can vary by project. If not specified in the project scope of work and/or documentation (e.g., Work Plan, Sampling Analysis Plan (SAP), or Quality Assurance Project Plan (QAPP)), consult with the appropriate regulatory agency for guidance, if applicable.
- Interferences on the test can be caused by any contaminant that can cause an oil sheen on water. The samples will be carefully observed for characteristic appearance or odors which may indicate a possible contaminant other than coal tar or petroleum substances.
- Sunlight and low temperatures may interfere with headspace development.
- Water and soil particles may interfere with PID and FID measurements.
- Decontamination of screening equipment is required to prevent cross-contamination.
- Contact the local one call system prior to digging to have public utilities identified at sampling locations. Privately owned underground utilities, if present, typically will not be identified by the one call system and contracting with a private utility locator may be necessary.

## 3.0 Responsibilities

The Project Manager, in conjunction with the client, develops the site specific scope of work (e.g., Work Plan, SAP, etc.).

Experienced Field Technicians are responsible for the proper sample identification, field screening procedures, field equipment and calibration, quality control procedures, and documentation.

Equipment Technicians are responsible for maintaining equipment in working order and aiding in troubleshooting equipment issues.

The role of the Field Safety Representative is to oversee on-site safety activities.

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent

sample contact with the skin and eyes. When screening soils contaminated with corrosive materials, emergency eye flushing facilities should be available.

Consult the applicable Safety Data Sheet to review hazards and appropriate PPE to minimize exposure.

## 5.0 Equipment, Reagents, and Supplies

- Photoionization detector (PID)
- Flame ionization detector (FID)
- Squirt bottle with tap water
- Waterproof ink pen or pencil
- Polyethylene bags
- Chemical resistant gloves (e.g., nitrile)
- Stainless steel spoon
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

## 6.0 Procedure

The field screening techniques for soils are as follows: visual examination, odor, headspace organic vapor screening, and oil sheen. The results of these four screening procedures may be used to screen soil samples for possible contamination.

### 6.1 Calibration

The PID or FID shall be calibrated or checked against a known concentration of a calibration gas standard prior to collection of field measurements. Calibration of the PID or FID shall follow the recommended procedures as described in the manufacturer's operation manual or as per the applicable Barr SOP.

Regular calibration checks (bump tests) are expected to be performed by the field technician a minimum of once per day of use in the field. It is recommended that bump tests be conducted around mid-day and at the end of the day. More frequent bump testing may be completed if warranted by field conditions. The bump testing results should be recorded in the field log book or field log data sheets.

If problems occur during calibration, during bump tests, or if the unit will not stay calibrated, the field technician should document the issue in the field notes then contact the equipment technician or project manager for assistance.

### 6.2 Screening Techniques

The field screening techniques for soils are as follows: visual examination, odor, headspace organic vapor screening, and oil sheen. The results of these four screening procedures may be used to screen soil samples for possible contamination. To prevent sample cross-contamination, the screening equipment is carefully cleaned before and after working with each sample per Barr's SOP 'Decontamination of Sampling Equipment'.

#### 6.2.1 Visual Examination

A visual examination of the soil sample will include noting any discoloration of the soil or visible oiliness or tar.

#### 6.2.2 Odor

The field technician will note odor only if noticed incidentally while handling the soil sample. Field technicians will not unduly expose themselves to sample odors. Odor will be described as trace, light, moderate, or strong, and appropriate description of the type of odor, if evident.

### 6.2.3 Headspace Organic Vapor Screening

The polyethylene bag headspace method recommended by the Minnesota Pollution Control Agency will be used in the field to screen soils suspected to contain volatile organic compounds. The screening method is intended to be used in conjunction with other “real time” observations.

The following equipment is required to conduct headspace organic vapor screening: PID or FID, polyethylene bag, log book or record sheet, and appropriate PPE. Soil samples collected from a split-barrel sampler or a direct-push (i.e., Geoprobe) sample liner will be collected immediately after opening the barrel or liner. If the sample is collected from an excavation wall, soil pile, or backhoe bucket, it will be collected from a freshly exposed surface.

- Half-fill the bag with the sample to be analyzed using a stainless-steel spoon or a gloved hand and immediately seal it. Agitate the bag for 15 seconds and manually break up any soil clumps within the bag.
- Allow headspace development for approximately 10 minutes. The sample should be kept in a shaded area out of direct sunlight. Ambient temperatures during headspace development should be recorded. When ambient temperatures are below 50°F, headspace development should be conducted inside a heated vehicle or building. After completing the headspace development, agitate the bag for an additional 15 seconds.
- Quickly puncture the bag with the sampling probe of the PID or FID at a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particles.
- Record the highest PID or FID meter response as the headspace concentration. The maximum response will likely occur between 0 to 5 seconds.
- When using a FID, it may be necessary to correct for methane. In this case, take a reading first with the carbon filter, then without. This will require two duplicate bag samples. The second reading less the first is the headspace adjusted for methane. Adjusted readings less than zero are considered zero. Methane correction is not necessary if a PID is used.

### 6.2.4 Oil Sheen Test

The oil sheen or hydrocarbon test is a method used to immediately determine the approximate magnitude of coal tar or petroleum contamination in soil by observation of the sample in the field. The test is useful in soils which do not have a high binding capacity with petroleum compounds or polycyclic aromatic hydrocarbons (PAHs) (i.e., petroleum compounds or PAHs are free on the surface of the soil particles and can be released by a stream of water).

The equipment required to conduct the oil sheen test includes: a stainless-steel spoon, a squirt bottle filled with tap water, a log book or field log data sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan.

The procedure for conducting the oil sheen test consists of obtaining approximately 50 grams (about 30 cc) of representative soil with the spoon and then directing a stream of water onto the soil in the spoon with the squirt bottle until the soil is saturated and water begins to collect around the soil. The amount of oil sheen present on the water is determined by observation and the results of the test are reported as a magnitude of oil sheen observed: none, trace, light, moderate, heavy or rainbow. The test results, sample location, and observations of the sample's appearance and odor are recorded in the log book or field log data sheet.

The specific soil types at the area of investigation should be accounted for when performing the oil sheen test. The best results are obtained in silts, sands, and/or gravels with low organic content. The results obtained from clay soils may appear deceptively low. Typical descriptions of each test result are provided in the table below.

<b>Oil Sheen Test Result</b>	<b>Description</b>
None	No sheen detected.
Trace	Possible or faint oil sheen observed (may not continue to generate sheen as additional water is added).
Light	Obvious sheen that may not cover entire water surface
Moderate	Definite oil sheen that covers entire surface, but "rainbow colors" not distinguishable.
Heavy	Definite oil film or product that does not display rainbow colors.
Rainbow	Definite oil sheen, film or product that displays rainbow colors.

### 6.3 Data Reduction/Calculations

No data reduction or calculations are associated with this procedure.

### 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

Field background readings are measured for the headspace organic vapor screening. PID and FID readings should be duplicated every 20 field samples.

## 8.0 Records

The field technician(s) will document the field screening activities and measurements in a project dedicated field logbook or on field log data sheets.

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Field Sampling Report
- Field Log Data Sheet

Field documentation are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual."

Other Barr SOP subjects referenced within this SOP: PID and FID equipment, decontamination of sampling equipment, and investigative derived waste.

## 9.0 References

PID and FID operation manuals.






# Standard Operating Procedure Monitoring Well Development Oversight

Revision 6

October 22, 2019

Approved By:

<u>John W. Juntilla</u>		<u>10/22/19</u>
Print	Technical Reviewer Signature	Date
<u>Terri A. Olson</u>		<u>10/22/19</u>
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

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Date: \_\_\_\_\_

# Monitoring Well Development Oversight

## 1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe oversight provided on monitoring well development or redevelopment. These procedures are performed with the objective of obtaining representative groundwater information and water quality samples from aquifers.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Well development should be completed by an appropriately licensed or registered well contractor unless allowed by rules governing wells and borings.
- Best practice is to have a minimum of one week pass between monitoring well development and monitoring well sampling unless there are other project requirements.
- If well will be sampled for per- and polyfluorinated alkyl substances (PFAS), special consideration must be taken to avoid accidental contamination of the well during the development process - see Barr's SOP 'Collection of Per- and Polyfluorinated Alkyl Substances (PFAS) Samples'.

## 3.0 Responsibilities

Experienced Field Technicians are responsible for overseeing the well development, quality control procedures, and documentation.

The role of the Field Safety Representative is to oversee on-site safety activities.

The well drilling contractors are typically responsible for the development of monitoring wells at the time of installation and have the necessary tools, equipment, chemicals, applicable licenses or registrations that may be required to perform the development work. Successful development of a new well may be a requirement of the drilling specifications.

## 4.0 Safety

Barr staff is responsible for conducting the aspects of the job safely. When applicable, refer to the appropriate Project Health and Safety Plan (PHASP) to understand the hazards associated with suspected contamination, symptoms of exposure, methods to minimize exposure, personal protective equipment (PPE), and personal air monitoring required when using this SOP. Minimum protection of one pair of chemical resistant gloves (e.g., nitrile) and safety glasses with side shields should be worn to prevent sample contact with the skin and eyes. When working with liquids contaminated with corrosive materials, emergency eye flushing facilities should be available.

## 5.0 Equipment, Reagents, and Supplies

- Pumps^ (e.g., submersible or peristaltic)
- Pump discharge hose/tubing
- Chemical resistant gloves (e.g., nitrile)
- Surge block (optional)
- Turbidimeter (optional)
- Water level indicator or interface probe
- Bailers
- Water quality meter (optional)
- Items listed in Section 8.0 Records
- Decontamination supplies (see Decon SOP)

\* See Barr's PFAS SOP for a list of prohibited and acceptable items.

## 6.0 Procedure

These procedures are used to remove the fine-grained materials from a well or well bore as a result of boring or well construction. Monitoring wells must be developed to provide water free of suspended solids and to yield representative samples. Well development should result in a well that yields visibly clear groundwater.

### 6.1 Calibration

If used, the water quality meter and turbidimeter will be calibrated as per the applicable Barr SOP. The meters will undergo calibration checks, at a minimum, before and after sampling. The calibration check will be documented on a calibration form (as appropriate) and/or in the field notebook. Any significant issues found during the calibration check will be noted in the field notebook and the Equipment Technicians will be notified.

### 6.2 Development

Successful development methods include bailing, surging, pumping/over-pumping, and jetting with water. The basic principle behind each method is to create reversals of water flow into and out of the well screen (and/or bore hole) to break-down any potential mud cake or disturbed zones where fine-grained particles may be concentrated at the borehole-formation interface, and to draw the finer materials into the well or borehole for removal. This process also helps remove fine fraction formation materials in proximity to the borehole wall, leaving behind a "natural" pack of coarser-grained materials.

#### 6.2.1 Bailing

In relatively clean, permeable formations where water flows freely into the borehole, bailing is an effective development technique. Let the bailer fall down the well until it strikes the surface of the groundwater which produces an outward surge. Rapidly withdraw the bailer to create a drawdown and/or after the bailer hits the groundwater lower it to the bottom of the well and agitate it with rapid short strokes. Continue bailing with repeated up and down "surging motions" until water bailed from the well is free from suspended particles.

*Note: During this process, if the well goes dry, stop bailing and let the well recharge before continuing.*

#### 6.2.2 Surge Block

A surge block is a tool used to break up bridging of fine grained material by inducing agitation and inducing flow into and out of the well and aquifer formation. Bridging is the tendency for particles moving towards a well under unidirectional flow (pumping) to develop a blockage that restricts subsequent

particles to move into a well. Surge block is used alternately with either a pump or bailer. Let the surge block fall down the well until it strikes the groundwater surface. This creates a vigorous outward surge; rapidly retrieve the surge block. Lower the surge block to the top of the well intake and begin a pumping action with a typical stroke of approximately 3 feet and gradually work downward through the screened interval. Remove the surge block at regular intervals to discard the loosened suspended particles by either bailing or pumping. Continue the cycle of surging/bailing/pumping until satisfactory development has been attained.

### 6.2.3 Pumping/Over-pumping

In both pumping techniques, the groundwater flow is induced to flow into the well and the fine particulate material moves into the well and is discharged by the pump. In the case of over-pumping, the pump is operated at a capacity that substantially exceeds the ability of the formation to deliver water. Once pumping has begun, start the surging action by lowering and raising the hose/pumping apparatus through the screened interval. Bailing or bailing and surging may be combined with pumping for efficient well development. Continue pumping until such time as satisfactory development has been attained based on field observation of visibly clear water produced. If an analytical measure is needed, use turbidity meter readings to document initial turbidity and final turbidity readings. Well stabilization parameters may also be measured and documented pre- and post-development.

If pumping/over-pumping is completed by air lifting, the air compressor must be of an oil-less type or fitted with an oil trap capable of removing compressor oil from the air stream to avoid contaminating the well or boring.

*Note: The types of pumps used are described in Barr's SOPs 'Collection of Groundwater Samples from a Temporary or Permanent Monitoring Well (Includes Well Purging and Stabilization)' or 'Collection of Groundwater Samples using Low-Flow Purging and Sampling'.*

### 6.2.4 High Velocity Jetting

Development by high velocity jetting may be completed with either water or air. In practice, jetting with water is typically followed by or simultaneously occurring air-lift pumping/over pumping to remove the fine materials. The jetting procedure consists of operating a horizontal water jet(s) inside of the well screen so high velocity streams of water shoot through the screen openings into the sand pack/formation. The jetting tool is worked similar to a surge block. The jetting tool ideally will have four openings located 90 degrees apart and should be worked up and down the screened interval while being rotated. At a minimum, the amount of water introduced during jetting and, if feasible, an additional 10 well volumes of water should be purged from the well.

## 6.3 Data Reduction/Calculations

The calculations for well volume and volume of water to be purged are included in Barr's SOP 'Collection of Groundwater Samples from a Temporary or Permanent Monitoring Well (Includes Well Purging and Stabilization)'.

## 6.4 Disposal

Waste generated by this process will be disposed of in accordance with Federal, State and Local regulations and Barr's SOP 'Investigative Derived Waste'. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

## 7.0 Quality Control and Quality Assurance (QA/QC)

QA/QC objectives (e.g., turbidity, well recovery rate, water quality parameters) are specific to each project and/or well. Discuss QA/QC procedures with the project team prior to well development.

## 8.0 Records

The field technician(s) will document the method of development, any deviations from this SOP, volume of water purged, and any volume of water introduced to the well (e.g., high velocity jetting, flushing).

Examples of common field documentation are available in Barr's "Compendium of Field Documentation". Field documentation specific to this SOP are listed below:

- Field Log Cover Sheet
- Field Log Data Sheet

The field documents are provided to a Barr Data Management Administrator for storage on the internal Barr network.

Additional records information can be found in Barr's "Records Management System Manual".

Other Barr SOP subjects referenced within this SOP: water quality meter, turbidimeter, well recovery rate testing, collection of PFAS samples, decontamination of sampling equipment, groundwater purging/sampling, low-flow purging/sampling, and investigative derived waste.

## 9.0 References

American Society for Testing and Materials (ASTM), D5521/D5521M-13. 2013. *Standard Guide for Development of Groundwater Monitoring Wells in Granular Aquifers*.

Environmental Protection Agency, Offices of Waste Programs Enforcement and Solid Waste and Emergency Response. 1986. *RCRA Ground-Water Monitoring Technical Enforcement Document*.

Johnson Filtration Systems. 1986. *Groundwater and Wells*.

National Water Well Association. *Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells*.





**Standard Operating Procedure**  
**Routine Level Semivolatile Organic Compounds (SVOC),**  
**Polycyclic Aromatic Hydrocarbons (PAH), Diesel Range**  
**Organics (DRO), and Total Petroleum Hydrocarbons (TPH)**  
**Data Evaluation**

Revision 7

January 2, 2020

Approved By:

<u>Marta Nelson</u>		<u>01/02/20</u>
Print	Technical Reviewer    Signature	Date
<u>Terri Olson</u>		<u>01/02/20</u>
Print	QA Manager    Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

# Routine Level Semivolatile Organic Compounds (SVOC), Polycyclic Aromatic Hydrocarbons (PAH), Diesel Range Organics (DRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

## 1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of semivolatile organic compounds data provided by laboratories to be used in Barr Engineering Co. (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine SVOC (including PAHs and phenols), TPH at various carbon ranges (e.g., TPH as fuel oil, TPH as motor oil, TPH as jet fuel), and DRO data evaluation for analyses by the following technologies:

- Gas Chromatography/Flame Ionization Detector (GC/FID)
  - Method examples: EPA 8015, EPA 8100, WI DRO
- Gas Chromatography/Mass Spectrometry (GC/MS)
  - Method example: EPA 625, EPA 8270
- Gas Chromatography/Mass Spectrometry-Selective Ion Monitoring (GC/MS-SIM)
  - Method example: EPA 8270
- High Performance Liquid Chromatography (HPLC)
  - Method example: EPA 610, EPA 8310
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

The letter indicator for the various EPA method revisions have been intentional omitted. Multiple versions of the approved methods would be applicable for review under this SOP. In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review. Laboratories may not provide all the review elements in this SOP, review only those that are provided.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

### 3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

### 4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

#### 4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136, WI DRO method, and the Test Methods for Evaluating Solid Waste (SW-846) are used as guidance for the recommended holding time and preservation acceptance criteria listed in Table 1.

Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
SVOC/PAH/TPH	Aqueous	≤ 6° C	Ice	7 days extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6° C	Ice	14 days extraction/ addl. 40 days analysis
DRO	Aqueous	≤ 6° C	Ice, HCl < 2 pH; sodium azide for carbonate aquifer	7 days extraction/47 days collection to analysis 48 hours if not HCl or sodium azide preserved
	Sediment/Soil	≤ 6° C	Ice	10 days solvent addition/ 47 days collection to extraction and analysis
TCLP SVOC	Various	--	NA	14 days TCLP extraction/7 days extraction/addl. 40 days analysis

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an 'H' ("Recommended sample preservation, extraction or analysis holding time was exceeded."). Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subject to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

## 4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG). Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination	
Sample Result	Recommended Action for Associated Data
Non-detect	No action required
< 5x blank concentration	Qualify with 'UB'
≥ 5x blank concentration	Use professional judgment

UB = The analyte is detected in one of the associated laboratory, equipment, field or trip blank samples and is considered non-detect at the concentration reported by the laboratory.

*Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)*

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with 'J' ("Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.") or 'R' ("The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.").

### 4.3 Deuterated Monitoring Compounds (DMC) and Surrogates

DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are only used for the SVOC GC/MS analysis. Table 3 presents the recommended DMCs with their associated target compounds.

Table 3 – DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
<i>2,4-Dichlorophenol-d<sub>3</sub></i>	2,4-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol	2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol
<i>2-Chlorophenol-d<sub>4</sub></i>	2-Chlorophenol	
<i>2-Nitrophenol-d<sub>4</sub></i>	Isophorone	2-Nitrophenol
<i>4-6-Dinitro-2-methylphenol-d<sub>2</sub></i>	4,6-Dinitro-2-methylphenol	
<i>4-Chloroaniline-d<sub>4</sub></i>	4-Chloroaniline Hexachlorocyclopentadiene	3,3'-Dichlorobenzidine
<i>4-Methylphenol-d<sub>8</sub></i>	2-Methylphenol 4-Methylphenol	2,4-Dimethylphenol
<i>4-Nitrophenol-d<sub>4</sub></i>	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol	4-Nitrophenol 4-Nitroaniline
<i>Acenaphthylene-d<sub>8</sub></i>	Naphthalene 2-Methylnaphthalene 2-Chloronaphthalene	Acenaphthylene Acenaphthene
<i>Anthracene-d<sub>10</sub></i>	Hexachlorobenzene Atrazine	Phenanthrene Anthracene
<i>Benzo(a)pyrene-d<sub>12</sub></i>	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
<i>Bis-(2-chloroethyl) ether-d<sub>8</sub></i>	Bis-(2-chloroethyl) ether 2,2'-oxybis(1-chloropropane)*	bis(2-Chloroethoxy) methane
<i>Dimethylphthalate-d<sub>6</sub></i>	Caprolactum 1,1'-Biphenyl Dimethylphthalate Diethylphthalate	Di-n-butylphthalate Butylbenzylphthalate bis(2-ethylhexyl)phthalate Di-n-octylphthalate
<i>Fluorene-d<sub>10</sub></i>	Dibenzofuran Fluorene 4-Chlorophenyl-phenylether	4-Bromophenyl-phenylether Carbazole

(Table 3 continued on next page)



Table 3 – DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
<i>Nitrobenzene-d5</i>	Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene	2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosdiphenylamine
<i>Phenol-d5</i>	Benzaldehyde	Phenol
<i>Pyrene-d10</i>	Fluoranthrene Pyrene	Benzo(a)anthracene Chrysene
SIM DMC and Associated Target Compounds		
<i>Fluoranthene-d10</i>	Fluoranthene Pyrene Benzo(a)anthracene Chrysene Benzo(b)fluoranthene	Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
<i>2-Methylnaphthalene-d10</i>	Naphthalene 2-Methylnaphthalene Acenaphthylene Acenaphthene	Fluorene Pentachlorophenol Phenanthrene Anthracene

\* = Chemical name changed by Integrated Risk Information System (IRIS) on November 30, 2007 from Bis(2-chloroisopropyl)ether to Bis(2-chloro-1-methylethyl)ether (common name). 2,2'-oxybis(1-chloropropane) is CAS index name.

Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other DMC or surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. All samples (blanks, spiked samples, project samples, QC samples) should contain DMC or surrogates. If a sample does not contain DMC or surrogates or the method does not require surrogates (WI DRO), professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of DMC or surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each DMC or surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Surrogates are not required for some methods (e.g., WI DRO). If used for WI DRO, the method requires that the surrogates must not elute within the WI DRO window (C<sub>10</sub>-C<sub>28</sub>). If the laboratory report includes a surrogate spike recovery for WI DRO, use professional judgment to assess the data.

Table 4 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

<b>Table 4 – Guidelines for Single DMC or Surrogate</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
%R > Upper Limit	Qualify with 'J+'	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
%R within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.  
The analyte may or may not be present in the sample.

Table 5 includes guidance where multiple surrogates are analyzed per analytical fraction.

<b>Table 5 – Guidelines for Multiple DMC or Surrogates</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with 'J+'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.  
The analyte may or may not be present in the sample.

#### 4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (20 or less samples of the same matrix - WI DRO requires an additional LCSD analyzed at the end of 20 samples).
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision

(when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

<b>Table 6 – Guidelines for Laboratory Control Samples</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
%R > Upper Limit	Qualify with 'J+' or use professional judgment	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
RPD > Upper Limit	Qualify with 'J' or use professional judgment	
%R and RPD within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

#### 4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Ideally, blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The MS/MSD duplicate pairs may be substituted for laboratory duplicates. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with U, UB, <, or R. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

<b>Table 7 – Guidelines for Laboratory Duplicates</b>	
<b>% RPD</b>	<b>Recommended Action for Associated Data</b>
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with 'J'

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

#### 4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with U, UB, <, or R. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs  $\leq 30\%$  for aqueous samples and  $\leq 40\%$  for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times ( $>5x$ ) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times ( $>5x$ ) the RL, professional judgment should be used to determine if qualification is appropriate.

<b>Table 8 – Guidelines for Field Duplicates</b>	
<b>% RPD</b>	<b>Recommended Action for Associated Data</b>
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with 'J'

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

#### 4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to DRO in the WI method)

- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added ( $>4x$ ), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

<b>Table 9 – Guidelines for Matrix Spikes</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
%R > Upper Limit	Qualify with 'J+' or use professional judgment	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
RPD > Upper Limit	Qualify with 'J' or use professional judgment	
%R and RPD within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

#### 4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately



documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

## 5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data evaluation may include the completion of a Routine Level Quality Control Report. This may be a report produced via EQuIS DQM (Environmental Quality Information System Data Quality Module) or a hardcopy as found in Barr's "Compendium of Data Quality Assessment Documentation". Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented. If using EQuIS DQM, reason codes will also be applied. The reason codes are defined in the software. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

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The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information. Documentation of the data evaluation may include but is not limited to an email to the project team, data evaluation summary report, technical memo, or section within a project report.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

## 7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3.*

Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review.*

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

## Attachment 1

### Revision History

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 10
		Attachments	Added Attachment 3
3.2	04/2011	Document Wide	Added analytical methods to applicability section.
		Attachments	Updated Attachment 1 and 2 to include current forms.
4.0	04/06/12	Document Wide	Major revision
5.0	06/17/13	Cover page	Added Calgary office
		I	Added waste rock and drill cores to examples of product sample
		III, IV, V, VI, VII	Added 'project specific requirements' as possible criteria source
		VI	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		VI	Clarified field duplicate criteria as < one value and not a range
		IX	Added statement regarding multiple qualifiers
6	01/19/16	Document Wide	SOP restructuring, new format
7	01/02/20	Document Wide	Updated for new qualifiers



# Standard Operating Procedure

## Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

Revision 7

January 2, 2020

Approved By:

Michael Dupay		01/02/20
Print	Technical Reviewer Signature	Date
Terri Olson		01/02/20
Print	QA Manager Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

Initials: \_\_\_\_\_

Date: \_\_\_\_\_

# Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

## 1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of VOC, GRO, and TPH data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine VOC (including BTEX), GRO, and TPH (in the approximate gasoline carbon range, C<sub>6</sub>-C<sub>10</sub>) data evaluation for analyses by the following technologies:

- Gas Chromatography/Flame Ionization Detector (GC/FID)
  - Method examples: EPA 8015, WI GRO (GRO)
- Gas Chromatography/Photoionization Detector (GC/PID)
  - Method example: EPA 8021, WI GRO (PVOC)
- Gas Chromatography/Electrolytic Conductivity Detector (GC/ELCD)
  - Method example: EPA 8021
- Gas Chromatography/Mass Spectrometry (GC/MS)
  - Method example: EPA 624, EPA 8260
- Gas Chromatography/Mass Spectrometry-Selective Ion Monitoring (GC/MS-SIM)
  - Method example: EPA 8260
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

The letter indicator for the various EPA method revisions have been intentional omitted. Multiple versions of the approved methods would be applicable for review under this SOP. In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review. Laboratories may not provide all the review elements in this SOP, review only those that are provided.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

## 2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

### 3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

### 4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report evaluation. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

#### 4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136, WI GRO method, and the Test Methods for Evaluating Solid Waste (SW-846) are used as guidance for the recommended holding time and preservation acceptance criteria listed in Table 1.

Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
VOC/PVOC	Aqueous	≤ 6 °C	HCl < 2 pH	14 days
	Aqueous	≤ 6 °C	Unpreserved	7 days
	Sediment/Soil	≤ 6 °C	1:1 soil:solvent (e.g., 10 g soil:10 mL MeOH in lab pre-weighed vial)	14 days
GRO (WI Method)	Aqueous	≤ 6 °C	HCl < 2 pH	14 days
	Sediment/Soil	≤ 6 °C	1:1 soil:solvent (e.g., 10 g soil:10 mL MeOH in lab pre-weighed vial)	21 days

(Table 1 continued on next page)



Table 1 – Recommended Holding Times and Preservation				
Compound	Matrix	Temp.	Preservative	Maximum Hold Time
TPH	Aqueous	≤ 6 °C	HCl or H <sub>2</sub> SO <sub>4</sub> < 2 pH	7 day extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6 °C	Zero headspace*	14 days extraction/ addl. 40 days analysis
TCLP	Various	≤ 6 °C	No preservative	14 days TCLP extraction/ addl. 14 days analysis

\* = Alternatively, samples may be collected as per the VOC analysis.

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an 'H' ("Recommended sample preservation, extraction or analysis holding time was exceeded."). Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subject to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

## 4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG) – laboratories should analyze a method blank at least once every 12 hours. Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Trip blanks should be placed in each transport cooler containing VOC sample containers prior to shipment into the field and remain with the associated VOC samples submitted to the laboratory for VOC analysis; including sample storage through analysis.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination	
Sample Result	Recommended Action for Associated Data
Non-detect	No action required
< 5x blank concentration	Qualify with 'UB'
≥ 5x blank concentration	Use professional judgment

UB = The analyte is detected in one of the associated laboratory, equipment, field or trip blank samples and is considered non-detect at the concentration reported by the laboratory.

*Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)*

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with 'J' ("Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.") or 'R' ("The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.").

#### 4.3 Deuterated Monitoring Compounds (DMC) and Surrogates

DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are only used for the VOC GC/MS analysis. Table 3 presents the recommended DMCs with their associated target compounds.

Table 3 –DMC and Associated Target Compounds		
DMC (alphabetical)	Associated Target Compounds	
1,1,2,2-Tetrachloroethane- <i>d</i> <sub>2</sub>	1,1,2,2-Tetrachloroethane	1,2-Dibromo-3-chloropropane
1,1-Dichloroethane- <i>d</i> <sub>2</sub>	trans-1,2-Dichloroethene 1,1-Dichloroethene	cis-1,2-Dichloroethene
1,2-Dichlorobenzene- <i>d</i> <sub>4</sub>	Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene	1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene
1,2-Dichloroethane- <i>d</i> <sub>4</sub>	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl-tert-butyl ether	1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
1,2-Dichloropropane- <i>d</i> <sub>6</sub>	Cyclohexane Methylcyclohexane	1,2-Dichloropropane Bromodichloromethane
1,4-Dioxane- <i>d</i> <sub>8</sub>	1,4-Dioxane	
2-Butanone- <i>d</i> <sub>5</sub>	Acetone	2-Butanone
2-Hexanone- <i>d</i> <sub>5</sub>	4-Methyl-2-pentanone	2-Hexanone
Benzene- <i>d</i> <sub>6</sub>	Benzene	
Chloroethane- <i>d</i> <sub>5</sub>	Dichlorodifluoromethane Chloromethane Bromomethane	Chloroethane Carbon disulfide
Chloroform- <i>d</i>	1,1-Dichloroethane Bromochloromethane Chloroform	Dibromochloromethane Bromoform
Toluene- <i>d</i> <sub>8</sub>	Trichloroethene Toluene Tetrachloroethene Ethylbenzene	o-Xylene m,p-Xylene Styrene Isopropylbenzene
trans-1,3-Dichloropropene- <i>d</i> <sub>4</sub>	cis-1,3-Dichloropropene trans-1,3-Dichloropropene	1,1,2-Trichloroethane
Vinyl Chloride- <i>d</i> <sub>3</sub>	Vinyl chloride	

Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other DMCs or surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. All samples (blanks, spiked samples, project samples, QC samples) should contain DMCs or surrogates. If a sample does not contain DMC or surrogates or the method does not require surrogates (e.g., WI GRO), professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of the DMC or surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each DMC or surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

While not required for WI GRO analyses, surrogates are required for PVOC. The method minimum surrogate recovery is 80%; there is no method maximum recovery. Use professional judgment when evaluating surrogates for WI GRO samples.

Table 4 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

<b>Table 4 – Guidelines for Single DMC or Surrogate</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
%R > Upper Limit	Qualify with 'J+'	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
%R within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.  
The analyte may or may not be present in the sample.

Table 5 includes guidance where multiple surrogates are analyzed per analytical fraction.

<b>Table 5 – Guidelines for Multiple DMC or Surrogates</b>		
<b>Criteria</b>	<b>Recommended Action for Associated Data</b>	
	<b>Detect</b>	<b>Non-Detect</b>
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with 'J+'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.  
The analyte may or may not be present in the sample.

#### 4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (typically 20 or less samples of the same matrix - WI GRO requires an additional LCSD analyzed at the end of 20 samples)
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 6 – Guidelines for Laboratory Control Samples		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R > Upper Limit	Qualify with 'J+' or use professional judgment	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
RPD > Upper Limit	Qualify with 'J' or use professional judgment	
%R and RPD within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

#### 4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Ideally, blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The MS/MSD duplicate pairs may be substituted for laboratory duplicates. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with U, UB, <, or R. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times ( $>5x$ ) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times ( $>5x$ ) the RL, professional judgment should be used to determine if qualification is appropriate.

<b>Table 7 – Guidelines for Laboratory Duplicates</b>	
<b>% RPD</b>	<b>Recommended Action for Associated Data</b>
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with 'J'

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

#### 4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with U, UB, <, or R. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs  $\leq 30\%$  for aqueous samples and  $\leq 40\%$  for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times ( $>5x$ ) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times ( $>5x$ ) the RL, professional judgment should be used to determine if qualification is appropriate.

<b>Table 8 – Guidelines for Field Duplicates</b>	
<b>% RPD</b>	<b>Recommended Action for Associated Data</b>
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are $\leq 5x$ RL, no action is required
RPD > Upper Limit	Both results are $> 5x$ RL, consider qualifying with 'J'

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.



#### 4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to GRO in the WI method)
- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added ( $>4x$ ), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

Table 9 – Guidelines for Matrix Spikes		
Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R > Upper Limit	Qualify with 'J+' or use professional judgment	No qualification
%R < Lower Limit	Qualify with 'J-' or 'R', use professional judgment	
RPD > Upper Limit	Qualify with 'J' or use professional judgment	
%R and RPD within Limits	No qualification	

J+ = The result is an estimated quantity and may be biased high.

J- = The result is an estimated quantity and may be biased low.

J = Estimated detected value. Either certain QC criteria were not met or the concentration is between the laboratory's detection and quantitation limits.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

#### 4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

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## **Appendix B**


### **Boring Log and Well Construction Report**



Route To: Watershed/Wastewater ☐ Waste Management ☐  
Remediation/Redevelopment ☒ Other ☐

Page 1 of 1

Facility/Project Name <b>Post-Incident Site Investigation</b>		License/Permit/Monitoring Number <b>NA</b>		Boring Number <b>IW-1</b>	
Boring Drilled By: Name of crew chief (first, last) and Firm <b>Adam Reimer Twin Ports Testing</b>		Date Drilling Started <b>11/10/2023</b>		Date Drilling Completed <b>11/10/2023</b>	
WI Unique Well No.		DNR Well ID No.		Common Well Name <b>IW-1</b>	
		Final Static Water Level		Surface Elevation <b>658.2 Feet</b>	
				Borehole Diameter <b>8.3 inches</b>	
Local Grid Origin <input type="checkbox"/> (estimated: <input type="checkbox"/> ) or Boring Location <input checked="" type="checkbox"/>		State Plane <b>562,097 N, 1,448,797 E S/C/N</b>		Local Grid Location <input type="checkbox"/> N <input type="checkbox"/> E <input type="checkbox"/> S <input type="checkbox"/> W	
NE 1/4 of NW 1/4 of Section 36, T 49 N, R 14 W		Lat _____ Long _____		Feet _____ Feet _____	
Facility ID <b>816009590</b>		County <b>Douglas</b>		County Code <b>16</b>	
				Civil Town/City/ or Village <b>Superior</b>	

Sample		Blow Counts	Depth In Feet	Soil/Rock Description And Geologic Origin For Each Major Unit	U S C S	Graphic Log	Well Diagram	PID (ppm)	Soil Properties					RQD/ Comments
Number and Type	Length Att. & Recovered (in)								Compressive Strength	Moisture Content	G/S/F %	Color	Plasticity Index	
	60 48			FILL: silty sand with gravel; brown; moist; angular gravel	CH			0.0						
				FILL: fat clay; red brown; firm; moist				0.0						
				FAT CLAY: (CH) black staining; moist; firm; high plasticity	CH			0.9						
				soft asphalt chunks at 1.5 feet				0.8						
	60 60		5	FAT CLAY: (CH) red brown; moist; very firm; high plasticity	CH			0.7						
				Some mottling around 5.0 feet				0.6						
	60 60		10		CH			1.2						
								1.2						
			15		CH			1.5						
				End of boring 15.0 feet										
				Analytical Soil Samples Collected: IW-1_1.5-2.0 ft: PAHs and PVOCS										

I hereby certify that the information on this form is true and correct to the best of my knowledge.

Signature <i>Jes Pedersen</i>	Firm <b>Barr Engineering Co</b>	Tel: Fax:
----------------------------------	------------------------------------	--------------

This form is authorized by Chapters 281, 283, 289, 291, 292, 293, 295, and 299, Wis. Stats. Completion of this form is mandatory. Failure to file this form may result in forfeiture of between \$10 and \$25,000, or imprisonment for up to one year, depending on the program and conduct involved. Personally identifiable information on this form is not intended to be used for any other purpose. NOTE: See instructions for more information, including where the completed form should be sent.

Route To: Watershed/Wastewater ☐ Waste Management ☐  
Remediation/Redevelopment ☒ Other ☐

MONITORING WELL CONSTRUCTION  
Form 4400-113A Rev. 7-98

Facility/Project Name Post-Incident Site Investigation	Local Grid Location of Well ft. <input type="checkbox"/> N. <input type="checkbox"/> E. ft. <input type="checkbox"/> S. <input type="checkbox"/> W.	Well Name IW-1
Facility License, Permit or Monitoring No. NA	Local Grid Origin <input type="checkbox"/> (estimated: <input type="checkbox"/> ) or Well Location <input checked="" type="checkbox"/> Lat. " ' " Long. " ' " or	Wis. Unique Well No. DNR Well Number
Facility ID 816009590	St. Plane 562,097 ft. N. 1,448,797 ft. E. S/C/N	Date Well Installed 11/10/2023
Type of Well Well Code 11/mw	Section Location of Waste/Source NE 1/4 of NW 1/4 of Sec. 36 T. 49 N. R. 14 <input type="checkbox"/> E <input checked="" type="checkbox"/> W	Well Installed By: (Person's Name and Firm) Adam Reimer
Distance from Waste/ Source ft.	Location of Well Relative to Waste/Source u <input type="checkbox"/> Upgradient s <input type="checkbox"/> Sidegradient d <input type="checkbox"/> Downgradient n <input checked="" type="checkbox"/> Not Known	Twin Ports Testing
Enf. Stds. Apply <input type="checkbox"/>	Gov. Lot Number	

A. Protective pipe, top elevation	661.24 ft. MSL	1. Cap and lock? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
B. Well casing, top elevation	660.92 ft. MSL	2. Protective cover pipe: a. Inside diameter: 6.0 in. b. Length: 5.0 ft. c. Material: Steel <input checked="" type="checkbox"/> 04 Other <input type="checkbox"/>
C. Land surface elevation	658.2 ft. MSL	d. Additional protection? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No If yes, describe: Bumper posts
D. Surface seal, bottom	655.2 ft. MSL or 3.0 ft.	3. Surface seal: Bentonite <input checked="" type="checkbox"/> 30 Concrete <input type="checkbox"/> 01 Other <input type="checkbox"/>
12. USCS classification of soil near screen: GP <input type="checkbox"/> GM <input type="checkbox"/> GC <input type="checkbox"/> GW <input type="checkbox"/> SW <input type="checkbox"/> SP <input type="checkbox"/> SM <input type="checkbox"/> SC <input type="checkbox"/> ML <input type="checkbox"/> MH <input type="checkbox"/> CL <input type="checkbox"/> CH <input checked="" type="checkbox"/> Bedrock <input type="checkbox"/>		4. Material between well casing and protective pipe: Bentonite <input checked="" type="checkbox"/> 30 Other <input type="checkbox"/>
13. Sieve analysis attached?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	5. Annular space seal: a. Granular/Chipped Bentonite <input checked="" type="checkbox"/> 33 b. Lbs/gal mud weight . . . Bentonite-sand slurry <input type="checkbox"/> 35 c. Lbs/gal mud weight . . . Bentonite slurry <input type="checkbox"/> 31 d. % Bentonite . . . Bentonite-cement grout <input type="checkbox"/> 50 e. Ft <sup>3</sup> volume added for any of the above f. How installed: Tremie <input checked="" type="checkbox"/> 01 Tremie pumped <input type="checkbox"/> 02 Gravity <input type="checkbox"/> 08
14. Drilling method used:	Rotary <input type="checkbox"/> 50 Hollow Stem Auger <input checked="" type="checkbox"/> 41 Other <input type="checkbox"/>	6. Bentonite seal: a. Bentonite granules <input type="checkbox"/> 33 b. <input type="checkbox"/> 1/4 in. <input checked="" type="checkbox"/> 3/8 in. <input type="checkbox"/> 1/2 in. Bentonite chips <input checked="" type="checkbox"/> 32 c. Other <input type="checkbox"/>
15. Drilling fluid used: Water <input type="checkbox"/> 02 Air <input type="checkbox"/> 01 Drilling Mud <input type="checkbox"/> 03 None <input checked="" type="checkbox"/> 99		7. Fine sand material: Manufacturer, product name & mesh size a. #10 Fine Sand b. Volume added 0.25 ft <sup>3</sup>
16. Drilling additives used? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No Describe		8. Filter pack material: Manufacturer, product name & mesh size a. Red Flint #30 b. Volume added 4 ft <sup>3</sup>
17. Source of water (attach analysis, if required):		9. Well casing: Flush threaded PVC schedule 40 <input checked="" type="checkbox"/> 23 Flush threaded PVC schedule 80 <input type="checkbox"/> 24 Other <input type="checkbox"/>
E. Bentonite seal, top	658.2 ft. MSL or 0.0 ft.	10. Screen material: PVC a. Screen Type: Factory cut <input type="checkbox"/> 11 Continuous slot <input checked="" type="checkbox"/> 01 Other <input type="checkbox"/>
F. Fine sand, top	655.2 ft. MSL or 3.0 ft.	b. Manufacturer c. Slot size: 0.010 in. d. Slotted length: 10.0 ft.
G. Filter pack, top	654.7 ft. MSL or 3.5 ft.	11. Backfill material (below filter pack): None <input checked="" type="checkbox"/> 14 Other <input type="checkbox"/>
H. Screen joint, top	654.7 ft. MSL or 3.5 ft.	
I. Well bottom	643.7 ft. MSL or 14.5 ft.	
J. Filter pack, bottom	643.7 ft. MSL or 14.5 ft.	
K. Borehole, bottom	643.2 ft. MSL or 15.0 ft.	
L. Borehole, diameter	8.3 in.	
M. O.D. well casing	2.38 in.	
N. I.D. well casing	2.00 in.	

I hereby certify that the information on this form is true and correct to the best of my knowledge.

Signature *Adam Reimer*

Firm Barr Engineering Co

Tel:  
Fax:

Please complete both Forms 4400-113A and 4400-113B and return them to the appropriate DNR office and bureau. Completion of these reports is required by chs. 160, 281, 283, 289, 291, 292, 293, 295, and 299, Wis. Stats., and ch. NR 141, Wis. Adm. Code. In accordance with chs. 281, 289, 291, 292, 293, 295, and 299, Wis. Stats., failure to file these forms may result in a forfeiture of between \$10 and \$25,000, or imprisonment for up to one year, depending on the program and conduct involved. Personally identifiable information on these forms is not intended to be used for any other purpose. NOTE: See the instructions for more information, including where the completed forms should be sent.

Route to: Watershed/Wastewater ☐ Waste Management ☐  
Remediation/Redevelopment ☒ Other ☐

Facility/Project Name SRC - Post-Incident Site Investigation	County Name Douglas	Well Name IW-1
Facility License, Permit or Monitoring Number 816009590	County Code 1 6	Wis. Unique Well Number _____
		DNR Well ID Number _____

1. Can this well be purged dry? ☒ Yes ☐ No

2. Well development method

- surged with bailer and bailed ☐ 4 1  
 surged with bailer and pumped ☒ 6 1  
 surged with block and bailed ☐ 4 2  
 surged with block and pumped ☐ 6 2  
 surged with block, bailed and pumped ☐ 7 0  
 compressed air ☐ 2 0  
 bailed only ☐ 1 0  
 pumped only ☐ 5 1  
 pumped slowly ☐ 5 0  
 Other ☐

3. Time spent developing well \_\_\_\_\_ 6 5 min.

4. Depth of well (from top of well casing) \_\_\_\_\_ 1 6 . 7 ft.

5. Inside diameter of well \_\_\_\_\_ 2 . 0 0 in.

6. Volume of water in filter pack and well casing \_\_\_\_\_ 1 . 8 gal.

7. Volume of water removed from well \_\_\_\_\_ 1 0 . 0 gal.

8. Volume of water added (if any) \_\_\_\_\_ N/A gal.

9. Source of water added N/A

10. Analysis performed on water added? ☐ Yes ☒ No  
(If yes, attach results)

17. Additional comments on development:

Well surged with bailer for 30 minutes and pumped for 30 minute before going dry

	Before Development	After Development
11. Depth to Water (from top of well casing)	a. _____ 5 . 7 2 ft.	_____ 1 6 . 6 8 ft.
Date	b. $\frac{0}{m} \frac{3}{m} / \frac{1}{d} \frac{2}{d} / \frac{2}{y} \frac{0}{y} \frac{2}{y} \frac{4}{y}$	$\frac{0}{m} \frac{3}{m} / \frac{1}{d} \frac{2}{d} / \frac{2}{y} \frac{0}{y} \frac{2}{y} \frac{4}{y}$
Time	c. $\frac{0}{h} \frac{9}{m} : \frac{0}{m} \frac{5}{m}$ <input checked="" type="checkbox"/> a.m. <input type="checkbox"/> p.m.	$\frac{1}{h} \frac{0}{m} : \frac{1}{h} \frac{0}{m}$ <input checked="" type="checkbox"/> a.m. <input type="checkbox"/> p.m.
12. Sediment in well bottom	_____ 0 0 : 0 inches	_____ 0 0 : 0 inches
13. Water clarity	Clear <input type="checkbox"/> 1 0 Turbid <input checked="" type="checkbox"/> 1 5 (Describe) reddish brown	Clear <input checked="" type="checkbox"/> 2 0 Turbid <input checked="" type="checkbox"/> 2 5 (Describe) clear

Fill in if drilling fluids were used and well is at solid waste facility:

14. Total suspended \_\_\_\_\_ N/A mg/l \_\_\_\_\_ N/A mg/l  
solids

15. COD \_\_\_\_\_ N/A mg/l \_\_\_\_\_ N/A mg/l

16. Well developed by: Name (first, last) and Firm

First Name: Jes Last Name: Pedersen

Firm: Barr Engineering Co.

Name and Address of Facility Contact/Owner/Responsible Party

First Name: Joseph Last Name: Pearson

Facility/Firm: Superior Refining Company LLC (SRC)

Street: 2400 Stinson Avenue

City/State/Zip: Superior, WI 54880

I hereby certify that the above information is true and correct to the best of my knowledge.

Signature:

Print Name: Jes Pedersen

Firm: Barr Engineering Co.





## **Appendix C**

### **Photo Log**

**Appendix C**  
**Site Investigation Report – Groundwater**

**Photograph Log**

Superior, Wisconsin  
November 2023, March/April 2024

Photo #	Comments
1	Photo 1: Proposed IW-1 location; photo facing north; photo taken on 11/10/2023.
2	Photo 2: Well installation at IW-1; photo facing north; photo taken on 11/10/2023.
3	Photo 3: IW-1 after installation; photo facing north; photo taken on 11/10/2023.
4	Photo 4: IW-1 after bollard installation; photo facing northeast; photo taken on 11/16/2023.
5	Photo 5: IW-1 development water immediately after surging, photo facing east; photo taken on 3/12/2024.
6	Photo 6: IW-1 development water clearing up after pumping, photo facing east; photo taken on 3/12/2024.
7	Photo 7: IW-1 during sampling event; photo facing east; photo taken on 3/27/2024.
8	Photo 8: IW-1 during sampling event; photo facing east; photo taken on 4/24/2024.





Photo 1: Proposed IW-1 location; photo facing north; photo taken on 11/10/2023.



Photo 2: Well installation at IW-1; photo facing north; photo taken on 11/10/2023.





Photo 3: IW-1 after installation; photo facing north; photo taken on 11/10/2023.



Photo 4: IW-1 after bollard installation; photo facing northeast; photo taken on 11/16/2023.





Photo 5: IW-1 development water immediately after surging, photo facing east; photo taken on 3/12/2024.



Photo 6: IW-1 development water clearing up after pumping, photo facing east; photo taken on 3/12/2024.





Photo 7: IW-1 during sampling event; photo facing east; photo taken on 3/27/2024.



Photo 8: IW-1 during sampling event; photo facing east; photo taken on 4/24/2024.



## **Appendix D**

### **Laboratory Reports**





# Analytical Laboratory Report

Report ID: S60291.01(01)+QC02  
Generated on 04/19/2024

**Report to**  
Attention: David Beattie  
Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802  
  
Phone: 218-348-9051    FAX:  
Email: David.Beattie@cenovus.com


**Report produced by**  
Merit Laboratories, Inc.  
2680 East Lansing Drive  
East Lansing, MI 48823  
  
Phone: (517) 332-0167    FAX: (517) 332-6333  
  
Contacts for report questions:  
John Lavery (johnlavery@meritlabs.com)  
Barbara Ball (bball@meritlabs.com)

Additional Contacts: Matthew Turner, Joseph Pearson, Lynette Carney, Guy Partch, Kaitlin Montz, Terri A. Olson, Barr Data Manager

**Report Summary**  
Lab Sample ID(s): S60291.01-S60291.05  
Project: SRC 49161497.04 100 101  
Collected Date(s): 03/27/2024  
Submitted Date/Time: 03/28/2024 10:25  
Sampled by: JSP  
P.O. #: PO

**Table of Contents**

- Cover Page (Page 1)
- General Report Notes (Page 2)
- Report Narrative (Page 2)
- Laboratory Accreditations (Page 3)
- Qualifier Descriptions (Page 3)
- Glossary of Abbreviations (Page 3)
- Method Summary (Page 4)
- Parameter Summary (Page 5)
- Sample Summary (Page 6)
- QC Report (Pages 17-37)

  
Maya Murshak  
Technical Director



# Analytical Laboratory Report

## General Report Notes

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Analytical results relate only to the samples tested, in the condition received by the laboratory.

Methods may be modified for improved performance.

Results reported on a dry weight basis where applicable.

'Not detected' indicates that parameter was not found at a level equal to or greater than the reporting limit (RL).

When MDL results are provided, then 'Not detected' indicates that parameter was not found at a level equal to or greater than the MDL.

40 CFR Part 136 Table II Required Containers, Preservation Techniques and Holding Times for the Clean Water Act specify that samples for acrolein and acrylonitrile, and 2-chloroethylvinyl ether need to be preserved at a pH in the range of 4 to 5 or if not preserved, analyzed within 3 days of sampling.

QA/QC corresponding to this analytical report is a separate document with the same Merit ID reference and is available upon request.

Starred (\*) analytes are not NY NELAP accredited.

Samples are held by the lab for 30 days from the final report date unless a written request to hold longer is provided by the client.

Report shall not be reproduced except in full, without the written approval of Merit Laboratories, Inc.

Limits for drinking water samples, are listed as the MCL Limits (Maximum Contaminant Level Concentrations)

PFAS requirement: Section 9.3.8 of U.S. EPA Method 537.1 states "If the method analyte(s) found in the Field Sample is present in the FRB at a concentration greater than 1/3 the MRL, then all samples collected with that FRB are invalid and must be recollected and reanalyzed."

Samples submitted without an accompanying FRB may not be acceptable for compliance purposes.

Wisconsin PFAs analysis: MDL = LOD; RL = LOQ. LOD and LOQ are adjusted for dilution.

All accreditations/certifications held by this laboratory are listed on page 3. Not all accreditations/certifications are applicable to this report.

For a specific list of accredited analytes, please feel free to contact the laboratory or visit <https://www.meritlabs.com/certifications>.

## Report Narrative

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There is no additional narrative for this analytical report

## Laboratory Accreditations (For Reference Only)

Authority	Accreditation ID
Michigan DEQ	#9956
DOD ELAP & ISO/IEC 17025:2017	#69699 PJLA Testing
WBENC	#2005110032
Ohio VAP	#CL0002
Indiana DOH	#C-MI-07
New York NELAC	#11814
North Carolina DENR	#680
North Carolina DOH	#26702
Pennsylvania DEP	#68-05884
Wisconsin DNR	FID# 399147320

## Qualifier Descriptions

Qualifier	Description
!	Result is outside of stated limit criteria
B	Compound also found in associated method blank
E	Concentration exceeds calibration range
F	Analysis run outside of holding time
G	Estimated result due to extraction run outside of holding time
H	Sample submitted and run outside of holding time
I	Matrix interference with internal standard
J	Estimated value less than reporting limit, but greater than MDL
L	Elevated reporting limit due to low sample amount
M	Result reported to MDL not RDL
O	Analysis performed by outside laboratory. See attached report.
R	Preliminary result
S	Surrogate recovery outside of control limits
T	No correction for total solids
X	Elevated reporting limit due to matrix interference
Y	Elevated reporting limit due to high target concentration
b	Value detected less than reporting limit, but greater than MDL
e	Reported value estimated due to interference
j	Analyte also found in associated method blank
o	Associated EIS outside of control limits
p	Benzo(b)Fluoranthene and Benzo(k)Fluoranthene integrated as one peak.
q	Qualifier ion ratio outside of control limits
x	Preserved from bulk sample

## Glossary of Abbreviations

Abbreviation	Description
RL/RDL	Reporting Limit
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
SW	EPA SW 846 (Soil and Wastewater) Methods
E	EPA Methods
SM	Standard Methods
LN	Linear
BR	Branched



# Analytical Laboratory Report

## Method Summary

Method	Version
N/A	Not Applicable
WI SPE	PFAS by LCMSMS Per Wisconsin DNR Document EA-19-0001

## Parameter Summary

Parameter	Synonym	Cas #
PFBA	Perfluorobutanoic Acid	375-22-4
PFPeA	Perfluoropentanoic Acid	2706-90-3
4:2 FTSA	4:2 Fluorotelomer Sulfonic Acid	757124-72-4
PFHxA	Perfluorohexanoic Acid	307-24-4
PFBS	Perfluorobutane sulfonic Acid	375-73-5
PFHpA	Perfluoroheptanoic Acid	375-85-9
PFPeS	Perfluoropentane Sulfonic Acid	2706-91-4
6:2 FTSA	6:2 Fluorotelomer Sulfonic Acid	27619-97-2
PFOA	Perfluorooctanoic Acid	335-67-1
PFHxS	Perfluorohexane Sulfonic Acid	355-46-4
PFHxS-LN	Perfluorohexane Sulfonic Acid - LN	355-46-4-LN
PFHxS-BR	Perfluorohexane Sulfonic Acid - BR	355-46-4-BR
PFNA	Perfluorononanoic Acid	375-95-1
8:2 FTSA	8:2 Fluorotelomer Sulfonic Acid	39108-34-4
PFHpS	Perfluoroheptane Sulfonic Acid	375-92-8
PFDA	Perfluorodecanoic Acid	335-76-2
N-MeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9
EtFOSAA	N-Ethyl Perfluorooctane Sulfonamidoacetic Acid	2991-50-6
PFOS	Perfluorooctane Sulfonic Acid	1763-23-1
PFOS-LN	Perfluorooctane Sulfonic Acid - LN	1763-23-1-LN
PFOS-BR	Perfluorooctane Sulfonic Acid - BR	1763-23-1-BR
PFUnDA	Perfluoroundecanoic Acid	2058-94-8
PFNS	Perfluorononane Sulfonic Acid	68259-12-1
PFDoDA	Perfluorododecanoic Acid	307-55-1
PFDS	Perfluorodecane Sulfonic Acid	335-77-3
PFTTrDA	Perfluorotridecanoic Acid	72629-94-8
FOSA	Perfluorooctane Sulfonamide	754-91-6
PFTeDA	Perfluorotetradecanoic Acid	376-06-7
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanone1-sulfonic acid	756426-58-1
ADONA	4,8-dioxa-3H-perfluorononanoic acid	919005-14-4
HFPO-DA	Hexafluoropropylene oxide dimer	13252-13-6
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
NMeFOSAM	N-Methylperfluorooctanesulfonamide	31506-32-8
NEtFOSAM	N-Ethylperfluorooctanesulfonamide	4151-50-2
NMeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
NEtFOSE	N-Ethylperfluorooctanesulfonamidoethanol	1691-99-2





# Analytical Laboratory Report

## Sample Summary (5 samples)

Sample ID	Sample Tag	Matrix	Collected Date/Time
S60291.01	FB-01	Water	03/27/24 09:30
S60291.02	RB-01	Water	03/27/24 09:55
S60291.03	RB-02	Water	03/27/24 10:10
S60291.04	FD-01	Water	03/27/24 00:01
S60291.05	IW-1	Groundwater	03/27/24 10:40



# Analytical Laboratory Report

Lab Sample ID: S60291.01  
Sample Tag: FB-01  
Collected Date/Time: 03/27/2024 09:30  
Matrix: Water  
COC Reference: 597206

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
1	250mL Plastic	Trizma	Yes	3.3	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	04/04/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	316.69/33.26	WI SPE	04/04/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 16:38, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	0.36	1.8	0.25	ng/L	0.0141	375-22-4	J
PFPeA*	Not detected	1.8	0.20	ng/L	0.0141	2706-90-3	
4:2 FTSA*	Not detected	1.8	0.32	ng/L	0.0141	757124-72-4	
PFHxA*	Not detected	1.8	0.23	ng/L	0.0141	307-24-4	
PFBS*	Not detected	1.8	0.17	ng/L	0.0141	375-73-5	
PFHpA*	Not detected	1.8	0.39	ng/L	0.0141	375-85-9	
PFPeS*	Not detected	1.8	0.21	ng/L	0.0141	2706-91-4	
6:2 FTSA*	Not detected	1.8	0.48	ng/L	0.0141	27619-97-2	
PFOA*	Not detected	1.8	0.37	ng/L	0.0141	335-67-1	
PFHxS*	Not detected	1.8	0.55	ng/L	0.0141	355-46-4	
PFHxS-LN*	Not detected	1.8	0.55	ng/L	0.0141	355-46-4-LN	
PFHxS-BR*	Not detected	1.8	0.55	ng/L	0.0141	355-46-4-BR	
PFNA*	Not detected	1.8	0.37	ng/L	0.0141	375-95-1	
8:2 FTSA*	Not detected	1.8	0.63	ng/L	0.0141	39108-34-4	
PFHpS*	Not detected	1.8	0.45	ng/L	0.0141	375-92-8	
PFDA*	Not detected	1.8	0.48	ng/L	0.0141	335-76-2	
N-MeFOSAA*	0.30	1.8	0.30	ng/L	0.0141	2355-31-9	J
EtFOSAA*	Not detected	1.8	0.58	ng/L	0.0141	2991-50-6	
PFOS*	Not detected	1.8	0.32	ng/L	0.0141	1763-23-1	
PFOS-LN*	Not detected	1.8	0.32	ng/L	0.0141	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.32	ng/L	0.0141	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.41	ng/L	0.0141	2058-94-8	
PFNS*	Not detected	1.8	0.48	ng/L	0.0141	68259-12-1	
PFDODA*	Not detected	1.8	0.89	ng/L	0.0141	307-55-1	
PFDS*	Not detected	1.8	0.54	ng/L	0.0141	335-77-3	
PFTTrDA*	Not detected	1.8	0.61	ng/L	0.0141	72629-94-8	
FOSA*	Not detected	1.8	0.51	ng/L	0.0141	754-91-6	
PFTeDA*	Not detected	1.8	0.72	ng/L	0.0141	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.69	ng/L	0.0141	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.38	ng/L	0.0141	756426-58-1	
ADONA*	0.33	1.8	0.32	ng/L	0.0141	919005-14-4	J
HFPO-DA*	Not detected	1.8	0.28	ng/L	0.0141	13252-13-6	
PFDoS*	Not detected	1.8	0.54	ng/L	0.0141	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.54	ng/L	0.0141	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.54	ng/L	0.0141	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S60291.01 (continued)

Sample Tag: FB-01

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 16:38, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.63	ng/L	0.0141	24448-09-7	
NEtFOSE*	Not detected	1.8	0.69	ng/L	0.0141	1691-99-2	



# Analytical Laboratory Report

Lab Sample ID: S60291.02  
Sample Tag: RB-01  
Collected Date/Time: 03/27/2024 09:55  
Matrix: Water  
COC Reference: 597206

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
1	250mL Plastic	Trizma	Yes	3.3	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	04/04/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	304.56/33.33	WI SPE	04/04/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 16:54, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	0.39	1.8	0.26	ng/L	0.0147	375-22-4	J
PFPeA*	Not detected	1.8	0.21	ng/L	0.0147	2706-90-3	
4:2 FTSA*	Not detected	1.8	0.34	ng/L	0.0147	757124-72-4	
PFHxA*	Not detected	1.8	0.24	ng/L	0.0147	307-24-4	
PFBS*	Not detected	1.8	0.18	ng/L	0.0147	375-73-5	
PFHpA*	Not detected	1.8	0.41	ng/L	0.0147	375-85-9	
PFPeS*	Not detected	1.8	0.22	ng/L	0.0147	2706-91-4	
6:2 FTSA*	Not detected	1.8	0.50	ng/L	0.0147	27619-97-2	
PFOA*	Not detected	1.8	0.38	ng/L	0.0147	335-67-1	
PFHxS*	Not detected	1.8	0.57	ng/L	0.0147	355-46-4	
PFHxS-LN*	Not detected	1.8	0.57	ng/L	0.0147	355-46-4-LN	
PFHxS-BR*	Not detected	1.8	0.57	ng/L	0.0147	355-46-4-BR	
PFNA*	Not detected	1.8	0.38	ng/L	0.0147	375-95-1	
8:2 FTSA*	Not detected	1.8	0.66	ng/L	0.0147	39108-34-4	
PFHpS*	Not detected	1.8	0.47	ng/L	0.0147	375-92-8	
PFDA*	Not detected	1.8	0.50	ng/L	0.0147	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.31	ng/L	0.0147	2355-31-9	
EtFOSAA*	Not detected	1.8	0.60	ng/L	0.0147	2991-50-6	
PFOS*	Not detected	1.8	0.34	ng/L	0.0147	1763-23-1	
PFOS-LN*	Not detected	1.8	0.34	ng/L	0.0147	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.34	ng/L	0.0147	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.43	ng/L	0.0147	2058-94-8	
PFNS*	Not detected	1.8	0.50	ng/L	0.0147	68259-12-1	
PFDODA*	Not detected	1.8	0.93	ng/L	0.0147	307-55-1	
PFDS*	Not detected	1.8	0.56	ng/L	0.0147	335-77-3	
PFTTrDA*	Not detected	1.8	0.63	ng/L	0.0147	72629-94-8	
FOSA*	Not detected	1.8	0.53	ng/L	0.0147	754-91-6	
PFTeDA*	Not detected	1.8	0.75	ng/L	0.0147	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.72	ng/L	0.0147	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.40	ng/L	0.0147	756426-58-1	
ADONA*	Not detected	1.8	0.34	ng/L	0.0147	919005-14-4	
HFPO-DA*	Not detected	1.8	0.29	ng/L	0.0147	13252-13-6	
PFDoS*	Not detected	1.8	0.56	ng/L	0.0147	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.56	ng/L	0.0147	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.56	ng/L	0.0147	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S60291.02 (continued)

Sample Tag: RB-01

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 16:54, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.66	ng/L	0.0147	24448-09-7	
NEtFOSE*	Not detected	1.8	0.72	ng/L	0.0147	1691-99-2	





# Analytical Laboratory Report

Lab Sample ID: S60291.03  
Sample Tag: RB-02  
Collected Date/Time: 03/27/2024 10:10  
Matrix: Water  
COC Reference: 597206

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
1	250mL Plastic	Trizma	Yes	3.3	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	04/04/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	311.41/33.28	WI SPE	04/04/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 17:10, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	0.39	1.8	0.26	ng/L	0.0144	375-22-4	J
PFPeA*	Not detected	1.8	0.20	ng/L	0.0144	2706-90-3	
4:2 FTSA*	Not detected	1.8	0.33	ng/L	0.0144	757124-72-4	
PFHxA*	Not detected	1.8	0.23	ng/L	0.0144	307-24-4	
PFBS*	Not detected	1.8	0.17	ng/L	0.0144	375-73-5	
PFHpA*	Not detected	1.8	0.40	ng/L	0.0144	375-85-9	
PFPeS*	Not detected	1.8	0.22	ng/L	0.0144	2706-91-4	
6:2 FTSA*	Not detected	1.8	0.49	ng/L	0.0144	27619-97-2	
PFOA*	Not detected	1.8	0.37	ng/L	0.0144	335-67-1	
PFHxS*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4	
PFHxS-LN*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4-LN	
PFHxS-BR*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4-BR	
PFNA*	Not detected	1.8	0.37	ng/L	0.0144	375-95-1	
8:2 FTSA*	Not detected	1.8	0.65	ng/L	0.0144	39108-34-4	
PFHpS*	Not detected	1.8	0.46	ng/L	0.0144	375-92-8	
PFDA*	Not detected	1.8	0.49	ng/L	0.0144	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.30	ng/L	0.0144	2355-31-9	
EtFOSAA*	Not detected	1.8	0.59	ng/L	0.0144	2991-50-6	
PFOS*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1	
PFOS-LN*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.42	ng/L	0.0144	2058-94-8	
PFNS*	Not detected	1.8	0.49	ng/L	0.0144	68259-12-1	
PFDODA*	Not detected	1.8	0.91	ng/L	0.0144	307-55-1	
PFDS*	Not detected	1.8	0.55	ng/L	0.0144	335-77-3	
PFTTrDA*	Not detected	1.8	0.62	ng/L	0.0144	72629-94-8	
FOSA*	Not detected	1.8	0.52	ng/L	0.0144	754-91-6	
PFTeDA*	Not detected	1.8	0.73	ng/L	0.0144	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.71	ng/L	0.0144	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.39	ng/L	0.0144	756426-58-1	
ADONA*	Not detected	1.8	0.33	ng/L	0.0144	919005-14-4	
HFPO-DA*	Not detected	1.8	0.29	ng/L	0.0144	13252-13-6	
PFDoS*	Not detected	1.8	0.55	ng/L	0.0144	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.55	ng/L	0.0144	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.55	ng/L	0.0144	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S60291.03 (continued)

Sample Tag: RB-02

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 17:10, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.65	ng/L	0.0144	24448-09-7	
NEtFOSE*	Not detected	1.8	0.71	ng/L	0.0144	1691-99-2	



# Analytical Laboratory Report

Lab Sample ID: S60291.04  
Sample Tag: FD-01  
Collected Date/Time: 03/27/2024 00:01  
Matrix: Water  
COC Reference: 597206

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
3	250mL Plastic	Trizma	Yes	3.3	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	04/04/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	313.24/33.18	WI SPE	04/04/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 17:27, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	32	1.8	0.26	ng/L	0.0143	375-22-4	
PFPeA*	190	1.8	0.20	ng/L	0.0143	2706-90-3	
4:2 FTSA*	0.74	1.8	0.33	ng/L	0.0143	757124-72-4	IJ
PFHxA*	74	1.8	0.23	ng/L	0.0143	307-24-4	
PFBS*	3.9	1.8	0.17	ng/L	0.0143	375-73-5	
PFHpA*	14	1.8	0.40	ng/L	0.0143	375-85-9	
PFPeS*	2.0	1.8	0.21	ng/L	0.0143	2706-91-4	
6:2 FTSA*	18	1.8	0.49	ng/L	0.0143	27619-97-2	
PFOA*	3.8	1.8	0.37	ng/L	0.0143	335-67-1	
PFHxS*	4.2	1.8	0.56	ng/L	0.0143	355-46-4	
PFHxS-LN*	2.7	1.8	0.56	ng/L	0.0143	355-46-4-LN	
PFHxS-BR*	1.7	1.8	0.56	ng/L	0.0143	355-46-4-BR	J
PFNA*	0.59	1.8	0.37	ng/L	0.0143	375-95-1	J
8:2 FTSA*	Not detected	1.8	0.64	ng/L	0.0143	39108-34-4	
PFHpS*	Not detected	1.8	0.46	ng/L	0.0143	375-92-8	
PFDA*	Not detected	1.8	0.49	ng/L	0.0143	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.30	ng/L	0.0143	2355-31-9	
EtFOSAA*	Not detected	1.8	0.59	ng/L	0.0143	2991-50-6	
PFOS*	0.50	1.8	0.33	ng/L	0.0143	1763-23-1	J
PFOS-LN*	Not detected	1.8	0.33	ng/L	0.0143	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.33	ng/L	0.0143	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.41	ng/L	0.0143	2058-94-8	
PFNS*	Not detected	1.8	0.49	ng/L	0.0143	68259-12-1	
PFDODA*	Not detected	1.8	0.90	ng/L	0.0143	307-55-1	
PFDS*	Not detected	1.8	0.54	ng/L	0.0143	335-77-3	
PFTTrDA*	Not detected	1.8	0.61	ng/L	0.0143	72629-94-8	
FOSA*	Not detected	1.8	0.51	ng/L	0.0143	754-91-6	
PFTeDA*	Not detected	1.8	0.73	ng/L	0.0143	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.70	ng/L	0.0143	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.39	ng/L	0.0143	756426-58-1	
ADONA*	Not detected	1.8	0.33	ng/L	0.0143	919005-14-4	
HFPO-DA*	Not detected	1.8	0.29	ng/L	0.0143	13252-13-6	
PFDoS*	Not detected	1.8	0.54	ng/L	0.0143	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.54	ng/L	0.0143	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.54	ng/L	0.0143	4151-50-2	

I-Matrix interference with internal standard J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S60291.04 (continued)

Sample Tag: FD-01

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 17:27, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.64	ng/L	0.0143	24448-09-7	
NEtFOSE*	Not detected	1.8	0.70	ng/L	0.0143	1691-99-2	



# Analytical Laboratory Report

Lab Sample ID: S60291.05  
Sample Tag: IW-1  
Collected Date/Time: 03/27/2024 10:40  
Matrix: Groundwater  
COC Reference: 597206

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
3	250mL Plastic	Trizma	Yes	3.3	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	04/04/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	311.63/33.34	WI SPE	04/04/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 17:43, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	32	1.8	0.26	ng/L	0.0144	375-22-4	
PFPeA*	200	1.8	0.20	ng/L	0.0144	2706-90-3	
4:2 FTSA*	0.81	1.8	0.33	ng/L	0.0144	757124-72-4	J
PFHxA*	74	1.8	0.23	ng/L	0.0144	307-24-4	
PFBS*	3.8	1.8	0.17	ng/L	0.0144	375-73-5	
PFHpA*	14	1.8	0.40	ng/L	0.0144	375-85-9	
PFPeS*	1.8	1.8	0.22	ng/L	0.0144	2706-91-4	
6:2 FTSA*	19	1.8	0.49	ng/L	0.0144	27619-97-2	
PFOA*	3.8	1.8	0.37	ng/L	0.0144	335-67-1	
PFHxS*	4.2	1.8	0.56	ng/L	0.0144	355-46-4	
PFHxS-LN*	2.7	1.8	0.56	ng/L	0.0144	355-46-4-LN	
PFHxS-BR*	1.8	1.8	0.56	ng/L	0.0144	355-46-4-BR	J
PFNA*	0.53	1.8	0.37	ng/L	0.0144	375-95-1	J
8:2 FTSA*	Not detected	1.8	0.65	ng/L	0.0144	39108-34-4	
PFHpS*	Not detected	1.8	0.46	ng/L	0.0144	375-92-8	
PFDA*	Not detected	1.8	0.49	ng/L	0.0144	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.30	ng/L	0.0144	2355-31-9	
EtFOSAA*	Not detected	1.8	0.59	ng/L	0.0144	2991-50-6	
PFOS*	0.50	1.8	0.33	ng/L	0.0144	1763-23-1	J
PFOS-LN*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.42	ng/L	0.0144	2058-94-8	
PFNS*	Not detected	1.8	0.49	ng/L	0.0144	68259-12-1	
PFDODA*	Not detected	1.8	0.91	ng/L	0.0144	307-55-1	
PFDS*	Not detected	1.8	0.55	ng/L	0.0144	335-77-3	
PFTTrDA*	Not detected	1.8	0.62	ng/L	0.0144	72629-94-8	
FOSA*	Not detected	1.8	0.52	ng/L	0.0144	754-91-6	
PFTeDA*	Not detected	1.8	0.73	ng/L	0.0144	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.71	ng/L	0.0144	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.39	ng/L	0.0144	756426-58-1	
ADONA*	Not detected	1.8	0.33	ng/L	0.0144	919005-14-4	
HFPO-DA*	Not detected	1.8	0.29	ng/L	0.0144	13252-13-6	
PFDoS*	Not detected	1.8	0.55	ng/L	0.0144	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.55	ng/L	0.0144	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.55	ng/L	0.0144	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL





# Analytical Laboratory Report

Lab Sample ID: S60291.05 (continued)

Sample Tag: IW-1

WI 33 PFAs, Method: WI SPE, Run Date: 04/04/24 17:43, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.65	ng/L	0.0144	24448-09-7	
NEtFOSE*	Not detected	1.8	0.71	ng/L	0.0144	1691-99-2	



# Quality Control Report

Report ID: S60291.01(01)+QC02  
Generated on 05/10/2024

Report to

Attention: David Beattie  
Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802

Phone: 218-348-9051    FAX:

Report Produced by

Merit Laboratories  
2680 East Lansing Drive  
East Lansing, MI 48823

Phone: (517) 332-0167    FAX: (517) 332-6333

Report Summary

Lab Sample ID(s): S60291.01-S60291.05  
Project: SRC 49161497.04 100 101  
Submitted Date/Time: 03/28/2024 10:25  
Sampled by: JSP  
P.O. #: 8401702587

QC Report Sections

Cover Page (Page 17)  
Analysis Summary (Pages 18-22)  
Prep Batch Summary (Page 23)  
Surrogates per QC Sample (Page 24)  
Internal Standards per Lab Sample (Pages 25-29)  
Internal Standards per QC Sample (Pages 30-33)  
Batch QC Results (Pages 34-37)

QC Report Narrative

Blank re-processed to show J-values

Report Flag Descriptions

\*: QC result is outside of indicated control limits  
W: Surrogate result not applicable due to sample dilution

I certify that this data package is in compliance with the terms and conditions of the program, and project, and contractual requirements both technically and for completeness. Release of the data contained in this hardcopy data package and its computer-readable data submitted has been authorized by the Quality Assurance Manager and his/her designee, as verified by the following signature.

Barbara Ball  
Quality Assurance Manager

QC Report - Analysis Summary

Lab Sample ID: S60291.01  
Sample Tag: FB-01  
Collected Date/Time: 03/27/2024 09:30  
Matrix: Water  
COC Reference: 597206

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	04/04/24 16:38	CI240404WISPE	WS240404W1	Yes	BLK/LCS/MS/DUP

QC Report - Analysis Summary

Lab Sample ID: S60291.02  
Sample Tag: RB-01  
Collected Date/Time: 03/27/2024 09:55  
Matrix: Water  
COC Reference: 597206

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	04/04/24 16:54	CI240404WISPE	WS240404W1	Yes	BLK/LCS/MS/DUP

## QC Report - Analysis Summary

Lab Sample ID: S60291.03

Sample Tag: RB-02

Collected Date/Time: 03/27/2024 10:10

Matrix: Water

COC Reference: 597206

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b><i>Organics - Volatiles</i></b>						
WI 33 PFAs	WI SPE	04/04/24 17:10	CI240404WISPE	WS240404W1	Yes	BLK/LCS/MS/DUP



QC Report - Analysis Summary

Lab Sample ID: S60291.04  
Sample Tag: FD-01  
Collected Date/Time: 03/27/2024 00:01  
Matrix: Water  
COC Reference: 597206

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	04/04/24 17:27	CI240404WISPE	WS240404W1	Yes	BLK/LCS/MS/DUP

QC Report - Analysis Summary

Lab Sample ID: S60291.05  
Sample Tag: IW-1  
Collected Date/Time: 03/27/2024 10:40  
Matrix: Groundwater  
COC Reference: 597206

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	04/04/24 17:43	CI240404WISPE	WS240404W1	Yes	BLK/LCS/MS/DUP

## QC Report - Prep Batch Summary

### Organics - Volatiles, Prep Batch ID: WS240404W1

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

Sample ID	Analysis	Method	Run Date/Time	Batch ID
S60291.01	WI 33 PFAs	WI SPE	04/04/24 16:38	CI240404WISPE
S60291.02	WI 33 PFAs	WI SPE	04/04/24 16:54	CI240404WISPE
S60291.03	WI 33 PFAs	WI SPE	04/04/24 17:10	CI240404WISPE
S60291.04	WI 33 PFAs	WI SPE	04/04/24 17:27	CI240404WISPE
S60291.05	WI 33 PFAs	WI SPE	04/04/24 17:43	CI240404WISPE

## QC Report - Surrogates per QC Sample

### Organics - Volatiles, Prep Batch ID: WS240404W1

QC Types: BLK/LCS/MS/DUP

#### Blank (BLK)

Lab Sample ID: CI240404WISPE.BLK240404

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 15:32, Prep Date: 04/04/2024, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Laboratory Control Sample (LCS)

Lab Sample ID: CI240404WISPE.LCS240404

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 16:21, Prep Date: 04/04/2024, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Matrix Spike (MS)

Lab Sample ID: CI240404WISPE.6043101M, Parent Sample ID: S60431.01

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 21:15, Prep Date: 04/04/2024, Matrix: WW, Dilution: 0.0145

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Duplicate (DUP)

Lab Sample ID: CI240404WISPE.6043201D, Parent Sample ID: S60432.01

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 22:53, Prep Date: 04/04/2024, Matrix: WW, Dilution: 0.014

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S60291.01

Sample Tag: FB-01

Collected Date/Time: 03/27/2024 09:30

Matrix: Water

COC Reference: 597206

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 16:38, Matrix: WW, Dilution: 0.0141

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		109.7	25	150.0
M2-6:2FTSA		74.0	25	150.0
M2-8:2FTSA		105.5	25	150.0
M2PFTeDA		79.4	25	150.0
M3PFBS		102.3	25	150.0
M3PFHxS		106.3	25	150.0
M4PFHpA		108.4	25	150.0
M5PFHxA		106.1	25	150.0
M5PFPeA		104.8	25	150.0
M6PFDA		95.2	25	150.0
M7PFUnDA		96.4	25	150.0
M8FOSA		104.0	10	150.0
M8PFOA		105.0	25	150.0
M8PFOS		97.3	25	150.0
M9-PFNA		105.3	25	150.0
MPFBA		108.0	25	150.0
MPFDoDA		90.7	25	150.0
d3N-MeFOSAA		99.1	25	150.0
d5EtFOSAA		94.3	25	150.0
MHFPODA		103.1	25	150.0
d-N-EtFOSA-M		69.5	10	150.0
d-N-MeFOSA-M		86.9	10	150.0
d7-N-MeFOSE-M		87.7	10	150.0
d9-N-EtFOSE-M		73.7	10	150.0



# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S60291.02

Sample Tag: RB-01

Collected Date/Time: 03/27/2024 09:55

Matrix: Water

COC Reference: 597206

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 16:54, Matrix: WW, Dilution: 0.0147

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		117.6	25	150.0
M2-6:2FTSA		70.6	25	150.0
M2-8:2FTSA		122.0	25	150.0
M2PFTeDA		90.1	25	150.0
M3PFBS		110.4	25	150.0
M3PFHxS		108.1	25	150.0
M4PFHpA		115.9	25	150.0
M5PFHxA		111.3	25	150.0
M5PFPeA		107.8	25	150.0
M6PFDA		108.9	25	150.0
M7PFUnDA		111.2	25	150.0
M8FOSA		107.0	10	150.0
M8PFOA		107.7	25	150.0
M8PFOS		110.2	25	150.0
M9-PFNA		110.4	25	150.0
MPFBA		112.7	25	150.0
MPFDoDA		109.6	25	150.0
d3N-MeFOSAA		115.2	25	150.0
d5EtFOSAA		110.3	25	150.0
MHFPODA		107.4	25	150.0
d-N-EtFOSA-M		73.2	10	150.0
d-N-MeFOSA-M		78.1	10	150.0
d7-N-MeFOSE-M		91.5	10	150.0
d9-N-EtFOSE-M		81.7	10	150.0

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S60291.03

Sample Tag: RB-02

Collected Date/Time: 03/27/2024 10:10

Matrix: Water

COC Reference: 597206

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 17:10, Matrix: WW, Dilution: 0.0144

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		117.5	25	150.0
M2-6:2FTSA		63.0	25	150.0
M2-8:2FTSA		114.7	25	150.0
M2PFTeDA		79.3	25	150.0
M3PFBS		105.8	25	150.0
M3PFHxS		109.0	25	150.0
M4PFHpA		113.7	25	150.0
M5PFHxA		111.4	25	150.0
M5PFPeA		109.0	25	150.0
M6PFDA		104.5	25	150.0
M7PFUnDA		105.9	25	150.0
M8FOSA		104.2	10	150.0
M8PFOA		104.1	25	150.0
M8PFOS		105.5	25	150.0
M9-PFNA		109.9	25	150.0
MPFBA		114.5	25	150.0
MPFDoDA		95.6	25	150.0
d3N-MeFOSAA		100.7	25	150.0
d5EtFOSAA		102.3	25	150.0
MHFPODA		108.5	25	150.0
d-N-EtFOSA-M		69.4	10	150.0
d-N-MeFOSA-M		79.0	10	150.0
d7-N-MeFOSE-M		89.7	10	150.0
d9-N-EtFOSE-M		76.4	10	150.0

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S60291.04

Sample Tag: FD-01

Collected Date/Time: 03/27/2024 00:01

Matrix: Water

COC Reference: 597206

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 17:27, Matrix: WW, Dilution: 0.0143

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA	*	150.2	25	150.0
M2-6:2FTSA		78.6	25	150.0
M2-8:2FTSA		105.0	25	150.0
M2PFTeDA		81.9	25	150.0
M3PFBS		97.5	25	150.0
M3PFHxS		100.6	25	150.0
M4PFHpA		105.9	25	150.0
M5PFHxA		102.4	25	150.0
M5PFPeA		88.5	25	150.0
M6PFDA		94.0	25	150.0
M7PFUnDA		95.1	25	150.0
M8FOSA		93.0	10	150.0
M8PFOA		97.9	25	150.0
M8PFOS		100.6	25	150.0
M9-PFNA		100.4	25	150.0
MPFBA		89.7	25	150.0
MPFDoDA		87.4	25	150.0
d3N-MeFOSAA		97.3	25	150.0
d5EtFOSAA		100.4	25	150.0
MHFPODA		100.4	25	150.0
d-N-EtFOSA-M		72.1	10	150.0
d-N-MeFOSA-M		79.4	10	150.0
d7-N-MeFOSE-M		85.7	10	150.0
d9-N-EtFOSE-M		72.8	10	150.0

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S60291.05

Sample Tag: IW-1

Collected Date/Time: 03/27/2024 10:40

Matrix: Groundwater

COC Reference: 597206

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 17:43, Matrix: WW, Dilution: 0.0144

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		147.4	25	150.0
M2-6:2FTSA		75.5	25	150.0
M2-8:2FTSA		109.6	25	150.0
M2PFTeDA		65.8	25	150.0
M3PFBS		99.2	25	150.0
M3PFHxS		99.1	25	150.0
M4PFHpA		109.6	25	150.0
M5PFHxA		105.6	25	150.0
M5PFPeA		90.8	25	150.0
M6PFDA		93.8	25	150.0
M7PFUnDA		85.8	25	150.0
M8FOSA		99.6	10	150.0
M8PFOA		101.1	25	150.0
M8PFOS		96.7	25	150.0
M9-PFNA		104.3	25	150.0
MPFBA		93.2	25	150.0
MPFDoDA		76.2	25	150.0
d3N-MeFOSAA		91.1	25	150.0
d5EtFOSAA		85.7	25	150.0
MHFPODA		100.0	25	150.0
d-N-EtFOSA-M		59.3	10	150.0
d-N-MeFOSA-M		74.7	10	150.0
d7-N-MeFOSE-M		69.6	10	150.0
d9-N-EtFOSE-M		62.0	10	150.0

# QC Report - Internal Standards per QC Sample

**Organics - Volatiles, Prep Batch ID: WS240404W1**

QC Types: BLK/LCS/MS/DUP

## Blank (BLK)

Lab Sample ID: CI240404WISPE.BLK240404

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 15:32, Prep Date: 04/04/2024, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		107.3	25	150.0
M2-6:2FTSA		76.0	25	150.0
M2-8:2FTSA		113.6	25	150.0
M2PFTeDA		83.9	25	150.0
M3PFBS		104.3	25	150.0
M3PFHxS		108.6	25	150.0
M4PFHpA		109.2	25	150.0
M5PFHxA		106.3	25	150.0
M5PFPeA		105.6	25	150.0
M6PFDA		99.6	25	150.0
M7PFUnDA		90.8	25	150.0
M8FOSA		104.1	10	150.0
M8PFOA		103.9	25	150.0
M8PFOS		105.7	25	150.0
M9-PFNA		108.0	25	150.0
MPFBA		108.9	25	150.0
MPFDoDA		87.8	25	150.0
d3N-MeFOSAA		100.5	25	150.0
d5EtFOSAA		97.0	25	150.0
MHFPODA		104.7	25	150.0
d-N-EtFOSA-M		68.2	10	150.0
d-N-MeFOSA-M		84.2	10	150.0
d7-N-MeFOSE-M		85.9	10	150.0
d9-N-EtFOSE-M		75.6	10	150.0



# QC Report - Internal Standards per QC Sample

## Laboratory Control Sample (LCS)

Lab Sample ID: CI240404WISPE.LCS240404

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 16:21, Prep Date: 04/04/2024, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		103.9	25	150.0
M2-6:2FTSA		72.8	25	150.0
M2-8:2FTSA		110.5	25	150.0
M2PFTeDA		83.1	25	150.0
M3PFBS		101.8	25	150.0
M3PFHxS		101.4	25	150.0
M4PFHpA		107.3	25	150.0
M5PFHxA		104.1	25	150.0
M5PFPeA		103.2	25	150.0
M6PFDA		95.3	25	150.0
M7PFUnDA		95.3	25	150.0
M8FOSA		98.3	10	150.0
M8PFOA		100.1	25	150.0
M8PFOS		102.5	25	150.0
M9-PFNA		101.2	25	150.0
MPFBA		107.5	25	150.0
MPFDoDA		95.4	25	150.0
d3N-MeFOSAA		99.7	25	150.0
d5EtFOSAA		98.0	25	150.0
MHFPODA		102.8	25	150.0
d-N-EtFOSA-M		71.2	10	150.0
d-N-MeFOSA-M		76.0	10	150.0
d7-N-MeFOSE-M		92.4	10	150.0
d9-N-EtFOSE-M		77.4	10	150.0

# QC Report - Internal Standards per QC Sample

## Matrix Spike (MS)

Lab Sample ID: CI240404WISPE.6043101M, Parent Sample ID: S60431.01

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 21:15, Prep Date: 04/04/2024, Matrix: WW, Dilution: 0.0145

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA	*	437.2	25	150.0
M2-6:2FTSA	*	206.9	25	150.0
M2-8:2FTSA	*	422.8	25	150.0
M2PFTeDA		94.2	25	150.0
M3PFBS		85.6	25	150.0
M3PFHxS		92.7	25	150.0
M4PFHpA		86.9	25	150.0
M5PFHxA		88.8	25	150.0
M5PFPeA		66.7	25	150.0
M6PFDA		102.6	25	150.0
M7PFUnDA		103.6	25	150.0
M8FOSA		94.2	10	150.0
M8PFOA		85.3	25	150.0
M8PFOS		90.2	25	150.0
M9-PFNA		91.5	25	150.0
MPFBA		71.6	25	150.0
MPFDoDA		102.5	25	150.0
d3N-MeFOSAA		147.3	25	150.0
d5EtFOSAA	*	163.4	25	150.0
MHFPODA		72.2	25	150.0
d-N-EtFOSA-M		75.5	10	150.0
d-N-MeFOSA-M		84.1	10	150.0
d7-N-MeFOSE-M		73.2	10	150.0
d9-N-EtFOSE-M		74.0	10	150.0

# QC Report - Internal Standards per QC Sample

## Duplicate (DUP)

Lab Sample ID: CI240404WISPE.6043201D, Parent Sample ID: S60432.01

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 22:53, Prep Date: 04/04/2024, Matrix: WW, Dilution: 0.014

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA	*	183.2	25	150.0
M2-6:2FTSA		91.6	25	150.0
M2-8:2FTSA		123.5	25	150.0
M2PFTeDA		74.0	25	150.0
M3PFBS		98.0	25	150.0
M3PFHxS		96.5	25	150.0
M4PFHpA		99.2	25	150.0
M5PFHxA		99.0	25	150.0
M5PFPeA		91.2	25	150.0
M6PFDA		101.0	25	150.0
M7PFUnDA		96.0	25	150.0
M8FOSA		90.0	10	150.0
M8PFOA		99.0	25	150.0
M8PFOS		97.3	25	150.0
M9-PFNA		103.5	25	150.0
MPFBA		91.7	25	150.0
MPFDoDA		83.5	25	150.0
d3N-MeFOSAA		96.6	25	150.0
d5EtFOSAA		91.7	25	150.0
MHFPODA		92.8	25	150.0
d-N-EtFOSA-M		70.4	10	150.0
d-N-MeFOSA-M		78.3	10	150.0
d7-N-MeFOSE-M		74.9	10	150.0
d9-N-EtFOSE-M		72.4	10	150.0

# QC Report - Batch QC Results

## Organics - Volatiles, Prep Batch ID: WS240404W1

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

### Blank (BLK)

Lab Sample ID: CI240404WISPE.BLK240404

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 15:32, Prep Date: 04/04/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Conc	RDL	MDL	Units
PFBA	J*	0.4797	2.00	0.29	ng/l
PFPeA	J*	0.4819	2.00	0.22	ng/l
PFBS	J*	0.3496	2.00	0.19	ng/l
4:2 FTSA		ND	2.00	0.37	ng/l
PFHxA	J*	0.3242	2.00	0.26	ng/l
PFPeS		ND	2.00	0.24	ng/l
HFPO-DA		ND	2.00	0.3	ng/l
PFHxS-BR		ND	2.00	0.62	ng/l
PFHpA		ND	2.00	0.45	ng/l
PFHxS		ND	2.00	0.62	ng/l
PFHxS-LN		ND	2.00	0.62	ng/l
ADONA		ND	2.00	0.37	ng/l
6:2 FTSA		ND	2.00	0.54	ng/l
PFOA		ND	2.00	0.42	ng/l
PFHpS		ND	2.00	0.51	ng/l
PFOS-BR		ND	2.00	0.37	ng/l
PFOS		ND	2.00	0.37	ng/l
PFOS-LN		ND	2.00	0.37	ng/l
PFNA		ND	2.00	0.42	ng/l
9CL-PF3ONS		ND	2.00	0.43	ng/l
PFNS		ND	2.00	0.54	ng/l
8:2 FTSA		ND	2.00	0.72	ng/l
PFDA		ND	2.00	0.54	ng/l
N-MeFOSAA		ND	2.00	0.34	ng/l
PFDS		ND	2.00	0.61	ng/l
PFUnDA		ND	2.00	0.46	ng/l
EtFOSAA		ND	2.00	0.66	ng/l
FOSA		ND	2.00	0.58	ng/l
11CL-PF3OUdS		ND	2.00	0.78	ng/l
PFDoDA		ND	2.00	1.0	ng/l
PFDOS		ND	2.00	0.61	ng/l
PFTTrDA		ND	2.00	0.69	ng/l
NMeFOSE		ND	2.00	0.72	ng/l
NMeFOSAM		ND	2.00	0.61	ng/l
PFTeDA		ND	2.00	0.82	ng/l
NEtFOSAM		ND	2.00	0.61	ng/l
NEtFOSE		ND	2.00	0.78	ng/l

### Laboratory Control Sample (LCS)

Lab Sample ID: CI240404WISPE.LCS240404

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 16:21, Prep Date: 04/04/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCS Conc	% Rec	LCL	UCL
PFBA		0.00	4.00	4.51	112.8	50	150
PFPeA		0.00	4.00	4.32	108.0	50	150
PFBS		0.00	4.00	4.55	113.8	50	150
4:2 FTSA		0.00	4.00	3.99	99.8	50	150

# QC Report - Batch QC Results

## Organics - Volatiles, Prep Batch ID: WS240404W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

## Laboratory Control Sample (LCS) (continued)

Lab Sample ID: CI240404WISPE.LCS240404

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 16:21, Prep Date: 04/04/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCS Conc	% Rec	LCL	UCL
PFHxA		0.00	4.00	4.47	111.8	50	150
PFPeS		0.00	4.00	4.70	117.5	50	150
HFPO-DA		0.00	4.00	3.95	98.8	50	150
PFHpA		0.00	4.00	4.67	116.8	50	150
PFHxS		0.00	4.00	4.68	117.0	50	150
ADONA		0.00	4.00	4.79	119.8	50	150
6:2 FTSA		0.00	4.00	3.76	94.0	50	150
PFOA		0.00	4.00	4.48	112.0	50	150
PFHpS		0.00	4.00	4.25	106.3	50	150
PFOS		0.00	4.00	4.51	112.8	50	150
PFNA		0.00	4.00	4.42	110.5	50	150
9CL-PF3ONS		0.00	4.00	4.14	103.5	50	150
PFNS		0.00	4.00	4.17	104.3	50	150
8:2 FTSA		0.00	4.00	4.38	109.5	50	150
PFDA		0.00	4.00	4.41	110.3	50	150
N-MeFOSAA		0.00	4.00	3.95	98.8	50	150
PFDS		0.00	4.00	4.16	104.0	50	150
PFUnDA		0.00	4.00	4.59	114.8	50	150
EtFOSAA		0.00	4.00	4.65	116.3	50	150
FOSA		0.00	4.00	4.22	105.5	50	150
11CL-PF3OUdS		0.00	4.00	4.19	104.8	50	150
PFDODA		0.00	4.00	4.54	113.5	50	150
PFDOS		0.00	4.00	3.34	83.5	50	150
PFTTrDA		0.00	4.00	4.33	108.3	50	150
NMeFOSE		0.00	4.00	4.37	109.3	50	150
NMeFOSAM		0.00	4.00	4.35	108.7	50	150
PFTeDA		0.00	4.00	4.68	117.0	50	150
NEtFOSAM		0.00	4.00	4.50	112.5	50	150
NEtFOSE		0.00	4.00	4.89	122.2	50	150

## Matrix Spike (MS)

Lab Sample ID: CI240404WISPE.6043101M, Parent Sample ID: S60431.01

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 21:15, Prep Date: 04/04/2024, Matrix: WW, Dilution: 0.0145

Analyte	Flags	Orig Conc	Spike	MS Conc	% Rec	LCL	UCL
PFBA	*	35	3.63	41	165.3	50	150
PFPeA	*	120	3.63	130	275.5	50	150
4:2 FTSA		0.43	3.63	3.4	81.8	50	150
PFHxA		65	3.63	69	110.2	50	150
PFBS	*	5.1	3.63	23	493.1	50	150
PFHpA	*	39	3.63	53	385.7	50	150
PFPeS		3.7	3.63	7.6	107.4	50	150
6:2 FTSA		24	3.63	29	137.7	50	150
PFOA		21	3.63	24	82.6	50	150
PFHxS		28	3.63	31	82.6	50	150
PFNA		5.6	3.63	9.3	101.9	50	150
8:2 FTSA		4.6	3.63	9.2	126.7	50	150



# QC Report - Batch QC Results

## Organics - Volatiles, Prep Batch ID: WS240404W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

## Matrix Spike (MS) (continued)

Lab Sample ID: CI240404WISPE.6043101M, Parent Sample ID: S60431.01

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 21:15, Prep Date: 04/04/2024, Matrix: WW, Dilution: 0.0145

Analyte	Flags	Orig Conc	Spike	MS Conc	% Rec	LCL	UCL
PFHpS		0.83	3.63	4.1	90.1	50	150
PFDA		0.84	3.63	4.4	98.1	50	150
N-MeFOSAA		0	3.63	3.8	104.7	50	150
EtFOSAA		0	3.63	3.7	101.9	50	150
PFOS		28	3.63	32	110.2	50	150
PFUnDA		0	3.63	4.5	124.0	50	150
PFNS		0	3.63	4.0	110.2	50	150
PFDODA		0	3.63	4.3	118.5	50	150
PFDS		0	3.63	4.2	115.7	50	150
PFTTrDA		0	3.63	4.0	110.2	50	150
FOSA		0	3.63	4.1	112.9	50	150
PFTeDA		0	3.63	4.4	121.2	50	150
11CL-PF3OUdS		0	3.63	3.4	93.7	50	150
9CL-PF3ONS		0	3.63	3.7	101.9	50	150
ADONA		0	3.63	4.4	121.2	50	150
HFPO-DA		0	3.63	3.8	104.7	50	150
PFDOS		0	3.63	2.8	77.1	50	150
NMeFOSAM		0	3.63	3.8	104.7	50	150
NEtFOSAM		0	3.63	3.9	107.4	50	150
NMeFOSE		0	3.63	3.9	107.4	50	150
NEtFOSE		0	3.63	3.8	104.7	50	150

## Duplicate (DUP)

Lab Sample ID: CI240404WISPE.6043201D, Parent Sample ID: S60432.01

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 22:53, Prep Date: 04/04/2024, Matrix: WW, Dilution: 0.014

Analyte	Flags	Orig Conc	Dup Conc	RPD	RPD CL
PFBA		42	43	2.4	30.0
PFPeA		85	85	0.0	30.0
4:2 FTSA		ND	ND	NC	30.0
PFHxA		35	35	0.0	30.0
PFBS		ND	ND	NC	30.0
PFHpA		7.5	7.4	1.3	30.0
PFPeS		ND	ND	NC	30.0
6:2 FTSA		2.9	3.0	3.4	30.0
PFOA	J	0.97	1.0	3.0	30.0
PFHxS		ND	ND	NC	30.0
PFHxS-LN		ND	ND	NC	30.0
PFHxS-BR		ND	ND	NC	30.0
PFNA		ND	ND	NC	30.0
8:2 FTSA		ND	ND	NC	30.0
PFHpS		ND	ND	NC	30.0
PFDA		ND	ND	NC	30.0
N-MeFOSAA		ND	ND	NC	30.0
EtFOSAA		ND	ND	NC	30.0
PFOS		ND	ND	NC	30.0
PFOS-LN		ND	ND	NC	30.0

## QC Report - Batch QC Results

### Organics - Volatiles, Prep Batch ID: WS240404W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

### Duplicate (DUP) (continued)

Lab Sample ID: CI240404WISPE.6043201D, Parent Sample ID: S60432.01

Run in Batch: CI240404WISPE, Run Date: 04/04/2024 22:53, Prep Date: 04/04/2024, Matrix: WW, Dilution: 0.014

Analyte	Flags	Orig Conc	Dup Conc	RPD	RPD CL
PFOS-BR		ND	ND	NC	30.0
PFUnDA		ND	ND	NC	30.0
PFNS		ND	ND	NC	30.0
PFDoDA		ND	ND	NC	30.0
PFDS		ND	ND	NC	30.0
PFTTrDA		ND	ND	NC	30.0
FOSA		ND	ND	NC	30.0
PFTeDA		ND	ND	NC	30.0
11CL-PF3OUdS		ND	ND	NC	30.0
9CL-PF3ONS		ND	ND	NC	30.0
ADONA		ND	ND	NC	30.0
HFPO-DA		ND	ND	NC	30.0
PFDOS		ND	ND	NC	30.0
NMeFOSAM		ND	ND	NC	30.0
NEtFOSAM		ND	ND	NC	30.0
NMeFOSE		ND	ND	NC	30.0
NEtFOSE		ND	ND	NC	30.0

# Merit Laboratories Login Checklist

Lab Set ID:S60291

Client:BARR (Barr Engineering)

Project: SRC 49161497.04 100 101

Submitted:03/28/2024 10:25 Login User: MMC

Attention: David Beattie

Address: Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802

Phone: 218-348-9051

FAX:

Email: David.Beattie@cenovus.com

Selection	Description	Note
<b>Sample Receiving</b>		
01.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples are received at 4C +/- 2C Thermometer # IR 3.3
02.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Received on ice/ cooling process begun
03.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples shipped FedEx
04.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Samples left in 24 hr. drop box
05.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Are there custody seals/tape or is the drop box locked
<b>Chain of Custody</b>		
06.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	COC adequately filled out
07.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	COC signed and relinquished to the lab
08.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Sample tag on bottles match COC
09.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Subcontracting needed? Subcontacted to:
<b>Preservation</b>		
10.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Do sample have correct chemical preservation
11.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Completed pH checks on preserved samples? (no VOAs)
12.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Did any samples need to be preserved in the lab?
<b>Bottle Conditions</b>		
13.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	All bottles intact
14.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Appropriate analytical bottles are used
15.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Merit bottles used
16.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Sufficient sample volume received
17.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Samples require laboratory filtration
18.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples submitted within holding time
19.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Do water VOC or TOX bottles contain headspace

Corrective action for all exceptions is to call the client and to notify the project manager.

Client Review By: \_\_\_\_\_ Date: \_\_\_\_\_



# Barr Engineering Co. Chain of Custody

## Sample Origination State

☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ NV ☐ TX ☐ UT ☒ WI ☐ WY ☐ Other: \_\_\_\_\_

REPORT TO	INVOICE TO
Company: <u>Barr Engineering</u>	Company: <u>Barr Engineering</u>
Address: <u>325 S. Lake Ave</u>	Address: _____
Address: <u>Duluth, MN 55802</u>	Address: _____
Name: <u>Lynette Carney</u>	Name: <u>SAME</u>
email: <u>LCarney@barr.com</u>	email: _____
Copy to: <u>BarrDM@barr.com</u>	P.O. _____
Project Name: <u>SRL</u>	Barr Project No: <u>49161497.04 100101</u>

Location	Sample Depth			Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code
	Start	Stop	Unit (m./ft. or in.)			
1. <u>FB-01</u>	—	—	—	<u>03/27/2024</u>	<u>09:30</u>	<u>WQ</u>
2. <u>RB-01</u>	—	—	—	<u>03/27/2024</u>	<u>09:55</u>	<u>WQ</u>
3. <u>RB-02</u>	—	—	—	<u>03/27/2024</u>	<u>10:10</u>	<u>WQ</u>
4. <u>FD-01</u>	—	—	—	<u>03/27/2024</u>	—	<u>WQ</u>
5. <u>IW-1</u>	—	—	—	<u>03/27/2024</u>	<u>10:40</u>	<u>GW</u>
6.						
7.						
8.						
9.						
10.						

Analysis Requested											
Water						Soil					

COC Number: **Nº 597206**

COC 1 of 1

Matrix Code:	Preservative Code:
GW = Groundwater	A = None
SW = Surface Water	B = HCl
DW = Drinking Water	C = HNO <sub>3</sub>
PW = Pore Water	D = H <sub>2</sub> SO <sub>4</sub>
WW = Waste Water	E = NaOH
WQ = TB, FB, EB, etc.	F = MeOH
W = Unspecified	G = NaHSO <sub>4</sub>
S = Soil/Solid	H = Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>
SD = Sediment	I = Ascorbic Acid
SQ = MeOH blank	J = Zn Acetate
OTH = Other (Oil, etc.)	K = Other

Preservative Code: \_\_\_\_\_

Field Filtered Y/N: \_\_\_\_\_

BARR USE ONLY		Relinquished by:	On Ice?	Date	Time	Received by:	Date	Time
Sampled by: <u>JSP</u>	Relinquished by: <u>JSP</u>	<u>N</u>	<u>03/27/24</u>	<u>14:30</u>				
Barr Proj. Manager: <u>LMC</u>	Relinquished by: <u>FedEx</u>	<u>N</u>	<u>3/28/24</u>	<u>1025</u>				
Barr DQ Manager: <u>TAO</u>	Samples Shipped VIA: <input type="checkbox"/> Ground Courier <input type="checkbox"/> Air Carrier				Air Bill Number:			
Lab Name: <u>Merit</u>	<input type="checkbox"/> Sampler <input type="checkbox"/> Other: _____							
Lab Location: <u>East Lansing, MI</u>	Lab WO: _____				Temperature on Receipt (°C): <u>3.3</u>	Custody Seal Intact? <input type="checkbox"/> Y <input type="checkbox"/> N <input type="checkbox"/> None	Requested Due Date: <u>3/28/24 1025</u>	

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Scan and email: a copy to BarrDM@barr.com for tracking and filing procedures

HRLGSTDFORMS(Chain of Custody Form 2022 RLG Rev. 10/14/2021)

# WI PFAS (33)

Perfluorobutanoic acid (PFBA)
Perfluorobutanesulfonic acid (PFBS)
Perfluoropentanoic acid (PFPeA)
Perfluoropentanesulfonic acid (PFPeS)
Perfluorohexanoic acid (PFHxA)
Perfluorohexanesulfonic acid (PFHxS)
Perfluoroheptanoic acid (PFHpA)
Perfluoroheptane sulfonic acid (PFHpS)
Perfluorooctanoic acid (PFOA)
Perfluorooctanesulfonic acid (PFOS)
Perfluorononanoic acid (PFNA)
Perfluorononanesulfonic acid (PFNS)
Perfluorodecanoic acid (PFDA)
Perfluorodecane sulfonic acid (PFDS)
Perfluoroundecanoic acid (PFUnA)
Perfluorododecanoic acid (PFDoA)
Perfluorododecanesulfonic Acid (PFDoS or PFDoDS)
Perfluorooctanesulfonamide (PFOSA)
Perfluorotridecanoic acid (PFTTrDA)
Perfluorotetradecanoic acid (PFTDA or PFTA or PFTeDA)
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)
Hexfluoropropylene oxide dimer acid (HFPO-DA) aka Gen-X
<sup>11</sup> chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) - F53B Minor
<sup>9</sup> chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) - F53B Major
4,8-dioxa-3H-perfluorononanoic acid (DONA; NaDONA in MDH)
N-Methyl perfluorooctane sulfonamide (MeFOSA)
N-Ethyl perfluorooctane sulfonamide (EtFOSA)
N-Methyl perfluorooctane sulfonamidoethanol (MeFOSE)
N-Ethyl perfluorooctane sulfonamidoethanol (EtFOSE)
4:2 FTS
6:2 FTS
8:2 FTS





# Analytical Laboratory Report

Report ID: S61354.01(01)+QC02  
Generated on 05/17/2024

**Report to**  
Attention: David Beattie  
Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802  
  
Phone: 218-348-9051    FAX:  
Email: David.Beattie@cenovus.com


**Report produced by**  
Merit Laboratories, Inc.  
2680 East Lansing Drive  
East Lansing, MI 48823  
  
Phone: (517) 332-0167    FAX: (517) 332-6333  
  
Contacts for report questions:  
John Lavery (johnlavery@meritlabs.com)  
Barbara Ball (bball@meritlabs.com)

Additional Contacts: Matthew Turner, Joseph Pearson, Lynette Carney, Guy Partch, Kaitlin Montz, Terri A. Olson, Barr Data Manager

**Report Summary**  
Lab Sample ID(s): S61354.01-S61354.05  
Project: SRC 49161497.04 100 101  
Collected Date(s): 04/24/2024  
Submitted Date/Time: 04/25/2024 09:40  
Sampled by: JSP  
P.O. #: 8401702587

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- Parameter Summary (Page 5)
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- QC Report (Pages 17-42)

  
Maya Murshak  
Technical Director



# Analytical Laboratory Report

## General Report Notes

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Analytical results relate only to the samples tested, in the condition received by the laboratory.

Methods may be modified for improved performance.

Results reported on a dry weight basis where applicable.

'Not detected' indicates that parameter was not found at a level equal to or greater than the reporting limit (RL).

When MDL results are provided, then 'Not detected' indicates that parameter was not found at a level equal to or greater than the MDL.

40 CFR Part 136 Table II Required Containers, Preservation Techniques and Holding Times for the Clean Water Act specify that samples for acrolein and acrylonitrile, and 2-chloroethylvinyl ether need to be preserved at a pH in the range of 4 to 5 or if not preserved, analyzed within 3 days of sampling.

QA/QC corresponding to this analytical report is a separate document with the same Merit ID reference and is available upon request.

Starred (\*) analytes are not NY NELAP accredited.

Samples are held by the lab for 30 days from the final report date unless a written request to hold longer is provided by the client.

Report shall not be reproduced except in full, without the written approval of Merit Laboratories, Inc.

Limits for drinking water samples, are listed as the MCL Limits (Maximum Contaminant Level Concentrations)

PFAS requirement: Section 9.3.8 of U.S. EPA Method 537.1 states "If the method analyte(s) found in the Field Sample is present in the FRB at a concentration greater than 1/3 the MRL, then all samples collected with that FRB are invalid and must be recollected and reanalyzed."

Samples submitted without an accompanying FRB may not be acceptable for compliance purposes.

Wisconsin PFAs analysis: MDL = LOD; RL = LOQ. LOD and LOQ are adjusted for dilution.

All accreditations/certifications held by this laboratory are listed on page 3. Not all accreditations/certifications are applicable to this report.

For a specific list of accredited analytes, please feel free to contact the laboratory or visit <https://www.meritlabs.com/certifications>.

## Report Narrative

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There is no additional narrative for this analytical report



# Analytical Laboratory Report

## Laboratory Accreditations (For Reference Only)

Authority	Accreditation ID
Michigan DEQ	#9956
DOD ELAP & ISO/IEC 17025:2017	#69699 PJLA Testing
WBENC	#2005110032
Ohio VAP	#CL0002
Indiana DOH	#C-MI-07
New York NELAC	#11814
North Carolina DENR	#680
North Carolina DOH	#26702
Pennsylvania DEP	#68-05884
Wisconsin DNR	FID# 399147320

## Qualifier Descriptions

Qualifier	Description
!	Result is outside of stated limit criteria
B	Compound also found in associated method blank
E	Concentration exceeds calibration range
F	Analysis run outside of holding time
G	Estimated result due to extraction run outside of holding time
H	Sample submitted and run outside of holding time
I	Matrix interference with internal standard
J	Estimated value less than reporting limit, but greater than MDL
L	Elevated reporting limit due to low sample amount
M	Result reported to MDL not RDL
O	Analysis performed by outside laboratory. See attached report.
R	Preliminary result
S	Surrogate recovery outside of control limits
T	No correction for total solids
X	Elevated reporting limit due to matrix interference
Y	Elevated reporting limit due to high target concentration
b	Value detected less than reporting limit, but greater than MDL
e	Reported value estimated due to interference
j	Analyte also found in associated method blank
o	Associated EIS outside of control limits
p	Benzo(b)Fluoranthene and Benzo(k)Fluoranthene integrated as one peak.
q	Qualifier ion ratio outside of control limits
x	Preserved from bulk sample

## Glossary of Abbreviations

Abbreviation	Description
RL/RDL	Reporting Limit
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
SW	EPA SW 846 (Soil and Wastewater) Methods
E	EPA Methods
SM	Standard Methods
LN	Linear
BR	Branched



# Analytical Laboratory Report

## Method Summary

Method	Version
N/A	Not Applicable
WI SPE	PFAS by LCMSMS Per Wisconsin DNR Document EA-19-0001

## Parameter Summary

Parameter	Synonym	Cas #
PFBA	Perfluorobutanoic Acid	375-22-4
PFPeA	Perfluoropentanoic Acid	2706-90-3
4:2 FTSA	4:2 Fluorotelomer Sulfonic Acid	757124-72-4
PFHxA	Perfluorohexanoic Acid	307-24-4
PFBS	Perfluorobutane sulfonic Acid	375-73-5
PFHpA	Perfluoroheptanoic Acid	375-85-9
PFPeS	Perfluoropentane Sulfonic Acid	2706-91-4
6:2 FTSA	6:2 Fluorotelomer Sulfonic Acid	27619-97-2
PFOA	Perfluorooctanoic Acid	335-67-1
PFHxS	Perfluorohexane Sulfonic Acid	355-46-4
PFHxS-LN	Perfluorohexane Sulfonic Acid - LN	355-46-4-LN
PFHxS-BR	Perfluorohexane Sulfonic Acid - BR	355-46-4-BR
PFNA	Perfluorononanoic Acid	375-95-1
8:2 FTSA	8:2 Fluorotelomer Sulfonic Acid	39108-34-4
PFHpS	Perfluoroheptane Sulfonic Acid	375-92-8
PFDA	Perfluorodecanoic Acid	335-76-2
N-MeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9
EtFOSAA	N-Ethyl Perfluorooctane Sulfonamidoacetic Acid	2991-50-6
PFOS	Perfluorooctane Sulfonic Acid	1763-23-1
PFOS-LN	Perfluorooctane Sulfonic Acid - LN	1763-23-1-LN
PFOS-BR	Perfluorooctane Sulfonic Acid - BR	1763-23-1-BR
PFUnDA	Perfluoroundecanoic Acid	2058-94-8
PFNS	Perfluorononane Sulfonic Acid	68259-12-1
PFDoDA	Perfluorododecanoic Acid	307-55-1
PFDS	Perfluorodecane Sulfonic Acid	335-77-3
PFTTrDA	Perfluorotridecanoic Acid	72629-94-8
FOSA	Perfluorooctane Sulfonamide	754-91-6
PFTeDA	Perfluorotetradecanoic Acid	376-06-7
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanone1-sulfonic acid	756426-58-1
ADONA	4,8-dioxa-3H-perfluorononanoic acid	919005-14-4
HFPO-DA	Hexafluoropropylene oxide dimer	13252-13-6
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
NMeFOSAM	N-Methylperfluorooctanesulfonamide	31506-32-8
NEtFOSAM	N-Ethylperfluorooctanesulfonamide	4151-50-2
NMeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
NEtFOSE	N-Ethylperfluorooctanesulfonamidoethanol	1691-99-2





# Analytical Laboratory Report

## Sample Summary (5 samples)

Sample ID	Sample Tag	Matrix	Collected Date/Time
S61354.01	FB-01	Water	04/24/24 09:25
S61354.02	RB-01	Water	04/24/24 09:35
S61354.03	RB-02	Water	04/24/24 09:45
S61354.04	FD-01	Water	04/24/24 00:01
S61354.05	IW-1	Groundwater	04/24/24 10:15



# Analytical Laboratory Report

Lab Sample ID: S61354.01  
Sample Tag: FB-01  
Collected Date/Time: 04/24/2024 09:25  
Matrix: Water  
COC Reference: 597209

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
1	250mL Plastic	Trizma	Yes	2.0	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	05/03/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	317.23/33.30	WI SPE	05/03/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 17:02, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	Not detected	1.8	0.25	ng/L	0.0141	375-22-4	
PFPeA*	0.33	1.8	0.20	ng/L	0.0141	2706-90-3	J
4:2 FTSA*	Not detected	1.8	0.32	ng/L	0.0141	757124-72-4	
PFHxA*	Not detected	1.8	0.23	ng/L	0.0141	307-24-4	
PFBS*	Not detected	1.8	0.17	ng/L	0.0141	375-73-5	
PFHpA*	Not detected	1.8	0.39	ng/L	0.0141	375-85-9	
PFPeS*	Not detected	1.8	0.21	ng/L	0.0141	2706-91-4	
6:2 FTSA*	Not detected	1.8	0.48	ng/L	0.0141	27619-97-2	
PFOA*	Not detected	1.8	0.37	ng/L	0.0141	335-67-1	
PFHxS*	Not detected	1.8	0.55	ng/L	0.0141	355-46-4	
PFHxS-LN*	Not detected	1.8	0.55	ng/L	0.0141	355-46-4-LN	
PFHxS-BR*	Not detected	1.8	0.55	ng/L	0.0141	355-46-4-BR	
PFNA*	Not detected	1.8	0.37	ng/L	0.0141	375-95-1	
8:2 FTSA*	Not detected	1.8	0.63	ng/L	0.0141	39108-34-4	
PFHpS*	Not detected	1.8	0.45	ng/L	0.0141	375-92-8	
PFDA*	Not detected	1.8	0.48	ng/L	0.0141	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.30	ng/L	0.0141	2355-31-9	
EtFOSAA*	Not detected	1.8	0.58	ng/L	0.0141	2991-50-6	
PFOS*	Not detected	1.8	0.32	ng/L	0.0141	1763-23-1	
PFOS-LN*	Not detected	1.8	0.32	ng/L	0.0141	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.32	ng/L	0.0141	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.41	ng/L	0.0141	2058-94-8	
PFNS*	Not detected	1.8	0.48	ng/L	0.0141	68259-12-1	
PFDODA*	Not detected	1.8	0.89	ng/L	0.0141	307-55-1	
PFDS*	Not detected	1.8	0.54	ng/L	0.0141	335-77-3	
PFTTrDA*	Not detected	1.8	0.61	ng/L	0.0141	72629-94-8	
FOSA*	Not detected	1.8	0.51	ng/L	0.0141	754-91-6	
PFTeDA*	Not detected	1.8	0.72	ng/L	0.0141	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.69	ng/L	0.0141	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.38	ng/L	0.0141	756426-58-1	
ADONA*	0.34	1.8	0.32	ng/L	0.0141	919005-14-4	J
HFPO-DA*	Not detected	1.8	0.28	ng/L	0.0141	13252-13-6	
PFDoS*	Not detected	1.8	0.54	ng/L	0.0141	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.54	ng/L	0.0141	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.54	ng/L	0.0141	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S61354.01 (continued)

Sample Tag: FB-01

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 17:02, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.63	ng/L	0.0141	24448-09-7	
NEtFOSE*	Not detected	1.8	0.69	ng/L	0.0141	1691-99-2	



# Analytical Laboratory Report

Lab Sample ID: S61354.02  
Sample Tag: RB-01  
Collected Date/Time: 04/24/2024 09:35  
Matrix: Water  
COC Reference: 597209

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
1	250mL Plastic	Trizma	Yes	2.0	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	05/03/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	310.28/33.30	WI SPE	05/03/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 17:19, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	Not detected	1.8	0.26	ng/L	0.0144	375-22-4	
PFPeA*	Not detected	1.8	0.20	ng/L	0.0144	2706-90-3	
4:2 FTSA*	Not detected	1.8	0.33	ng/L	0.0144	757124-72-4	
PFHxA*	Not detected	1.8	0.23	ng/L	0.0144	307-24-4	
PFBS*	Not detected	1.8	0.17	ng/L	0.0144	375-73-5	
PFHpA*	Not detected	1.8	0.40	ng/L	0.0144	375-85-9	
PFPeS*	Not detected	1.8	0.22	ng/L	0.0144	2706-91-4	
6:2 FTSA*	Not detected	1.8	0.49	ng/L	0.0144	27619-97-2	
PFOA*	Not detected	1.8	0.37	ng/L	0.0144	335-67-1	
PFHxS*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4	
PFHxS-LN*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4-LN	
PFHxS-BR*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4-BR	
PFNA*	Not detected	1.8	0.37	ng/L	0.0144	375-95-1	
8:2 FTSA*	Not detected	1.8	0.65	ng/L	0.0144	39108-34-4	
PFHpS*	Not detected	1.8	0.46	ng/L	0.0144	375-92-8	
PFDA*	Not detected	1.8	0.49	ng/L	0.0144	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.30	ng/L	0.0144	2355-31-9	
EtFOSAA*	Not detected	1.8	0.59	ng/L	0.0144	2991-50-6	
PFOS*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1	
PFOS-LN*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.42	ng/L	0.0144	2058-94-8	
PFNS*	Not detected	1.8	0.49	ng/L	0.0144	68259-12-1	
PFDODA*	Not detected	1.8	0.91	ng/L	0.0144	307-55-1	
PFDS*	Not detected	1.8	0.55	ng/L	0.0144	335-77-3	
PFTTrDA*	Not detected	1.8	0.62	ng/L	0.0144	72629-94-8	
FOSA*	Not detected	1.8	0.52	ng/L	0.0144	754-91-6	
PFTeDA*	Not detected	1.8	0.73	ng/L	0.0144	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.71	ng/L	0.0144	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.39	ng/L	0.0144	756426-58-1	
ADONA*	0.35	1.8	0.33	ng/L	0.0144	919005-14-4	J
HFPO-DA*	Not detected	1.8	0.29	ng/L	0.0144	13252-13-6	
PFDoS*	Not detected	1.8	0.55	ng/L	0.0144	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.55	ng/L	0.0144	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.55	ng/L	0.0144	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S61354.02 (continued)

Sample Tag: RB-01

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 17:19, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.65	ng/L	0.0144	24448-09-7	
NEtFOSE*	Not detected	1.8	0.71	ng/L	0.0144	1691-99-2	





# Analytical Laboratory Report

Lab Sample ID: S61354.03  
Sample Tag: RB-02  
Collected Date/Time: 04/24/2024 09:45  
Matrix: Water  
COC Reference: 597209

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
1	250mL Plastic	Trizma	Yes	2.0	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	05/03/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	328.88/33.32	WI SPE	05/03/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 17:35, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	Not detected	1.7	0.24	ng/L	0.0135	375-22-4	
PFPeA*	Not detected	1.7	0.19	ng/L	0.0135	2706-90-3	
4:2 FTSA*	Not detected	1.7	0.31	ng/L	0.0135	757124-72-4	
PFHxA*	0.24	1.7	0.22	ng/L	0.0135	307-24-4	J
PFBS*	0.30	1.7	0.16	ng/L	0.0135	375-73-5	J
PFHpA*	Not detected	1.7	0.38	ng/L	0.0135	375-85-9	
PFPeS*	Not detected	1.7	0.20	ng/L	0.0135	2706-91-4	
6:2 FTSA*	Not detected	1.7	0.46	ng/L	0.0135	27619-97-2	
PFOA*	Not detected	1.7	0.35	ng/L	0.0135	335-67-1	
PFHxS*	Not detected	1.7	0.53	ng/L	0.0135	355-46-4	
PFHxS-LN*	Not detected	1.7	0.53	ng/L	0.0135	355-46-4-LN	
PFHxS-BR*	Not detected	1.7	0.53	ng/L	0.0135	355-46-4-BR	
PFNA*	Not detected	1.7	0.35	ng/L	0.0135	375-95-1	
8:2 FTSA*	Not detected	1.7	0.61	ng/L	0.0135	39108-34-4	
PFHpS*	Not detected	1.7	0.43	ng/L	0.0135	375-92-8	
PFDA*	Not detected	1.7	0.46	ng/L	0.0135	335-76-2	
N-MeFOSAA*	0.39	1.7	0.28	ng/L	0.0135	2355-31-9	J
EtFOSAA*	Not detected	1.7	0.55	ng/L	0.0135	2991-50-6	
PFOS*	Not detected	1.7	0.31	ng/L	0.0135	1763-23-1	
PFOS-LN*	Not detected	1.7	0.31	ng/L	0.0135	1763-23-1-LN	
PFOS-BR*	Not detected	1.7	0.31	ng/L	0.0135	1763-23-1-BR	
PFUnDA*	Not detected	1.7	0.39	ng/L	0.0135	2058-94-8	
PFNS*	Not detected	1.7	0.46	ng/L	0.0135	68259-12-1	
PFDODA*	Not detected	1.7	0.85	ng/L	0.0135	307-55-1	
PFDS*	Not detected	1.7	0.51	ng/L	0.0135	335-77-3	
PFTTrDA*	Not detected	1.7	0.58	ng/L	0.0135	72629-94-8	
FOSA*	Not detected	1.7	0.49	ng/L	0.0135	754-91-6	
PFTeDA*	Not detected	1.7	0.69	ng/L	0.0135	376-06-7	
11Cl-PF3OUdS*	Not detected	1.7	0.66	ng/L	0.0135	763051-92-9	
9Cl-PF3ONS*	Not detected	1.7	0.36	ng/L	0.0135	756426-58-1	
ADONA*	Not detected	1.7	0.31	ng/L	0.0135	919005-14-4	
HFPO-DA*	Not detected	1.7	0.27	ng/L	0.0135	13252-13-6	
PFDoS*	Not detected	1.7	0.51	ng/L	0.0135	79780-39-5	
NMeFOSAM*	Not detected	1.7	0.51	ng/L	0.0135	31506-32-8	
NEtFOSAM*	Not detected	1.7	0.51	ng/L	0.0135	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S61354.03 (continued)

Sample Tag: RB-02

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 17:35, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.7	0.61	ng/L	0.0135	24448-09-7	
NEtFOSE*	Not detected	1.7	0.66	ng/L	0.0135	1691-99-2	



# Analytical Laboratory Report

Lab Sample ID: S61354.04  
Sample Tag: FD-01  
Collected Date/Time: 04/24/2024 00:01  
Matrix: Water  
COC Reference: 597209

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
3	250mL Plastic	Trizma	Yes	2.0	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	05/03/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	317.81/33.27	WI SPE	05/03/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 17:51, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	35	1.8	0.25	ng/L	0.0141	375-22-4	
PFPeA*	230	1.8	0.20	ng/L	0.0141	2706-90-3	
4:2 FTSA*	0.86	1.8	0.32	ng/L	0.0141	757124-72-4	IJ
PFHxA*	86	1.8	0.23	ng/L	0.0141	307-24-4	
PFBS*	4.1	1.8	0.17	ng/L	0.0141	375-73-5	
PFHpA*	15	1.8	0.39	ng/L	0.0141	375-85-9	
PFPeS*	2.0	1.8	0.21	ng/L	0.0141	2706-91-4	
6:2 FTSA*	32	1.8	0.48	ng/L	0.0141	27619-97-2	
PFOA*	3.5	1.8	0.37	ng/L	0.0141	335-67-1	
PFHxS*	5.2	1.8	0.55	ng/L	0.0141	355-46-4	
PFHxS-LN*	3.4	1.8	0.55	ng/L	0.0141	355-46-4-LN	
PFHxS-BR*	2.1	1.8	0.55	ng/L	0.0141	355-46-4-BR	
PFNA*	0.52	1.8	0.37	ng/L	0.0141	375-95-1	J
8:2 FTSA*	Not detected	1.8	0.63	ng/L	0.0141	39108-34-4	
PFHpS*	Not detected	1.8	0.45	ng/L	0.0141	375-92-8	
PFDA*	Not detected	1.8	0.48	ng/L	0.0141	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.30	ng/L	0.0141	2355-31-9	
EtFOSAA*	Not detected	1.8	0.58	ng/L	0.0141	2991-50-6	
PFOS*	0.55	1.8	0.32	ng/L	0.0141	1763-23-1	J
PFOS-LN*	Not detected	1.8	0.32	ng/L	0.0141	1763-23-1-LN	
PFOS-BR*	0.38	1.8	0.32	ng/L	0.0141	1763-23-1-BR	J
PFUnDA*	Not detected	1.8	0.41	ng/L	0.0141	2058-94-8	
PFNS*	Not detected	1.8	0.48	ng/L	0.0141	68259-12-1	
PFDODA*	Not detected	1.8	0.89	ng/L	0.0141	307-55-1	
PFDS*	Not detected	1.8	0.54	ng/L	0.0141	335-77-3	
PFTTrDA*	Not detected	1.8	0.61	ng/L	0.0141	72629-94-8	
FOSA*	Not detected	1.8	0.51	ng/L	0.0141	754-91-6	
PFTeDA*	Not detected	1.8	0.72	ng/L	0.0141	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.69	ng/L	0.0141	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.38	ng/L	0.0141	756426-58-1	
ADONA*	0.34	1.8	0.32	ng/L	0.0141	919005-14-4	Jq
HFPO-DA*	Not detected	1.8	0.28	ng/L	0.0141	13252-13-6	
PFDoS*	Not detected	1.8	0.54	ng/L	0.0141	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.54	ng/L	0.0141	31506-32-8	

I-Matrix interference with internal standard J-Estimated value less than reporting limit, but greater than MDL  
q-Qualifier ion ratio outside of control limits



# Analytical Laboratory Report

Lab Sample ID: S61354.04 (continued)  
Sample Tag: FD-01

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 17:51, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NEtFOSAM*	Not detected	1.8	0.54	ng/L	0.0141	4151-50-2	
NMeFOSE*	Not detected	1.8	0.63	ng/L	0.0141	24448-09-7	
NEtFOSE*	Not detected	1.8	0.69	ng/L	0.0141	1691-99-2	



# Analytical Laboratory Report

Lab Sample ID: S61354.05  
Sample Tag: IW-1  
Collected Date/Time: 04/24/2024 10:15  
Matrix: Groundwater  
COC Reference: 597209

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
3	250mL Plastic	Trizma	Yes	2.0	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	05/03/24 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	318.28/33.30	WI SPE	05/03/24 10:00	PTW	

Organics

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 18:24, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	35	1.8	0.25	ng/L	0.014	375-22-4	
PFPeA*	230	1.8	0.20	ng/L	0.014	2706-90-3	
4:2 FTSA*	0.96	1.8	0.32	ng/L	0.014	757124-72-4	J
PFHxA*	84	1.8	0.22	ng/L	0.014	307-24-4	
PFBS*	3.7	1.8	0.17	ng/L	0.014	375-73-5	
PFHpA*	15	1.8	0.39	ng/L	0.014	375-85-9	
PFPeS*	1.9	1.8	0.21	ng/L	0.014	2706-91-4	
6:2 FTSA*	31	1.8	0.48	ng/L	0.014	27619-97-2	
PFOA*	3.5	1.8	0.36	ng/L	0.014	335-67-1	
PFHxS*	4.8	1.8	0.55	ng/L	0.014	355-46-4	
PFHxS-LN*	3.0	1.8	0.55	ng/L	0.014	355-46-4-LN	
PFHxS-BR*	2.0	1.8	0.55	ng/L	0.014	355-46-4-BR	
PFNA*	0.51	1.8	0.36	ng/L	0.014	375-95-1	J
8:2 FTSA*	Not detected	1.8	0.63	ng/L	0.014	39108-34-4	
PFHpS*	Not detected	1.8	0.45	ng/L	0.014	375-92-8	
PFDA*	Not detected	1.8	0.48	ng/L	0.014	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.29	ng/L	0.014	2355-31-9	
EtFOSAA*	Not detected	1.8	0.57	ng/L	0.014	2991-50-6	
PFOS*	0.66	1.8	0.32	ng/L	0.014	1763-23-1	J
PFOS-LN*	0.35	1.8	0.32	ng/L	0.014	1763-23-1-LN	J
PFOS-BR*	0.38	1.8	0.32	ng/L	0.014	1763-23-1-BR	J
PFUnDA*	Not detected	1.8	0.41	ng/L	0.014	2058-94-8	
PFNS*	Not detected	1.8	0.48	ng/L	0.014	68259-12-1	
PFDODA*	Not detected	1.8	0.88	ng/L	0.014	307-55-1	
PFDS*	Not detected	1.8	0.53	ng/L	0.014	335-77-3	
PFTTrDA*	Not detected	1.8	0.60	ng/L	0.014	72629-94-8	
FOSA*	Not detected	1.8	0.50	ng/L	0.014	754-91-6	
PFTeDA*	Not detected	1.8	0.71	ng/L	0.014	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.69	ng/L	0.014	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.38	ng/L	0.014	756426-58-1	
ADONA*	Not detected	1.8	0.32	ng/L	0.014	919005-14-4	
HFPO-DA*	Not detected	1.8	0.28	ng/L	0.014	13252-13-6	
PFDoS*	Not detected	1.8	0.53	ng/L	0.014	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.53	ng/L	0.014	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.53	ng/L	0.014	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL





Analytical Laboratory Report

Lab Sample ID: S61354.05 (continued)  
Sample Tag: IW-1

WI 33 PFAs, Method: WI SPE, Run Date: 05/06/24 18:24, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.63	ng/L	0.014	24448-09-7	
NEtFOSE*	Not detected	1.8	0.69	ng/L	0.014	1691-99-2	



# Quality Control Report

Report ID: S61354.01(01)+QC02  
Generated on 05/17/2024

Report to  
Attention: David Beattie  
Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802  
  
Phone: 218-348-9051    FAX:

Report Produced by  
Merit Laboratories  
2680 East Lansing Drive  
East Lansing, MI 48823  
  
Phone: (517) 332-0167    FAX: (517) 332-6333

Report Summary  
Lab Sample ID(s): S61354.01-S61354.05  
Project: SRC 49161497.04 100 101  
Submitted Date/Time: 04/25/2024 09:40  
Sampled by: JSP  
P.O. #: 8401702587

QC Report Sections  
Cover Page (Page 17)  
Analysis Summary (Pages 18-22)  
Prep Batch Summary (Page 23)  
Surrogates per QC Sample (Page 24)  
Internal Standards per Lab Sample (Pages 25-29)  
Internal Standards per QC Sample (Pages 30-36)  
Batch QC Results (Pages 37-42)

QC Report Narrative  
DOD QC removed from Prep Batch WS240503W1 per client request

Report Flag Descriptions  
\*: QC result is outside of indicated control limits  
W: Surrogate result not applicable due to sample dilution

I certify that this data package is in compliance with the terms and conditions of the program, and project, and contractual requirements both technically and for completeness. Release of the data contained in this hardcopy data package and its computer-readable data submitted has been authorized by the Quality Assurance Manager and his/her designee, as verified by the following signature.

  
Barbara Ball  
Quality Assurance Manager

QC Report - Analysis Summary

Lab Sample ID: S61354.01  
Sample Tag: FB-01  
Collected Date/Time: 04/24/2024 09:25  
Matrix: Water  
COC Reference: 597209

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	05/06/24 17:02	CI240506WISPE	WS240503W1	Yes	BLK/LCS/MS/DUP

QC Report - Analysis Summary

Lab Sample ID: S61354.02  
Sample Tag: RB-01  
Collected Date/Time: 04/24/2024 09:35  
Matrix: Water  
COC Reference: 597209

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	05/06/24 17:19	CI240506WISPE	WS240503W1	Yes	BLK/LCS/MS/DUP

QC Report - Analysis Summary

Lab Sample ID: S61354.03  
Sample Tag: RB-02  
Collected Date/Time: 04/24/2024 09:45  
Matrix: Water  
COC Reference: 597209

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	05/06/24 17:35	CI240506WISPE	WS240503W1	Yes	BLK/LCS/MS/DUP

QC Report - Analysis Summary

Lab Sample ID: S61354.04  
Sample Tag: FD-01  
Collected Date/Time: 04/24/2024 00:01  
Matrix: Water  
COC Reference: 597209

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	05/06/24 17:51	CI240506WISPE	WS240503W1	Yes	BLK/LCS/MS/DUP



QC Report - Analysis Summary

Lab Sample ID: S61354.05  
Sample Tag: IW-1  
Collected Date/Time: 04/24/2024 10:15  
Matrix: Groundwater  
COC Reference: 597209

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	05/06/24 18:24	CI240506WISPE	WS240503W1	Yes	BLK/LCS/MS/DUP

## QC Report - Prep Batch Summary

### Organics - Volatiles, Prep Batch ID: WS240503W1

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

Sample ID	Analysis	Method	Run Date/Time	Batch ID
S61354.01	WI 33 PFAs	WI SPE	05/06/24 17:02	CI240506WISPE
S61354.02	WI 33 PFAs	WI SPE	05/06/24 17:19	CI240506WISPE
S61354.03	WI 33 PFAs	WI SPE	05/06/24 17:35	CI240506WISPE
S61354.04	WI 33 PFAs	WI SPE	05/06/24 17:51	CI240506WISPE
S61354.05	WI 33 PFAs	WI SPE	05/06/24 18:24	CI240506WISPE

## QC Report - Surrogates per QC Sample

### Organics - Volatiles, Prep Batch ID: WS240503W1

QC Types: BLK/LCS/MS/DUP

#### Blank (BLK)

Lab Sample ID: CI240506WISPE.BLK240503

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 15:41, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Blank (BLK)

Lab Sample ID: CI240508WISPE.BLK240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:17, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Laboratory Control Sample (LCS)

Lab Sample ID: CI240506WISPE.LCS240503

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 16:30, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Laboratory Control Sample (LCS)

Lab Sample ID: CI240508WISPE.LCS240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:33, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Laboratory Control Sample Duplicate (LCSD)

Lab Sample ID: CI240508WISPE.LCSD240508, Parent Sample ID: CI240508WISPE.LCS240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:50, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Matrix Spike (MS)

Lab Sample ID: CI240506WISPE.6135404M, Parent Sample ID: S61354.04

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 18:07, Prep Date: 05/03/2024, Matrix: WW, Dilution: 0.0138

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

#### Duplicate (DUP)

Lab Sample ID: CI240506WISPE.6135405D, Parent Sample ID: S61354.05

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 18:40, Prep Date: 05/03/2024, Matrix: WW, Dilution: 0.0139

Surrogate	Flags	%Rec	LCL	UCL
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No Surrogates

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S61354.01

Sample Tag: FB-01

Collected Date/Time: 04/24/2024 09:25

Matrix: Water

COC Reference: 597209

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 17:02, Matrix: WW, Dilution: 0.0141

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		87.0	25	150.0
M2-6:2FTSA		58.3	25	150.0
M2-8:2FTSA		77.2	25	150.0
M2PFTeDA		64.5	25	150.0
M3PFBS		94.7	25	150.0
M3PFHxS		89.0	25	150.0
M4PFHpA		102.5	25	150.0
M5PFHxA		100.0	25	150.0
M5PFPeA		99.3	25	150.0
M6PFDA		90.0	25	150.0
M7PFUnDA		85.4	25	150.0
M8FOSA		95.1	10	150.0
M8PFOA		96.9	25	150.0
M8PFOS		82.8	25	150.0
M9-PFNA		92.1	25	150.0
MPFBA		102.8	25	150.0
MPFDoDA		78.7	25	150.0
d3N-MeFOSAA		85.0	25	150.0
d5EtFOSAA		78.3	25	150.0
MHFPODA		99.8	25	150.0
d-N-EtFOSA-M		51.5	10	150.0
d-N-MeFOSA-M		62.1	10	150.0
d7-N-MeFOSE-M		67.0	10	150.0
d9-N-EtFOSE-M		58.0	10	150.0

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S61354.02

Sample Tag: RB-01

Collected Date/Time: 04/24/2024 09:35

Matrix: Water

COC Reference: 597209

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 17:19, Matrix: WW, Dilution: 0.0144

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		91.9	25	150.0
M2-6:2FTSA		55.2	25	150.0
M2-8:2FTSA		86.0	25	150.0
M2PFTeDA		79.9	25	150.0
M3PFBS		100.8	25	150.0
M3PFHxS		95.6	25	150.0
M4PFHpA		106.9	25	150.0
M5PFHxA		104.3	25	150.0
M5PFPeA		104.7	25	150.0
M6PFDA		96.9	25	150.0
M7PFUnDA		96.4	25	150.0
M8FOSA		97.3	10	150.0
M8PFOA		101.3	25	150.0
M8PFOS		92.7	25	150.0
M9-PFNA		97.6	25	150.0
MPFBA		108.4	25	150.0
MPFDoDA		103.4	25	150.0
d3N-MeFOSAA		97.4	25	150.0
d5EtFOSAA		92.3	25	150.0
MHFPODA		105.1	25	150.0
d-N-EtFOSA-M		66.7	10	150.0
d-N-MeFOSA-M		76.7	10	150.0
d7-N-MeFOSE-M		80.3	10	150.0
d9-N-EtFOSE-M		72.8	10	150.0

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S61354.03

Sample Tag: RB-02

Collected Date/Time: 04/24/2024 09:45

Matrix: Water

COC Reference: 597209

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 17:35, Matrix: WW, Dilution: 0.0135

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		92.3	25	150.0
M2-6:2FTSA		53.1	25	150.0
M2-8:2FTSA		88.6	25	150.0
M2PFTeDA		65.3	25	150.0
M3PFBS		103.5	25	150.0
M3PFHxS		102.1	25	150.0
M4PFHpA		108.8	25	150.0
M5PFHxA		106.6	25	150.0
M5PFPeA		105.8	25	150.0
M6PFDA		97.7	25	150.0
M7PFUnDA		84.4	25	150.0
M8FOSA		102.2	10	150.0
M8PFOA		104.0	25	150.0
M8PFOS		97.0	25	150.0
M9-PFNA		99.5	25	150.0
MPFBA		76.9	25	150.0
MPFDoDA		76.8	25	150.0
d3N-MeFOSAA		84.7	25	150.0
d5EtFOSAA		80.9	25	150.0
MHFPODA		109.5	25	150.0
d-N-EtFOSA-M		57.9	10	150.0
d-N-MeFOSA-M		66.9	10	150.0
d7-N-MeFOSE-M		74.4	10	150.0
d9-N-EtFOSE-M		64.2	10	150.0



# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S61354.04

Sample Tag: FD-01

Collected Date/Time: 04/24/2024 00:01

Matrix: Water

COC Reference: 597209

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 17:51, Matrix: WW, Dilution: 0.0141

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA	*	170.8	25	150.0
M2-6:2FTSA		66.3	25	150.0
M2-8:2FTSA		103.9	25	150.0
M2PFTeDA		65.3	25	150.0
M3PFBS		100.4	25	150.0
M3PFHxS		96.1	25	150.0
M4PFHpA		107.6	25	150.0
M5PFHxA		104.6	25	150.0
M5PFPeA		82.9	25	150.0
M6PFDA		97.2	25	150.0
M7PFUnDA		90.5	25	150.0
M8FOSA		95.5	10	150.0
M8PFOA		103.5	25	150.0
M8PFOS		94.2	25	150.0
M9-PFNA		98.4	25	150.0
MPFBA		73.3	25	150.0
MPFDoDA		75.5	25	150.0
d3N-MeFOSAA		94.0	25	150.0
d5EtFOSAA		87.5	25	150.0
MHFPODA		102.5	25	150.0
d-N-EtFOSA-M		55.1	10	150.0
d-N-MeFOSA-M		61.8	10	150.0
d7-N-MeFOSE-M		69.2	10	150.0
d9-N-EtFOSE-M		58.6	10	150.0

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S61354.05

Sample Tag: IW-1

Collected Date/Time: 04/24/2024 10:15

Matrix: Groundwater

COC Reference: 597209

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 18:24, Matrix: WW, Dilution: 0.014

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		146.8	25	150.0
M2-6:2FTSA		61.5	25	150.0
M2-8:2FTSA		92.8	25	150.0
M2PFTeDA		75.8	25	150.0
M3PFBS		103.2	25	150.0
M3PFHxS		98.3	25	150.0
M4PFHpA		107.4	25	150.0
M5PFHxA		105.7	25	150.0
M5PFPeA		81.6	25	150.0
M6PFDA		96.6	25	150.0
M7PFUnDA		89.9	25	150.0
M8FOSA		100.7	10	150.0
M8PFOA		100.9	25	150.0
M8PFOS		87.1	25	150.0
M9-PFNA		98.2	25	150.0
MPFBA		88.1	25	150.0
MPFDoDA		86.2	25	150.0
d3N-MeFOSAA		89.2	25	150.0
d5EtFOSAA		79.4	25	150.0
MHFPODA		104.8	25	150.0
d-N-EtFOSA-M		61.4	10	150.0
d-N-MeFOSA-M		70.0	10	150.0
d7-N-MeFOSE-M		73.3	10	150.0
d9-N-EtFOSE-M		64.5	10	150.0

# QC Report - Internal Standards per QC Sample

**Organics - Volatiles, Prep Batch ID: WS240503W1**

QC Types: BLK/LCS/MS/DUP

## Blank (BLK)

Lab Sample ID: CI240506WISPE.BLK240503

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 15:41, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		87.2	25	150.0
M2-6:2FTSA		49.4	25	150.0
M2-8:2FTSA		85.2	25	150.0
M2PFTeDA		79.0	25	150.0
M3PFBS		96.3	25	150.0
M3PFHxS		97.2	25	150.0
M4PFHpA		100.4	25	150.0
M5PFHxA		99.0	25	150.0
M5PFPeA		99.2	25	150.0
M6PFDA		92.7	25	150.0
M7PFUnDA		89.9	25	150.0
M8FOSA		99.0	10	150.0
M8PFOA		97.0	25	150.0
M8PFOS		87.7	25	150.0
M9-PFNA		94.3	25	150.0
MPFBA		101.4	25	150.0
MPFDoDA		86.0	25	150.0
d3N-MeFOSAA		90.0	25	150.0
d5EtFOSAA		84.8	25	150.0
MHFPODA		101.6	25	150.0
d-N-EtFOSA-M		72.3	10	150.0
d-N-MeFOSA-M		76.0	10	150.0
d7-N-MeFOSE-M		84.5	10	150.0
d9-N-EtFOSE-M		81.1	10	150.0

# QC Report - Internal Standards per QC Sample

## Blank (BLK)

Lab Sample ID: CI240508WISPE.BLK240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:17, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		99.6	25	150.0
M2-6:2FTSA		80.5	25	150.0
M2-8:2FTSA		97.3	25	150.0
M2PFTeDA		83.2	25	150.0
M3PFBS		96.8	25	150.0
M3PFHxS		96.8	25	150.0
M4PFHpA		100.3	25	150.0
M5PFHxA		99.5	25	150.0
M5PFPeA		98.1	25	150.0
M6PFDA		94.6	25	150.0
M7PFUnDA		94.2	25	150.0
M8FOSA		93.8	10	150.0
M8PFOA		102.9	25	150.0
M8PFOS		95.0	25	150.0
M9-PFNA		98.8	25	150.0
MPFBA		101.0	25	150.0
MPFDoDA		90.3	25	150.0
d3N-MeFOSAA		89.3	25	150.0
d5EtFOSAA		89.2	25	150.0
MHFPODA		98.4	25	150.0
d-N-EtFOSA-M		71.8	10	150.0
d-N-MeFOSA-M		74.6	10	150.0
d7-N-MeFOSE-M		82.0	10	150.0
d9-N-EtFOSE-M		81.3	10	150.0

# QC Report - Internal Standards per QC Sample

## Laboratory Control Sample (LCS)

Lab Sample ID: CI240506WISPE.LCS240503

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 16:30, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		87.0	25	150.0
M2-6:2FTSA		58.1	25	150.0
M2-8:2FTSA		84.6	25	150.0
M2PFTeDA		81.5	25	150.0
M3PFBS		102.5	25	150.0
M3PFHxS		98.3	25	150.0
M4PFHpA		108.1	25	150.0
M5PFHxA		106.1	25	150.0
M5PFPeA		104.4	25	150.0
M6PFDA		96.1	25	150.0
M7PFUnDA		92.1	25	150.0
M8FOSA		98.0	10	150.0
M8PFOA		102.7	25	150.0
M8PFOS		99.2	25	150.0
M9-PFNA		95.8	25	150.0
MPFBA		107.7	25	150.0
MPFDoDA		89.6	25	150.0
d3N-MeFOSAA		94.9	25	150.0
d5EtFOSAA		88.4	25	150.0
MHFPODA		106.8	25	150.0
d-N-EtFOSA-M		68.8	10	150.0
d-N-MeFOSA-M		70.8	10	150.0
d7-N-MeFOSE-M		79.0	10	150.0
d9-N-EtFOSE-M		72.0	10	150.0

# QC Report - Internal Standards per QC Sample

## Laboratory Control Sample (LCS)

Lab Sample ID: CI240508WISPE.LCS240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:33, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		111.6	25	150.0
M2-6:2FTSA		100.2	25	150.0
M2-8:2FTSA		97.7	25	150.0
M2PFTeDA		83.7	25	150.0
M3PFBS		101.5	25	150.0
M3PFHxS		104.3	25	150.0
M4PFHpA		105.4	25	150.0
M5PFHxA		102.9	25	150.0
M5PFPeA		102.3	25	150.0
M6PFDA		99.4	25	150.0
M7PFUnDA		99.9	25	150.0
M8FOSA		92.9	10	150.0
M8PFOA		107.4	25	150.0
M8PFOS		96.9	25	150.0
M9-PFNA		105.2	25	150.0
MPFBA		106.1	25	150.0
MPFDoDA		94.5	25	150.0
d3N-MeFOSAA		94.4	25	150.0
d5EtFOSAA		91.5	25	150.0
MHFPODA		102.7	25	150.0
d-N-EtFOSA-M		65.1	10	150.0
d-N-MeFOSA-M		70.8	10	150.0
d7-N-MeFOSE-M		81.1	10	150.0
d9-N-EtFOSE-M		75.6	10	150.0



# QC Report - Internal Standards per QC Sample

## Laboratory Control Sample Duplicate (LCSD)

Lab Sample ID: CI240508WISPE.LCSD240508, Parent Sample ID: CI240508WISPE.LCS240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:50, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		91.2	25	150.0
M2-6:2FTSA		74.4	25	150.0
M2-8:2FTSA		83.6	25	150.0
M2PFTeDA		62.4	25	150.0
M3PFBS		85.7	25	150.0
M3PFHxS		87.0	25	150.0
M4PFHpA		96.3	25	150.0
M5PFHxA		97.3	25	150.0
M5PFPeA		95.5	25	150.0
M6PFDA		83.7	25	150.0
M7PFUnDA		82.0	25	150.0
M8FOSA		82.3	10	150.0
M8PFOA		95.3	25	150.0
M8PFOS		72.3	25	150.0
M9-PFNA		91.4	25	150.0
MPFBA		99.7	25	150.0
MPFDoDA		78.1	25	150.0
d3N-MeFOSAA		78.3	25	150.0
d5EtFOSAA		71.6	25	150.0
MHFPODA		98.6	25	150.0
d-N-EtFOSA-M		50.9	10	150.0
d-N-MeFOSA-M		61.7	10	150.0
d7-N-MeFOSE-M		81.2	10	150.0
d9-N-EtFOSE-M		70.9	10	150.0

# QC Report - Internal Standards per QC Sample

## Matrix Spike (MS)

Lab Sample ID: CI240506WISPE.6135404M, Parent Sample ID: S61354.04

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 18:07, Prep Date: 05/03/2024, Matrix: WW, Dilution: 0.0138

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA	*	170.6	25	150.0
M2-6:2FTSA		74.2	25	150.0
M2-8:2FTSA		102.5	25	150.0
M2PFTeDA		92.1	25	150.0
M3PFBS		103.6	25	150.0
M3PFHxS		102.3	25	150.0
M4PFHpA		108.6	25	150.0
M5PFHxA		107.6	25	150.0
M5PFPeA		81.8	25	150.0
M6PFDA		103.9	25	150.0
M7PFUnDA		95.4	25	150.0
M8FOSA		103.0	10	150.0
M8PFOA		106.2	25	150.0
M8PFOS		101.1	25	150.0
M9-PFNA		105.0	25	150.0
MPFBA		87.5	25	150.0
MPFDoDA		94.8	25	150.0
d3N-MeFOSAA		103.5	25	150.0
d5EtFOSAA		94.1	25	150.0
MHFPODA		103.1	25	150.0
d-N-EtFOSA-M		77.6	10	150.0
d-N-MeFOSA-M		78.7	10	150.0
d7-N-MeFOSE-M		86.0	10	150.0
d9-N-EtFOSE-M		75.4	10	150.0

# QC Report - Internal Standards per QC Sample

## Duplicate (DUP)

Lab Sample ID: CI240506WISPE.6135405D, Parent Sample ID: S61354.05

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 18:40, Prep Date: 05/03/2024, Matrix: WW, Dilution: 0.0139

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA	*	165.7	25	150.0
M2-6:2FTSA		81.1	25	150.0
M2-8:2FTSA		86.8	25	150.0
M2PFTeDA		82.2	25	150.0
M3PFBS		103.4	25	150.0
M3PFHxS		102.4	25	150.0
M4PFHpA		110.5	25	150.0
M5PFHxA		108.1	25	150.0
M5PFPeA		81.7	25	150.0
M6PFDA		97.4	25	150.0
M7PFUnDA		94.7	25	150.0
M8FOSA		98.5	10	150.0
M8PFOA		107.0	25	150.0
M8PFOS		97.2	25	150.0
M9-PFNA		101.3	25	150.0
MPFBA		88.0	25	150.0
MPFDoDA		90.2	25	150.0
d3N-MeFOSAA		99.8	25	150.0
d5EtFOSAA		90.1	25	150.0
MHFPODA		102.5	25	150.0
d-N-EtFOSA-M		71.5	10	150.0
d-N-MeFOSA-M		70.9	10	150.0
d7-N-MeFOSE-M		74.6	10	150.0
d9-N-EtFOSE-M		69.8	10	150.0

# QC Report - Batch QC Results

**Organics - Volatiles, Prep Batch ID: WS240503W1**

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

## Blank (BLK)

Lab Sample ID: CI240506WISPE.BLK240503

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 15:41, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Conc	RDL	MDL	Units
PFBA		ND	2.00	0.29	ng/l
PFPeA		ND	2.00	0.22	ng/l
PFBS		ND	2.00	0.19	ng/l
4:2 FTSA		ND	2.00	0.37	ng/l
PFHxA		ND	2.00	0.26	ng/l
PFPeS		ND	2.00	0.24	ng/l
HFPO-DA		ND	2.00	0.3	ng/l
PFHxS-BR		ND	2.00	0.62	ng/l
PFHxS		ND	2.00	0.62	ng/l
PFHpA		ND	2.00	0.45	ng/l
PFHxS-LN		ND	2.00	0.62	ng/l
ADONA		ND	2.00	0.37	ng/l
6:2 FTSA		ND	2.00	0.54	ng/l
PFOA		ND	2.00	0.42	ng/l
PFHpS		ND	2.00	0.51	ng/l
PFOS		ND	2.00	0.37	ng/l
PFOS-BR		ND	2.00	0.37	ng/l
PFOS-LN		ND	2.00	0.37	ng/l
PFNA		ND	2.00	0.42	ng/l
9CL-PF3ONS		ND	2.00	0.43	ng/l
PFNS		ND	2.00	0.54	ng/l
8:2 FTSA		ND	2.00	0.72	ng/l
PFDA		ND	2.00	0.54	ng/l
N-MeFOSAA		ND	2.00	0.34	ng/l
EtFOSAA		ND	2.00	0.66	ng/l
PFDS		ND	2.00	0.61	ng/l
PFUnDA		ND	2.00	0.46	ng/l
FOSA		ND	2.00	0.58	ng/l
11CL-PF3OUdS		ND	2.00	0.78	ng/l
PFDODA		ND	2.00	1.0	ng/l
PFDOS		ND	2.00	0.61	ng/l
PFTTrDA		ND	2.00	0.69	ng/l
NMeFOSE		ND	2.00	0.72	ng/l
NMeFOSAM		ND	2.00	0.61	ng/l
PFTeDA		ND	2.00	0.82	ng/l
NEtFOSAM		ND	2.00	0.61	ng/l
NEtFOSE		ND	2.00	0.78	ng/l

## Blank (BLK)

Lab Sample ID: CI240508WISPE.BLK240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:17, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Conc	RDL	MDL	Units
PFBA		ND	2.00	0.29	ng/l
PFPeA		ND	2.00	0.22	ng/l
PFBS		ND	2.00	0.19	ng/l
4:2 FTSA		ND	2.00	0.37	ng/l

# QC Report - Batch QC Results

## Organics - Volatiles, Prep Batch ID: WS240503W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

## Blank (BLK) (continued)

Lab Sample ID: CI240508WISPE.BLK240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:17, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Conc	RDL	MDL	Units
PFHxA		ND	2.00	0.26	ng/l
PFPeS		ND	2.00	0.24	ng/l
HFPO-DA		ND	2.00	0.3	ng/l
PFHxS-BR		ND	2.00	0.62	ng/l
PfHpA		ND	2.00	0.45	ng/l
PFHxS		ND	2.00	0.62	ng/l
PFHxS-LN		ND	2.00	0.62	ng/l
ADONA		ND	2.00	0.37	ng/l
6:2 FTSA		ND	2.00	0.54	ng/l
PFOA		ND	2.00	0.42	ng/l
PfHpS		ND	2.00	0.51	ng/l
PFOS-BR		ND	2.00	0.37	ng/l
PFOS		ND	2.00	0.37	ng/l
PFOS-LN		ND	2.00	0.37	ng/l
PFNA		ND	2.00	0.42	ng/l
9CL-PF3ONS		ND	2.00	0.43	ng/l
PFNS		ND	2.00	0.54	ng/l
8:2 FTSA		ND	2.00	0.72	ng/l
PFDA		ND	2.00	0.54	ng/l
N-MeFOSAA		ND	2.00	0.34	ng/l
PFDS		ND	2.00	0.61	ng/l
EtFOSAA		ND	2.00	0.66	ng/l
PFUnDA		ND	2.00	0.46	ng/l
FOSA		ND	2.00	0.58	ng/l
11CL-PF3OUdS		ND	2.00	0.78	ng/l
PFDODA		ND	2.00	1.0	ng/l
PFDOS		ND	2.00	0.61	ng/l
PFTTrDA		ND	2.00	0.69	ng/l
NMeFOSE		ND	2.00	0.72	ng/l
NMeFOSAM		ND	2.00	0.61	ng/l
PFTeDA		ND	2.00	0.82	ng/l
NEtFOSAM		ND	2.00	0.61	ng/l
NEtFOSE		ND	2.00	0.78	ng/l

## Laboratory Control Sample (LCS)

Lab Sample ID: CI240506WISPE.LCS240503

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 16:30, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCS Conc	% Rec	LCL	UCL
PFBA		0.00	4.00	4.52	113.0	50	150
PFPeA		0.00	4.00	4.59	114.8	50	150
PFBS		0.00	4.00	4.42	110.5	50	150
4:2 FTSA		0.00	4.00	3.75	93.8	50	150
PFHxA		0.00	4.00	4.62	115.5	50	150
PFPeS		0.00	4.00	4.42	110.5	50	150
HFPO-DA		0.00	4.00	4.06	101.5	50	150
PFHxS		0.00	4.00	4.79	119.8	50	150

# QC Report - Batch QC Results

## Organics - Volatiles, Prep Batch ID: WS240503W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

## Laboratory Control Sample (LCS) (continued)

Lab Sample ID: CI240506WISPE.LCS240503

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 16:30, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCS Conc	% Rec	LCL	UCL
PFHpA		0.00	4.00	4.23	105.8	50	150
ADONA		0.00	4.00	5.02	125.5	50	150
6:2 FTSA		0.00	4.00	3.54	88.5	50	150
PFOA		0.00	4.00	4.53	113.3	50	150
PFHpS		0.00	4.00	4.31	107.7	50	150
PFOS		0.00	4.00	4.66	116.5	50	150
PFNA		0.00	4.00	4.63	115.8	50	150
9CL-PF3ONS		0.00	4.00	4.37	109.3	50	150
PFNS		0.00	4.00	4.41	110.3	50	150
8:2 FTSA		0.00	4.00	4.10	102.5	50	150
PFDA		0.00	4.00	4.67	116.8	50	150
N-MeFOSAA		0.00	4.00	4.47	111.8	50	150
EtFOSAA		0.00	4.00	4.43	110.8	50	150
PFDS		0.00	4.00	4.51	112.8	50	150
PFUnDA		0.00	4.00	4.61	115.3	50	150
FOSA		0.00	4.00	4.70	117.5	50	150
11CL-PF3OUdS		0.00	4.00	4.13	103.3	50	150
PFDoDA		0.00	4.00	4.68	117.0	50	150
PFDOS		0.00	4.00	3.55	88.8	50	150
PFTTrDA		0.00	4.00	4.33	108.3	50	150
NMeFOSE		0.00	4.00	5.58	139.5	50	150
NMeFOSAM		0.00	4.00	4.84	121.0	50	150
PFTeDA		0.00	4.00	4.71	117.8	50	150
NEtFOSAM		0.00	4.00	4.44	111.0	50	150
NEtFOSE		0.00	4.00	4.32	108.0	50	150

## Laboratory Control Sample (LCS)

Lab Sample ID: CI240508WISPE.LCS240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:33, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCS Conc	% Rec	LCL	UCL
PFBA		0.00	4.00	4.42	110.5	50	150
PFPeA		0.00	4.00	4.36	109.0	50	150
PFBS		0.00	4.00	4.37	109.3	50	150
4:2 FTSA		0.00	4.00	3.80	95.0	50	150
PFHxA		0.00	4.00	4.39	109.7	50	150
PFPeS		0.00	4.00	4.29	107.3	50	150
HFPO-DA		0.00	4.00	4.33	108.3	50	150
PFHpA		0.00	4.00	4.26	106.5	50	150
PFHxS		0.00	4.00	4.24	106.0	50	150
ADONA		0.00	4.00	4.15	103.8	50	150
6:2 FTSA		0.00	4.00	4.01	100.3	50	150
PFOA		0.00	4.00	4.20	105.0	50	150
PFHpS		0.00	4.00	4.52	113.0	50	150
PFOS		0.00	4.00	4.43	110.8	50	150
PFNA		0.00	4.00	4.22	105.5	50	150
9CL-PF3ONS		0.00	4.00	4.48	112.0	50	150



# QC Report - Batch QC Results

Organics - Volatiles, Prep Batch ID: WS240503W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

## Laboratory Control Sample (LCS) (continued)

Lab Sample ID: CI240508WISPE.LCS240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:33, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCS Conc	% Rec	LCL	UCL
PFNS		0.00	4.00	4.45	111.3	50	150
8:2 FTSA		0.00	4.00	4.34	108.5	50	150
PFDA		0.00	4.00	4.50	112.5	50	150
N-MeFOSAA		0.00	4.00	4.52	113.0	50	150
PFDS		0.00	4.00	4.21	105.3	50	150
EtFOSAA		0.00	4.00	4.04	101.0	50	150
PFUnDA		0.00	4.00	4.35	108.7	50	150
FOSA		0.00	4.00	4.36	109.0	50	150
11CL-PF3OUdS		0.00	4.00	4.08	102.0	50	150
PFDoDA		0.00	4.00	4.23	105.8	50	150
PFDOS		0.00	4.00	3.50	87.5	50	150
PFTTrDA		0.00	4.00	4.27	106.7	50	150
NMeFOSE		0.00	4.00	4.34	108.5	50	150
NMeFOSAM		0.00	4.00	4.50	112.5	50	150
PFTeDA		0.00	4.00	4.62	115.5	50	150
NEtFOSAM		0.00	4.00	4.77	119.2	50	150
NEtFOSE		0.00	4.00	3.74	93.5	50	150

## Laboratory Control Sample Duplicate (LCSD)

Lab Sample ID: CI240508WISPE.LCSD240508, Parent Sample ID: CI240508WISPE.LCS240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:50, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCSD Conc	% Rec	LCL	UCL	LCS Conc	RPD	RPD CL
PFBA	0		4.00	4.44	111.0	50	150	4.42	0.5	30.0
PFPeA	0		4.00	4.33	108.3	50	150	4.36	0.7	30.0
PFBS	0		4.00	4.27	106.7	50	150	4.37	2.3	30.0
4:2 FTSA	0		4.00	4.32	108.0	50	150	3.80	12.8	30.0
PFHxA	0		4.00	4.23	105.8	50	150	4.39	3.7	30.0
PFPeS	0		4.00	4.00	100.0	50	150	4.29	7.0	30.0
HFPO-DA	0		4.00	3.98	99.5	50	150	4.33	8.4	30.0
PFHpA	0		4.00	4.27	106.7	50	150	4.26	0.2	30.0
PFHxS	0		4.00	4.36	109.0	50	150	4.24	2.8	30.0
ADONA	0		4.00	4.43	110.8	50	150	4.15	6.5	30.0
6:2 FTSA	0		4.00	4.02	100.5	50	150	4.01	0.2	30.0
PFOA	0		4.00	4.20	105.0	50	150	4.20	0.0	30.0
PFHpS	0		4.00	3.99	99.8	50	150	4.52	12.5	30.0
PFOS	0		4.00	4.64	116.0	50	150	4.43	4.6	30.0
PFNA	0		4.00	4.52	113.0	50	150	4.22	6.9	30.0
9CL-PF3ONS	0		4.00	4.32	108.0	50	150	4.48	3.6	30.0
PFNS	0		4.00	4.01	100.3	50	150	4.45	10.4	30.0
8:2 FTSA	0		4.00	3.79	94.8	50	150	4.34	13.5	30.0
PFDA	0		4.00	4.36	109.0	50	150	4.50	3.2	30.0
N-MeFOSAA	0		4.00	4.21	105.3	50	150	4.52	7.1	30.0
PFDS	0		4.00	3.57	89.3	50	150	4.21	16.5	30.0
EtFOSAA	0		4.00	3.57	89.3	50	150	4.04	12.4	30.0
PFUnDA	0		4.00	4.55	113.8	50	150	4.35	4.5	30.0
FOSA	0		4.00	4.37	109.3	50	150	4.36	0.2	30.0

# QC Report - Batch QC Results

Organics - Volatiles, Prep Batch ID: WS240503W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

## Laboratory Control Sample Duplicate (LCSD) (continued)

Lab Sample ID: CI240508WISPE.LCSD240508, Parent Sample ID: CI240508WISPE.LCS240508

Run in Batch: CI240508WISPE, Run Date: 05/08/2024 15:50, Prep Date: 05/03/2024, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCSD Conc	% Rec	LCL	UCL	LCS Conc	RPD	RPD CL
11CL-PF3OUdS		0	4.00	3.92	98.0	50	150	4.08	4.0	30.0
PFDODA		0	4.00	4.22	105.5	50	150	4.23	0.2	30.0
PFDOS		0	4.00	2.68	67.0	50	150	3.50	26.5	30.0
PFTTrDA		0	4.00	4.19	104.8	50	150	4.27	1.9	30.0
NMeFOSE		0	4.00	4.04	101.0	50	150	4.34	7.2	30.0
NMeFOSAM		0	4.00	4.34	108.5	50	150	4.50	3.6	30.0
PFTeDA		0	4.00	4.29	107.3	50	150	4.62	7.4	30.0
NEtFOSAM		0	4.00	4.42	110.5	50	150	4.77	7.6	30.0
NEtFOSE		0	4.00	4.07	101.8	50	150	3.74	8.5	30.0

## Matrix Spike (MS)

Lab Sample ID: CI240506WISPE.6135404M, Parent Sample ID: S61354.04

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 18:07, Prep Date: 05/03/2024, Matrix: WW, Dilution: 0.0138

Analyte	Flags	Orig Conc	Spike	MS Conc	% Rec	LCL	UCL
PFBA		35	3.45	39	115.9	50	150
PFPeA	*	230	3.45	230	0.0	50	150
4:2 FTSA		0.86	3.45	4.0	91.0	50	150
PFHxA		86	3.45	88	58.0	50	150
PFBS		4.1	3.45	7.6	101.4	50	150
PFHpA		15	3.45	19	115.9	50	150
PFPeS		2.0	3.45	5.7	107.2	50	150
6:2 FTSA		32	3.45	34	58.0	50	150
PFOA		3.5	3.45	7.4	113.0	50	150
PFHxS		5.2	3.45	8.7	101.4	50	150
PFNA		0.52	3.45	4.1	103.8	50	150
8:2 FTSA		0	3.45	3.5	101.4	50	150
PFHpS		0	3.45	3.8	110.1	50	150
PFDA		0	3.45	3.9	113.0	50	150
N-MeFOSAA		0	3.45	3.8	110.1	50	150
EtFOSAA		0	3.45	4.3	124.6	50	150
PFOS		0.55	3.45	4.3	108.7	50	150
PFUnDA		0	3.45	4.0	115.9	50	150
PFNS		0	3.45	4.0	115.9	50	150
PFDODA		0	3.45	4.2	121.7	50	150
PFDS		0	3.45	3.7	107.2	50	150
PFTTrDA		0	3.45	3.9	113.0	50	150
FOSA		0	3.45	4.0	115.9	50	150
PFTeDA		0	3.45	4.2	121.7	50	150
11CL-PF3OUdS		0	3.45	3.5	101.4	50	150
9CL-PF3ONS		0	3.45	3.6	104.3	50	150
ADONA		0.34	3.45	4.2	111.9	50	150
HFPO-DA		0	3.45	3.6	104.3	50	150
PFDOS		0	3.45	3.2	92.8	50	150
NMeFOSAM		0	3.45	4.4	127.5	50	150
NEtFOSAM		0	3.45	3.8	110.1	50	150
NMeFOSE		0	3.45	4.1	118.8	50	150

# QC Report - Batch QC Results

## Organics - Volatiles, Prep Batch ID: WS240503W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS/MS/DUP

### Matrix Spike (MS) (continued)

Lab Sample ID: CI240506WISPE.6135404M, Parent Sample ID: S61354.04

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 18:07, Prep Date: 05/03/2024, Matrix: WW, Dilution: 0.0138

Analyte	Flags	Orig Conc	Spike	MS Conc	% Rec	LCL	UCL
NETFOSE		0	3.45	4.3	124.6	50	150

### Duplicate (DUP)

Lab Sample ID: CI240506WISPE.6135405D, Parent Sample ID: S61354.05

Run in Batch: CI240506WISPE, Run Date: 05/06/2024 18:40, Prep Date: 05/03/2024, Matrix: WW, Dilution: 0.0139

Analyte	Flags	Orig Conc	Dup Conc	RPD	RPD CL
PFBA		35	34	2.9	30.0
PFPeA		230	230	0.0	30.0
4:2 FTSA	J	0.96	0.88	8.7	30.0
PFHxA		84	83	1.2	30.0
PFBS		3.7	3.8	2.7	30.0
PFHpA		15	15	0.0	30.0
PFPeS		1.9	2.0	5.1	30.0
6:2 FTSA		31	30	3.3	30.0
PFOA		3.5	3.4	2.9	30.0
PFHxS		4.8	4.7	2.1	30.0
PFHxS-LN		3.0	3.0	0.0	30.0
PFHxS-BR		2.0	2.0	0.0	30.0
PFNA	J	0.51	0.51	0.0	30.0
8:2 FTSA		ND	ND	NC	30.0
PFHpS		ND	ND	NC	30.0
PFDA		ND	ND	NC	30.0
N-MeFOSAA		ND	ND	NC	30.0
EtFOSAA		ND	ND	NC	30.0
PFOS	J	0.66	0.50	27.6	30.0
PFOS-LN	*	0.35	ND	200.0	30.0
PFOS-BR	*	0.38	ND	200.0	30.0
PFUnDA		ND	ND	NC	30.0
PFNS		ND	ND	NC	30.0
PFDODA		ND	ND	NC	30.0
PFDS		ND	ND	NC	30.0
PFTTrDA		ND	ND	NC	30.0
FOSA		ND	ND	NC	30.0
PFTeDA		ND	ND	NC	30.0
11CL-PF3OUdS		ND	ND	NC	30.0
9CL-PF3ONS		ND	ND	NC	30.0
ADONA		ND	ND	NC	30.0
HFPO-DA		ND	ND	NC	30.0
PFDOS		ND	ND	NC	30.0
NMeFOSAM		ND	ND	NC	30.0
NEtFOSAM		ND	ND	NC	30.0
NMeFOSE		ND	ND	NC	30.0
NEtFOSE		ND	ND	NC	30.0

# Merit Laboratories Login Checklist

Lab Set ID:S61354

Client:BARR (Barr Engineering)

Project: SRC 49161497.04 100 101

Submitted:04/25/2024 09:40 Login User: MMC

Attention: David Beattie

Address: Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802

Phone: 218-348-9051 FAX:  
Email: David.Beattie@cenovus.com

Selection	Description	Note
<b>Sample Receiving</b>		
01.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples are received at 4C +/- 2C Thermometer # IR 2.0
02.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Received on ice/ cooling process begun
03.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples shipped FedEx
04.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Samples left in 24 hr. drop box
05.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Are there custody seals/tape or is the drop box locked
<b>Chain of Custody</b>		
06.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	COC adequately filled out
07.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	COC signed and relinquished to the lab
08.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Sample tag on bottles match COC
09.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Subcontracting needed? Subcontracted to:
<b>Preservation</b>		
10.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Do sample have correct chemical preservation
11.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Completed pH checks on preserved samples? (no VOAs)
12.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Did any samples need to be preserved in the lab?
<b>Bottle Conditions</b>		
13.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	All bottles intact
14.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Appropriate analytical bottles are used
15.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Merit bottles used
16.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Sufficient sample volume received
17.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Samples require laboratory filtration
18.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples submitted within holding time
19.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Do water VOC or TOX bottles contain headspace

Corrective action for all exceptions is to call the client and to notify the project manager.

Client Review By: \_\_\_\_\_ Date: \_\_\_\_\_



# Barr Engineering Co. Chain of Custody

## Sample Origination State

☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ NV ☐ TX ☐ UT ☒ WI ☐ WY ☐ Other: \_\_\_\_\_

REPORT TO		INVOICE TO	
Company: <u>Barr Engineering</u>	Company: <u>Barr Engineering</u>		
Address: <u>325 S. Lake Ave</u>	Address:		
Address: <u>Duluth, MN 55802</u>	Address:		
Name: <u>Lynette Carney</u>	Name: <u>SAME</u>		
email: <u>LCarney@barr.com</u>	email:		
Copy to: <u>BarrDM@barr.com</u>	P.O.		
Project Name: <u>SR</u>	Barr Project No: <u>49161497.04 100 101</u>		

Location	Sample Depth			Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code
	Start	Stop	Unit (m./ft. or in.)			
1. <u>FB-01</u>	<u>      </u>	<u>      </u>		<u>04/24/2024</u>	<u>09:25</u>	<u>WQ</u>
2. <u>RB-01</u>	<u>      </u>	<u>      </u>		<u>04/24/2024</u>	<u>09:35</u>	<u>WQ</u>
3. <u>RB-02</u>	<u>      </u>	<u>      </u>		<u>04/24/2024</u>	<u>09:45</u>	<u>WQ</u>
4. <u>FD-01</u>	<u>      </u>	<u>      </u>		<u>04/24/2024</u>	<u>      </u>	<u>WQ</u>
5. <u>IW-1</u>	<u>      </u>	<u>      </u>		<u>04/24/2024</u>	<u>10:15</u>	<u>GW</u>
6.						
7.						
8.						
9.						
10.						

		Analysis Requested												COC Number: <b>Nº 597209</b>
		Water						Soil						
Perform MS/MSD Y / N	Total Number Of Containers													COC <u>1</u> of <u>1</u>
													Matrix Code: GW = Groundwater SW = Surface Water DW = Drinking Water PW = Pore Water WW = Waste Water WQ = TB, FB, EB, etc. W = Unspecified S = Soil/Solid SD = Sediment SQ = MeOH blank OTH = Other (Oil, etc.)	
													Preservative Code: A = None B = HCl C = HNO <sub>3</sub> D = H <sub>2</sub> SO <sub>4</sub> E = NaOH F = MeOH G = NaHSO <sub>4</sub> H = Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> I = Ascorbic Acid J = Zn Acetate K = Other	
													Preservative Code	
													Field Filtered Y/N	
													see attached list for WI PFAS(33) <u>61354.01</u>	
													<u>.02</u>	
													<u>.03</u>	
													<u>.04</u>	
													<u>.05</u>	

BARR USE ONLY		Relinquished by: <u>[Signature]</u>		On Ice? <input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Date: <u>04/24/2024</u>	Time: <u>12:41</u>	Received by:	Date:	Time:
Sampled by: <u>ISP</u>		Relinquished by: <u>Fed Ex</u>		On Ice? <input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Date: <u>4/25/24</u>	Time: <u>0940</u>	Received by: <u>[Signature]</u>	Date: <u>4/25/24</u>	Time: <u>0940</u>
Barr Proj. Manager: <u>LML</u>		Samples Shipped VIA: <input type="checkbox"/> Ground Courier <input checked="" type="checkbox"/> Air Carrier		Air Bill Number: <u>8177 1640 3167</u>		Requested Due Date: <input checked="" type="checkbox"/> Standard Turn Around Time			
Barr DQ Manager: <u>TAO</u>		<input type="checkbox"/> Sampler <input type="checkbox"/> Other: _____		Custody Seal Intact? <input type="checkbox"/> Y <input type="checkbox"/> N <input type="checkbox"/> None		<input type="checkbox"/> Rush _____			
Lab Name: <u>Merit</u>		Lab WO: _____		Temperature on Receipt (°C): <u>2.0</u>					
Lab Location: <u>East Lansing, MI</u>									

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Scan and email: a copy to BarrDM@barr.com for tracking and filing procedures

# WI PFAS (33)

Perfluorobutanoic acid (PFBA)	
Perfluorobutanesulfonic acid (PFBS)	
Perfluoropentanoic acid (PFPeA)	
Perfluoropentanesulfonic acid (PFPeS)	
Perfluorohexanoic acid (PFHxA)	
Perfluorohexanesulfonic acid (PFHxS)	
Perfluoroheptanoic acid (PFHpA)	
Perfluoroheptane sulfonic acid (PFHpS)	
Perfluorooctanoic acid (PFOA)	
Perfluorooctanesulfonic acid (PFOS)	
Perfluorononanoic acid (PFNA)	
Perfluorononanesulfonic acid (PFNS)	
Perfluorodecanoic acid (PFDA)	
Perfluorodecane sulfonic acid (PFDS)	
Perfluoroundecanoic acid (PFUnA)	
Perfluorododecanoic acid (PFDoA)	
Perfluorododecanesulfonic Acid (PFDoS or PFDoDS)	
Perfluorooctanesulfonamide (PFOSA)	
Perfluorotridecanoic acid (PFTTrDA)	
Perfluorotetradecanoic acid (PFTDA or PFTA or PFTeDA)	
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	
Hexafluoropropylene oxide dimer acid (HFPO-DA) aka Gen-X	
<sup>11</sup> Cl-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) - F53B Minor	
<sup>9</sup> Cl-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) - F53B Major	
4,8-dioxa-3H-perfluorononanoic acid (DONA; NaDONA in MDH)	
N-Methyl perfluorooctane sulfonamide (MeFOSA)	
N-Ethyl perfluorooctane sulfonamide (EtFOSA)	
N-Methyl perfluorooctane sulfonamidoethanol (MeFOSE)	
N-Ethyl perfluorooctane sulfonamidoethanol (EtFOSE)	
4:2 FTS	
6:2 FTS	
8:2 FTS	





April 03, 2024

Terri Olson  
Barr Engineering Company  
4300 MarketPointe Drive  
Suite 200  
Minneapolis, MN 55435

RE: Project: 49161497.04 100 101 SRC  
Pace Project No.: 10687707

Dear Terri Olson:

Enclosed are the analytical results for sample(s) received by the laboratory on March 28, 2024. The results relate only to the samples included in this report. Results reported herein conform to the applicable TNI/NELAC Standards and the laboratory's Quality Manual, where applicable, unless otherwise noted in the body of the report.

The test results provided in this final report were generated by each of the following laboratories within the Pace Network:

- Pace Analytical Services - Minneapolis

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

A handwritten signature in blue ink, appearing to read "Martha Hansen".

Martha Hansen  
martha.hansen@pacelabs.com  
(612)607-6451  
Project Manager

Enclosures

cc: Barr DM, Barr Engineering  
Accounts Payable, Barr Engineering



## REPORT OF LABORATORY ANALYSIS

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## CERTIFICATIONS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

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**Pace Analytical Services, LLC - Minneapolis MN**

1700 Elm Street SE, Minneapolis, MN 55414

A2LA Certification #: 2926.01

Alabama Certification #: 40770

Alaska Contaminated Sites Certification #: 17-009

Alaska DW Certification #: MN00064

Arizona Certification #: AZ0014

Arkansas DW Certification #: MN00064

Arkansas WW Certification #: 88-0680

California Certification #: 2929

Colorado Certification #: MN00064

Connecticut Certification #: PH-0256

EPA Region 8 Tribal Water Systems+Wyoming DW

Certification #: via MN 027-053-137

Florida Certification #: E87605

Georgia Certification #: 959

GMP+ Certification #: GMP050884

Hawaii Certification #: MN00064

Idaho Certification #: MN00064

Illinois Certification #: 200011

Indiana Certification #: C-MN-01

Iowa Certification #: 368

Kansas Certification #: E-10167

Kentucky DW Certification #: 90062

Kentucky WW Certification #: 90062

Louisiana DEQ Certification #: AI-03086

Louisiana DW Certification #: MN00064

Maine Certification #: MN00064

Maryland Certification #: 322

Michigan Certification #: 9909

Minnesota Certification #: 027-053-137

Minnesota Dept of Ag Approval: via MN 027-053-137

Minnesota Petrofund Registration #: 1240

Mississippi Certification #: MN00064

Missouri Certification #: 10100

Montana Certification #: CERT0092

Nebraska Certification #: NE-OS-18-06

Nevada Certification #: MN00064

New Hampshire Certification #: 2081

New Jersey Certification #: MN002

New York Certification #: 11647

North Carolina DW Certification #: 27700

North Carolina WW Certification #: 530

North Dakota Certification (A2LA) #: R-036

North Dakota Certification (MN) #: R-036

Ohio DW Certification #: 41244

Ohio VAP Certification (1700) #: CL101

Oklahoma Certification #: 9507

Oregon Primary Certification #: MN300001

Oregon Secondary Certification #: MN200001

Pennsylvania Certification #: 68-00563

Puerto Rico Certification #: MN00064

South Carolina Certification #: 74003001

Tennessee Certification #: TN02818

Texas Certification #: T104704192

Utah Certification #: MN00064

Vermont Certification #: VT-027053137

Virginia Certification #: 460163

Washington Certification #: C486

West Virginia DEP Certification #: 382

West Virginia DW Certification #: 9952 C

Wisconsin Certification #: 999407970

Wyoming UST Certification #: via A2LA 2926.01

USDA Permit #: P330-19-00208

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## REPORT OF LABORATORY ANALYSIS

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## SAMPLE SUMMARY

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10687707001	FB-01	Water	03/27/24 09:30	03/28/24 11:00
10687707002	FD-01	Water	03/27/24 00:00	03/28/24 11:00
10687707003	IW-1	Water	03/27/24 10:40	03/28/24 11:00
10687707004	TB-01	Water	03/27/24 09:25	03/28/24 11:00

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## SAMPLE ANALYTE COUNT

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10687707001	FB-01	EPA 8270E by SIM	GY1	20	PASI-M
		EPA 8260D	TKL	10	PASI-M
10687707002	FD-01	EPA 8270E by SIM	GY1	20	PASI-M
		EPA 8260D	TKL	10	PASI-M
10687707003	IW-1	EPA 8270E by SIM	GY1	20	PASI-M
		EPA 8260D	TKL	10	PASI-M
10687707004	TB-01	EPA 8260D	TKL	10	PASI-M

PASI-M = Pace Analytical Services - Minneapolis

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

Sample: FB-01 Lab ID: 10687707001 Collected: 03/27/24 09:30 Received: 03/28/24 11:00 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8270E MSSV PAH by SIM</b>									
Analytical Method: EPA 8270E by SIM Preparation Method: EPA 3510C									
Pace Analytical Services - Minneapolis									
Acenaphthene	0.015J	ug/L	0.039	0.0045	1	03/29/24 15:59	04/01/24 14:00	83-32-9	
Acenaphthylene	<0.0041	ug/L	0.039	0.0041	1	03/29/24 15:59	04/01/24 14:00	208-96-8	
Anthracene	<0.0072	ug/L	0.039	0.0072	1	03/29/24 15:59	04/01/24 14:00	120-12-7	
Benzo(a)anthracene	<0.0045	ug/L	0.039	0.0045	1	03/29/24 15:59	04/01/24 14:00	56-55-3	
Benzo(a)pyrene	<0.0048	ug/L	0.039	0.0048	1	03/29/24 15:59	04/01/24 14:00	50-32-8	
Benzo(b)fluoranthene	<0.0073	ug/L	0.039	0.0073	1	03/29/24 15:59	04/01/24 14:00	205-99-2	
Benzo(g,h,i)perylene	<0.0098	ug/L	0.039	0.0098	1	03/29/24 15:59	04/01/24 14:00	191-24-2	
Benzo(k)fluoranthene	<0.0080	ug/L	0.039	0.0080	1	03/29/24 15:59	04/01/24 14:00	207-08-9	
Chrysene	<0.0078	ug/L	0.039	0.0078	1	03/29/24 15:59	04/01/24 14:00	218-01-9	
Dibenz(a,h)anthracene	<0.010	ug/L	0.039	0.010	1	03/29/24 15:59	04/01/24 14:00	53-70-3	
Fluoranthene	<0.014	ug/L	0.039	0.014	1	03/29/24 15:59	04/01/24 14:00	206-44-0	
Fluorene	<0.0052	ug/L	0.039	0.0052	1	03/29/24 15:59	04/01/24 14:00	86-73-7	
Indeno(1,2,3-cd)pyrene	<0.0097	ug/L	0.039	0.0097	1	03/29/24 15:59	04/01/24 14:00	193-39-5	
1-Methylnaphthalene	<0.0053	ug/L	0.039	0.0053	1	03/29/24 15:59	04/01/24 14:00	90-12-0	
2-Methylnaphthalene	<0.0072	ug/L	0.039	0.0072	1	03/29/24 15:59	04/01/24 14:00	91-57-6	
Naphthalene	<0.015	ug/L	0.039	0.015	1	03/29/24 15:59	04/01/24 14:00	91-20-3	
Phenanthrene	<0.013	ug/L	0.039	0.013	1	03/29/24 15:59	04/01/24 14:00	85-01-8	
Pyrene	<0.012	ug/L	0.039	0.012	1	03/29/24 15:59	04/01/24 14:00	129-00-0	
<b>Surrogates</b>									
2-Fluorobiphenyl (S)	68	%.	34-125		1	03/29/24 15:59	04/01/24 14:00	321-60-8	
p-Terphenyl-d14 (S)	78	%.	38-139		1	03/29/24 15:59	04/01/24 14:00	1718-51-0	
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D									
Pace Analytical Services - Minneapolis									
Benzene	<0.21	ug/L	1.0	0.21	1		03/28/24 15:32	71-43-2	
Ethylbenzene	<0.11	ug/L	1.0	0.11	1		03/28/24 15:32	100-41-4	
Methyl-tert-butyl ether	<0.13	ug/L	1.0	0.13	1		03/28/24 15:32	1634-04-4	
Toluene	<0.21	ug/L	1.0	0.21	1		03/28/24 15:32	108-88-3	
1,2,4-Trimethylbenzene	<0.13	ug/L	1.0	0.13	1		03/28/24 15:32	95-63-6	
1,3,5-Trimethylbenzene	<0.11	ug/L	1.0	0.11	1		03/28/24 15:32	108-67-8	
Xylene (Total)	<0.42	ug/L	3.0	0.42	1		03/28/24 15:32	1330-20-7	
<b>Surrogates</b>									
1,2-Dichlorobenzene-d4 (S)	103	%.	75-125		1		03/28/24 15:32	2199-69-1	
4-Bromofluorobenzene (S)	99	%.	75-125		1		03/28/24 15:32	460-00-4	
Toluene-d8 (S)	111	%.	75-125		1		03/28/24 15:32	2037-26-5	

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

Sample: FD-01 Lab ID: 10687707002 Collected: 03/27/24 00:00 Received: 03/28/24 11:00 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8270E MSSV PAH by SIM</b>									
Analytical Method: EPA 8270E by SIM Preparation Method: EPA 3510C									
Pace Analytical Services - Minneapolis									
Acenaphthene	<0.0045	ug/L	0.039	0.0045	1	03/29/24 15:59	04/01/24 14:22	83-32-9	
Acenaphthylene	<0.0041	ug/L	0.039	0.0041	1	03/29/24 15:59	04/01/24 14:22	208-96-8	
Anthracene	<0.0072	ug/L	0.039	0.0072	1	03/29/24 15:59	04/01/24 14:22	120-12-7	
Benzo(a)anthracene	<0.0045	ug/L	0.039	0.0045	1	03/29/24 15:59	04/01/24 14:22	56-55-3	
Benzo(a)pyrene	<0.0048	ug/L	0.039	0.0048	1	03/29/24 15:59	04/01/24 14:22	50-32-8	
Benzo(b)fluoranthene	<0.0073	ug/L	0.039	0.0073	1	03/29/24 15:59	04/01/24 14:22	205-99-2	
Benzo(g,h,i)perylene	<0.0098	ug/L	0.039	0.0098	1	03/29/24 15:59	04/01/24 14:22	191-24-2	
Benzo(k)fluoranthene	<0.0080	ug/L	0.039	0.0080	1	03/29/24 15:59	04/01/24 14:22	207-08-9	
Chrysene	<0.0078	ug/L	0.039	0.0078	1	03/29/24 15:59	04/01/24 14:22	218-01-9	
Dibenz(a,h)anthracene	<0.010	ug/L	0.039	0.010	1	03/29/24 15:59	04/01/24 14:22	53-70-3	
Fluoranthene	<0.014	ug/L	0.039	0.014	1	03/29/24 15:59	04/01/24 14:22	206-44-0	
Fluorene	<0.0052	ug/L	0.039	0.0052	1	03/29/24 15:59	04/01/24 14:22	86-73-7	
Indeno(1,2,3-cd)pyrene	<0.0097	ug/L	0.039	0.0097	1	03/29/24 15:59	04/01/24 14:22	193-39-5	
1-Methylnaphthalene	<0.0053	ug/L	0.039	0.0053	1	03/29/24 15:59	04/01/24 14:22	90-12-0	
2-Methylnaphthalene	<0.0072	ug/L	0.039	0.0072	1	03/29/24 15:59	04/01/24 14:22	91-57-6	
Naphthalene	<0.015	ug/L	0.039	0.015	1	03/29/24 15:59	04/01/24 14:22	91-20-3	
Phenanthrene	<0.013	ug/L	0.039	0.013	1	03/29/24 15:59	04/01/24 14:22	85-01-8	
Pyrene	<0.012	ug/L	0.039	0.012	1	03/29/24 15:59	04/01/24 14:22	129-00-0	
<b>Surrogates</b>									
2-Fluorobiphenyl (S)	64	%.	34-125		1	03/29/24 15:59	04/01/24 14:22	321-60-8	
p-Terphenyl-d14 (S)	83	%.	38-139		1	03/29/24 15:59	04/01/24 14:22	1718-51-0	
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D									
Pace Analytical Services - Minneapolis									
Benzene	<0.21	ug/L	1.0	0.21	1		03/28/24 16:54	71-43-2	
Ethylbenzene	<0.11	ug/L	1.0	0.11	1		03/28/24 16:54	100-41-4	
Methyl-tert-butyl ether	<0.13	ug/L	1.0	0.13	1		03/28/24 16:54	1634-04-4	
Toluene	<0.21	ug/L	1.0	0.21	1		03/28/24 16:54	108-88-3	
1,2,4-Trimethylbenzene	<0.13	ug/L	1.0	0.13	1		03/28/24 16:54	95-63-6	
1,3,5-Trimethylbenzene	<0.11	ug/L	1.0	0.11	1		03/28/24 16:54	108-67-8	
Xylene (Total)	<0.42	ug/L	3.0	0.42	1		03/28/24 16:54	1330-20-7	
<b>Surrogates</b>									
1,2-Dichlorobenzene-d4 (S)	101	%.	75-125		1		03/28/24 16:54	2199-69-1	
4-Bromofluorobenzene (S)	100	%.	75-125		1		03/28/24 16:54	460-00-4	
Toluene-d8 (S)	109	%.	75-125		1		03/28/24 16:54	2037-26-5	

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## ANALYTICAL RESULTS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

Sample: IW-1 Lab ID: 10687707003 Collected: 03/27/24 10:40 Received: 03/28/24 11:00 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8270E MSSV PAH by SIM</b>									
Analytical Method: EPA 8270E by SIM Preparation Method: EPA 3510C									
Pace Analytical Services - Minneapolis									
Acenaphthene	<0.0045	ug/L	0.039	0.0045	1	03/29/24 15:59	04/01/24 14:44	83-32-9	
Acenaphthylene	<0.0041	ug/L	0.039	0.0041	1	03/29/24 15:59	04/01/24 14:44	208-96-8	
Anthracene	<0.0072	ug/L	0.039	0.0072	1	03/29/24 15:59	04/01/24 14:44	120-12-7	
Benzo(a)anthracene	<0.0045	ug/L	0.039	0.0045	1	03/29/24 15:59	04/01/24 14:44	56-55-3	
Benzo(a)pyrene	<0.0048	ug/L	0.039	0.0048	1	03/29/24 15:59	04/01/24 14:44	50-32-8	
Benzo(b)fluoranthene	<0.0073	ug/L	0.039	0.0073	1	03/29/24 15:59	04/01/24 14:44	205-99-2	
Benzo(g,h,i)perylene	<0.0098	ug/L	0.039	0.0098	1	03/29/24 15:59	04/01/24 14:44	191-24-2	
Benzo(k)fluoranthene	<0.0080	ug/L	0.039	0.0080	1	03/29/24 15:59	04/01/24 14:44	207-08-9	
Chrysene	<0.0078	ug/L	0.039	0.0078	1	03/29/24 15:59	04/01/24 14:44	218-01-9	
Dibenz(a,h)anthracene	<0.010	ug/L	0.039	0.010	1	03/29/24 15:59	04/01/24 14:44	53-70-3	
Fluoranthene	<0.014	ug/L	0.039	0.014	1	03/29/24 15:59	04/01/24 14:44	206-44-0	
Fluorene	<0.0052	ug/L	0.039	0.0052	1	03/29/24 15:59	04/01/24 14:44	86-73-7	
Indeno(1,2,3-cd)pyrene	<0.0097	ug/L	0.039	0.0097	1	03/29/24 15:59	04/01/24 14:44	193-39-5	
1-Methylnaphthalene	<0.0053	ug/L	0.039	0.0053	1	03/29/24 15:59	04/01/24 14:44	90-12-0	
2-Methylnaphthalene	<0.0072	ug/L	0.039	0.0072	1	03/29/24 15:59	04/01/24 14:44	91-57-6	
Naphthalene	<0.015	ug/L	0.039	0.015	1	03/29/24 15:59	04/01/24 14:44	91-20-3	
Phenanthrene	<0.013	ug/L	0.039	0.013	1	03/29/24 15:59	04/01/24 14:44	85-01-8	
Pyrene	<0.012	ug/L	0.039	0.012	1	03/29/24 15:59	04/01/24 14:44	129-00-0	
<b>Surrogates</b>									
2-Fluorobiphenyl (S)	66	%.	34-125		1	03/29/24 15:59	04/01/24 14:44	321-60-8	
p-Terphenyl-d14 (S)	80	%.	38-139		1	03/29/24 15:59	04/01/24 14:44	1718-51-0	
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D									
Pace Analytical Services - Minneapolis									
Benzene	<0.21	ug/L	1.0	0.21	1		03/28/24 17:10	71-43-2	
Ethylbenzene	<0.11	ug/L	1.0	0.11	1		03/28/24 17:10	100-41-4	
Methyl-tert-butyl ether	<0.13	ug/L	1.0	0.13	1		03/28/24 17:10	1634-04-4	
Toluene	<0.21	ug/L	1.0	0.21	1		03/28/24 17:10	108-88-3	
1,2,4-Trimethylbenzene	<0.13	ug/L	1.0	0.13	1		03/28/24 17:10	95-63-6	
1,3,5-Trimethylbenzene	<0.11	ug/L	1.0	0.11	1		03/28/24 17:10	108-67-8	
Xylene (Total)	<0.42	ug/L	3.0	0.42	1		03/28/24 17:10	1330-20-7	
<b>Surrogates</b>									
1,2-Dichlorobenzene-d4 (S)	101	%.	75-125		1		03/28/24 17:10	2199-69-1	
4-Bromofluorobenzene (S)	99	%.	75-125		1		03/28/24 17:10	460-00-4	
Toluene-d8 (S)	107	%.	75-125		1		03/28/24 17:10	2037-26-5	

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

Sample: TB-01 Lab ID: 10687707004 Collected: 03/27/24 09:25 Received: 03/28/24 11:00 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D									
Pace Analytical Services - Minneapolis									
Benzene	<0.21	ug/L	1.0	0.21	1		03/28/24 15:16	71-43-2	
Ethylbenzene	<0.11	ug/L	1.0	0.11	1		03/28/24 15:16	100-41-4	
Methyl-tert-butyl ether	<0.13	ug/L	1.0	0.13	1		03/28/24 15:16	1634-04-4	
Toluene	<0.21	ug/L	1.0	0.21	1		03/28/24 15:16	108-88-3	
1,2,4-Trimethylbenzene	<0.13	ug/L	1.0	0.13	1		03/28/24 15:16	95-63-6	
1,3,5-Trimethylbenzene	<0.11	ug/L	1.0	0.11	1		03/28/24 15:16	108-67-8	
Xylene (Total)	<0.42	ug/L	3.0	0.42	1		03/28/24 15:16	1330-20-7	
<b>Surrogates</b>									
1,2-Dichlorobenzene-d4 (S)	101	%.	75-125		1		03/28/24 15:16	2199-69-1	
4-Bromofluorobenzene (S)	98	%.	75-125		1		03/28/24 15:16	460-00-4	
Toluene-d8 (S)	111	%.	75-125		1		03/28/24 15:16	2037-26-5	

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## QUALITY CONTROL DATA

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

QC Batch:	938370	Analysis Method:	EPA 8260D
QC Batch Method:	EPA 8260D	Analysis Description:	8260D MSV UST-WATER
		Laboratory:	Pace Analytical Services - Minneapolis

Associated Lab Samples: 10687707001, 10687707002, 10687707003, 10687707004

METHOD BLANK: 4914769 Matrix: Water

Associated Lab Samples: 10687707001, 10687707002, 10687707003, 10687707004

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
1,2,4-Trimethylbenzene	ug/L	<0.13	1.0	03/28/24 14:43	
1,3,5-Trimethylbenzene	ug/L	<0.11	1.0	03/28/24 14:43	
Benzene	ug/L	<0.21	1.0	03/28/24 14:43	
Ethylbenzene	ug/L	<0.11	1.0	03/28/24 14:43	
Methyl-tert-butyl ether	ug/L	<0.13	1.0	03/28/24 14:43	
Toluene	ug/L	<0.21	1.0	03/28/24 14:43	
Xylene (Total)	ug/L	<0.42	3.0	03/28/24 14:43	
1,2-Dichlorobenzene-d4 (S)	%	101	75-125	03/28/24 14:43	
4-Bromofluorobenzene (S)	%	98	75-125	03/28/24 14:43	
Toluene-d8 (S)	%	109	75-125	03/28/24 14:43	

LABORATORY CONTROL SAMPLE &amp; LCSD: 4914770

4914771

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
1,2,4-Trimethylbenzene	ug/L	20	20.8	21.0	104	105	75-125	1	20	
1,3,5-Trimethylbenzene	ug/L	20	20.4	20.7	102	104	75-125	1	20	
Benzene	ug/L	20	20.9	20.9	105	105	75-125	0	20	
Ethylbenzene	ug/L	20	21.3	20.8	107	104	75-125	2	20	
Methyl-tert-butyl ether	ug/L	20	22.8	22.1	114	110	75-125	3	20	
Toluene	ug/L	20	20.9	20.2	104	101	75-125	4	20	
Xylene (Total)	ug/L	60	63.9	61.0	107	102	75-125	5	20	
1,2-Dichlorobenzene-d4 (S)	%				100	99	75-125			
4-Bromofluorobenzene (S)	%				103	101	75-125			
Toluene-d8 (S)	%				103	100	75-125			

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## QUALITY CONTROL DATA

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

QC Batch: 938473

Analysis Method: EPA 8270E by SIM

QC Batch Method: EPA 3510C

Analysis Description: 8270E Water PAH by SIM MSSV

Laboratory: Pace Analytical Services - Minneapolis

Associated Lab Samples: 10687707001, 10687707002, 10687707003

METHOD BLANK: 4915123

Matrix: Water

Associated Lab Samples: 10687707001, 10687707002, 10687707003

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
1-Methylnaphthalene	ug/L	<0.0054	0.040	04/01/24 11:48	
2-Methylnaphthalene	ug/L	<0.0074	0.040	04/01/24 11:48	
Acenaphthene	ug/L	<0.0046	0.040	04/01/24 11:48	
Acenaphthylene	ug/L	<0.0042	0.040	04/01/24 11:48	
Anthracene	ug/L	<0.0073	0.040	04/01/24 11:48	
Benzo(a)anthracene	ug/L	<0.0046	0.040	04/01/24 11:48	
Benzo(a)pyrene	ug/L	<0.0049	0.040	04/01/24 11:48	
Benzo(b)fluoranthene	ug/L	<0.0074	0.040	04/01/24 11:48	
Benzo(g,h,i)perylene	ug/L	<0.010	0.040	04/01/24 11:48	
Benzo(k)fluoranthene	ug/L	<0.0081	0.040	04/01/24 11:48	
Chrysene	ug/L	<0.0080	0.040	04/01/24 11:48	
Dibenz(a,h)anthracene	ug/L	<0.010	0.040	04/01/24 11:48	
Fluoranthene	ug/L	<0.014	0.040	04/01/24 11:48	
Fluorene	ug/L	<0.0053	0.040	04/01/24 11:48	
Indeno(1,2,3-cd)pyrene	ug/L	<0.0099	0.040	04/01/24 11:48	
Naphthalene	ug/L	<0.015	0.040	04/01/24 11:48	
Phenanthrene	ug/L	<0.013	0.040	04/01/24 11:48	
Pyrene	ug/L	<0.013	0.040	04/01/24 11:48	
2-Fluorobiphenyl (S)	%	72	34-125	04/01/24 11:48	
p-Terphenyl-d14 (S)	%	84	38-139	04/01/24 11:48	

LABORATORY CONTROL SAMPLE &amp; LCSD: 4915124

4915125

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
1-Methylnaphthalene	ug/L	1	0.79	0.69	79	69	42-125	14	20	
2-Methylnaphthalene	ug/L	1	0.76	0.68	76	68	39-125	12	20	
Acenaphthene	ug/L	1	0.72	0.69	72	69	45-125	4	20	
Acenaphthylene	ug/L	1	0.73	0.68	73	68	47-125	6	20	
Anthracene	ug/L	1	0.81	0.77	81	77	50-125	5	20	
Benzo(a)anthracene	ug/L	1	0.83	0.84	83	84	60-125	2	20	
Benzo(a)pyrene	ug/L	1	0.77	0.79	77	79	60-125	3	20	
Benzo(b)fluoranthene	ug/L	1	0.81	0.82	81	82	54-125	1	20	
Benzo(g,h,i)perylene	ug/L	1	0.81	0.84	81	84	46-125	3	20	
Benzo(k)fluoranthene	ug/L	1	0.84	0.89	84	89	57-125	5	20	
Chrysene	ug/L	1	0.84	0.83	84	83	60-125	1	20	
Dibenz(a,h)anthracene	ug/L	1	0.77	0.72	77	72	32-125	8	20	
Fluoranthene	ug/L	1	0.78	0.74	78	74	55-125	5	20	
Fluorene	ug/L	1	0.72	0.70	72	70	50-125	4	20	
Indeno(1,2,3-cd)pyrene	ug/L	1	0.79	0.79	79	79	56-125	1	20	

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## QUALITY CONTROL DATA

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

LABORATORY CONTROL SAMPLE & LCSD: 4915124			4915125							
Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
Naphthalene	ug/L	1	0.71	0.66	71	66	43-125	6	20	
Phenanthrene	ug/L	1	0.79	0.76	79	76	56-125	4	20	
Pyrene	ug/L	1	0.85	0.81	85	81	59-125	4	20	
2-Fluorobiphenyl (S)	%.				77	69	34-125			
p-Terphenyl-d14 (S)	%.				85	83	38-139			

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## QUALIFIERS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above LOD.

J - The reported result is an estimated value.

LOD - Limit of Detection adjusted for dilution factor, percent moisture, initial weight and final volume.

LOQ - Limit of Quantitation adjusted for dilution factor, percent moisture, initial weight and final volume.

DL - Adjusted Method Detection Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Analyte was not detected and is reported as less than the LOD or as defined by the customer.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### BATCH QUALIFIERS

Batch: 938370

[M5] A matrix spike/matrix spike duplicate was not performed for this batch due to insufficient sample volume.

Batch: 938748

[M5] A matrix spike/matrix spike duplicate was not performed for this batch due to insufficient sample volume.

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161497.04 100 101 SRC

Pace Project No.: 10687707

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10687707001	FB-01	EPA 3510C	938473	EPA 8270E by SIM	938748
10687707002	FD-01	EPA 3510C	938473	EPA 8270E by SIM	938748
10687707003	IW-1	EPA 3510C	938473	EPA 8270E by SIM	938748
10687707001	FB-01	EPA 8260D	938370		
10687707002	FD-01	EPA 8260D	938370		
10687707003	IW-1	EPA 8260D	938370		
10687707004	TB-01	EPA 8260D	938370		

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# Barr Engineering Co. Chain of Custody

## Sample Origination State

☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ NV ☐ TX ☐ UT ☒ WI ☐ VA

WO#: 10687707



10687707

ested

Soil

COC Number: **Nº 597205**

COC 1 of 1

## REPORT TO

## INVOICE TO

Company: Barr Engineering  
Address: 325 S. Lake Ave  
Address: Duluth MN, 55802  
Name: Lynette Corney  
email: LCorney@barr.com  
Copy to: BarrDM@barr.com  
Project Name: SR

Company: Barr Engineering  
Address:                       
Address:                       
Name: SAME  
email:                       
P.O.                       
Barr Project No: 49161497.04 100 101

## Matrix Code:

## Preservative Code:

GW = Groundwater A = None  
SW = Surface Water B = HCl  
DW = Drinking Water C = HNO<sub>3</sub>  
PW = Pore Water D = H<sub>2</sub>SO<sub>4</sub>  
WW = Waste Water E = NaOH  
WQ = TB, FB, EB, etc. F = MeOH  
W = Unspecified G = NaHSO<sub>4</sub>  
S = Soil/Solid H = Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>  
SD = Sediment I = Ascorbic Acid  
SQ = MeOH blank J = Zn Acetate  
OTH = Other (Oil, etc.) K = Other

Location	Sample Depth			Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform MS/MSD Y/N	Total Number Of Containers	PAHs A B N N	Pb/Cd A B N N	% Solids	Preservative Code	Field Filtered Y/N
	Start	Stop	Unit (m./ft. or in.)										
1. <u>FB-01</u>	<u>          </u>	<u>          </u>	<u>          </u>	<u>03/27/2024</u>	<u>09:30</u>	<u>WQ</u>	<u>N</u>	<u>5</u>	<u>X</u>	<u>X</u>		<u>001</u>	
2. <u>FD-01</u>	<u>          </u>	<u>          </u>	<u>          </u>	<u>03/24/2024</u>	<u>          </u>	<u>WQ</u>	<u>N</u>	<u>5</u>	<u>X</u>	<u>X</u>		<u>002</u>	
3. <u>IW-1</u>	<u>          </u>	<u>          </u>	<u>          </u>	<u>03/27/2024</u>	<u>10:40</u>	<u>GW</u>	<u>N</u>	<u>5</u>	<u>X</u>	<u>X</u>		<u>003</u>	
4. <u>TB-01</u>	<u>          </u>	<u>          </u>	<u>          </u>	<u>03/27/2024</u>	<u>09:25</u>	<u>WQ</u>	<u>N</u>	<u>2</u>	<u>X</u>			<u>004</u>	
5.													
6.													
7.													
8.													
9.												<u>EZ 3083587</u>	
10.													

## BARR USE ONLY

Sampled by: JSP

Barr Proj. Manager: LMC

Barr DQ Manager: TAO

Lab Name: Pace

Lab Location: Duluth, MN

Relinquished by: Jr Pahn

On Ice? ☒ Y ☐ N

Date 3/27/24

Time 1457

Received by: Stellaacid Pace

Date 3/27/24

Time 14:57

Relinquished by: Stellaacid Pace

On Ice? ☒ Y ☐ N

Date 3/27/24

Time 1457

Received by: Stellaacid Pace

Date 3/27/24

Time 1100

Samples Shipped VIA: ☐ Ground Courier

☐ Air Carrier

Air Bill Number:                     

☒ Sampler

☐ Other:                     

Requested Due Date:

☒ Standard Turn Around Time

☐ Rush (mm/dd/yyyy)

Lab WO:                     

Temperature on Receipt (°C): 25

Custody Seal Intact? ☐ Y ☐ N ☐ None

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Scan and email: a copy to BarrDM@barr.com for tracking and filing procedures

# ENV-FRM-MIN4-0150 v15\_Sample Condition Upon Receipt

CLIENT NAME: Barr

PROJECT #:

COURIER: ☐ Client ☐ Commercial ☐ FedEx ☒ Pace  
☐ SpeedDee ☐ UPS ☐ USPS

WO#: 10687707

PM: MKH

Due Date: 04/11/24

CLIENT: BARR

TRACKING NUMBER: ☐ See Exceptions form ENV-FRM-MIN4-0142

Custody Seal on Coole/Box Present: ☒ YES ☐ NO Seals Intact: ☒ YES ☐ NO Biological Tissue Frozen: ☐ YES ☐ NO ☐ N/A

Packing Material: ☐ Bubble Bags ☐ Bubble Wrap ☐ None ☐ Other Temp Blank: ☒ YES ☐ NO Type of Ice: ☐ Blue ☐ Dry ☒ Wet

Thermometer: ☐ T1 (0461) ☒ T2 (0436) ☐ T3 (0459) ☐ T4 (0402) ☐ T5 (0178) ☐ T6 (0235)  
☐ T7 (0042) ☐ T8 (0775) ☐ T9 (0727) ☐ 01339252 (1710) ☐ Melted ☐ None

Did Samples Originate in West Virginia: <input type="checkbox"/> YES <input checked="" type="checkbox"/> NO	Were All Container Temps taken: <input type="checkbox"/> YES <input type="checkbox"/> NO <input checked="" type="checkbox"/> N/A
Correction Factor: <u>1.2</u> Cooler Temp Read w/Temp Blank: <u>12</u> °C	Average Corrected Temp (no Temp Blank Only): _____ °C
NOTE: Temp should be above freezing to 6°C. Cooler Temp Corrected w/Temp Blank: <u>16</u> °C	<input type="checkbox"/> See Exceptions Form ENV-FRM-MIN4-0142 <input type="checkbox"/> 1 Container

USDA Regulated Soil: <input checked="" type="checkbox"/> N/A <input type="checkbox"/> Water Sample/Other (describe): _____	Initials & Date of Person Examining Contents: <u>3/28/24 MS</u>
Did Samples originate from one of the following states (check maps) – AL, AR, AZ, CA, FL, GA, ID, LA, MS, NC, NM, NY, OK, OR, SC, TN, TX, or VA: <input type="checkbox"/> YES <input type="checkbox"/> NO	Did samples originate from a foreign source (international, including Hawaii and Puerto Rico): <input type="checkbox"/> YES <input type="checkbox"/> NO
NOTE: If YES to either question, fill out a Regulated Soil Checklist (ENV-FRM-MIN4-0154) and include with SCUR/COC paperwork.	

LOCATION (check one): <input type="checkbox"/> DULUTH <input checked="" type="checkbox"/> MINNEAPOLIS <input type="checkbox"/> VIRGINIA	YES	NO	N/A	COMMENT(S)								
Chain of Custody Present and Filled Out?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1.								
Chain of Custody Relinquished?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2.								
Sampler Name and/or Signature on COC?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3.								
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. If Fecal: <input type="checkbox"/> <8 hrs <input type="checkbox"/> >8 hr, <24 hr <input type="checkbox"/> No								
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	5. <input type="checkbox"/> BOD / cBOD <input type="checkbox"/> Fecal coliform <input type="checkbox"/> Hex Chrom <input type="checkbox"/> HPC <input type="checkbox"/> Nitrate <input type="checkbox"/> Nitrite <input type="checkbox"/> Ortho Phos <input type="checkbox"/> Total coliform/E. coli <input type="checkbox"/> Other: _____								
Rush Turn Around Time Requested?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	6.								
Sufficient Sample Volume?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7.								
Correct Containers Used?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8.								
– Pace Containers Used?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>									
Containers Intact?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9.								
Field Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	10. Is sediment visible in the dissolved container: <input type="checkbox"/> YES <input type="checkbox"/> NO								
Is sufficient information available to reconcile the samples to the COC? NOTE: If ID/Date/Time don't match fill out section 11. Matrix: <input type="checkbox"/> Oil <input type="checkbox"/> Soil <input checked="" type="checkbox"/> Water <input type="checkbox"/> Other	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	11. If NO, write ID/Date/Time of container below: <input type="checkbox"/> See Exceptions form ENV-FRM-MIN4-0142								
All containers needing acid/base preservation have been checked? All containers needing preservation are found to be in compliance with EPA recommendation? (HNO <sub>3</sub> , H <sub>2</sub> SO <sub>4</sub> , < 2 pH, NaOH > 9 Sulfide, NaOH > 10 Cyanide) Exceptions: VOA, Coliform, TOC/DOC, Oil & Grease, DRO/8015 (water) and Dioxins/PKAS	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	12. Sample #: <input type="checkbox"/> HNO <sub>3</sub> <input type="checkbox"/> H <sub>2</sub> SO <sub>4</sub> <input type="checkbox"/> NaOH <input type="checkbox"/> Zinc Acetate Positive for Residual Chlorine: <input type="checkbox"/> YES <input type="checkbox"/> NO pH Paper Lot # <table border="1"> <tr> <th>Residual Chlorine</th> <th>0-6 Roll</th> <th>0-6 Strip</th> <th>0-14 Strip</th> </tr> <tr> <td></td> <td></td> <td></td> <td></td> </tr> </table> <input type="checkbox"/> See Exceptions form ENV-FRM-MIN4-0142	Residual Chlorine	0-6 Roll	0-6 Strip	0-14 Strip				
Residual Chlorine	0-6 Roll	0-6 Strip	0-14 Strip									
NOTE: If adding preservative to a container, it must be added to associated field and equipment blanks—verify with PM first.												
Headspace in Methyl Mercury Container?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	13.								
Extra labels present on soil VOA or WIDRO containers?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	14.								
Headspace in VOA Vials (greater than 6mm)?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> See Exceptions form ENV-FRM-MIN4-0142								
Trip Blanks Present?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15. <u>2 total</u>								
Trip Blank Custody Seals Present?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Pace Trip Blank Lot # (if purchased): <u>456</u> <u>157627</u>								

## CLIENT NOTIFICATION / RESOLUTION

FIELD DATA REQUIRED: ☐ YES ☐ NO

Person Contacted: \_\_\_\_\_ Date & Time: \_\_\_\_\_

Comments / Resolution: \_\_\_\_\_

Project Manager Review: [Signature]

Date: 3/28/24

NOTE: When there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e., out of hold, incorrect preservative, out of temp, incorrect containers).

Labeled By: MLS Line: 2



May 08, 2024

Terri Olson  
Barr Engineering Company  
4300 MarketPointe Drive  
Suite 200  
Minneapolis, MN 55435

RE: Project: 49161497.04 100 101 SRC  
Pace Project No.: 10690769

Dear Terri Olson:

Enclosed are the analytical results for sample(s) received by the laboratory on April 25, 2024. The results relate only to the samples included in this report. Results reported herein conform to the applicable TNI/NELAC Standards and the laboratory's Quality Manual, where applicable, unless otherwise noted in the body of the report.

The test results provided in this final report were generated by each of the following laboratories within the Pace Network:

- Pace Analytical Services - Minneapolis

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Martha Hansen  
martha.hansen@pacelabs.com  
(612)607-6451  
Project Manager

Enclosures

cc: Barr DM, Barr Engineering  
Accounts Payable, Barr Engineering



## REPORT OF LABORATORY ANALYSIS

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## CERTIFICATIONS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

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### Pace Analytical Services, LLC - Minneapolis MN

1700 Elm Street SE, Minneapolis, MN 55414

Alabama Certification #: 40770

Alaska Contaminated Sites Certification #: 17-009

Alaska DW Certification #: MN00064

Arizona Certification #: AZ0014

Arkansas DW Certification #: MN00064

Arkansas WW Certification #: 88-0680

California Certification #: 2929

Colorado Certification #: MN00064

Connecticut Certification #: PH-0256

DoD Certification via A2LA #: 2926.01

EPA Region 8 Tribal Water Systems+Wyoming DW  
Certification #: via MN 027-053-137

Florida Certification #: E87605

Georgia Certification #: 959

GMP+ Certification #: GMP050884

Hawaii Certification #: MN00064

Idaho Certification #: MN00064

Illinois Certification #: 200011

Indiana Certification #: C-MN-01

Iowa Certification #: 368

ISO/IEC 17025 Certification via A2LA #: 2926.01

Kansas Certification #: E-10167

Kentucky DW Certification #: 90062

Kentucky WW Certification #: 90062

Louisiana DEQ Certification #: AI-03086

Louisiana DW Certification #: MN00064

Maine Certification #: MN00064

Maryland Certification #: 322

Michigan Certification #: 9909

Minnesota Certification #: 027-053-137

Minnesota Dept of Ag Approval: via MN 027-053-137

Minnesota Petrofund Registration #: 1240

Mississippi Certification #: MN00064

Missouri Certification #: 10100

Montana Certification #: CERT0092

Nebraska Certification #: NE-OS-18-06

Nevada Certification #: MN00064

New Hampshire Certification #: 2081

New Jersey Certification #: MN002

New York Certification #: 11647

North Carolina DW Certification #: 27700

North Carolina WW Certification #: 530

North Dakota Certification (A2LA) #: R-036

North Dakota Certification (MN) #: R-036

Ohio DW Certification #: 41244

Ohio VAP Certification (1700) #: CL101

Oklahoma Certification #: 9507

Oregon Primary Certification #: MN300001

Oregon Secondary Certification #: MN200001

Pennsylvania Certification #: 68-00563

Puerto Rico Certification #: MN00064

South Carolina Certification #: 74003001

Tennessee Certification #: TN02818

Texas Certification #: T104704192

Utah Certification #: MN00064

Vermont Certification #: VT-027053137

Virginia Certification #: 460163

Washington Certification #: C486

West Virginia DEP Certification #: 382

West Virginia DW Certification #: 9952 C

Wisconsin Certification #: 999407970

Wyoming UST Certification via A2LA #: 2926.01

USDA Permit #: P330-19-00208

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## SAMPLE SUMMARY

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10690769001	FB-1	Water	04/24/24 09:25	04/25/24 10:50
10690769002	FD-01	Water	04/24/24 00:00	04/25/24 10:50
10690769003	IW-01	Water	04/24/24 10:15	04/25/24 10:50
10690769004	TB-01	Water	04/24/24 09:20	04/25/24 10:50

## REPORT OF LABORATORY ANALYSIS

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## SAMPLE ANALYTE COUNT

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10690769001	FB-1	EPA 8270E by SIM	GY1	20	PASI-M
		EPA 8260D	TKL	10	PASI-M
10690769002	FD-01	EPA 8270E by SIM	GY1	20	PASI-M
		EPA 8260D	TKL	10	PASI-M
10690769003	IW-01	EPA 8270E by SIM	GY1	20	PASI-M
		EPA 8260D	TKL	10	PASI-M
10690769004	TB-01	EPA 8260D	TKL	10	PASI-M

PASI-M = Pace Analytical Services - Minneapolis

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## ANALYTICAL RESULTS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

Sample: FB-1 Lab ID: 10690769001 Collected: 04/24/24 09:25 Received: 04/25/24 10:50 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8270E MSSV PAH by SIM</b>									
Analytical Method: EPA 8270E by SIM Preparation Method: EPA 3510C									
Pace Analytical Services - Minneapolis									
Acenaphthene	<0.0046	ug/L	0.040	0.0046	1	04/29/24 11:35	05/01/24 16:07	83-32-9	
Acenaphthylene	<0.0041	ug/L	0.040	0.0041	1	04/29/24 11:35	05/01/24 16:07	208-96-8	
Anthracene	<0.0073	ug/L	0.040	0.0073	1	04/29/24 11:35	05/01/24 16:07	120-12-7	
Benzo(a)anthracene	<0.0046	ug/L	0.040	0.0046	1	04/29/24 11:35	05/01/24 16:07	56-55-3	
Benzo(a)pyrene	<0.0049	ug/L	0.040	0.0049	1	04/29/24 11:35	05/01/24 16:07	50-32-8	
Benzo(b)fluoranthene	<0.0073	ug/L	0.040	0.0073	1	04/29/24 11:35	05/01/24 16:07	205-99-2	
Benzo(g,h,i)perylene	<0.0099	ug/L	0.040	0.0099	1	04/29/24 11:35	05/01/24 16:07	191-24-2	
Benzo(k)fluoranthene	<0.0080	ug/L	0.040	0.0080	1	04/29/24 11:35	05/01/24 16:07	207-08-9	
Chrysene	<0.0079	ug/L	0.040	0.0079	1	04/29/24 11:35	05/01/24 16:07	218-01-9	
Dibenz(a,h)anthracene	<0.010	ug/L	0.040	0.010	1	04/29/24 11:35	05/01/24 16:07	53-70-3	
Fluoranthene	<0.014	ug/L	0.040	0.014	1	04/29/24 11:35	05/01/24 16:07	206-44-0	
Fluorene	<0.0053	ug/L	0.040	0.0053	1	04/29/24 11:35	05/01/24 16:07	86-73-7	
Indeno(1,2,3-cd)pyrene	<0.0098	ug/L	0.040	0.0098	1	04/29/24 11:35	05/01/24 16:07	193-39-5	
1-Methylnaphthalene	<0.0054	ug/L	0.040	0.0054	1	04/29/24 11:35	05/01/24 16:07	90-12-0	
2-Methylnaphthalene	<0.0073	ug/L	0.040	0.0073	1	04/29/24 11:35	05/01/24 16:07	91-57-6	
Naphthalene	<0.015	ug/L	0.040	0.015	1	04/29/24 11:35	05/01/24 16:07	91-20-3	
Phenanthrene	<0.013	ug/L	0.040	0.013	1	04/29/24 11:35	05/01/24 16:07	85-01-8	
Pyrene	<0.013	ug/L	0.040	0.013	1	04/29/24 11:35	05/01/24 16:07	129-00-0	
<b>Surrogates</b>									
2-Fluorobiphenyl (S)	86	%.	34-125		1	04/29/24 11:35	05/01/24 16:07	321-60-8	
p-Terphenyl-d14 (S)	91	%.	38-139		1	04/29/24 11:35	05/01/24 16:07	1718-51-0	
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D									
Pace Analytical Services - Minneapolis									
Benzene	<0.21	ug/L	1.0	0.21	1		05/03/24 01:34	71-43-2	
Ethylbenzene	<0.11	ug/L	1.0	0.11	1		05/03/24 01:34	100-41-4	
Methyl-tert-butyl ether	<0.13	ug/L	1.0	0.13	1		05/03/24 01:34	1634-04-4	
Toluene	<0.21	ug/L	1.0	0.21	1		05/03/24 01:34	108-88-3	
1,2,4-Trimethylbenzene	<0.13	ug/L	1.0	0.13	1		05/03/24 01:34	95-63-6	
1,3,5-Trimethylbenzene	<0.11	ug/L	1.0	0.11	1		05/03/24 01:34	108-67-8	
Xylene (Total)	<0.42	ug/L	3.0	0.42	1		05/03/24 01:34	1330-20-7	
<b>Surrogates</b>									
1,2-Dichlorobenzene-d4 (S)	100	%.	75-125		1		05/03/24 01:34	2199-69-1	
4-Bromofluorobenzene (S)	100	%.	75-125		1		05/03/24 01:34	460-00-4	
Toluene-d8 (S)	101	%.	75-125		1		05/03/24 01:34	2037-26-5	

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

Sample: FD-01 Lab ID: 10690769002 Collected: 04/24/24 00:00 Received: 04/25/24 10:50 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8270E MSSV PAH by SIM</b>									
Analytical Method: EPA 8270E by SIM Preparation Method: EPA 3510C									
Pace Analytical Services - Minneapolis									
Acenaphthene	<0.0044	ug/L	0.038	0.0044	1	04/29/24 11:35	05/01/24 16:29	83-32-9	
Acenaphthylene	<0.0040	ug/L	0.038	0.0040	1	04/29/24 11:35	05/01/24 16:29	208-96-8	
Anthracene	<0.0071	ug/L	0.038	0.0071	1	04/29/24 11:35	05/01/24 16:29	120-12-7	
Benzo(a)anthracene	<0.0044	ug/L	0.038	0.0044	1	04/29/24 11:35	05/01/24 16:29	56-55-3	
Benzo(a)pyrene	<0.0047	ug/L	0.038	0.0047	1	04/29/24 11:35	05/01/24 16:29	50-32-8	
Benzo(b)fluoranthene	<0.0071	ug/L	0.038	0.0071	1	04/29/24 11:35	05/01/24 16:29	205-99-2	
Benzo(g,h,i)perylene	<0.0096	ug/L	0.038	0.0096	1	04/29/24 11:35	05/01/24 16:29	191-24-2	
Benzo(k)fluoranthene	<0.0078	ug/L	0.038	0.0078	1	04/29/24 11:35	05/01/24 16:29	207-08-9	
Chrysene	<0.0077	ug/L	0.038	0.0077	1	04/29/24 11:35	05/01/24 16:29	218-01-9	
Dibenz(a,h)anthracene	<0.010	ug/L	0.038	0.010	1	04/29/24 11:35	05/01/24 16:29	53-70-3	
Fluoranthene	<0.013	ug/L	0.038	0.013	1	04/29/24 11:35	05/01/24 16:29	206-44-0	
Fluorene	<0.0051	ug/L	0.038	0.0051	1	04/29/24 11:35	05/01/24 16:29	86-73-7	
Indeno(1,2,3-cd)pyrene	<0.0095	ug/L	0.038	0.0095	1	04/29/24 11:35	05/01/24 16:29	193-39-5	
1-Methylnaphthalene	<0.0052	ug/L	0.038	0.0052	1	04/29/24 11:35	05/01/24 16:29	90-12-0	
2-Methylnaphthalene	<0.0071	ug/L	0.038	0.0071	1	04/29/24 11:35	05/01/24 16:29	91-57-6	
Naphthalene	<0.015	ug/L	0.038	0.015	1	04/29/24 11:35	05/01/24 16:29	91-20-3	
Phenanthrene	<0.013	ug/L	0.038	0.013	1	04/29/24 11:35	05/01/24 16:29	85-01-8	
Pyrene	<0.012	ug/L	0.038	0.012	1	04/29/24 11:35	05/01/24 16:29	129-00-0	
<b>Surrogates</b>									
2-Fluorobiphenyl (S)	71	%.	34-125		1	04/29/24 11:35	05/01/24 16:29	321-60-8	
p-Terphenyl-d14 (S)	81	%.	38-139		1	04/29/24 11:35	05/01/24 16:29	1718-51-0	
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D									
Pace Analytical Services - Minneapolis									
Benzene	<0.21	ug/L	1.0	0.21	1		05/03/24 04:49	71-43-2	
Ethylbenzene	0.12J	ug/L	1.0	0.11	1		05/03/24 04:49	100-41-4	
Methyl-tert-butyl ether	<0.13	ug/L	1.0	0.13	1		05/03/24 04:49	1634-04-4	
Toluene	<0.21	ug/L	1.0	0.21	1		05/03/24 04:49	108-88-3	
1,2,4-Trimethylbenzene	0.20J	ug/L	1.0	0.13	1		05/03/24 04:49	95-63-6	
1,3,5-Trimethylbenzene	<0.11	ug/L	1.0	0.11	1		05/03/24 04:49	108-67-8	
Xylene (Total)	<0.42	ug/L	3.0	0.42	1		05/03/24 04:49	1330-20-7	
<b>Surrogates</b>									
1,2-Dichlorobenzene-d4 (S)	100	%.	75-125		1		05/03/24 04:49	2199-69-1	
4-Bromofluorobenzene (S)	99	%.	75-125		1		05/03/24 04:49	460-00-4	
Toluene-d8 (S)	102	%.	75-125		1		05/03/24 04:49	2037-26-5	

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## ANALYTICAL RESULTS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

Sample: IW-01 Lab ID: 10690769003 Collected: 04/24/24 10:15 Received: 04/25/24 10:50 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8270E MSSV PAH by SIM</b>									
Analytical Method: EPA 8270E by SIM Preparation Method: EPA 3510C									
Pace Analytical Services - Minneapolis									
Acenaphthene	<0.0045	ug/L	0.039	0.0045	1	04/29/24 11:35	05/01/24 16:51	83-32-9	
Acenaphthylene	<0.0041	ug/L	0.039	0.0041	1	04/29/24 11:35	05/01/24 16:51	208-96-8	
Anthracene	<0.0071	ug/L	0.039	0.0071	1	04/29/24 11:35	05/01/24 16:51	120-12-7	
Benzo(a)anthracene	<0.0045	ug/L	0.039	0.0045	1	04/29/24 11:35	05/01/24 16:51	56-55-3	
Benzo(a)pyrene	<0.0048	ug/L	0.039	0.0048	1	04/29/24 11:35	05/01/24 16:51	50-32-8	
Benzo(b)fluoranthene	<0.0072	ug/L	0.039	0.0072	1	04/29/24 11:35	05/01/24 16:51	205-99-2	
Benzo(g,h,i)perylene	<0.0097	ug/L	0.039	0.0097	1	04/29/24 11:35	05/01/24 16:51	191-24-2	
Benzo(k)fluoranthene	<0.0079	ug/L	0.039	0.0079	1	04/29/24 11:35	05/01/24 16:51	207-08-9	
Chrysene	<0.0077	ug/L	0.039	0.0077	1	04/29/24 11:35	05/01/24 16:51	218-01-9	
Dibenz(a,h)anthracene	<0.010	ug/L	0.039	0.010	1	04/29/24 11:35	05/01/24 16:51	53-70-3	
Fluoranthene	<0.014	ug/L	0.039	0.014	1	04/29/24 11:35	05/01/24 16:51	206-44-0	
Fluorene	<0.0052	ug/L	0.039	0.0052	1	04/29/24 11:35	05/01/24 16:51	86-73-7	
Indeno(1,2,3-cd)pyrene	<0.0096	ug/L	0.039	0.0096	1	04/29/24 11:35	05/01/24 16:51	193-39-5	
1-Methylnaphthalene	<0.0053	ug/L	0.039	0.0053	1	04/29/24 11:35	05/01/24 16:51	90-12-0	
2-Methylnaphthalene	<0.0072	ug/L	0.039	0.0072	1	04/29/24 11:35	05/01/24 16:51	91-57-6	
Naphthalene	<0.015	ug/L	0.039	0.015	1	04/29/24 11:35	05/01/24 16:51	91-20-3	
Phenanthrene	<0.013	ug/L	0.039	0.013	1	04/29/24 11:35	05/01/24 16:51	85-01-8	
Pyrene	<0.012	ug/L	0.039	0.012	1	04/29/24 11:35	05/01/24 16:51	129-00-0	
<b>Surrogates</b>									
2-Fluorobiphenyl (S)	74	%.	34-125		1	04/29/24 11:35	05/01/24 16:51	321-60-8	
p-Terphenyl-d14 (S)	78	%.	38-139		1	04/29/24 11:35	05/01/24 16:51	1718-51-0	
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D									
Pace Analytical Services - Minneapolis									
Benzene	<0.21	ug/L	1.0	0.21	1		05/03/24 05:06	71-43-2	
Ethylbenzene	<0.11	ug/L	1.0	0.11	1		05/03/24 05:06	100-41-4	
Methyl-tert-butyl ether	<0.13	ug/L	1.0	0.13	1		05/03/24 05:06	1634-04-4	
Toluene	<0.21	ug/L	1.0	0.21	1		05/03/24 05:06	108-88-3	
1,2,4-Trimethylbenzene	<0.13	ug/L	1.0	0.13	1		05/03/24 05:06	95-63-6	
1,3,5-Trimethylbenzene	<0.11	ug/L	1.0	0.11	1		05/03/24 05:06	108-67-8	
Xylene (Total)	<0.42	ug/L	3.0	0.42	1		05/03/24 05:06	1330-20-7	
<b>Surrogates</b>									
1,2-Dichlorobenzene-d4 (S)	102	%.	75-125		1		05/03/24 05:06	2199-69-1	
4-Bromofluorobenzene (S)	101	%.	75-125		1		05/03/24 05:06	460-00-4	
Toluene-d8 (S)	101	%.	75-125		1		05/03/24 05:06	2037-26-5	

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## ANALYTICAL RESULTS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

Sample: TB-01 Lab ID: 10690769004 Collected: 04/24/24 09:20 Received: 04/25/24 10:50 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D									
Pace Analytical Services - Minneapolis									
Benzene	<0.21	ug/L	1.0	0.21	1		05/03/24 00:45	71-43-2	
Ethylbenzene	<0.11	ug/L	1.0	0.11	1		05/03/24 00:45	100-41-4	
Methyl-tert-butyl ether	<0.13	ug/L	1.0	0.13	1		05/03/24 00:45	1634-04-4	
Toluene	<0.21	ug/L	1.0	0.21	1		05/03/24 00:45	108-88-3	
1,2,4-Trimethylbenzene	<0.13	ug/L	1.0	0.13	1		05/03/24 00:45	95-63-6	
1,3,5-Trimethylbenzene	<0.11	ug/L	1.0	0.11	1		05/03/24 00:45	108-67-8	
Xylene (Total)	<0.42	ug/L	3.0	0.42	1		05/03/24 00:45	1330-20-7	
<b>Surrogates</b>									
1,2-Dichlorobenzene-d4 (S)	100	%.	75-125		1		05/03/24 00:45	2199-69-1	
4-Bromofluorobenzene (S)	99	%.	75-125		1		05/03/24 00:45	460-00-4	
Toluene-d8 (S)	100	%.	75-125		1		05/03/24 00:45	2037-26-5	

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

QC Batch:	943833	Analysis Method:	EPA 8260D
QC Batch Method:	EPA 8260D	Analysis Description:	8260D MSV UST-WATER
		Laboratory:	Pace Analytical Services - Minneapolis

Associated Lab Samples: 10690769001, 10690769002, 10690769003, 10690769004

METHOD BLANK: 4939872 Matrix: Water

Associated Lab Samples: 10690769001, 10690769002, 10690769003, 10690769004

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
1,2,4-Trimethylbenzene	ug/L	<0.13	1.0	05/03/24 00:29	
1,3,5-Trimethylbenzene	ug/L	<0.11	1.0	05/03/24 00:29	
Benzene	ug/L	<0.21	1.0	05/03/24 00:29	
Ethylbenzene	ug/L	<0.11	1.0	05/03/24 00:29	
Methyl-tert-butyl ether	ug/L	<0.13	1.0	05/03/24 00:29	
Toluene	ug/L	<0.21	1.0	05/03/24 00:29	
Xylene (Total)	ug/L	<0.42	3.0	05/03/24 00:29	
1,2-Dichlorobenzene-d4 (S)	%	101	75-125	05/03/24 00:29	
4-Bromofluorobenzene (S)	%	100	75-125	05/03/24 00:29	
Toluene-d8 (S)	%	100	75-125	05/03/24 00:29	

LABORATORY CONTROL SAMPLE &amp; LCSD: 4939873 4939874

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
1,2,4-Trimethylbenzene	ug/L	20	19.7	19.5	99	98	75-125	1	20	
1,3,5-Trimethylbenzene	ug/L	20	19.9	19.6	99	98	75-125	1	20	
Benzene	ug/L	20	18.3	17.8	92	89	75-125	3	20	
Ethylbenzene	ug/L	20	19.1	18.6	96	93	75-125	3	20	
Methyl-tert-butyl ether	ug/L	20	19.4	19.3	97	97	75-125	0	20	
Toluene	ug/L	20	17.5	17.3	87	87	75-125	1	20	
Xylene (Total)	ug/L	60	57.6	56.4	96	94	75-125	2	20	
1,2-Dichlorobenzene-d4 (S)	%				100	101	75-125			
4-Bromofluorobenzene (S)	%				98	99	75-125			
Toluene-d8 (S)	%				97	97	75-125			

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## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

QC Batch: 943034

Analysis Method: EPA 8270E by SIM

QC Batch Method: EPA 3510C

Analysis Description: 8270E Water PAH by SIM MSSV

Laboratory:

Pace Analytical Services - Minneapolis

Associated Lab Samples: 10690769001, 10690769002, 10690769003

METHOD BLANK: 4936357

Matrix: Water

Associated Lab Samples: 10690769001, 10690769002, 10690769003

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
1-Methylnaphthalene	ug/L	<0.0054	0.040	05/01/24 11:17	
2-Methylnaphthalene	ug/L	<0.0074	0.040	05/01/24 11:17	
Acenaphthene	ug/L	<0.0046	0.040	05/01/24 11:17	
Acenaphthylene	ug/L	<0.0042	0.040	05/01/24 11:17	
Anthracene	ug/L	<0.0073	0.040	05/01/24 11:17	
Benzo(a)anthracene	ug/L	<0.0046	0.040	05/01/24 11:17	
Benzo(a)pyrene	ug/L	<0.0049	0.040	05/01/24 11:17	
Benzo(b)fluoranthene	ug/L	<0.0074	0.040	05/01/24 11:17	
Benzo(g,h,i)perylene	ug/L	<0.010	0.040	05/01/24 11:17	
Benzo(k)fluoranthene	ug/L	<0.0081	0.040	05/01/24 11:17	
Chrysene	ug/L	<0.0080	0.040	05/01/24 11:17	
Dibenz(a,h)anthracene	ug/L	<0.010	0.040	05/01/24 11:17	
Fluoranthene	ug/L	<0.014	0.040	05/01/24 11:17	
Fluorene	ug/L	<0.0053	0.040	05/01/24 11:17	
Indeno(1,2,3-cd)pyrene	ug/L	<0.0099	0.040	05/01/24 11:17	
Naphthalene	ug/L	<0.015	0.040	05/01/24 11:17	
Phenanthrene	ug/L	<0.013	0.040	05/01/24 11:17	
Pyrene	ug/L	<0.013	0.040	05/01/24 11:17	
2-Fluorobiphenyl (S)	%	74	34-125	05/01/24 11:17	
p-Terphenyl-d14 (S)	%	86	38-139	05/01/24 11:17	

LABORATORY CONTROL SAMPLE &amp; LCSD: 4936358

4936359

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
1-Methylnaphthalene	ug/L	1	0.81	0.73	81	73	42-125	10	20	
2-Methylnaphthalene	ug/L	1	0.89	0.85	89	85	39-125	4	20	
Acenaphthene	ug/L	1	0.85	0.84	85	84	45-125	2	20	
Acenaphthylene	ug/L	1	0.91	0.87	91	87	47-125	4	20	
Anthracene	ug/L	1	0.83	0.80	83	80	50-125	3	20	
Benzo(a)anthracene	ug/L	1	0.84	0.88	84	88	60-125	4	20	
Benzo(a)pyrene	ug/L	1	0.83	0.87	83	87	60-125	4	20	
Benzo(b)fluoranthene	ug/L	1	0.87	0.92	87	92	54-125	5	20	
Benzo(g,h,i)perylene	ug/L	1	0.85	0.87	85	87	46-125	2	20	
Benzo(k)fluoranthene	ug/L	1	0.80	0.80	80	80	57-125	0	20	
Chrysene	ug/L	1	0.81	0.81	81	81	60-125	0	20	
Dibenz(a,h)anthracene	ug/L	1	0.87	0.87	87	87	32-125	0	20	
Fluoranthene	ug/L	1	0.84	0.86	84	86	55-125	2	20	
Fluorene	ug/L	1	0.88	0.85	88	85	50-125	3	20	
Indeno(1,2,3-cd)pyrene	ug/L	1	0.90	0.86	90	86	56-125	5	20	

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## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

LABORATORY CONTROL SAMPLE & LCSD: 4936358			4936359							
Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
Naphthalene	ug/L	1	0.87	0.80	87	80	43-125	8	20	
Phenanthrene	ug/L	1	0.87	0.86	87	86	56-125	0	20	
Pyrene	ug/L	1	0.84	0.94	84	94	59-125	11	20	
2-Fluorobiphenyl (S)	%.				85	81	34-125			
p-Terphenyl-d14 (S)	%.				83	91	38-139			

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## QUALIFIERS

Project: 49161497.04 100 101 SRC

Pace Project No.: 10690769

### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above LOD.

J - The reported result is an estimated value.

LOD - Limit of Detection adjusted for dilution factor, percent moisture, initial weight and final volume.

LOQ - Limit of Quantitation adjusted for dilution factor, percent moisture, initial weight and final volume.

DL - Adjusted Method Detection Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Analyte was not detected and is reported as less than the LOD or as defined by the customer.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### BATCH QUALIFIERS

Batch: 943484

[M5] A matrix spike/matrix spike duplicate was not performed for this batch due to insufficient sample volume.

Batch: 943833

[M5] A matrix spike/matrix spike duplicate was not performed for this batch due to insufficient sample volume.

## REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161497.04 100 101 SRC  
Pace Project No.: 10690769

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10690769001	FB-1	EPA 3510C	943034	EPA 8270E by SIM	943484
10690769002	FD-01	EPA 3510C	943034	EPA 8270E by SIM	943484
10690769003	IW-01	EPA 3510C	943034	EPA 8270E by SIM	943484
10690769001	FB-1	EPA 8260D	943833		
10690769002	FD-01	EPA 8260D	943833		
10690769003	IW-01	EPA 8260D	943833		
10690769004	TB-01	EPA 8260D	943833		

REPORT OF LABORATORY ANALYSIS

EZ 3083821



# Barr Engineering Co. Chain of Custody

## Sample Origination State

☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ NV ☐ TX ☐ UT ☒ WI ☐ WY ☐ Other: \_\_\_\_\_

REPORT TO	INVOICE TO
Company: <u>Barr Engineering</u>	Company: <u>Barr Engineering</u>
Address: <u>325 S. Lake Ave</u>	Address: _____
Address: <u>Duluth, MN 55802</u>	Address: _____
Name: <u>Lynette Carney</u>	Name: <u>SAME</u>
email: <u>LCarney@barr.com</u>	email: _____
Copy to: <u>BarrDM@barr.com</u>	P.O. _____
Project Name: <u>SRC</u>	Barr Project No: <u>49161497.04 100101</u>

Location	Sample Depth			Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code
	Start	Stop	Unit (m./ft. or in.)			
1. <u>FB-01</u>	—	—		<u>04/24/2024</u>	<u>09:25</u>	<u>WQ</u>
2. <u>FD-01</u>	—	—		<u>04/24/2024</u>	—	<u>WQ</u>
3. <u>IW-1</u>	—	—		<u>04/24/2024</u>	<u>10:15</u>	<u>GW</u>
4. <u>TB-01</u>	—	—		<u>04/24/2024</u>	<u>09:20</u>	<u>WQ</u>
5.						
6.						
7.						
8.						
9.						
10.						

Analysis Requested		COC Number: <b>Nº 597208</b>	
Water	Soil	COC <u>1</u> of <u>1</u>	
PAHs		<b>Matrix Code:</b> GW = Groundwater SW = Surface Water DW = Drinking Water PW = Pore Water WW = Waste Water WQ = TB, FB, EB, etc. W = Unspecified S = Soil/Solid SD = Sediment SQ = MeOH blank OTH = Other (Oil, etc.)	
		<b>Preservative Code:</b> A = None B = HCl C = HNO <sub>3</sub> D = H <sub>2</sub> SO <sub>4</sub> E = NaOH F = MeOH G = NaHSO <sub>4</sub> H = Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> I = Ascorbic Acid J = Zn Acetate K = Other	
		Preservative Code	
		Field Filtered Y/N	

**WO#: 10690769**



10690769

BARR USE ONLY		Relinquished by: <u>J. Pace</u>		On Ice? <input checked="" type="checkbox"/> N	Date <u>04/24/2024</u>	Time <u>13:00</u>	Received by: <u>Shelagh Pace</u>	Date <u>4/24/24</u>	Time <u>13:00</u>
Sampled by: <u>JSP</u>		Relinquished by: <u>Shelagh Pace</u>		On Ice? <input checked="" type="checkbox"/> N	Date <u>4/24/24</u>	Time <u>13:00</u>	Received by: <u>Shelagh Pace</u>	Date <u>4/24/24</u>	Time <u>10:50</u>
Barr Proj. Manager: <u>X ZML</u>		Samples Shipped VIA: <input type="checkbox"/> Ground Courier <input type="checkbox"/> Air Carrier <input checked="" type="checkbox"/> Sampler		Air Bill Number: <u>40</u>		Requested Due Date: <input checked="" type="checkbox"/> Standard Turn Around Time <input type="checkbox"/> Rush			
Barr DQ Manager: <u>TAO</u>		Lab Name: <u>Pace</u>		Lab Location: <u>Duluth, MN</u>		Lab WO: _____		Temperature on Receipt (°C): <u>25</u>	
								Custody Seal Intact? <input checked="" type="checkbox"/> Y <input type="checkbox"/> N <input type="checkbox"/> None	

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Scan and email: a copy to BarrDM@barr.com for tracking and filing procedures

# ENV-FRM-MIN4-0150 v16 Sample Condition Upon Receipt

CLIENT NAME: Barr Engineering PROJECT #:

**WO# : 10690769**

COURIER: ☐ Client ☐ Commercial ☐ FedEx ☒ Pace  
☐ Speedee ☐ UPS ☐ USPS

PM: MKH

Due Date: 05/09/24

CLIENT: BARR

TRACKING NUMBER: ☐ See Exceptions form ENV-FRM-MIN4-0142

Custody Seal on Coole/Box Present: ☒ YES ☐ NO Seals Intact: ☒ YES ☐ NO Biological Tissue Frozen: ☐ YES ☐ NO ☒ N/A

Packing Material: ☐ Bubble Bags ☐ Bubble Wrap ☐ None ☐ Other Temp Blank: ☐ YES ☐ NO Type of Ice: ☐ Blue ☐ Dry ☐ Wet

Thermometer: ☒ T1 (0461) ☐ T2 (0436) ☐ T3 (0459) ☐ T4 (0402) ☐ T5 (0178) ☐ T6 (0235)  
☒ T7 (0042) ☐ T8 (0775) ☐ T9 (0727) ☐ 01339252 (1710) ☐ Melted ☐ None

Did Samples Originate in West Virginia: ☐ YES ☒ NO

Were All Container Temps taken: ☐ YES ☐ NO ☒ N/A

Correction Factor: -0.4 Cooler Temp Read w/Temp Blank: 2.9 °C  
Cooler Temp Corrected w/Temp Blank: 2.5 °C

Average Corrected Temp (no Temp Blank Only): \_\_\_\_\_ °C

NOTE: Temp should be above freezing to 6°C.

☐ See Exceptions Form ENV-FRM-MIN4-0142 ☐ 1 Container

USDA Regulated Soil: ☐ N/A ☒ Water Sample/Other (describe):

Initials & Date of Person Examining Contents: SMC 4/25/24

Did Samples originate from one of the following states (check maps) – AL, AR, AZ, CA, FL, GA, ID, LA, MS, NC, NM, NY, OK, OR, SC, TN, TX, or VA: ☐ YES ☐ NO

Did samples originate from a foreign source (international, including Hawaii and Puerto Rico): ☐ YES ☐ NO

NOTE: If YES to either question, fill out a Regulated Soil Checklist (ENV-FRM-MIN4-0154) and include with SCUR/COC paperwork.

LOCATION (check one): <input type="checkbox"/> DULUTH <input checked="" type="checkbox"/> MINNEAPOLIS <input type="checkbox"/> VIRGINIA	YES	NO	N/A	COMMENT(S)								
Chain of Custody Present and Filled Out?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1.								
Chain of Custody Relinquished?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2.								
Sampler Name and/or Signature on COC?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3.								
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. If Fecal: <input type="checkbox"/> <8 hrs <input type="checkbox"/> >8 hr, <24 hr <input type="checkbox"/> No								
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	5. <input type="checkbox"/> BOD / cBOD <input type="checkbox"/> Fecal coliform <input type="checkbox"/> Hex Chrom <input type="checkbox"/> HPC <input type="checkbox"/> Nitrate <input type="checkbox"/> Nitrite <input type="checkbox"/> Ortho Phos <input type="checkbox"/> Total coliform/E. coli <input type="checkbox"/> Other: _____								
Rush Turn Around Time Requested?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	6.								
Sufficient Sample Volume?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7.								
Correct Containers Used?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8.								
– Pace Containers Used?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>									
Containers Intact?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9.								
Field Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	10. Is sediment visible in the dissolved container: <input type="checkbox"/> YES <input type="checkbox"/> NO								
Is sufficient information available to reconcile the samples to the COC? NOTE: If ID/Date/Time don't match fill out section 11. Matrix: <input type="checkbox"/> Oil <input type="checkbox"/> Soil <input checked="" type="checkbox"/> Water <input type="checkbox"/> Other	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	11. If NO, write ID/Date/Time of container below: <input type="checkbox"/> See Exceptions form ENV-FRM-MIN4-0142								
All containers needing acid/base preservation have been checked? All containers needing preservation are found to be in compliance with EPA recommendation? (HNO <sub>3</sub> , H <sub>2</sub> SO <sub>4</sub> , < 2 pH, NaOH > 9 Sulfide, NaOH > 10 Cyanide) Exceptions: VOA, Coliform, TOC/DOC, Oil & Grease, DRO/8015 (water) and Dioxins/PFAS NOTE: If adding preservation to the container, verify with the PM first. Clients may require adding preservative to the field and equipment blanks when this occurs.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	12. Sample #: <input type="checkbox"/> HNO <sub>3</sub> <input type="checkbox"/> H <sub>2</sub> SO <sub>4</sub> <input type="checkbox"/> NaOH <input type="checkbox"/> Zinc Acetate Positive for Residual Chlorine: <input type="checkbox"/> YES <input type="checkbox"/> NO <p style="text-align: center;"><b>pH Paper Lot #</b></p> <table border="1" style="width: 100%;"> <tr> <th>Residual Chlorine</th> <th>0-6 Roll</th> <th>0-6 Strip</th> <th>0-14 Strip</th> </tr> <tr> <td> </td> <td> </td> <td> </td> <td> </td> </tr> </table> <input type="checkbox"/> See Exceptions form ENV-FRM-MIN4-0142	Residual Chlorine	0-6 Roll	0-6 Strip	0-14 Strip				
Residual Chlorine	0-6 Roll	0-6 Strip	0-14 Strip									
Headspace in Methyl Mercury Container?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	13.								
Extra labels present on soil VOA or WIDRO containers?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	14.								
Headspace in VOA Vials (greater than 6mm)?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> See Exceptions form ENV-FRM-MIN4-0142								
Trip Blanks Present?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15.								
Trip Blank Custody Seals Present?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Pace Trip Blank Lot # (if purchased): <u>020524-304R</u>								

CLIENT NOTIFICATION / RESOLUTION

FIELD DATA REQUIRED: ☐ YES ☐ NO

Person Contacted: \_\_\_\_\_ Date & Time: \_\_\_\_\_

Comments / Resolution: \_\_\_\_\_

Project Manager Review: [Signature]

Date: 4/25/24

NOTE: When there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEQ Certification Office (i.e., out of hold, incorrect preservative, out of temp, incorrect containers).

Labeled By: SMC

Line: 3





November 21, 2023

Terri Olson  
Barr Engineering Company  
4300 MarketPointe Drive  
Suite 200  
Minneapolis, MN 55435

RE: Project: 49161497.03 SRC Site Investiga  
Pace Project No.: 10676081

Dear Terri Olson:

Enclosed are the analytical results for sample(s) received by the laboratory on November 15, 2023. The results relate only to the samples included in this report. Results reported herein conform to the applicable TNI/NELAC Standards and the laboratory's Quality Manual, where applicable, unless otherwise noted in the body of the report.

The test results provided in this final report were generated by each of the following laboratories within the Pace Network:

- Pace Analytical Services - Minneapolis

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

A handwritten signature in blue ink, appearing to read "Martha Hansen".

Martha Hansen  
martha.hansen@pacelabs.com  
(612)607-6451  
Project Manager

Enclosures

cc: Barr DM, Barr Engineering  
Accounts Payable, Barr Engineering



## REPORT OF LABORATORY ANALYSIS

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## CERTIFICATIONS

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

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### Pace Analytical Services, LLC - Minneapolis MN

1700 Elm Street SE, Minneapolis, MN 55414

A2LA Certification #: 2926.01

Alabama Certification #: 40770

Alaska Contaminated Sites Certification #: 17-009

Alaska DW Certification #: MN00064

Arizona Certification #: AZ0014

Arkansas DW Certification #: MN00064

Arkansas WW Certification #: 88-0680

California Certification #: 2929

Colorado Certification #: MN00064

Connecticut Certification #: PH-0256

EPA Region 8 Tribal Water Systems+Wyoming DW  
Certification #: via MN 027-053-137

Florida Certification #: E87605

Georgia Certification #: 959

GMP+ Certification #: GMP050884

Hawaii Certification #: MN00064

Idaho Certification #: MN00064

Illinois Certification #: 200011

Indiana Certification #: C-MN-01

Iowa Certification #: 368

Kansas Certification #: E-10167

Kentucky DW Certification #: 90062

Kentucky WW Certification #: 90062

Louisiana DEQ Certification #: AI-03086

Louisiana DW Certification #: MN00064

Maine Certification #: MN00064

Maryland Certification #: 322

Michigan Certification #: 9909

Minnesota Certification #: 027-053-137

Minnesota Dept of Ag Approval: via MN 027-053-137

Minnesota Petrofund Registration #: 1240

Mississippi Certification #: MN00064

Missouri Certification #: 10100

Montana Certification #: CERT0092

Nebraska Certification #: NE-OS-18-06

Nevada Certification #: MN00064

New Hampshire Certification #: 2081

New Jersey Certification #: MN002

New York Certification #: 11647

North Carolina DW Certification #: 27700

North Carolina WW Certification #: 530

North Dakota Certification (A2LA) #: R-036

North Dakota Certification (MN) #: R-036

Ohio DW Certification #: 41244

Ohio VAP Certification (1700) #: CL101

Oklahoma Certification #: 9507

Oregon Primary Certification #: MN300001

Oregon Secondary Certification #: MN200001

Pennsylvania Certification #: 68-00563

Puerto Rico Certification #: MN00064

South Carolina Certification #: 74003001

Tennessee Certification #: TN02818

Texas Certification #: T104704192

Utah Certification #: MN00064

Vermont Certification #: VT-027053137

Virginia Certification #: 460163

Washington Certification #: C486

West Virginia DEP Certification #: 382

West Virginia DW Certification #: 9952 C

Wisconsin Certification #: 999407970

Wyoming UST Certification #: via A2LA 2926.01

USDA Permit #: P330-19-00208

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## REPORT OF LABORATORY ANALYSIS

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## SAMPLE SUMMARY

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10676081001	IW-1_1.5-2.0	Solid	11/10/23 10:30	11/15/23 11:10
10676081002	Trip Blank	Solid	11/10/23 00:00	11/15/23 11:10

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## SAMPLE ANALYTE COUNT

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10676081001	IW-1_1.5-2.0	ASTM D2974	JDL	1	PASI-M
		EPA 8270E by SIM	JLR	20	PASI-M
		EPA 8260D	SB2	10	PASI-M
10676081002	Trip Blank	EPA 8260D	SB2	10	PASI-M

PASI-M = Pace Analytical Services - Minneapolis

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## ANALYTICAL RESULTS

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

Sample: IW-1\_1.5-2.0 Lab ID: 10676081001 Collected: 11/10/23 10:30 Received: 11/15/23 11:10 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>Dry Weight / %M by ASTM D2974</b>									
Analytical Method: ASTM D2974									
Pace Analytical Services - Minneapolis									
Percent Moisture	24.7	%	0.10	0.10	1		11/20/23 14:11		N2
<b>8270E MSSV PAH by SIM</b>									
Analytical Method: EPA 8270E by SIM Preparation Method: EPA 3546									
Pace Analytical Services - Minneapolis									
Acenaphthene	<2.7	ug/kg	13.2	2.7	1	11/15/23 23:13	11/20/23 23:10	83-32-9	
Acenaphthylene	2.3J	ug/kg	13.2	1.4	1	11/15/23 23:13	11/20/23 23:10	208-96-8	
Anthracene	<1.2	ug/kg	13.2	1.2	1	11/15/23 23:13	11/20/23 23:10	120-12-7	
Benzo(a)anthracene	9.2J	ug/kg	13.2	2.2	1	11/15/23 23:13	11/20/23 23:10	56-55-3	
Benzo(a)pyrene	9.5J	ug/kg	13.2	1.5	1	11/15/23 23:13	11/20/23 23:10	50-32-8	
Benzo(b)fluoranthene	14.8	ug/kg	13.2	1.3	1	11/15/23 23:13	11/20/23 23:10	205-99-2	
Benzo(g,h,i)perylene	12.2J	ug/kg	13.2	2.5	1	11/15/23 23:13	11/20/23 23:10	191-24-2	
Benzo(k)fluoranthene	5.4J	ug/kg	13.2	1.3	1	11/15/23 23:13	11/20/23 23:10	207-08-9	
Chrysene	17.5	ug/kg	13.2	1.3	1	11/15/23 23:13	11/20/23 23:10	218-01-9	
Dibenz(a,h)anthracene	<1.6	ug/kg	13.2	1.6	1	11/15/23 23:13	11/20/23 23:10	53-70-3	
Fluoranthene	21.4	ug/kg	13.2	0.95	1	11/15/23 23:13	11/20/23 23:10	206-44-0	
Fluorene	5.8J	ug/kg	13.2	1.5	1	11/15/23 23:13	11/20/23 23:10	86-73-7	
Indeno(1,2,3-cd)pyrene	8.6J	ug/kg	13.2	1.1	1	11/15/23 23:13	11/20/23 23:10	193-39-5	
1-Methylnaphthalene	12.6J	ug/kg	13.2	3.1	1	11/15/23 23:13	11/20/23 23:10	90-12-0	
2-Methylnaphthalene	16.4	ug/kg	13.2	2.7	1	11/15/23 23:13	11/20/23 23:10	91-57-6	
Naphthalene	11.9J	ug/kg	13.2	1.4	1	11/15/23 23:13	11/20/23 23:10	91-20-3	
Phenanthrene	21.7	ug/kg	13.2	1.0	1	11/15/23 23:13	11/20/23 23:10	85-01-8	
Pyrene	16.6	ug/kg	13.2	2.0	1	11/15/23 23:13	11/20/23 23:10	129-00-0	
<b>Surrogates</b>									
2-Fluorobiphenyl (S)	83	%	54-125		1	11/15/23 23:13	11/20/23 23:10	321-60-8	
p-Terphenyl-d14 (S)	89	%	60-125		1	11/15/23 23:13	11/20/23 23:10	1718-51-0	
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D Preparation Method: EPA 5035/5030B									
Pace Analytical Services - Minneapolis									
Benzene	<10.5	ug/kg	31.1	10.5	1	11/20/23 11:27	11/21/23 02:58	71-43-2	
Ethylbenzene	<26.1	ug/kg	77.8	26.1	1	11/20/23 11:27	11/21/23 02:58	100-41-4	
Methyl-tert-butyl ether	<22.7	ug/kg	77.8	22.7	1	11/20/23 11:27	11/21/23 02:58	1634-04-4	
Toluene	<18.1	ug/kg	77.8	18.1	1	11/20/23 11:27	11/21/23 02:58	108-88-3	
1,2,4-Trimethylbenzene	<22.6	ug/kg	77.8	22.6	1	11/20/23 11:27	11/21/23 02:58	95-63-6	
1,3,5-Trimethylbenzene	<21.8	ug/kg	77.8	21.8	1	11/20/23 11:27	11/21/23 02:58	108-67-8	
Xylene (Total)	<44.2	ug/kg	233	44.2	1	11/20/23 11:27	11/21/23 02:58	1330-20-7	
<b>Surrogates</b>									
4-Bromofluorobenzene (S)	104	%	75-125		1	11/20/23 11:27	11/21/23 02:58	460-00-4	
Toluene-d8 (S)	105	%	75-125		1	11/20/23 11:27	11/21/23 02:58	2037-26-5	
1,2-Dichlorobenzene-d4 (S)	97	%	75-125		1	11/20/23 11:27	11/21/23 02:58	2199-69-1	

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

Sample: Trip Blank Lab ID: 10676081002 Collected: 11/10/23 00:00 Received: 11/15/23 11:10 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
<b>8260D MSV UST</b>									
Analytical Method: EPA 8260D Preparation Method: EPA 5035/5030B									
Pace Analytical Services - Minneapolis									
Benzene	<6.7	ug/kg	20.0	6.7	1	11/20/23 11:27	11/21/23 02:27	71-43-2	
Ethylbenzene	<16.8	ug/kg	50.0	16.8	1	11/20/23 11:27	11/21/23 02:27	100-41-4	
Methyl-tert-butyl ether	<14.6	ug/kg	50.0	14.6	1	11/20/23 11:27	11/21/23 02:27	1634-04-4	
Toluene	<11.6	ug/kg	50.0	11.6	1	11/20/23 11:27	11/21/23 02:27	108-88-3	
1,2,4-Trimethylbenzene	<14.5	ug/kg	50.0	14.5	1	11/20/23 11:27	11/21/23 02:27	95-63-6	
1,3,5-Trimethylbenzene	<14.0	ug/kg	50.0	14.0	1	11/20/23 11:27	11/21/23 02:27	108-67-8	
Xylene (Total)	<28.4	ug/kg	150	28.4	1	11/20/23 11:27	11/21/23 02:27	1330-20-7	
<b>Surrogates</b>									
4-Bromofluorobenzene (S)	108	%.	75-125		1	11/20/23 11:27	11/21/23 02:27	460-00-4	
Toluene-d8 (S)	103	%.	75-125		1	11/20/23 11:27	11/21/23 02:27	2037-26-5	
1,2-Dichlorobenzene-d4 (S)	96	%.	75-125		1	11/20/23 11:27	11/21/23 02:27	2199-69-1	

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**QUALITY CONTROL DATA**

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

QC Batch: 919339

Analysis Method: ASTM D2974

QC Batch Method: ASTM D2974

Analysis Description: Dry Weight / %M by ASTM D2974

Laboratory: Pace Analytical Services - Minneapolis

Associated Lab Samples: 10676081001

SAMPLE DUPLICATE: 4833717

Parameter	Units	10676081001 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	24.7	27.2	10	30	N2

SAMPLE DUPLICATE: 4834280

Parameter	Units	10676178010 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	2.5	2.5	1	30	N2

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## QUALITY CONTROL DATA

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

QC Batch:	919395	Analysis Method:	EPA 8260D
QC Batch Method:	EPA 5035/5030B	Analysis Description:	8260D MSV UST
		Laboratory:	Pace Analytical Services - Minneapolis

Associated Lab Samples: 10676081001, 10676081002

METHOD BLANK: 4834040 Matrix: Solid

Associated Lab Samples: 10676081001, 10676081002

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
1,2,4-Trimethylbenzene	ug/kg	<14.5	50.0	11/21/23 02:12	
1,3,5-Trimethylbenzene	ug/kg	<14.0	50.0	11/21/23 02:12	
Benzene	ug/kg	<6.7	20.0	11/21/23 02:12	
Ethylbenzene	ug/kg	<16.8	50.0	11/21/23 02:12	
Methyl-tert-butyl ether	ug/kg	<14.6	50.0	11/21/23 02:12	
Toluene	ug/kg	<11.6	50.0	11/21/23 02:12	
Xylene (Total)	ug/kg	<28.4	150	11/21/23 02:12	
1,2-Dichlorobenzene-d4 (S)	%	96	75-125	11/21/23 02:12	
4-Bromofluorobenzene (S)	%	106	75-125	11/21/23 02:12	
Toluene-d8 (S)	%	109	75-125	11/21/23 02:12	

LABORATORY CONTROL SAMPLE &amp; LCSD: 4834041

4834042

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
1,2,4-Trimethylbenzene	ug/kg	1000	1090	988	109	99	75-134	10	20	
1,3,5-Trimethylbenzene	ug/kg	1000	1100	1030	110	103	75-132	7	20	
Benzene	ug/kg	1000	1090	1030	109	103	72-125	6	20	
Ethylbenzene	ug/kg	1000	1130	1050	113	105	75-130	7	20	
Methyl-tert-butyl ether	ug/kg	1000	1130	1080	113	108	70-125	5	20	
Toluene	ug/kg	1000	1030	987	103	99	75-125	5	20	
Xylene (Total)	ug/kg	3000	3350	3120	112	104	75-126	7	20	
1,2-Dichlorobenzene-d4 (S)	%				103	103	75-125			
4-Bromofluorobenzene (S)	%				104	103	75-125			
Toluene-d8 (S)	%				96	96	75-125			

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## QUALITY CONTROL DATA

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

QC Batch:	918750	Analysis Method:	EPA 8270E by SIM
QC Batch Method:	EPA 3546	Analysis Description:	8270E Solid PAH by SIM MSSV
		Laboratory:	Pace Analytical Services - Minneapolis

Associated Lab Samples: 10676081001

METHOD BLANK: 4830588

Matrix: Solid

Associated Lab Samples: 10676081001

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
1-Methylnaphthalene	ug/kg	<2.4	10.0	11/20/23 12:00	
2-Methylnaphthalene	ug/kg	<2.0	10.0	11/20/23 12:00	
Acenaphthene	ug/kg	<2.0	10.0	11/20/23 12:00	
Acenaphthylene	ug/kg	<1.1	10.0	11/20/23 12:00	
Anthracene	ug/kg	<0.92	10.0	11/20/23 12:00	
Benzo(a)anthracene	ug/kg	<1.7	10.0	11/20/23 12:00	
Benzo(a)pyrene	ug/kg	<1.1	10.0	11/20/23 12:00	
Benzo(b)fluoranthene	ug/kg	<1.0	10.0	11/20/23 12:00	
Benzo(g,h,i)perylene	ug/kg	<1.9	10.0	11/20/23 12:00	
Benzo(k)fluoranthene	ug/kg	<1.0	10.0	11/20/23 12:00	
Chrysene	ug/kg	<1.0	10.0	11/20/23 12:00	
Dibenz(a,h)anthracene	ug/kg	<1.2	10.0	11/20/23 12:00	
Fluoranthene	ug/kg	<0.72	10.0	11/20/23 12:00	
Fluorene	ug/kg	<1.2	10.0	11/20/23 12:00	
Indeno(1,2,3-cd)pyrene	ug/kg	<0.80	10.0	11/20/23 12:00	
Naphthalene	ug/kg	<1.1	10.0	11/20/23 12:00	
Phenanthrene	ug/kg	<0.79	10.0	11/20/23 12:00	
Pyrene	ug/kg	<1.5	10.0	11/20/23 12:00	
2-Fluorobiphenyl (S)	%	81	54-125	11/20/23 12:00	
p-Terphenyl-d14 (S)	%	105	60-125	11/20/23 12:00	

LABORATORY CONTROL SAMPLE: 4830589

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1-Methylnaphthalene	ug/kg	100	83.8	84	41-125	
2-Methylnaphthalene	ug/kg	100	83.3	83	45-125	
Acenaphthene	ug/kg	100	80.3	80	56-125	
Acenaphthylene	ug/kg	100	80.8	81	54-125	
Anthracene	ug/kg	100	85.4	85	59-125	
Benzo(a)anthracene	ug/kg	100	94.2	94	55-125	
Benzo(a)pyrene	ug/kg	100	97.6	98	69-125	
Benzo(b)fluoranthene	ug/kg	100	107	107	54-125	
Benzo(g,h,i)perylene	ug/kg	100	98.5	99	63-125	
Benzo(k)fluoranthene	ug/kg	100	94.9	95	65-125	
Chrysene	ug/kg	100	96.1	96	62-125	
Dibenz(a,h)anthracene	ug/kg	100	101	101	64-125	
Fluoranthene	ug/kg	100	98.5	98	69-125	
Fluorene	ug/kg	100	84.0	84	61-125	
Indeno(1,2,3-cd)pyrene	ug/kg	100	103	103	54-125	

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## QUALITY CONTROL DATA

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

LABORATORY CONTROL SAMPLE: 4830589

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Naphthalene	ug/kg	100	81.3	81	49-125	
Phenanthrene	ug/kg	100	84.0	84	60-125	
Pyrene	ug/kg	100	91.4	91	69-125	
2-Fluorobiphenyl (S)	%			81	54-125	
p-Terphenyl-d14 (S)	%			100	60-125	

MATRIX SPIKE &amp; MATRIX SPIKE DUPLICATE: 4830590 4830591

Parameter	Units	20296465002 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
1-Methylnaphthalene	ug/kg	<0.0024 mg/kg	97.6	96.3	73.1	82.7	75	86	30-125	12	30	
2-Methylnaphthalene	ug/kg	<0.0020 mg/kg	97.6	96.3	74.0	82.2	76	85	30-150	10	30	
Acenaphthene	ug/kg	<0.0020 mg/kg	97.6	96.3	70.3	78.1	72	81	51-125	10	30	
Acenaphthylene	ug/kg	<1.1	97.6	96.3	71.2	78.0	73	81	50-125	9	30	
Anthracene	ug/kg	<0.00092 mg/kg	97.6	96.3	80.0	82.8	82	86	39-136	3	30	
Benzo(a)anthracene	ug/kg	<0.0017 mg/kg	97.6	96.3	87.7	88.9	89	92	30-131	1	30	
Benzo(a)pyrene	ug/kg	<0.0011 mg/kg	97.6	96.3	92.5	93.4	95	97	30-150	1	30	
Benzo(b)fluoranthene	ug/kg	<0.0010 mg/kg	97.6	96.3	103	95.5	105	99	30-150	7	30	
Benzo(g,h,i)perylene	ug/kg	<0.0019 mg/kg	97.6	96.3	91.7	93.0	94	97	30-146	1	30	
Benzo(k)fluoranthene	ug/kg	<0.0010 mg/kg	97.6	96.3	87.2	93.0	89	97	41-130	6	30	
Chrysene	ug/kg	<0.0010 mg/kg	97.6	96.3	89.9	92.1	92	96	30-135	2	30	
Dibenz(a,h)anthracene	ug/kg	<0.0012 mg/kg	97.6	96.3	94.8	95.9	97	100	50-129	1	30	
Fluoranthene	ug/kg	<0.00072 mg/kg	97.6	96.3	93.1	93.9	95	98	30-150	1	30	
Fluorene	ug/kg	<0.0012 mg/kg	97.6	96.3	75.3	82.3	77	85	56-125	9	30	
Indeno(1,2,3-cd)pyrene	ug/kg	<0.00080 mg/kg	97.6	96.3	95.2	96.7	98	100	30-148	2	30	
Naphthalene	ug/kg	<0.0011 mg/kg	97.6	96.3	72.2	79.9	74	83	30-125	10	30	
Phenanthrene	ug/kg	<0.00078 mg/kg	97.6	96.3	77.9	80.1	80	83	30-143	3	30	
Pyrene	ug/kg	<0.0015 mg/kg	97.6	96.3	86.8	87.1	89	90	30-150	0	30	
2-Fluorobiphenyl (S)	%						72	81	54-125			
p-Terphenyl-d14 (S)	%						95	99	60-125			

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## QUALITY CONTROL DATA

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 4830592 4830593												
		20296299024	MS Spike	MSD Spike	MS	MSD	MS	MSD	% Rec		Max	
Parameter	Units	Result	Conc.	Conc.	Result	Result	% Rec	% Rec	Limits	RPD	RPD	Qual
1-Methylnaphthalene	ug/kg	0.11	99.1	97	289	235	183	132	30-125	20	30	M1
		mg/kg										
2-Methylnaphthalene	ug/kg	0.081	99.1	97	253	193	174	116	30-150	27	30	M1
		mg/kg										
Acenaphthene	ug/kg	0.0099	99.1	97	101	98.5	91	91	51-125	2	30	
		mg/kg										
Acenaphthylene	ug/kg	3.5J	99.1	97	88.2	84.7	85	84	50-125	4	30	
Anthracene	ug/kg	0.0095J	99.1	97	112	102	104	96	39-136	9	30	
		mg/kg										
Benzo(a)anthracene	ug/kg	0.0028J	99.1	97	101	98.6	99	99	30-131	2	30	
		mg/kg										
Benzo(a)pyrene	ug/kg	0.0034J	99.1	97	95.3	93.2	93	93	30-150	2	30	
		mg/kg										
Benzo(b)fluoranthene	ug/kg	0.016	99.1	97	113	111	98	98	30-150	2	30	
		mg/kg										
Benzo(g,h,i)perylene	ug/kg	0.0094J	99.1	97	109	105	100	99	30-146	3	30	
		mg/kg										
Benzo(k)fluoranthene	ug/kg	0.0011J	99.1	97	93.0	89.6	93	91	41-130	4	30	
		mg/kg										
Chrysene	ug/kg	0.055	99.1	97	153	132	99	79	30-135	15	30	
		mg/kg										
Dibenz(a,h)anthracene	ug/kg	<0.0011	99.1	97	100	100	101	103	50-129	0	30	
		mg/kg										
Fluoranthene	ug/kg	0.014	99.1	97	119	109	106	98	30-150	9	30	
		mg/kg										
Fluorene	ug/kg	0.24	99.1	97	689	410	452	174	56-125	51	30	M1,R1
		mg/kg										
Indeno(1,2,3-cd)pyrene	ug/kg	0.0024J	99.1	97	103	103	101	104	30-148	0	30	
		mg/kg										
Naphthalene	ug/kg	0.014	99.1	97	129	129	116	119	30-125	0	30	
		mg/kg										
Phenanthrene	ug/kg	0.10	99.1	97	392	231	292	132	30-143	52	30	M1,R1
		mg/kg										
Pyrene	ug/kg	0.020	99.1	97	123	112	104	95	30-150	9	30	
		mg/kg										
2-Fluorobiphenyl (S)	%						81	81	54-125			
p-Terphenyl-d14 (S)	%						96	97	60-125			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

## REPORT OF LABORATORY ANALYSIS

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## QUALIFIERS

Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above LOD.

J - Estimated concentration at or above the LOD and below the LOQ.

LOD - Limit of Detection adjusted for dilution factor, percent moisture, initial weight and final volume.

LOQ - Limit of Quantitation adjusted for dilution factor, percent moisture, initial weight and final volume.

DL - Adjusted Method Detection Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected at or above the adjusted LOD.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### BATCH QUALIFIERS

Batch: 919604

[M5] A matrix spike/matrix spike duplicate was not performed for this batch due to insufficient sample volume.

### ANALYTE QUALIFIERS

M1 Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.

N2 The lab does not hold NELAC/TNI accreditation for this parameter but other accreditations/certifications may apply. A complete list of accreditations/certifications is available upon request.

R1 RPD value was outside control limits.

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA CROSS REFERENCE TABLE

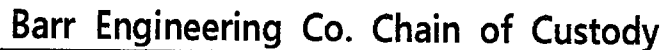
Project: 49161497.03 SRC Site Investiga

Pace Project No.: 10676081

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10676081001	IW-1_1.5-2.0	ASTM D2974	919339		
10676081001	IW-1_1.5-2.0	EPA 3546	918750	EPA 8270E by SIM	919224
10676081001	IW-1_1.5-2.0	EPA 5035/5030B	919395	EPA 8260D	919604
10676081002	Trip Blank	EPA 5035/5030B	919395	EPA 8260D	919604

## REPORT OF LABORATORY ANALYSIS

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☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ NV ☐ TX ☐ UT ☒ WI ☐ WY ☐ Other: \_\_\_\_\_

REPORT TO	INVOICE TO
Company: <i>Barr Engineering</i>	Company: <i>Barr Engineering</i>
Address: <i>375 S. Lake Ave.</i>	Address:
Address: <i>Duluth, MN 55802</i>	Address:
Name: <i>Lynette Carney</i>	Name:
email: <i>LCarney@barr.com</i>	email:
Copy to: <i>BarrDM@barr.com / jct@barr.com</i>	P.O.
Project Name: <i>SRL Site Investigation</i>	Barr Project No: <del><i>49161799-03</i></del> <i>49161497-03</i>

Location	Sample Depth			Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code
	Start	Stop	Unit (m./ft. or in.)			
1. IW-1	1.5	2.0	ft	11/10/2023	10:30	S
2. Trip Blank	-	-	-	11/10/2023	-	SQ
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						

[illegible]

**Distribution - White-Original:** Accompanies Shipment to Laboratory; **Yellow Copy:** Include in Field Documents; **Scan and email:** a copy to BarrDM@barr.com for tracking and filing procedures

H:\RLG\STDFORMS\Chain of Custody Form 2022 BLS Rev 10/14/2021

Effective Date: 4/14/2023

Sample Condition  
Upon Receipt

Client Name:

Barr Eng

Project #:

WO#: 10676081

PM: MKH

Due Date: 12/01/23

CLIENT: BARR

Courier: ☐ FedEx ☐ UPS ☐ USPS ☐ Client  
☐ Pace ☐ Speedee ☐ Commercial

Tracking Number:

☐ See Exceptions  
ENV-FRM-MIN4-0142Custody Seal on Cooler/Box Present? ☒ Yes ☐ No Seals Intact? ☒ Yes ☐ NoBiological Tissue Frozen? ☐ Yes ☐ No ☒ N/APacking Material: ☒ Bubble Wrap ☐ Bubble Bags ☐ None ☐ OtherTemp Blank? ☒ Yes ☐ NoThermometer: ☐ T1 (0461) ☐ T2 (0436) ☒ T3 (0459) ☐ T4 (0402) ☐ T5 (0178)  
☐ T6 (0235) ☐ T7 (0042) ☐ T8 (0775) ☐ T9 (0727) ☐ 01339252/1710Type of Ice: ☒ Wet ☐ Blue ☐ Dry ☐ None  
☐ MeltedDid Samples Originate in West Virginia? ☐ Yes ☒ NoWere All Container Temps Taken? ☐ Yes ☐ No ☒ N/A

Temp should be above freezing to 6 °C

Cooler temp Read w/Temp Blank: 4.8 °C

Average Corrected Temp

(no temp blank only): °C

Correction Factor: +0.1

Cooler Temp Corrected w/temp blank: 4.9 °C

☐ See Exceptions ENV-FRM-MIN4-0142 ☐ 1 ContainerUSDA Regulated Soil: ☐ N/A, water sample/other: \_\_\_\_\_

Date/Initials of Person Examining Contents: CRL 11/15/23

Did samples originate in a quarantine zone within the United States: AL, AR, AZ CA, FL, GA, ID, LA, MS, NC, NM, NY, OK, OR, SC, TN, TX, or VA (check maps)? ☐ Yes ☒ NoDid samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☒ No

If Yes to either question, fill out a Regulated Soil Checklist (ENV-FRM-MIN4-0154) and include with SCUR/COC paperwork.

Location (Check one):	Duluth	Minneapolis	Virginia	COMMENTS
Chain of Custody Present and Filled Out?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2.
Sampler Name and/or Signature on COC?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/> N/A	3.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. If fecal: <input type="checkbox"/> <8 hrs <input type="checkbox"/> >8 hr, <24 <input type="checkbox"/> No
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	5. <input type="checkbox"/> Fecal Coliform <input type="checkbox"/> HPC <input type="checkbox"/> Total Coliform/E.coli <input type="checkbox"/> BOD/cBOD <input type="checkbox"/> Hex Chrom <input type="checkbox"/> Turbidity <input type="checkbox"/> Nitrate <input type="checkbox"/> Nitrite <input type="checkbox"/> Orthophos <input type="checkbox"/> Other
Rush Turn Around Time Requested?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	6.
Sufficient Sample Volume?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7.
Correct Containers Used?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> N/A	8.
-Pace Containers Used?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9.
Containers Intact?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. Is sediment visible in the dissolved container? <input type="checkbox"/> Yes <input type="checkbox"/> No
Field Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/> N/A	11. If no, write ID/Date/Time of container below: <input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142
Is sufficient information available to reconcile the samples to the COC?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	12. Sample # <input type="checkbox"/> NaOH <input type="checkbox"/> HNO3 <input type="checkbox"/> H2SO4 <input type="checkbox"/> Zinc Acetate Positive for Residual Chlorine? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142 pH Paper Lot # Residual Chlorine 0-6 Roll 0-6 Strip 0-14 Strip
Matrix: <input type="checkbox"/> Water <input checked="" type="checkbox"/> Soil <input type="checkbox"/> Oil <input type="checkbox"/> Other				
All containers needing acid/base preservation have been checked?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/> N/A	
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO3, H2SO4, <2pH, NaOH >9 Sulfide, NaOH >10 Cyanide)	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/> N/A	
Exceptions: VOA Coliform, TOC/DOC Oil and Grease, DRO/8015 (water) and Bioxins/PFAS (*If adding preservative to a container, it must be added to associated field and equipment blanks--verify with PM first.)	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> N/A	
Headspace in Methyl Mercury Container?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/> N/A	13.
Extra labels present on soil VOA or WIDRO containers?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/> N/A	14. <input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142
Headspace in VOA Vials (greater than 6mm)?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/> N/A	
3 Trip Blanks Present?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15. 2 Pace Trip Blank Lot # (if purchased): 050823-3
Trip Blank Custody Seals Present?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

## CLIENT NOTIFICATION/RESOLUTION

Person Contacted:

Date/Time:

Comments/Resolution:

Field Data Required? ☐ Yes ☐ No

Project Manager Review:

Date: 11/15/23

NOTE: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e., out of hold, incorrect preservative, out of temp, incorrect containers).

Labeled By: CRL

Line: (3)



# Analytical Laboratory Report

Report ID: S51688.01(01)+QC02  
Generated on 08/09/2023

**Report to**  
Attention: Ryan Erickson  
Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802  
  
Phone: 218-529-7112    FAX:  
Email: [rerickson@barr.com](mailto:rerickson@barr.com)


**Report produced by**  
Merit Laboratories, Inc.  
2680 East Lansing Drive  
East Lansing, MI 48823  
  
Phone: (517) 332-0167    FAX: (517) 332-6333  
  
Contacts for report questions:  
[John Lavery \(johnlavery@meritlabs.com\)](mailto:JohnLavery@meritlabs.com)  
[Barbara Ball \(bball@meritlabs.com\)](mailto:BarbaraBall@meritlabs.com)

Additional Contacts: David Beattie, Brian Kwiatkoski, Katie Wolohan, Andy McCabe, Michael J. Powers, James Taraldsen, Aaron Laszewski, Joseph Pearson, Sara Binahmed-Menzies, Barr Data Manager

**Report Summary**  
Lab Sample ID(s): S51688.01-S51688.02  
Project: SRC GW 49161497.03 100 101  
Collected Date(s): 08/01/2023  
Submitted Date/Time: 08/02/2023 09:30  
Sampled by: KMJ3  
P.O. #: PO

**Table of Contents**

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- General Report Notes (Page 2)
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- Method Summary (Page 4)
- Sample Summary (Page 5)
- QC Report (Pages 10-20)

  
Maya Murshak  
Technical Director



# Analytical Laboratory Report

## General Report Notes

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Analytical results relate only to the samples tested, in the condition received by the laboratory.

Methods may be modified for improved performance.

Results reported on a dry weight basis where applicable.

'Not detected' indicates that parameter was not found at a level equal to or greater than the reporting limit (RL).

When MDL results are provided, then 'Not detected' indicates that parameter was not found at a level equal to or greater than the MDL.

40 CFR Part 136 Table II Required Containers, Preservation Techniques and Holding Times for the Clean Water Act specify that samples for acrolein and acrylonitrile, and 2-chloroethylvinyl ether need to be preserved at a pH in the range of 4 to 5 or if not preserved, analyzed within 3 days of sampling.

QA/QC corresponding to this analytical report is a separate document with the same Merit ID reference and is available upon request.

Full accreditation certificates are available upon request. Starred (\*) analytes are not NELAP accredited.

Samples are held by the lab for 30 days from the final report date unless a written request to hold longer is provided by the client.

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Limits for drinking water samples, are listed as the MCL Limits (Maximum Contaminant Level Concentrations)

PFAS requirement: Section 9.3.8 of U.S. EPA Method 537.1 states "If the method analyte(s) found in the Field Sample is present in the

FRB at a concentration greater than 1/3 the MRL, then all samples collected with that FRB are invalid and must be recollected and reanalyzed."

Samples submitted without an accompanying FRB may not be acceptable for compliance purposes.

Wisconsin PFAs analysis: MDL = LOD; RL = LOQ. LOD and LOQ are adjusted for dilution.

## Report Narrative

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There is no additional narrative for this analytical report



# Analytical Laboratory Report

## Laboratory Certifications

Authority	Certification ID
Michigan DEQ	#9956
DOD ELAP & ISO/IEC 17025:2017	#69699
WBENC	#2005110032
Ohio VAP	#CL0002
Indiana DOH	#C-MI-07
New York NELAC	#11814
North Carolina DENR	#680
North Carolina DOH	#26702
Pennsylvania DEP	#68-05884
Wisconsin DNR	FID# 399147320

## Qualifier Descriptions

Qualifier	Description
!	Result is outside of stated limit criteria
B	Compound also found in associated method blank
E	Concentration exceeds calibration range
F	Analysis run outside of holding time
G	Estimated result due to extraction run outside of holding time
H	Sample submitted and run outside of holding time
I	Matrix interference with internal standard
J	Estimated value less than reporting limit, but greater than MDL
L	Elevated reporting limit due to low sample amount
M	Result reported to MDL not RDL
O	Analysis performed by outside laboratory. See attached report.
R	Preliminary result
S	Surrogate recovery outside of control limits
T	No correction for total solids
X	Elevated reporting limit due to matrix interference
Y	Elevated reporting limit due to high target concentration
b	Value detected less than reporting limit, but greater than MDL
e	Reported value estimated due to interference
j	Analyte also found in associated method blank
p	Benzo(b)Fluoranthene and Benzo(k)Fluoranthene integrated as one peak.
x	Preserved from bulk sample

## Glossary of Abbreviations

Abbreviation	Description
RL/RDL	Reporting Limit
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
SW	EPA SW 846 (Soil and Wastewater) Methods
E	EPA Methods
SM	Standard Methods
LN	Linear
BR	Branched



## Method Summary

Method	Version
N/A	Not Applicable
WI SPE	PFAS by LCMSMS Per Wisconsin DNR Document EA-19-0001

## Parameter Summary

Parameter	Synonym	Cas #
PFBA	Perfluorobutanoic Acid	375-22-4
PFPeA	Perfluoropentanoic Acid	2706-90-3
4:2 FTSA	4:2 Fluorotelomer Sulfonic Acid	757124-72-4
PFHxA	Perfluorohexanoic Acid	307-24-4
PFBS	Perfluorobutane sulfonic Acid	375-73-5
PfHpA	Perfluoroheptanoic Acid	375-85-9
PFPeS	Perfluoropentane Sulfonic Acid	2706-91-4
6:2 FTSA	6:2 Fluorotelomer Sulfonic Acid	27619-97-2
PFOA	Perfluorooctanoic Acid	335-67-1
PFHxS	Perfluorohexane Sulfonic Acid	355-46-4
PFHxS-LN	Perfluorohexane Sulfonic Acid - LN	355-46-4-LN
PFHxS-BR	Perfluorohexane Sulfonic Acid - BR	355-46-4-BR
PFNA	Perfluorononanoic Acid	375-95-1
8:2 FTSA	8:2 Fluorotelomer Sulfonic Acid	39108-34-4
PFHpS	Perfluoroheptane Sulfonic Acid	375-92-8
PFDA	Perfluorodecanoic Acid	335-76-2
N-MeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9
EtFOSAA	N-Ethyl Perfluorooctane Sulfonamidoacetic Acid	2991-50-6
PFOS	Perfluorooctane Sulfonic Acid	1763-23-1
PFOS-LN	Perfluorooctane Sulfonic Acid - LN	1763-23-1-LN
PFOS-BR	Perfluorooctane Sulfonic Acid - BR	1763-23-1-BR
PFUnDA	Perfluoroundecanoic Acid	2058-94-8
PFNS	Perfluorononane Sulfonic Acid	68259-12-1
PFDoDA	Perfluorododecanoic Acid	307-55-1
PFDS	Perfluorodecane Sulfonic Acid	335-77-3
PFTTrDA	Perfluorotridecanoic Acid	72629-94-8
FOSA	Perfluorooctane Sulfonamide	754-91-6
PFTeDA	Perfluorotetradecanoic Acid	376-06-7
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanone1-sulfonic acid	756426-58-1
ADONA	4,8-dioxa-3H-perfluorononanoic acid	919005-14-4
HFPO-DA	Hexafluoropropylene oxide dimer	13252-13-6
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
NMeFOSAM	N-Methylperfluorooctanesulfonamide	31506-32-8
NEtFOSAM	N-Ethylperfluorooctanesulfonamide	4151-50-2
NMeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
NEtFOSE	N-Ethylperfluorooctanesulfonamidoethanol	1691-99-2



# Analytical Laboratory Report

## Sample Summary (2 samples)

Sample ID	Sample Tag	Matrix	Collected Date/Time
S51688.01	RB-Decon	Liquid	08/01/23 09:10
S51688.02	RB-PVC	Liquid	08/01/23 09:20



# Analytical Laboratory Report

Lab Sample ID: S51688.01

Sample Tag: RB-Decon

Collected Date/Time: 08/01/2023 09:10

Matrix: Liquid

COC Reference: 588196

## Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
3	250ml Plastic	Trizma	Yes	4.0	IR

## Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	08/04/23 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	304.51/32.87	WI SPE	08/04/23 10:00	PTW	

## Organics

WI 33 PFAs, Method: WI SPE, Run Date: 08/07/23 14:16, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	0.75	1.8	0.26	ng/L	0.0147	375-22-4	J
PFPeA*	0.46	1.8	0.21	ng/L	0.0147	2706-90-3	J
4:2 FTSA*	Not detected	1.8	0.34	ng/L	0.0147	757124-72-4	
PFHxA*	0.34	1.8	0.24	ng/L	0.0147	307-24-4	J
PFBS*	0.38	1.8	0.18	ng/L	0.0147	375-73-5	J
PFHpA*	Not detected	1.8	0.41	ng/L	0.0147	375-85-9	
PFPeS*	Not detected	1.8	0.22	ng/L	0.0147	2706-91-4	
6:2 FTSA*	Not detected	1.8	0.50	ng/L	0.0147	27619-97-2	
PFOA*	Not detected	1.8	0.38	ng/L	0.0147	335-67-1	
PFHxS*	Not detected	1.8	0.57	ng/L	0.0147	355-46-4	
PFHxS-LN*	Not detected	1.8	0.57	ng/L	0.0147	355-46-4-LN	
PFHxS-BR*	Not detected	1.8	0.57	ng/L	0.0147	355-46-4-BR	
PFNA*	Not detected	1.8	0.38	ng/L	0.0147	375-95-1	
8:2 FTSA*	Not detected	1.8	0.66	ng/L	0.0147	39108-34-4	
PFHpS*	Not detected	1.8	0.47	ng/L	0.0147	375-92-8	
PFDA*	Not detected	1.8	0.50	ng/L	0.0147	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.31	ng/L	0.0147	2355-31-9	
EtFOSAA*	Not detected	1.8	0.60	ng/L	0.0147	2991-50-6	
PFOS*	0.54	1.8	0.34	ng/L	0.0147	1763-23-1	J
PFOS-LN*	Not detected	1.8	0.34	ng/L	0.0147	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.34	ng/L	0.0147	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.43	ng/L	0.0147	2058-94-8	
PFNS*	Not detected	1.8	0.50	ng/L	0.0147	68259-12-1	
PFDODA*	Not detected	1.8	0.93	ng/L	0.0147	307-55-1	
PFDS*	Not detected	1.8	0.56	ng/L	0.0147	335-77-3	
PFTTrDA*	Not detected	1.8	0.63	ng/L	0.0147	72629-94-8	
FOSA*	Not detected	1.8	0.53	ng/L	0.0147	754-91-6	
PFTeDA*	Not detected	1.8	0.75	ng/L	0.0147	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.72	ng/L	0.0147	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.40	ng/L	0.0147	756426-58-1	
ADONA*	0.36	1.8	0.34	ng/L	0.0147	919005-14-4	J
HFPO-DA*	Not detected	1.8	0.29	ng/L	0.0147	13252-13-6	
PFDoS*	Not detected	1.8	0.56	ng/L	0.0147	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.56	ng/L	0.0147	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.56	ng/L	0.0147	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S51688.01 (continued)

Sample Tag: RB-Decon

WI 33 PFAs, Method: WI SPE, Run Date: 08/07/23 14:16, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	Not detected	1.8	0.66	ng/L	0.0147	24448-09-7	
NEtFOSE*	Not detected	1.8	0.72	ng/L	0.0147	1691-99-2	



# Analytical Laboratory Report

Lab Sample ID: S51688.02

Sample Tag: RB-PVC

Collected Date/Time: 08/01/2023 09:20

Matrix: Liquid

COC Reference: 588196

## Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
3	250ml Plastic	Trizma	Yes	4.0	IR

## Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
pH check for DW PFAs*	7	N/A	08/04/23 10:00	PTW	
Initial wt. (g) / Final wt. (g)*	311.16/33.01	WI SPE	08/04/23 10:00	PTW	

## Organics

WI 33 PFAs, Method: WI SPE, Run Date: 08/07/23 14:32, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	0.73	1.8	0.26	ng/L	0.0144	375-22-4	J
PFPeA*	Not detected	1.8	0.20	ng/L	0.0144	2706-90-3	
4:2 FTSA*	Not detected	1.8	0.33	ng/L	0.0144	757124-72-4	
PFHxA*	0.36	1.8	0.23	ng/L	0.0144	307-24-4	J
PFBS*	0.36	1.8	0.17	ng/L	0.0144	375-73-5	J
PFHpA*	Not detected	1.8	0.40	ng/L	0.0144	375-85-9	
PFPeS*	Not detected	1.8	0.22	ng/L	0.0144	2706-91-4	
6:2 FTSA*	Not detected	1.8	0.49	ng/L	0.0144	27619-97-2	
PFOA*	Not detected	1.8	0.37	ng/L	0.0144	335-67-1	
PFHxS*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4	
PFHxS-LN*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4-LN	
PFHxS-BR*	Not detected	1.8	0.56	ng/L	0.0144	355-46-4-BR	
PFNA*	Not detected	1.8	0.37	ng/L	0.0144	375-95-1	
8:2 FTSA*	Not detected	1.8	0.65	ng/L	0.0144	39108-34-4	
PFHpS*	Not detected	1.8	0.46	ng/L	0.0144	375-92-8	
PFDA*	Not detected	1.8	0.49	ng/L	0.0144	335-76-2	
N-MeFOSAA*	Not detected	1.8	0.30	ng/L	0.0144	2355-31-9	
EtFOSAA*	0.71	1.8	0.59	ng/L	0.0144	2991-50-6	J
PFOS*	0.36	1.8	0.33	ng/L	0.0144	1763-23-1	J
PFOS-LN*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1-LN	
PFOS-BR*	Not detected	1.8	0.33	ng/L	0.0144	1763-23-1-BR	
PFUnDA*	Not detected	1.8	0.42	ng/L	0.0144	2058-94-8	
PFNS*	Not detected	1.8	0.49	ng/L	0.0144	68259-12-1	
PFDODA*	Not detected	1.8	0.91	ng/L	0.0144	307-55-1	
PFDS*	Not detected	1.8	0.55	ng/L	0.0144	335-77-3	
PFTTrDA*	Not detected	1.8	0.62	ng/L	0.0144	72629-94-8	
FOSA*	Not detected	1.8	0.52	ng/L	0.0144	754-91-6	
PFTeDA*	Not detected	1.8	0.73	ng/L	0.0144	376-06-7	
11Cl-PF3OUdS*	Not detected	1.8	0.71	ng/L	0.0144	763051-92-9	
9Cl-PF3ONS*	Not detected	1.8	0.39	ng/L	0.0144	756426-58-1	
ADONA*	0.35	1.8	0.33	ng/L	0.0144	919005-14-4	J
HFPO-DA*	Not detected	1.8	0.29	ng/L	0.0144	13252-13-6	
PFDoS*	Not detected	1.8	0.55	ng/L	0.0144	79780-39-5	
NMeFOSAM*	Not detected	1.8	0.55	ng/L	0.0144	31506-32-8	
NEtFOSAM*	Not detected	1.8	0.55	ng/L	0.0144	4151-50-2	

J-Estimated value less than reporting limit, but greater than MDL



# Analytical Laboratory Report

Lab Sample ID: S51688.02 (continued)

Sample Tag: RB-PVC

WI 33 PFAs, Method: WI SPE, Run Date: 08/07/23 14:32, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
NMeFOSE*	0.69	1.8	0.65	ng/L	0.0144	24448-09-7	J
NEtFOSE*	Not detected	1.8	0.71	ng/L	0.0144	1691-99-2	

J-Estimated value less than reporting limit, but greater than MDL





# Quality Control Report

Report ID: S51688.01(01)+QC02  
Generated on 08/15/2023

## Report to

Attention: David Beattie  
Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802

Phone: 218-348-9051 FAX:

## Report Produced by

Merit Laboratories  
2680 East Lansing Drive  
East Lansing, MI 48823

Phone: (517) 332-0167 FAX: (517) 332-6333

## Report Summary

Lab Sample ID(s): S51688.01-S51688.02  
Project: SRC GW 49161497.03 100 101  
Submitted Date/Time: 08/02/2023 09:30  
Sampled by: KMJ3  
P.O. #: 8401610216-10

## QC Report Sections

Cover Page (Page 10)  
Analysis Summary (Pages 11-12)  
Prep Batch Summary (Page 13)  
Surrogates per QC Sample (Page 14)  
Internal Standards per Lab Sample (Pages 15-16)  
Internal Standards per QC Sample (Pages 17-18)  
Batch QC Results (Pages 19-20)

## Report Flag Descriptions

\*: QC result is outside of indicated control limits  
W: Surrogate result not applicable due to sample dilution

I certify that this data package is in compliance with the terms and conditions of the program, and project, and contractual requirements both technically and for completeness. Release of the data contained in this hardcopy data package and its computer-readable data submitted has been authorized by the Quality Assurance Manager and his/her designee, as verified by the following signature.

Barbara Ball  
Quality Assurance Manager

## QC Report - Analysis Summary

Lab Sample ID: S51688.01

Sample Tag: RB-Decon

Collected Date/Time: 08/01/2023 09:10

Matrix: Liquid

COC Reference: 588196

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b>Organics - Volatiles</b>						
WI 33 PFAs	WI SPE	08/07/23 14:16	CI230807WISPE	DS230804W1	Yes	BLK/LCS

## QC Report - Analysis Summary

Lab Sample ID: S51688.02

Sample Tag: RB-PVC

Collected Date/Time: 08/01/2023 09:20

Matrix: Liquid

COC Reference: 588196

Analysis	Method	Run Date/Time	Batch ID	Prep ID	Surr	QC Types
<b><i>Organics - Volatiles</i></b>						
WI 33 PFAs	WI SPE	08/07/23 14:32	CI230807WISPE	DS230804W1	Yes	BLK/LCS

## QC Report - Prep Batch Summary

### Organics - Volatiles, Prep Batch ID: DS230804W1

Surrogates: Yes, QC Types: BLK/LCS

Sample ID	Analysis	Method	Run Date/Time	Batch ID
S51688.01	WI 33 PFAs	WI SPE	08/07/23 14:16	CI230807WISPE
S51688.02	WI 33 PFAs	WI SPE	08/07/23 14:32	CI230807WISPE

## QC Report - Surrogates per QC Sample

**Organics - Volatiles, Prep Batch ID: DS230804W1**

QC Types: BLK/LCS

### Blank (BLK)

Lab Sample ID: CI230807WISPE.BLK230807

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 13:11, Prep Date: 08/04/2023, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
No Surrogates				

### Laboratory Control Sample (LCS)

Lab Sample ID: CI230807WISPE.LCS230807

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 13:27, Prep Date: 08/04/2023, Matrix: WW, Dilution: 1

Surrogate	Flags	%Rec	LCL	UCL
No Surrogates				

# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S51688.01

Sample Tag: RB-Decon

Collected Date/Time: 08/01/2023 09:10

Matrix: Liquid

COC Reference: 588196

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 14:16, Matrix: WW, Dilution: 0.0147

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		98.9	25	150.0
M2-6:2FTSA		74.7	25	150.0
M2-8:2FTSA		84.5	25	150.0
M2PFTeDA		75.8	25	150.0
M3PFBS		102.8	25	150.0
M3PFHxS		101.4	25	150.0
M4PFHpA		101.3	25	150.0
M5PFHxA		101.1	25	150.0
M5PFPeA		99.8	25	150.0
M6PFDA		93.3	25	150.0
M7PFUnDA		89.0	25	150.0
M8FOSA		90.2	10	150.0
M8PFOA		97.0	25	150.0
M8PFOS		96.0	25	150.0
M9-PFNA		96.3	25	150.0
MPFBA		107.0	25	150.0
MPFDoDA		77.0	25	150.0
d3N-MeFOSAA		94.0	25	150.0
d5EtFOSAA		89.6	25	150.0
MHFPODA		97.2	25	150.0
d-N-EtFOSA-M		67.9	10	150.0
d-N-MeFOSA-M		70.9	10	150.0
d7-N-MeFOSE-M		73.8	10	150.0
d9-N-EtFOSE-M		58.9	10	150.0



# QC Report - Internal Standards per Lab Sample

Lab Sample ID: S51688.02

Sample Tag: RB-PVC

Collected Date/Time: 08/01/2023 09:20

Matrix: Liquid

COC Reference: 588196

## Organics - Volatiles, Analysis: WI 33 PFAs

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 14:32, Matrix: WW, Dilution: 0.0144

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		114.5	25	150.0
M2-6:2FTSA		93.8	25	150.0
M2-8:2FTSA		100.2	25	150.0
M2PFTeDA		80.0	25	150.0
M3PFBS		104.9	25	150.0
M3PFHxS		105.6	25	150.0
M4PFHpA		102.5	25	150.0
M5PFHxA		103.7	25	150.0
M5PFPeA		102.6	25	150.0
M6PFDA		98.2	25	150.0
M7PFUnDA		97.3	25	150.0
M8FOSA		97.8	10	150.0
M8PFOA		101.1	25	150.0
M8PFOS		107.1	25	150.0
M9-PFNA		101.5	25	150.0
MPFBA		108.5	25	150.0
MPFDoDA		86.0	25	150.0
d3N-MeFOSAA		97.2	25	150.0
d5EtFOSAA		100.3	25	150.0
MHFPODA		101.8	25	150.0
d-N-EtFOSA-M		74.8	10	150.0
d-N-MeFOSA-M		78.0	10	150.0
d7-N-MeFOSE-M		72.9	10	150.0
d9-N-EtFOSE-M		63.6	10	150.0

# QC Report - Internal Standards per QC Sample

**Organics - Volatiles, Prep Batch ID: DS230804W1**

QC Types: BLK/LCS

## Blank (BLK)

Lab Sample ID: CI230807WISPE.BLK230807

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 13:11, Prep Date: 08/04/2023, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		94.6	25	150.0
M2-6:2FTSA		79.8	25	150.0
M2-8:2FTSA		85.9	25	150.0
M2PFTeDA		82.1	25	150.0
M3PFBS		104.5	25	150.0
M3PFHxS		104.4	25	150.0
M4PFHpA		102.0	25	150.0
M5PFHxA		103.1	25	150.0
M5PFPeA		103.9	25	150.0
M6PFDA		90.5	25	150.0
M7PFUnDA		89.2	25	150.0
M8FOSA		92.6	10	150.0
M8PFOA		99.0	25	150.0
M8PFOS		101.7	25	150.0
M9-PFNA		96.8	25	150.0
MPFBA		109.7	25	150.0
MPFDoDA		79.4	25	150.0
d3N-MeFOSAA		91.7	25	150.0
d5EtFOSAA		90.5	25	150.0
MHFPODA		99.8	25	150.0
d-N-EtFOSA-M		73.5	10	150.0
d-N-MeFOSA-M		72.3	10	150.0
d7-N-MeFOSE-M		83.8	10	150.0
d9-N-EtFOSE-M		67.6	10	150.0

# QC Report - Internal Standards per QC Sample

## Laboratory Control Sample (LCS)

Lab Sample ID: CI230807WISPE.LCS230807

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 13:27, Prep Date: 08/04/2023, Matrix: WW, Dilution: 1

Internal Standard	Flags	%Rec	LCL	UCL
M2-4:2FTSA		92.0	25	150.0
M2-6:2FTSA		83.3	25	150.0
M2-8:2FTSA		85.5	25	150.0
M2PFTeDA		87.2	25	150.0
M3PFBS		105.6	25	150.0
M3PFHxS		104.9	25	150.0
M4PFHpA		101.3	25	150.0
M5PFHxA		104.4	25	150.0
M5PFPeA		104.2	25	150.0
M6PFDA		87.1	25	150.0
M7PFUnDA		91.3	25	150.0
M8FOSA		95.8	10	150.0
M8PFOA		99.6	25	150.0
M8PFOS		100.6	25	150.0
M9-PFNA		97.3	25	150.0
MPFBA		110.8	25	150.0
MPFDoDA		84.7	25	150.0
d3N-MeFOSAA		93.3	25	150.0
d5EtFOSAA		84.3	25	150.0
MHFPODA		100.6	25	150.0
d-N-EtFOSA-M		82.0	10	150.0
d-N-MeFOSA-M		74.9	10	150.0
d7-N-MeFOSE-M		88.0	10	150.0
d9-N-EtFOSE-M		72.0	10	150.0

# QC Report - Batch QC Results

## Organics - Volatiles, Prep Batch ID: DS230804W1

Surrogates: Yes, QC Types: BLK/LCS

### Blank (BLK)

Lab Sample ID: CI230807WISPE.BLK230807

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 13:11, Prep Date: 08/04/2023, Matrix: WW, Dilution: 1

Analyte	Flags	Conc	RDL	MDL	Units
PFBA		ND	2.00	0.29	ng/l
PFPeA		ND	2.00	0.22	ng/l
4:2 FTSA		ND	2.00	0.37	ng/l
PFHxA	J*	0.2982	2.00	0.26	ng/l
PFBS		ND	2.00	0.19	ng/l
PfHpA		ND	2.00	0.45	ng/l
PFPeS		ND	2.00	0.24	ng/l
6:2 FTSA		ND	2.00	0.54	ng/l
PFOA		ND	2.00	0.42	ng/l
PFHxS		ND	2.00	0.62	ng/l
PFHxS-LN		ND	2.00	0.62	ng/l
PFHxS-BR		ND	2.00	0.62	ng/l
PFNA		ND	2.00	0.42	ng/l
8:2 FTSA		ND	2.00	0.72	ng/l
PfHpS		ND	2.00	0.51	ng/l
PFDA		ND	2.00	0.54	ng/l
N-MeFOSAA	J*	0.3597	2.00	0.34	ng/l
EtFOSAA		ND	2.00	0.66	ng/l
PFOS		ND	2.00	0.37	ng/l
PFOS-LN		ND	2.00	0.37	ng/l
PFOS-BR		ND	2.00	0.37	ng/l
PFUnDA		ND	2.00	0.46	ng/l
PFNS		ND	2.00	0.54	ng/l
PFDoDA		ND	2.00	1.0	ng/l
PFDS		ND	2.00	0.61	ng/l
PFTTrDA		ND	2.00	0.69	ng/l
FOSA		ND	2.00	0.58	ng/l
PFTeDA		ND	2.00	0.82	ng/l
11CL-PF3OUdS		ND	2.00	0.78	ng/l
9CL-PF3ONS		ND	2.00	0.43	ng/l
ADONA		ND	2.00	0.37	ng/l
HFPO-DA		ND	2.00	0.3	ng/l
PFDOS		ND	2.00	0.61	ng/l
NMeFOSAM		ND	2.00	0.61	ng/l
NEtFOSAM		ND	2.00	0.61	ng/l
NMeFOSE		ND	2.00	0.72	ng/l
NEtFOSE		ND	2.00	0.78	ng/l

### Laboratory Control Sample (LCS)

Lab Sample ID: CI230807WISPE.LCS230807

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 13:27, Prep Date: 08/04/2023, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCS Conc	% Rec	LCL	UCL
PFBA		0.00	20.0	17.7	88.5	50	150
PFPeA		0.00	20.0	18.1	90.5	50	150
PFBS		0.00	20.0	18.6	93.0	50	150
4:2 FTSA		0.00	20.0	16.7	83.5	50	150

# QC Report - Batch QC Results

## Organics - Volatiles, Prep Batch ID: DS230804W1 (continued)

Surrogates: Yes, QC Types: BLK/LCS

## Laboratory Control Sample (LCS) (continued)

Lab Sample ID: CI230807WISPE.LCS230807

Run in Batch: CI230807WISPE, Run Date: 08/07/2023 13:27, Prep Date: 08/04/2023, Matrix: WW, Dilution: 1

Analyte	Flags	Orig Conc	Spike	LCS Conc	% Rec	LCL	UCL
PFHxA		0.00	20.0	18.4	92.0	50	150
PFPeS		0.00	20.0	18.2	91.0	50	150
HFPO-DA		0.00	20.0	17.9	89.5	50	150
PFHxS		0.00	20.0	17.8	89.0	50	150
PFHpA		0.00	20.0	18.2	91.0	50	150
ADONA		0.00	20.0	18.9	94.5	50	150
6:2 FTSA		0.00	20.0	17.3	86.5	50	150
PFOA		0.00	20.0	18.5	92.5	50	150
PFHpS		0.00	20.0	16.6	83.0	50	150
PFOS		0.00	20.0	18.3	91.5	50	150
PFNA		0.00	20.0	18.5	92.5	50	150
9CL-PF3ONS		0.00	20.0	17.8	89.0	50	150
PFNS		0.00	20.0	17.7	88.5	50	150
8:2 FTSA		0.00	20.0	14.8	74.0	50	150
PFDA		0.00	20.0	19.1	95.5	50	150
N-MeFOSAA		0.00	20.0	16.3	81.5	50	150
EtFOSAA		0.00	20.0	18.4	92.0	50	150
PFDS		0.00	20.0	17.4	87.0	50	150
PFUnDA		0.00	20.0	18.9	94.5	50	150
FOSA		0.00	20.0	19.0	95.0	50	150
11CL-PF3OUdS		0.00	20.0	16.0	80.0	50	150
PFDoDA		0.00	20.0	18.3	91.5	50	150
PFDOS		0.00	20.0	19.1	95.5	50	150
PFTTrDA		0.00	20.0	18.3	91.5	50	150
NMeFOSAM		0.00	20.0	20.1	100.5	50	150
NMeFOSE		0.00	20.0	19.3	96.5	50	150
PFTeDA		0.00	20.0	18.8	94.0	50	150
NEtFOSAM		0.00	20.0	17.5	87.5	50	150
NEtFOSE		0.00	20.0	19.4	97.0	50	150

# Merit Laboratories Login Checklist

Lab Set ID:S51688

Client:BARR (Barr Engineering)

Project: SRC GW 49161497.03 100 101

Submitted:08/02/2023 09:30 Login User: MMC

Attention: David Beattie

Address: Barr Engineering  
325 South Lake Avenue  
Suite 700  
Duluth, MN 55802

Phone: 218-348-9051 FAX:  
Email: David.Beattie@cenovus.com

Selection	Description	Note
<b>Sample Receiving</b>		
01. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples are received at 4C +/- 2C Thermometer #	IR 4.0
02. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Received on ice/ cooling process begun	
03. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples shipped	FedEx
04. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Samples left in 24 hr. drop box	
05. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Are there custody seals/tape or is the drop box locked	
<b>Chain of Custody</b>		
06. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	COC adequately filled out	
07. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	COC signed and relinquished to the lab	
08. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Sample tag on bottles match COC	
09. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Subcontracting needed? Subcontacted to:	
<b>Preservation</b>		
10. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Do sample have correct chemical preservation	
11. <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Completed pH checks on preserved samples? (no VOAs)	
12. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Did any samples need to be preserved in the lab?	
<b>Bottle Conditions</b>		
13. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	All bottles intact	
14. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Appropriate analytical bottles are used	
15. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Merit bottles used	
16. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Sufficient sample volume received	
17. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Samples require laboratory filtration	
18. <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples submitted within holding time	
19. <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Do water VOC or TOX bottles contain headspace	

Corrective action for all exceptions is to call the client and to notify the project manager.

Client Review By: \_\_\_\_\_ Date: \_\_\_\_\_





# Barr Engineering Co. Chain of Custody

Sample Origination State

☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ TX ☐ UT ☒ WI ☐ Other: \_\_\_\_\_

REPORT TO		INVOICE TO	
Company: Barr Engineering Co.		Company: SRC	
Address: 325 S. Lake Ave		Address:	
Address: Duluth, MN 55862		Address:	
Name: Lynette Carney		Name:	
email: lcarney@barr.com		email:	
Copy to: BarrDM@barr.com		P.O.:	
Project Name: SRC GW		Barr Project No: 49161497.03 100101	

Location	Sample Depth			Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform	Total	Nu	2	K	N																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																	
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COC Number: **Nº 588196**

COC 1 of 1

<b>Matrix Code:</b>	<b>Preservative Code:</b>
GW = Groundwater	A = None
SW = Surface Water	B = HCl
WW = Waste Water	C = HNO <sub>3</sub>
DW = Drinking Water	D = H <sub>2</sub> SO <sub>4</sub>
S = Soil/Solid	E = NaOH
SD = Sediment	F = MeOH
O = Other	G = NaHSO <sub>4</sub>
	H = Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>
	I = Ascorbic Acid
	J = Zn Acetate
	K = Other

Preservative Code

Field Filtered Y/N

WI List of 33 PFAS  
↓  
51688.01  
.02

<b>BARR USE ONLY</b>		Relinquished by: <i>[Signature]</i>	On Ice? <input checked="" type="checkbox"/> Y <input type="checkbox"/> N	Date: 8/1/23	Time: 1215	Received by:	Date:	Time:
Sampled by: LMS3		Relinquished by: FedEx	On Ice? <input type="checkbox"/> Y <input type="checkbox"/> N	Date: 8/2/23	Time: 0930	Received by: <i>[Signature]</i>	Date: 8/2/23	Time: 0930
Barr Proj. Manager: LMC		Samples Shipped VIA: <input type="checkbox"/> Ground Courier <input type="checkbox"/> Air Carrier		Air Bill Number:		Requested Due Date:		
Barr DQ Manager: TAO		<input type="checkbox"/> Sampler <input type="checkbox"/> Other: _____				<input type="checkbox"/> Standard Turn Around Time		
Lab Name: Merit		Lab WO: _____		Temperature on Receipt (°C): 4.0		<input checked="" type="checkbox"/> Rush 5-day + AT		
Lab Location: East Lansing, MI				Custody Seal Intact? <input type="checkbox"/> Y <input type="checkbox"/> N <input type="checkbox"/> None		(mm/dd/yyyy)		

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