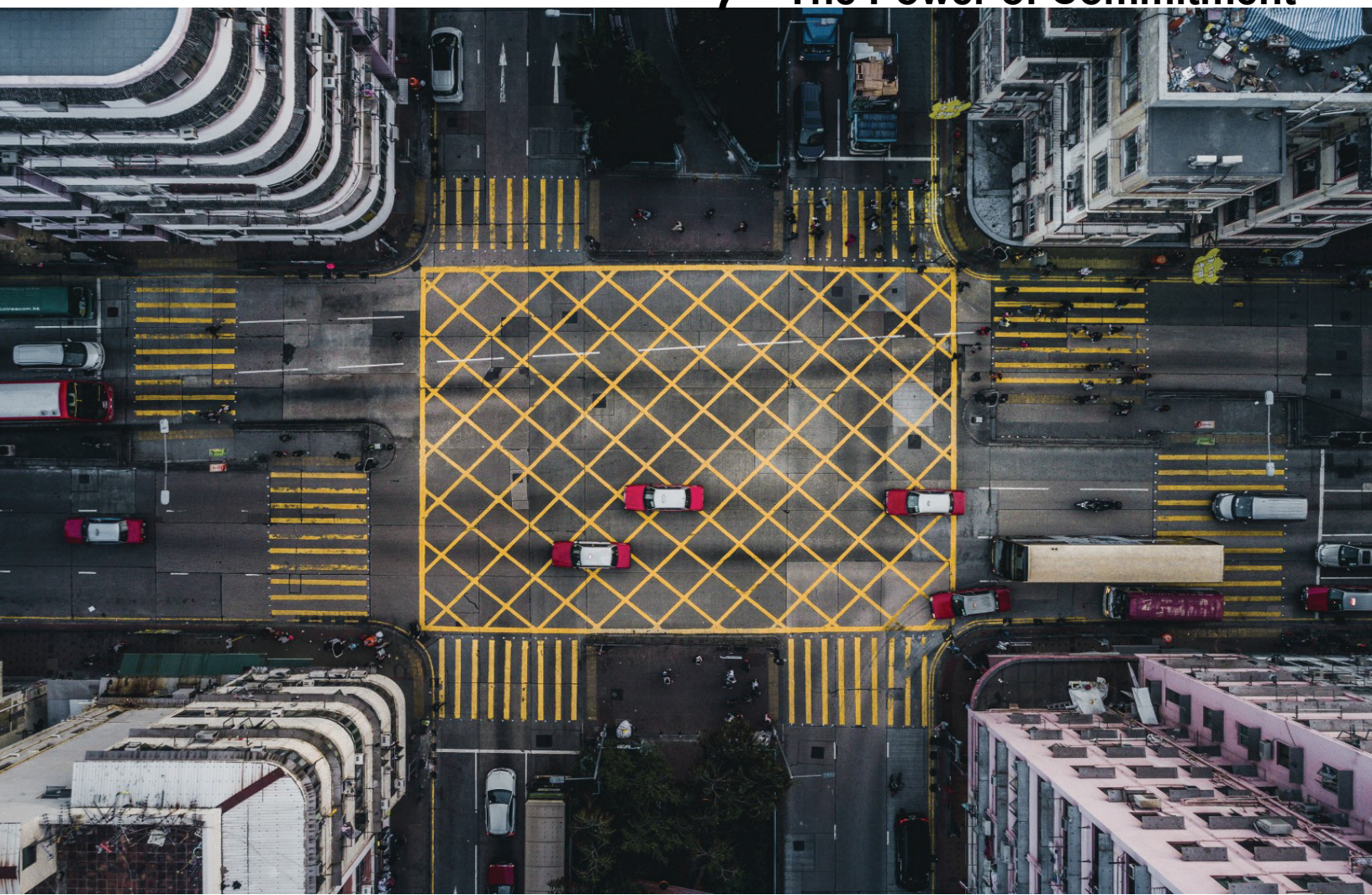
PFAS Initial Site Screening

RACER Nodular Facility – Saginaw, Michigan

RACER Trust, MID 041 793 340

06 November 2023

→ The Power of Commitment



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1. Introduction

This Report was prepared by GHD Services, Inc. (GHD) for Revitalizing Auto Communities Environmental Response Trust (RACER) to summarize the results of the 2023 Per- and Polyfluoroalkyl Substances (PFAS) and 1,4-dioxane sampling at the Former Nodular Industrial Lands (Site) in Saginaw, Michigan (Figure 1.1).

Resource Conservation and Recovery Act (RCRA) Corrective Action at the Site has been performed consistent with the Administrative Order on Consent (RCRA 05 2011 0023) (AOC) between RACER and the United States Environmental Protection Agency (U.S. EPA). The AOC was executed by RACER and U.S. EPA on September 29, 2011.

This sampling was conducted in response to U.S. EPA's request to sample for PFAS and 1,4-dioxane at the Site and in accordance with the Scope of Work to Complete PFAS and 1,4-Dioxane Sampling (Scope) (GHD, February 14, 2023) (Appendix A).

1.1 Request for PFAS and 1,4-dioxane Sampling

In an email dated February 4, 2021, U.S. EPA requested RACER sample and analyze groundwater for PFAS and 1,4-dioxane. In response to the request, GHD, on behalf of RACER submitted a response to comments document on March 18, 2021 identifying that data for 1,1,1-trichloroethane (1,1,1-TCA) can be used to determine whether future investigation of 1,4-dioxane may be necessary. The evaluation concluded that available data for the Site did not identify a significant source of 1,1,1-TCA or other chlorinated constituents that can co-occur with 1,4-dioxane, and as such additional evaluation and investigation of 1,4-dioxane is unnecessary. In the same response to comments document, RACER committed to evaluating known or potential uses/sources of PFAS at Nodular.

On April 28, 2021, GHD, on behalf of RACER, prepared and submitted a memorandum that presented "Potential Uses of Per- and Polyfluoroalkyl Substances (PFAS)" at the Site (part of Appendix A). The memorandum concluded that there is no known or suspected release of PFAS containing material to the environment that warrants investigation as part of RCRA Corrective Action. At that time (2021), RACER's position was that PFAS was not regulated by U.S. EPA as a hazardous waste, constituent, or substance, and therefore did not believe it was appropriate to expend funds to sample and analyze for PFAS at the Site.

On February 8, 2022, U.S. EPA presented RACER's proposed remedy for the Site to the Michigan Department of Environment, Great Lakes, and Energy's (EGLE's) Remediation Advisory Team. Out of the review, one of EGLE's requests was for RACER to evaluate groundwater for PFAS. In their November 14, 2022 letter, U.S. EPA also asked that RACER sample for PFAS at this Site.

After several communications with U.S. EPA in 2021 and 2022, and pursuant to U.S. EPA's November 14, 2022 letter, RACER agreed to sample groundwater for PFAS in order to allow for developing a path forward for U.S. EPA's remedy selection process. In addition, in the spirit of cooperation and to advance the RCRA Corrective action process, RACER agreed to sample for 1,4-dioxane.

2. Sampling Events

2.1 Sampling Activities

Following the agreement to sample for PFAS and 1,4-dioxane, RACER developed the Scope to evaluate the potential presence of PFAS and 1,4-dioxane. This initial Site screening of on-Site groundwater was completed through sampling of 7 on-Site monitoring wells. The select on-Site monitoring wells included: MW-04438R, MW-04336,

MW-05038, MW-05443, and MW-05452 which are on the downgradient property boundary; and MW-05036R and MW-8R which are in an area of former Plant operations. The Scope is presented in Appendix A.

Sampling was initiated on July 10 and 11, 2023 and included the collection of groundwater samples from 5 existing well locations (MW-04336, MW-04438R, MW-05036R, MW-05038, and MW-8R). These wells were previously sampled as part of the CMP monitoring and did not require redevelopment prior to sampling. MW-05452 was developed as it had not been sampled in recent years. MW-05443 was found to be damaged and could not be sampled. The riser and protective casing for MW-05443 was repaired on Friday August 4, 2023.

MW-05443 and MW-05452 were sampled on August 18, 2023. In addition, MW-05038 was also re-sampled to confirm results for the groundwater samples collected on July 10, 2023.

New high-density polyethylene (HDPE) tubing was used for sampling. Groundwater samples were collected using standard low flow procedures. Pumps utilized for the sampling did not contain Teflon, low-density polyethylene (LDPE), or Viton components. PFAS-specific sampling procedures presented in the Scope were followed. The sampling procedures are intended to prevent cross contamination of samples by PFAS. The sampling approach is consistent with U.S. EPA's Region 5 Analytical Services Branch PFAS Sampling Fact Sheet.

2.2 Analysis

In accordance with the Scope, the groundwater samples were analyzed by Eurofins Cleveland located in Barberton, Ohio, using the following methods:

– **PFAS**: EPA 537 modified ("Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)", Version 1.1, September 2009, EPA/600/R-08/092) for 28 PFAS analytes.
– **1,4-Dioxane**: SW 8270D SIM ("Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Third Edition, 1986, with subsequent revisions).

The Quality Assurance/Quality Control (QA/QC) samples collected included one field duplicate sample and one matrix spike/matrix spike duplicate (MS/MSD). A rinsate sample (equipment blank) was not required to be collected as dedicated sampling equipment was used.

One field blank and one trip blank per day were required in the Scope, however these QA/QC samples were inadvertently collected only on August 18, 2023.

The analytical reports are presented in Appendix B.

2.3 Validation

The analytical results for the samples collected on July 10 and 11, 2023 and on August 18, 2023 were validated and determined to be acceptable with qualifications. These qualifications are described in the final Data Validation Memorandum, provided in Appendix C, and they do not impact the usability of the data.

3. Results

3.1 Screening Levels

In accordance with the Scope, the groundwater results were compared to the EGLE Part 201 Residential Drinking Water and Groundwater-Surface Water Interface (GSI) Criteria (updated 10/12/2023). Quantities are listed in nanograms per litre (ng/L) which is equivalent to parts per trillion (ppt).

3.2 Results and Discussion

The analytical results for the samples collected in July and August 2023 are presented in Table 3.1 and Figure 3.1.

There were no exceedances of PFAS, with the exception of perfluorooctane sulfonic acid (PFOS) in one sample collected on July 10, 2023 at MW-05038. PFOS concentrations of 13 ng/L were detected in MW-05038 (and corresponding field duplicate sample), which marginally exceeded the EGLE GSI criteria of 12 ng/L. MW-05038 was resampled on August 18, 2023 and the concentration of PFOS was 12 ng/L. MW-05038 is located to the east of the Former Nodular Iron Facility, southeast of an off-Site storm retention pond but over 1,350 feet from the Saginaw River. There are no known PFAS sources in the vicinity. Further, PFOS was not detected in the monitoring wells on the western side of the Site (MW-04336 and MW-04438R), closer to the Saginaw River.

There were no exceedances of 1,4-dioxane.

4. Conclusion

No PFAS were detected above EGLE Residential Drinking Water at the Site.

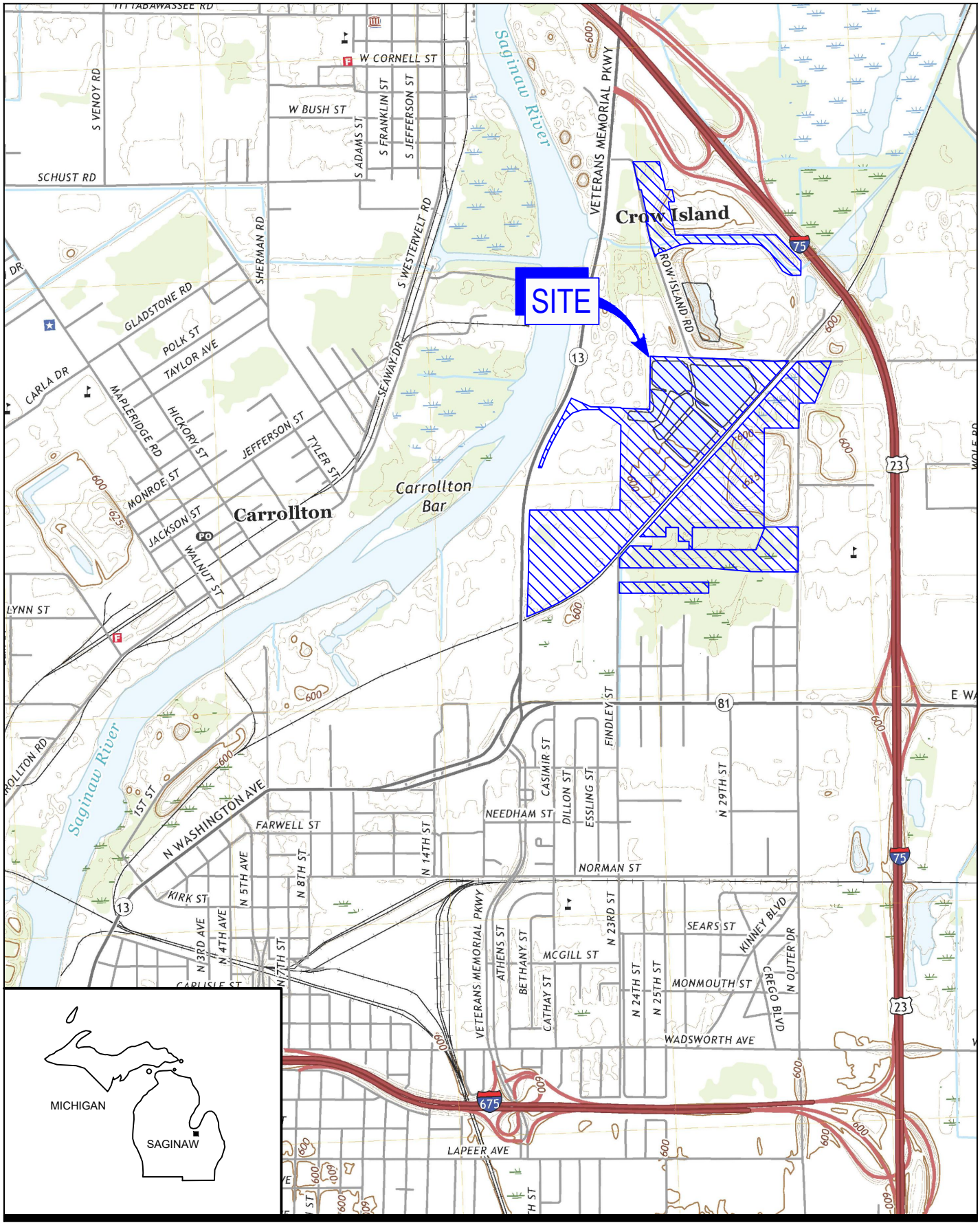
PFOS was detected at and just above EGLE GSI Criteria in MW-05038. MW-05038 is located over 1,350 feet from the Saginaw River.

1,4-dioxane was not detected above EGLE Residential Drinking Water or GSI Criteria at the Site.

Given the low level detected above and follow-up sample at the EGLE GSI Criteria at one location, no known PFAS sources in the vicinity, and distances from the Saginaw River, PFAS concentrations just above the EGLE GSI Criteria are not expected to be a surface water concern.

GHD believes that sufficient information has been collected and no further PFAS assessment is required.

Figures



0 1000 2000 ft

Coordinate System: MI83-SF

N

GHD

RACER
SAGINAW NODULAR INDUSTRIAL LAND
SAGINAW, MICHIGAN

Project No. **11208041**
Date **August 2023**

SITE LOCATION

FIGURE 1.1

Filename: N:\CA\Waterloo\Projects\662\11208041\Digital_Design\ACAD 2020\Figures\RPT006\11208041-GHD-00-00-RPT-EN-D102_WA-006.dwg
Plot Date: 15 August 2023 2:42 PM

Data Source: USGS QUADRANGLE MAP; SAGINAW, MI; 2019

LEGEND

APPROXIMATE LIMITS OF RACER PROPERTY AND LAND SUBJECT TO THE REQUIREMENTS OF RCRA CORRECTIVE ACTION

REGULATED WETLAND (PER WETLAND DELINEATION COMPLETED BY NISWANDER ENVIRONMENTAL [JULY 22, 2015])

0 250 500 1000 ft

Coordinate System: MI83-SF

N

GHD

RACER
SAGINAW NODULAR INDUSTRIAL LAND
SAGINAW, MICHIGAN

RACER PROPERTY

Project No. **11208041**
Date **August 2023**

FIGURE 1.2

Filename: N:\CA\Waterloo\Projects\662\11208041\Digital_Design\ACAD 2020\Figures\RPT006\11208041-GHD-00-00-RPT-EN-D103_WA-006.dwg
Plot Date: 31 August 2023 2:31 PM

Data Source: Source: U.S. Department of Agriculture (USDA) National Agriculture Imagery Program (NAIP). Acquired 3-Jul-2018.
TOPO - SANBORN, 1996.

MW-04757
MW-04257
MW-15457
PARKING
BUILDING
MW-05452
MW-04051
PARKING
MW-15450
MW-06951
RIVER
MW-04250
BUILDING
MW-03945
MW-04250R
MW-05443
MW-06445
SAGINAW
MW-04040
MW-04438R
MW-04438
MW-05038
MW-8
MW-8R
MW-04836R
MW-04836
MW-05036R
MW-05036
MW-04336
MW-04434
MW-04835
MW-04831

MW-04438R	07/11/2023
PFAS	
Hexafluoropropylene oxide dimer acid (HFPO-DA)	3.9 U
Perfluorobutane sulfonic acid (PFBS)	4.3
Perfluorohexane sulfonic acid (PFHxS)	1.9 U
Perfluorohexanoic acid (PFHxA)	3.2
Perfluorononanoic acid (PFNA)	0.41 J
Perfluorooctane sulfonic acid (PFOS)	1.9 U
Perfluorooctanoic acid (PFOA)	5.0
SVOC	
1,4-Dioxane	0.20 U

MW-05452	08/18/2023
PFAS	
Hexafluoropropylene oxide dimer acid (HFPO-DA)	4.0 U
Perfluorobutane sulfonic acid (PFBS)	0.67 J
Perfluorohexane sulfonic acid (PFHxS)	2.0 U
Perfluorohexanoic acid (PFHxA)	1.7 J
Perfluorononanoic acid (PFNA)	2.0 U
Perfluorooctane sulfonic acid (PFOS)	4.4
Perfluorooctanoic acid (PFOA)	3.8
SVOC	
1,4-Dioxane	0.96

MW-05038	07/10/2023	08/18/2023
PFAS		
Hexafluoropropylene oxide dimer acid (HFPO-DA)	3.8 U/3.9 U	3.9 U
Perfluorobutane sulfonic acid (PFBS)	0.69 J/0.84 J	0.80 J
Perfluorohexane sulfonic acid (PFHxS)	1.3 J/1.2 J	1.2 J
Perfluorohexanoic acid (PFHxA)	2.0 U/1.6 J	2.4
Perfluorononanoic acid (PFNA)	1.9 U/2.0 U	1.9 U
Perfluorooctane sulfonic acid (PFOS)	13 (b)/13 (b)	12
Perfluorooctanoic acid (PFOA)	4.9/5.2	5.1
SVOC		
1,4-Dioxane	3.9/4.1	2.8

MW-05443	08/18/2023
PFAS	
Hexafluoropropylene oxide dimer acid (HFPO-DA)	3.8 U
Perfluorobutane sulfonic acid (PFBS)	1.9 U
Perfluorohexane sulfonic acid (PFHxS)	1.9 U
Perfluorohexanoic acid (PFHxA)	2.9
Perfluorononanoic acid (PFNA)	1.9 U
Perfluorooctane sulfonic acid (PFOS)	1.9 U
Perfluorooctanoic acid (PFOA)	1.2 J
SVOC	
1,4-Dioxane	2.3

MW-8R	07/10/2023
PFAS	
Hexafluoropropylene oxide dimer acid (HFPO-DA)	3.8 U
Perfluorobutane sulfonic acid (PFBS)	1.4 J
Perfluorohexane sulfonic acid (PFHxS)	1.4 J
Perfluorohexanoic acid (PFHxA)	1.7 J
Perfluorononanoic acid (PFNA)	0.86 J
Perfluorooctane sulfonic acid (PFOS)	8.2
Perfluorooctanoic acid (PFOA)	4.8
SVOC	
1,4-Dioxane	5.2

MW-04336	07/11/2023
PFAS	
Hexafluoropropylene oxide dimer acid (HFPO-DA)	3.8 U
Perfluorobutane sulfonic acid (PFBS)	1.2 J
Perfluorohexane sulfonic acid (PFHxS)	1.9
Perfluorohexanoic acid (PFHxA)	4.8
Perfluorononanoic acid (PFNA)	1.9 U
Perfluorooctane sulfonic acid (PFOS)	1.9 U
Perfluorooctanoic acid (PFOA)	6.0
SVOC	
1,4-Dioxane	4.1

MW-05036R	07/11/2023
PFAS	
Hexafluoropropylene oxide dimer acid (HFPO-DA)	4.1 U
Perfluorobutane sulfonic acid (PFBS)	3.0
Perfluorohexane sulfonic acid (PFHxS)	1.3 J
Perfluorohexanoic acid (PFHxA)	3.2
Perfluorononanoic acid (PFNA)	0.60 J
Perfluorooctane sulfonic acid (PFOS)	6.1
Perfluorooctanoic acid (PFOA)	4.4
SVOC	
1,4-Dioxane	0.52

LEGEND

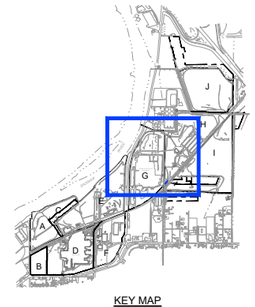
- APPROXIMATE PROPERTY BOUNDARY
- MW-8 ABANDONED MONITORING WELL LOCATION
- MW-06445 MONITORING WELL LOCATION
- MW-05443 PFAS AND 1,4-DIOXANE MONITORING LOCATION
- APPROXIMATE LOCATION OF FORMER PAINT SPRAY BUILDING AND FORMER NODULAR IRON PLANT WASTE PAINT SHED
- U NOT DETECTED AT THE ASSOCIATED REPORTING LIMIT
- J ESTIMATED CONCENTRATION
- EXCEEDANCE OF SCREENING LEVEL

IDENTIFICATION

MW-8R	07/10/2023
PFAS	
Hexafluoropropylene oxide dimer acid (HFPO-DA)	3.8 U
Perfluorobutane sulfonic acid (PFBS)	1.4 J
Perfluorohexane sulfonic acid (PFHxS)	1.4 J
Perfluorohexanoic acid (PFHxA)	1.7 J
Perfluorononanoic acid (PFNA)	0.86 J
Perfluorooctane sulfonic acid (PFOS)	8.2 — RESULT
Perfluorooctanoic acid (PFOA)	4.8
SVOC	
1,4-Dioxane	5.2 — PARAMETER

Screening Levels		
	EGLE Drinking Water	EGLE GSI
	a	b
Per/Polyfluoroalkyl Substances (PFAS) (ng/L)		
Hexafluoropropylene oxide dimer acid (HFPO-DA)	370	--
Perfluorobutane sulfonic acid (PFBS)	420	670,000
Perfluorohexane sulfonic acid (PFHxS)	51	210
Perfluorohexanoic acid (PFHxA)	400000	--
Perfluorononanoic acid (PFNA)	6	30
Perfluorooctane sulfonic acid (PFOS)	16	12
Perfluorooctanoic acid (PFOA)	8	170
Semi-Volatile Organic Compounds (SVOCs) (ug/L)		
1,4-Dioxane	7.2	280

a - EGLE Part 201 Residential Drinking Water Criteria (10/12/2023)
b - EGLE Part 201 Groundwater-Surface Water Interface (GSI) Criteria (10/12/2023)



KEY MAP

0 75 150 300 ft

Coordinate System: MI83-SF

N

GHD

RACER
SAGINAW NODULAR INDUSTRIAL LAND
SAGINAW, MICHIGAN

PFAS AND 1,4-DIOXANE RESULTS

Project No. **11208041**
Date **October 2023**

FIGURE 3.1

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Plot Date: 27 October 2023 3:36 PM

Tables

Table 3.1

PFAS and 1,4-Dioxane Results
RACER Nodular Industrial Land
Saginaw, MI
2023

Sample Location					MW-8R	MW-04336	MW-04438R	MW-05036R	MW-05038	MW-05038
Sample Identification					GW-11208041-071023-BW-003	GW-11208041-071123-BW-005	GW-11208041-071123-BW-004	GW-11208041-071123-BW-006	GW-11208041-071023-BW-001	GW-11208041-071023-BW-002
Sample Date					07/10/2023	07/11/2023	07/11/2023	07/11/2023	07/10/2023	07/10/2023
Sample Type										Duplicate
Parameters		Units	EGLE RDW	EGLE GSI						
Per/Polyfluoroalkyl Substances (PFAS)	**CAS**		a	b						
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propoxy]-propanoic acid (DONA)	919005-14-4	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	756426-58-1	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Fluorotelomer sulfonic acid (4:2)	757124-72-4	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Fluorotelomer sulfonic acid (6:2)	27619-97-2	ng/L	-	-	4.7 U	4.8 U	4.8 U	5.1 U	4.8 U	4.9 U
Fluorotelomer sulfonic acid (8:2)	39108-34-4	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Hexafluoropropylene oxide dimer acid (HFPO-DA)	13252-13-6	ng/L	370	-	3.8 U	3.8 U	3.9 U	4.1 U	3.8 U	3.9 U
N-Ethyl perfluorooctane sulfonamido acetic acid (N-EtFOSAA)	2991-50-6	ng/L	-	-	4.7 U	4.8 U	4.8 U	5.1 U	4.8 U	4.9 U
N-Methyl perfluorooctane sulfonamido acetic acid	2355-31-9	ng/L	-	-	4.7 U	4.8 U	4.8 U	5.1 U	4.8 U	4.9 U
Perfluorobutane sulfonic acid (PFBS)	375-73-5	ng/L	420	670,000	1.4 J	1.2 J	4.3	3.0	0.69 J	0.84 J
Perfluorobutanoic acid (PFBA)	375-22-4	ng/L	-	-	8.7	26	29	22	11	10
Perfluorodecanesulfonic acid (PFDS)	335-77-3	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Perfluorodecanoic acid (PFDA)	335-76-2	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Perfluorododecanoic acid (PFDoDA)	307-55-1	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Perfluoroheptane sulfonic acid (PFHpS)	375-92-8	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	0.27 J	0.28 J
Perfluoroheptanoic acid (PFHpA)	375-85-9	ng/L	-	-	1.3 J	2.5	3.4	2.5	1.1 J	1.1 J
Perfluorohexane sulfonic acid (PFHxS)	355-46-4	ng/L	51	210	1.4 J	1.9	1.9 U	1.3 J	1.3 J	1.2 J
Perfluorohexanoic acid (PFHxA)	307-24-4	ng/L	400,000	-	1.7 J	4.8	3.2	3.2	1.6 J	2.0 U
Perfluorononane sulfonic acid (PFNS)	474511-07-4	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Perfluorononanoic acid (PFNA)	375-95-1	ng/L	6	30	0.86 J	1.9 U	0.41 J	0.60 J	1.9 U	2.0 U
Perfluorooctane sulfonamide (FOSA)	754-91-6	ng/L	-	-	1.9 U	1.9 U	1.9 U	1.1 J	1.9 U	2.0 U
Perfluorooctanoic acid (PFOA)	335-67-1	ng/L	8	170	4.8	6.0	5.0	4.4	5.2	4.9
Perfluorooctane sulfonic acid (PFOS)	1763-23-1	ng/L	16	12	8.2	1.9 U	1.9 U	6.1	**13[b]**	**13[b]**
Perfluoropentane sulfonic acid (PFPeS)	2706-91-4	ng/L	-	-	1.9 U	0.63 J	1.9 U	2.0 U	1.9 U	2.0 U
Perfluoropentanoic acid (PFPeA)	2706-90-3	ng/L	-	-	1.9 U	4.2 J	2.7 J	2.0 U	1.9 U	1.2 J
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Perfluoroundecanoic acid (PFUnA)	2058-94-8	ng/L	-	-	1.9 U	1.9 U	1.9 U	2.0 U	1.9 U	2.0 U
Semi-Volatile Organic Compounds (SVOCs) SIM										
1,4-Dioxane	123-91-1	ug/L	7.2	280	5.2	4.1	0.20 U	0.52	3.9	4.1

Notes:

ng/L	Nanograms per Liter
U	Not detected at the associated reporting limit.
J	Estimated concentration.
SIM	Selective Ion Monitoring
13[b]	Boxed cell denotes exceedance of cleanup criteria screening level identified by superscript.
a	EGLE Part 201 Residential Drinking Water (RDW) Criteria (10/12/2023)
b	EGLE Part 201 Groundwater-Surface Water Interface (GSI) Criteria (10/12/2023)

Table 3.1

PFAS and 1,4-Dioxane Results
RACER Nodular Industrial Land
Saginaw, MI
2023

Sample Location					MW-05038	MW-05443	MW-05452
Sample Identification					GW-11208041-081823-BW-005	GW-11208041-081823-BW-001	GW-11208041-081823-BW-004
Sample Date			EGLE	EGLE	08/18/2023	08/18/2023	08/18/2023
Sample Type			RDW	GSI			
Parameters		Units					
Per/Polyfluoroalkyl Substances (PFAS)	**CAS**		a	b			
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	ng/L	-	-	1.9 U	1.9 U	2.0 U
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propoxy]-propanoic acid (DONA)	919005-14-4	ng/L	-	-	1.9 U	1.9 U	2.0 U
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	756426-58-1	ng/L	-	-	1.9 U	1.9 U	2.0 U
Fluorotelomer sulfonic acid (4:2)	757124-72-4	ng/L	-	-	1.9 U	1.9 U	2.0 U
Fluorotelomer sulfonic acid (6:2)	27619-97-2	ng/L	-	-	4.8 U	4.8 U	5.0 U
Fluorotelomer sulfonic acid (8:2)	39108-34-4	ng/L	-	-	1.9 U	1.9 U	2.0 U
Hexafluoropropylene oxide dimer acid (HFPO-DA)	13252-13-6	ng/L	370	-	3.9 U	3.8 U	4.0 U
N-Ethyl perfluorooctane sulfonamido acetic acid (N-EtFOSAA)	2991-50-6	ng/L	-	-	4.8 U	4.8 U	5.0 U
N-Methyl perfluorooctane sulfonamido acetic acid	2355-31-9	ng/L	-	-	4.8 U	4.8 U	5.0 U
Perfluorobutane sulfonic acid (PFBS)	375-73-5	ng/L	420	670,000	0.80 J	1.9 U	0.67 J
Perfluorobutanoic acid (PFBA)	375-22-4	ng/L	-	-	11	8.5 J	6.8 J
Perfluorodecanesulfonic acid (PFDS)	335-77-3	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluorodecanoic acid (PFDA)	335-76-2	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluorododecanoic acid (PFDoDA)	307-55-1	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluoroheptane sulfonic acid (PFHpS)	375-92-8	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluoroheptanoic acid (PFHpA)	375-85-9	ng/L	-	-	0.93 J	1.9 U	0.72 J
Perfluorohexane sulfonic acid (PFHxS)	355-46-4	ng/L	51	210	1.2 J	1.9 U	2.0 U
Perfluorohexanoic acid (PFHxA)	307-24-4	ng/L	400,000	-	2.4	2.9	1.7 J
Perfluorononane sulfonic acid (PFNS)	474511-07-4	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluorononanoic acid (PFNA)	375-95-1	ng/L	6	30	1.9 U	1.9 U	2.0 U
Perfluorooctane sulfonamide (FOSA)	754-91-6	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluorooctanoic acid (PFOA)	335-67-1	ng/L	8	170	5.1	1.2 J	3.8
Perfluorooctane sulfonic acid (PFOS)	1763-23-1	ng/L	16	12	12	1.9 U	4.4
Perfluoropentane sulfonic acid (PFPeS)	2706-91-4	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluoropentanoic acid (PFPeA)	2706-90-3	ng/L	-	-	1.8 J	2.8 J	1.4 J
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	ng/L	-	-	1.9 U	1.9 U	2.0 U
Perfluoroundecanoic acid (PFUnA)	2058-94-8	ng/L	-	-	1.9 U	1.9 U	2.0 U
Semi-Volatile Organic Compounds (SVOCs) SIM							
1,4-Dioxane	123-91-1	ug/L	7.2	280	2.8	2.3	0.96

Notes:

ng/L	Nanograms per Liter
U	Not detected at the associated reporting limit.
J	Estimated concentration.
SIM	Selective Ion Monitoring
13[b]	Boxed cell denotes exceedance of cleanup criteria screening level identified by superscript.
a	EGLE Part 201 Residential Drinking Water (RDW) Criteria (10/12/2023)
b	EGLE Part 201 Groundwater-Surface Water Interface (GSI) Criteria (10/12/2023)

Appendices

Appendix A

Scope of Work to Complete PFAS and 1,4-Dioxane Sampling

455 Phillip Street, Unit 100A
Waterloo, Ontario N2L 3X2
Canada
www.ghd.com

Your ref: EPA ID #MID 041 793 340
Our ref: 11208041-LTR-2

14 February 2023

Mr. Michael Beedle
Corrective Action Project Manager
U.S. EPA, Region 5
77 West Jackson Boulevard DW-8J
Chicago, Illinois
60604-3590

Scope of Work to Complete PFAS and 1,4-Dioxane Sampling
EPA ID #MID 041 793 340
RACER Nodular Facility – Saginaw Michigan

Dear Michael

This letter presents the Scope of Work (Scope) to complete Per and Polyfluoroalkyl (PFAS) and 1,4-dioxane sampling at the Revitalizing Auto Communities Environmental Response Trust (RACER) Former Nodular Industrial Lands (Site) in Saginaw, Michigan. This Scope was prepared in response to United States Environmental Protection Agency's (U.S. EPA's) email dated February 4, 2021, which requested RACER sample and analyze groundwater for PFAS and 1,4-dioxane and U.S. EPA's November 14, 2022, letter, which also asked that RACER sample for PFAS at this Site. The objective of this proposed Scope is to allow for evaluation of the presence of PFAS or 1,4-dioxane at the Site and the need for any follow-up sampling.

The following figures, tables and Attachments were prepared in support of the Scope:

Figure 1	Proposed Sampling Locations
Table 3.1	Analytical Methods
Table 3.2	Parameter List and Laboratory Limits
Table 3.3	Laboratory Precision and Accuracy Limits
Table 3.4	Summary of Sampling and Analysis Program
Table 3.5	Container, Preservation, Shipping, and Packaging Requirements
Attachment A	Potential Uses of Per- and Polyfluoroalkyl Substances (PFAS) Memorandum
Attachment B	Sampling Procedures
Attachment C	Laboratory Standard Operating Procedures
Attachment D	Scope of Work Approval Form

1. Background

The initial request to sample and analyze groundwater for PFAS and 1,4-dioxane was received via email from the United States Environmental Protection Agency (U.S. EPA) on February 4, 2021. In response to the request, GHD, on behalf of RACER, prepared and submitted a memorandum on April 28, 2021 (Attachment A) that presented "Potential Uses of Per- and Polyfluoroalkyl Substances (PFAS)" at the Site. The anecdotal potential use of PFAS at the Site was associated with the former Paint Spray Building (or Paint Storage Building), which contained a small paint spray booth. Based on painting operations identified at other locations at the Larger Facility, it is believed that the former Paint Spray Building was used for painting auto components for aesthetic purposes and/or for the storage of paints used for building/property maintenance purposes. Figure 1 displays the approximate location of the former Paint Spray Building. The memorandum concluded that there is no known or suspected release of PFAS containing material to the environment that warrants investigation as part of the Resource Conservation and Recovery Act (RCRA). At that time, RACER's position was that PFAS was not regulated by U.S. EPA as a hazardous waste, constituent, or substance, and therefore did not believe it was appropriate to expend funds to sample and analyze for PFAS at the Site.

In addition to U.S. EPA's request to sample groundwater for PFAS and 1,4-dioxane, on February 8, 2022, U.S. EPA presented RACER's proposed remedy for the Site to the Michigan Department of Environment, Great Lakes, and Energy's (EGLE's) Remediation Advisory Team. Out of the review, one of EGLE's requests was for RACER to evaluate groundwater for PFAS.

After several communications with U.S. EPA in 2021 and 2022, and pursuant to U.S. EPA's November 14, 2022, letter, RACER has agreed to sample groundwater for PFAS in order to allow for developing a path forward for U.S. EPA's remedy selection process. In addition, in the spirit of cooperation and to advance the RCRA Corrective action process, RACER has agreed to sample for 1,4-dioxane.

2. Proposed PFAS and 1,4-Dioxane Sampling Activities

To evaluate the potential presence of PFAS and 1,4-dioxane, an initial Site screening of on-Site groundwater will be completed through sampling of select on-Site monitoring wells. The select on-Site monitoring wells include: MW-04438R, MW-04336, MW-05038, MW-05443, and MW-05452 which are on the downgradient property boundary; and MW-05036R and MW-8R which are in an area of former Plant operations.

The locations of the select monitoring wells proposed for sampling are shown on Figure 1.

2.1 Sample Collection Procedure

Groundwater samples will be collected using standard low flow procedures. Only new/unused high-density polyethylene (HDPE) and silicon tubing will be utilized during sampling. Pumps utilized for the sampling will not contain Teflon, low-density polyethylene (LDPE), or Viton components. PFAS sampling procedures are presented in Attachment B (GHD Field Training Manual – Section 7.0 – Water Sampling and PFAS Addendum). The sampling procedures are intended to prevent cross contamination of samples by PFAS. In addition, due to the sensitivity of the PFAS analysis, the PFAS Sampling Checklist presented in Attachment B will be utilized.

Development/purge water, excess sampling water, and decontamination water/fluids will be containerized in 55-gallon drum(s).

3. Analytical Methods and Quality Control Samples

Eurofins TestAmerica will be the laboratory company supporting the environmental sample analyses for this project utilizing their facilities in West Sacramento, California (PFAS, 1,4-dioxane).

3.1 Laboratory Analytical Methods

Groundwater samples will be analyzed for specified chemical parameters by the project laboratory. The methods that will be used for sample analyses are presented in Table 3.1. Specific analytes, including 28 PFAS, and targeted quantitation limits for chemical parameters are presented in Table 3.2. The applicable Michigan Part 201 laboratory limits and criteria are also included in Table 3.2. The precision and accuracy criteria for laboratory analyses are provided in Table 3.3. The criteria listed in Tables 3.2 and 3.3 provide the data quality objectives for the project scope. Analytical data will be validated according to the procedures outlined in Section 3.2.

3.2 Quality Assurance/Quality Control Procedures

3.2.1 Field Quality Assurance/Quality Controls

Field Quality Assurance/Quality Control Procedures (QA/QC) samples will be collected during field sampling include equipment blank samples to determine the existence and magnitude of sample contamination resulting from ambient conditions or sampling procedures, and field duplicate sample to assess the overall precision of the sampling and analysis events. The specific QA/QC samples and collection frequency are summarized in Table 3.4. Equipment blank samples will be collected at a frequency of one per day, following sampling equipment decontamination procedures. Equipment blank samples will be collected by routing laboratory-provided deionized water through decontaminated sampling equipment. Equipment blank samples will be analyzed to check procedural contamination and/or ambient conditions and/or sample container contamination at the Site that may cause sample contamination. However, equipment blank samples will not be required for samples collected using dedicated sampling equipment or disposable sampling equipment. One field blank will be collected per day with PFAS samples.

Field duplicate samples will be collected at a minimum frequency of 1 per 10 or fewer investigative samples. Field duplicate samples will be analyzed to assess the precision of the field sample collection procedures.

Sufficient sample volume will be provided to the laboratory (as necessary) for MS/MSD analyses. The data from MS/MSD analyses provide an indication of the precision and accuracy of the analytical method relative to the sample matrix. Samples for MS/MSD analysis will be designated at a minimum frequency of 1 per 20 or fewer samples.

Samples will be collected and packed in laboratory supplied containers and transported in accordance with the container, preservation, shipping, and packaging requirements presented in Table 3.5.

3.2.2 Laboratory Quality Assurance/Quality Control

Laboratory QA/QC requirements for the analysis of groundwater samples includes analyzing method blanks, initial calibration verification standards, continuing calibration verification standards, MS/MSD samples, and laboratory Control Samples (LCS). The analysis frequency for these QA/QC samples is identified in the applicable laboratory SOP provided in Attachment C. The acceptance criteria for these QC checks will be consistent with the analytical methods provided in Table 3.1 and applicable laboratory SOP.

3.2.3 Laboratory Report Deliverables

Laboratory reports for samples collected will consist of the following data deliverables:

1. Case Narrative:
    i. Date of issuance
    ii. Project name and number
    iii. Any deviations from intended analytical strategy
    iv. Condition of samples “as received”
    v. Discussion of whether or not sample holding times were met
    vi. Discussion of technical problems or other observations that may have created analytical difficulties
    vii. Discussion of any laboratory quality control checks that failed to meet project criteria
2. Chemistry Data Package
    i. Dates of sample collection, receipt, preparation, and analysis
    ii. Cross-reference of laboratory to project sample identification numbers
    iii. Description of data qualifiers used
    iv. Methods of sample preparation and analysis
    v. Sample results in tabular format
    vi. Method blank data, surrogate data, LCS data, duplicate sample data, MS/MSD data,
    vii. Fully executed chain-of-custody document

3.3 Data Review and Validation

Upon receipt of the final data packages from the project laboratory the data will be reviewed and validated. The data review will evaluate the final analytical results, holding time period compliance, equipment blank sample data, field duplicate sample data, method blank data, LCS data, laboratory duplicate data, surrogate compound spike data, and MS/MSD sample data. Validation of the data will consist of evaluating the QA/QC data based on the applicable review criteria specified in “National Functional Guidelines for Inorganic Superfund Methods Data Review”, EPA 542-R-20-006, November 2020 and “National Functional Guidelines for Organic Superfund Methods Data Review”, EPA 540-R-20-005, November 2020. The results of the data review and validation process will be documented in memoranda that identify all limitations on the usability of the analytical data.

4. Reporting

Following receipt of the groundwater analytical results, a letter report will be prepared summarizing the completed field program, results, conclusions, and recommendations. Sample results will be compared to the August 2020 Michigan Drinking Water Maximum Contaminant Levels (MCLs) and the Michigan Department of Environment, Great Lakes, and Energy (EGLE) Part 201 Groundwater-Surface Water Interface (GSI) Criteria shown below.

Michigan Drinking Water MCLs and GSI Criteria for PFAS

Specific PFAS	Drinking Water MCL (parts per trillion)	GSI Criteria
PFNA	6	-
PFOA	8	12,000
PFHxA	400,000	-
PFOS	16	12

Specific PFAS	Drinking Water MCL (parts per trillion)	GSI Criteria
PFHxS	51	-
PFBS	420	
HFPO-DA	370	

For 1,4-dioxane, the analytical results will be compared to the EGLE Part 201 Groundwater Residential Drinking Water and GSI Generic Cleanup Criteria of 7.2 parts per billion (ppb) and 280 ppb, respectively.

A letter report will be submitted to U.S. EPA and will include recommendations on next steps, if required. In accordance with GHD's ISO 9001:2008 accreditation, all records will be stored in GHD's controlled filing system for a minimum 10-years including a backup and retention program.

5. Scope of Work Approval Form

If this scope of work is acceptable, please sign and return the form provided in Attachment D.

Should you have any questions or comments, please do not hesitate to call.

Regards

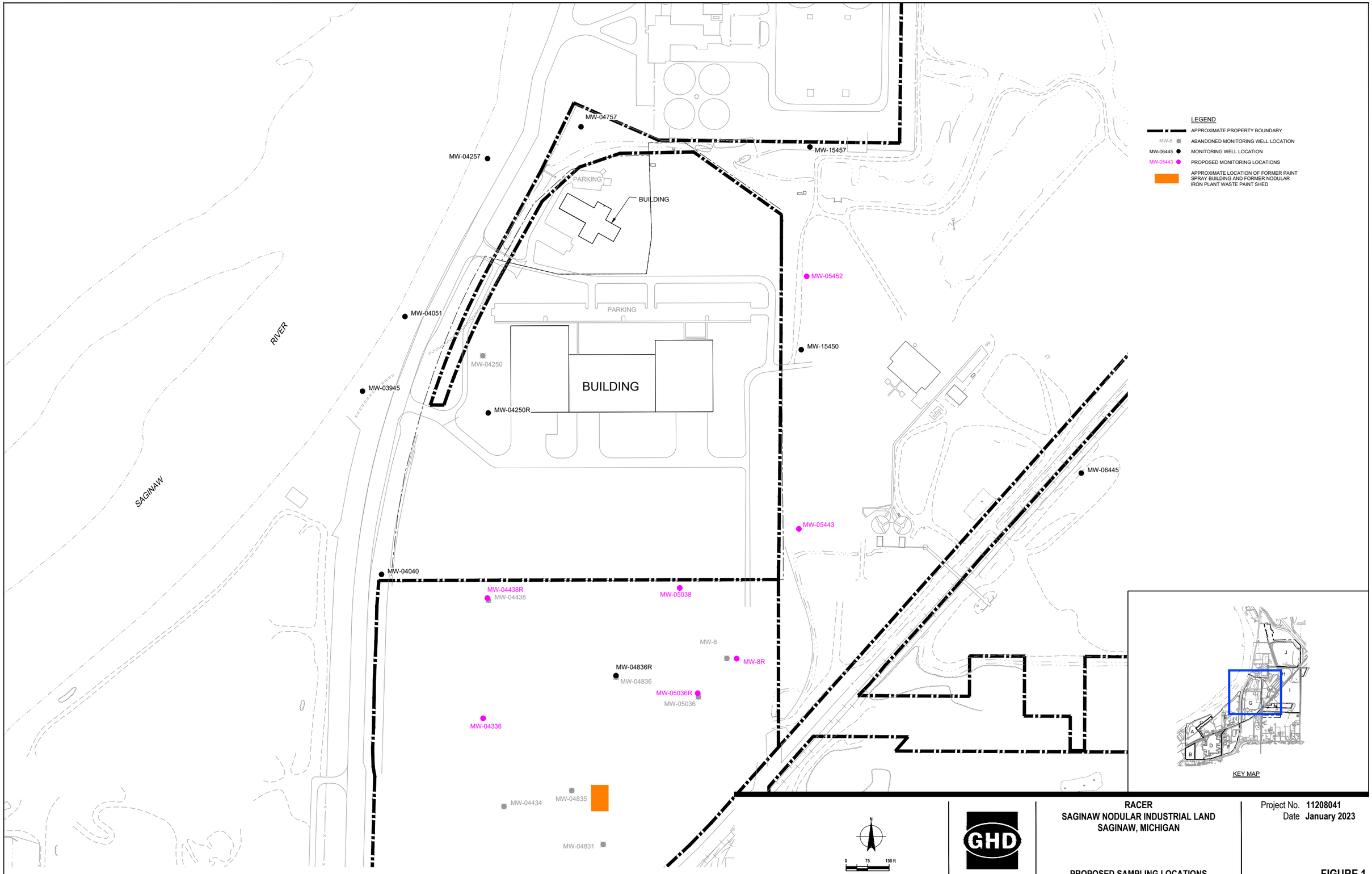
John-Eric Pardys
Project Manager

+1 519 340-4316
john-eric.pardys@ghd.com

Encl.

Copy to: Dave Favero (RACER)

Figure



RACER
SAGINAW NODULAR INDUSTRIAL LAND
SAGINAW, MICHIGAN

Project No. **11208041**
Date **January 2023**

PROPOSED SAMPLING LOCATIONS

FIGURE 1

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Tables

Table 3.1

Analytical Methods
PFAS and 1,4-Dioxane Sampling Plan
RACER Nodular Facility
Saginaw, Michigan

Parameter	Analytical Method[1]
Water Samples	
PFAS	EPA 537 modified
1,4-Dioxane	SW 8270D SIM

Notes:

[1] Method References:

EPA - "Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)", Version 1.1, September 2009, EPA/600/R-08/092

SW - "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Third Edition, 1986, with subsequent revisions

PFAS - Per- and Polyfluoroalkyl Substances

SIM - Selective Ion Monitoring

Table 3.2

Parameter List and Laboratory Limits
PFAS and 1,4-Dioxane Sampling Plan
RACER Nodular Facility
Saginaw, Michigan

PARAMETER	CHEMICAL ABSTRACT SERVICE NUMBER	EGLE Drinking water health-based MCL (ng/L)	EGLE GSI (ng/L)	AQUEOUS REPORTING LIMIT[1] (ng/L)	METHOD DETECTION LIMIT[2] (ng/L)
Perfluorobutanoic acid (PFBA)	375-22-4			5	2.4
Perfluoropentanoic acid (PFPeA)	2706-90-3			2	0.49
Perfluorohexanoic acid (PFHxA)	307-24-4	400000		2	0.58
Perfluoroheptanoic acid (PFHpA)	375-85-9			2	0.25
Perfluorooctanoic acid (PFOA)	335-67-1	8	12000	2	0.85
Perfluorononanoic acid (PFNA)	375-95-1	6		2	0.27
Perfluorodecanoic acid (PFDA)	335-76-2			2	0.31
Perfluoroundecanoic acid (PFUnA)	2058-94-8			2	1.1
Perfluorododecanoic acid (PFDoA)	307-55-1			2	0.55
Perfluorotridecanoic acid (PFTriA)	72629-94-8			2	1.3
Perfluorotetradecanoic acid (PFTeA)	376-06-7			2	0.73
Perfluorobutanesulfonic acid (PFBS)	375-73-5	420		2	0.2
Perfluoropentanesulfonic acid (PFPeS)	2706-91-4			2	0.3
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	51		2	0.57
Perfluoroheptanesulfonic Acid (PFHpS)	375-92-8			2	0.19
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	16	12	2	0.54
Perfluorononanesulfonic acid (PFNS)	68259-12-1			2	0.37
Perfluorodecanesulfonic acid (PFDS)	335-77-3			2	0.32
Perfluorooctanesulfonamide (FOSA)	754-91-6			2	0.98
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31-9			5	1.2
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50-6			5	1.3
4:2 FTS	757124-72-4			2	0.24
6:2 FTS	27619-97-2			5	2.5
8:2 FTS	39108-34-4			2	0.46
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	919005-14-4			2	0.4
HFPO-DA (GenX)	13252-13-6	370		4	1.5
F-53B Major	756426-58-1			2	0.24
F-53B Minor	763051-92-9			2	0.32
1,4-Dioxane	123-91-1	7.2 ug/l	280 ug/l	0.2 ug/l	0.1 ug/l

Notes:

[1] - Please note that these are targeted quantitation limits and are presented for guidance only. Actual quantitation limits are highly matrix dependent and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes.

[2] - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.

Table 3.3

Laboratory Precision and Accuracy Limits
PFAS Sampling Plan
RACER Nodular Facility
Saginaw, Michigan

Method	Analyte	LCS - LCL %R	LCS - UCL %R	LCS - RPD	MS - LCL %R	MS - UCL %R	MS - RPD
537 modified PFAS	Perfluorobutanoic acid (PFBA)	76	136	30	76	136	30
	Perfluoropentanoic acid (PFPeA)	71	131	30	71	131	30
	Perfluorohexanoic acid (PFHxA)	73	133	30	73	133	30
	Perfluoroheptanoic acid (PFHpA)	72	132	30	72	132	30
	Perfluorooctanoic acid (PFOA)	70	130	30	70	130	30
	Perfluorononanoic acid (PFNA)	75	135	30	75	135	30
	Perfluorodecanoic acid (PFDA)	76	136	30	76	136	30
	Perfluoroundecanoic acid (PFUnA)	68	128	30	68	128	30
	Perfluorododecanoic acid (PFDoA)	71	131	30	71	131	30
	Perfluorotridecanoic acid (PFTrDA)	71	131	30	71	131	30
	Perfluorotetradecanoic acid (PFTeA)	70	130	30	70	130	30
	Perfluorobutanesulfonic acid (PFBS)	67	127	30	67	127	30
	Perfluoropentanesulfonic acid (PFPeS)	66	126	30	66	126	30
	Perfluorohexanesulfonic acid (PFHxS)	59	119	30	59	119	30
	Perfluoroheptanesulfonic Acid (PFHpS)	76	136	30	76	136	30
	Perfluorooctanesulfonic acid (PFOS)	70	130	30	70	130	30
	Perfluorononanesulfonic acid (PFNS)	75	135	30	75	135	30
	Perfluorodecanesulfonic acid (PFDS)	71	131	30	71	131	30
	Perfluorooctanesulfonamide (FOSA)	73	133	30	73	133	30
	NMeFOSAA	76	136	30	76	136	30
	NEtFOSAA	76	136	30	76	136	30
	4:2 FTS	79	139	30	79	139	30
	6:2 FTS	59	175	30	59	175	30
	8:2 FTS	75	135	30	75	135	30
	4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	79	139	30	79	139	30
	HFPO-DA (GenX)	51	173	30	51	173	30
	9Cl-PF3ONS	75	135	30	75	135	30
	11Cl-PF3OUdS	54	114	30	54	114	30
SW 8270D SIM	1,4-Dioxane	40	140	20	40	140	20

Notes:

LCL	- Lower Control Limit
LCS	- Lab Control Sample
MS	- Matrix Spike
UCL	- Upper Control Limit
PFAS	- Per- and Polyfluoroalkyl Substances
%R	- Percent Recovery
RPD	- Relative Percent Difference
SIM	- Selective Ion Monitoring

Table 3.4

Summary of Sampling and Analysis Program
PFAS and 1,4-Dioxane Sampling Plan
RACER Nodular Facility
Saginaw, Michigan

Investigation Activity	Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Samples	Quality Control Samples			MS/MSD (2)	Total
					Equipment Blanks(1)	Field Blank	Field Dup		
PFAS Sampling	water	pH, temperature, conductivity, dissolved oxygen, oxygen reduction potential, turbidity	PFAS, 1,4-Dioxane	6	1	1 - PFAS only	1	1	11

Notes:

(1) - Equipment blank will be collected in the event that disposable sampling equipment is not being used.

(2) - Matrix Spike/Matrix Spike duplicate (MS/MSD) analyses are required for samples submitted for analyses are to be analyzed at a frequency of one per group of twenty (20) or fewer investigative samples for the activities detailed above. The MS/MSD is a pair a of two samples--spike and spike duplicate.

PFAS -- Per- and Polyfluoroalkyl Substances

Table 3.5

Container, Preservation, Shipping and Packaging Requirments
PFAS and 1,4-Dioxane Sampling Plan
RACER Nodular Facility
Saginaw, Michigan

Analyses	Sample Containers	Preservation	Maximum Holding Time from Sample Collection1	Volume of Sample	Shipping	Normal Packaging
Water						
PFAS	2 x 250 mL HDPE bottles	Iced, 0-6° C	14 days to extraction; 28 days from extraction to analysis	500 ml	Overnight	Bubble-wrap
1,4-Dioxane	2 x 250 mL amber glass	Iced, 0-6° C	7 days to extraction; 40 days from extraction to analysis	500 ml	Overnight	Bubble-wrap

Notes:

PFAS -- Per- and Polyfluoroalkyl Substances

Attachments

Attachment A

Potential Uses of Per- and Polyfluoroalkyl Substances (PFAS) Memorandum

Memorandum

April 28, 2021

To:	David Favero	Ref. No.:	11208041
From:	Nicole Knezevich, Francis C. Ramacciotti/kf/4	Tel:	215-853-3281
cc:	John-Eric Pardys, GHD		

Subject: **Potential Uses of Per- and Polyfluoroalkyl Substances (PFAS)**
Former Nodular Industrial Land
Saginaw, Michigan

1. Introduction

This Memorandum has been prepared by GHD Services, Inc. (GHD) for Revitalizing Auto Communities Environmental Response Trust (RACER) to summarize the information gathered on the potential uses of per- and polyfluoroalkyl substance (PFAS) at the Former Nodular Industrial Land (Site) in Saginaw, Michigan. Prior to the General Motors Corporation (GMC) bankruptcy, the Site was a portion of the larger Saginaw Metal Casting Operations (SMCO) Facility (Larger Facility) that encompassed approximately 700 acres. Following the bankruptcy, RACER retained approximately 315 acres and General Motors, LLC now owns the remaining portion (Grey Iron Plant). The Site includes a portion of the Former Nodular Iron Plant Area (Investigative Unit [IU] G), the Former Waste Water Treatment System and Stormwater Ditch (IU H), a portion of the former Classified Sand Staging Area (IU I), and an un-impacted strip of land the north of IU J.

PFAS has been termed an *emerging contaminant* because it presents unique issues associated with its distribution in the environment and the potential for adverse effects on ecological and human health. Because it is not a "hazardous constituent" for regulatory purposes according to United States Environmental Protection Agency (USEPA), it was not assessed in the Description of Current Conditions Report (DOCC) (EMCON, 1995)[1] or the DOCC Addendum Report (EMCON, 1997)[2] for the potential for a significant release from current or historical operations. This evaluation, as summarized in this memorandum, identified one potential historical PFAS-related manufacturing source. This supplemental information gathering was performed to ensure adequacy of the investigation and evaluation performed as part of the Resource Conservation and Recovery Act (RCRA) Corrective Action for the Facility. This Memorandum includes GHD's approach and findings regarding potential historical uses and/or releases, if any, from operations at the Site.

1 EMCON, 1995. RCRA Facility Investigation – Task 1: Description of Current Conditions Report – August 1995, General Motors Corporation, Saginaw Metal Casting Operations, Saginaw, Michigan, MID 041 793 340.

2 EMCON, 1997. RCRA Facility Investigation – Task 1: Description of Current Conditions Report Addendum – August 1995, General Motors Corporation, Saginaw Metal Casting Operations, Saginaw, Michigan, MID 041 793 340.

2. Approach

GHD conducted a review of historical documents for the Larger Facility to identify any former operations or processes at the Site that may have reasonably used PFAS containing materials. This evaluation was performed by reviewing historical uses identified in the DOCC and reviewing additional historical documents that were identified during this process (e.g., closure reports). The approach taken to evaluate potential uses of PFAS was consistent with that used to develop the DOCC and assessed general industry operations described in the literature as potentially using PFAS-containing materials.

Generally, there are limited uses of PFAS containing materials associated with casting of nodular iron for auto components at a foundry. Using the Interstate Technology & Regulatory Council (ITRC) History and Use of PFAS Fact Sheet (Section 4) (2020)[3], GHD identified four potential sources of PFAS in general/typical vehicle production, which are as follows:

- Textile and leather coatings for carpets and upholstery
- Coating for wires and cables
- Fume suppressants in metal plating
- Fire suppression systems that could contain aqueous film-forming foam (AFFF)
- PFAS may also be found in wipeable and anti-adhering paints and cleaning products (USEPA, 2021)[4]

However, many of the potential sources of PFAS in general/typical vehicle production are not applicable to the Site or Larger Facility. For instance, no carpets and upholstery nor wires and cables were manufactured or assembled and no metal plating operations occurred. In addition, no fire training or fires were or have been documented at the Larger Facility. Small painting operations for engine blocks and another auto components historically occurred at the Site and within the Larger Facility. Paint is sometimes attributed as a potential source of PFAS.

3. History of the Larger Facility

3.1 Larger Facility Operations

The Grey Iron Plant was initially constructed in 1906 and has been used for the manufacturing of automobile parts as well as mortar shell casings and aircraft engine parts for various war efforts. The former Nodular Iron Plant was not constructed until 1964, prior to which, the land was undeveloped. RACER's portion of the former Nodular Iron Plant is approximately 1,300 feet by 1,800 feet, for a total area of approximately 54 acres (Figure 1). The former Nodular Iron Plant and associated ancillary buildings were operational between 1966 and 1987 and cast auto components from nodular iron. The manufacturing process consisted

[3] ITRC, 2020. History and Use of Per- and Polyfluoroalkyl Substances (PFAS) found in the Environment. Updated August 2020. Available at: https://pfas-1.itrcweb.org/wp-content/uploads/2020/10/history_and_use_508_2020Aug_Final.pdf.

[4] USEPA, 2021. Basic Information on PFAS. Available at: https://www.epa.gov/pfas/basic-information-pfas [Accessed March, 2021].

of core mold making through the hot process, scrap iron melting, addition of calcium carbide to the molten iron, molten iron pouring, and rough cleaning and finishing.

The Site also contained a wastewater treatment plant (WWTP), which commenced operations in 1978. The WWTP was renovated and improved in 1988. The area consisted of classifiers, mixing tanks, flocculators, treatment tanks, and primary and secondary settling basins. The WWTP received water mostly from the Grey Iron Plant and recycled the water back to the Grey Iron Plant, located on the adjacent property, to provide water necessary for foundry operations. The Grey Iron Plant cast grey iron for engine blocks, cylinder heads, camshafts, brake rotors, brake drums, water pump housings, intake manifolds, flywheels using the same general manufacturing process as the former Nodular Iron Plant. The recycled water was pumped to emission units and dust collectors throughout the Grey Iron Plant to recover particulates from foundry emissions or used to quench the slag generated by the cupola melting facilities before being pumped back to the WWTP. The WWTP stopped receiving waters in September 2010 and was decommission/demolished in 2015/2016. No documented releases were identified at the WWTP and there were no PFAS sources identified from the historical activities at the Grey Iron Plant.

3.2 Potential PFAS Usage

Based on GHD's review, operations at the Site did not involve any type of leather or textile coating, metal plating or etching, or wire coating. No known past or current fire training areas were identified nor were there any known or potential/suspected fire-suppression systems containing AFFFs.

PFAS has also been associated with certain weather resistant stains and paints, such as top coats that may have been applied to vehicles. A former Paint Spray Building (or Paint Storage Building) was located southeast of the main former Nodular Iron Plant, which contained a small spray booth/paint hood and paint storage room. Therefore, the former Paint Spray Building (or Paint Storage Building) was considered a potential source of PFAS based on this anecdotal association and further evaluated below. By extension, hazardous waste drum storage areas that handled the disposal of paint residues (former Nodular Waste Paint Shed RCRA Hazardous Waste Drum Storage Area [Area of Interest (AOI) G.3]) could have also hypothetically been a potential source of PFAS. The former Nodular Waste Paint Shed RCRA Hazardous Waste and Drum Storage Area was a small (10 feet by 10 feet) covered, fenced, curbed concrete pad used to store hazardous waste drums located to the east of the former Paint Spray Building.

Based on GHD's review, potential uses of PFAS were identified in relation to the Former Paint Spray Building (or Paint Storage Building) and associated waste storage area (AOI G.3). The area where PFAS may have been used is described below and shown on the former Nodular Iron Plant (IUG G) (Figure 1) that includes the AOIs identified in the DOCC.

Former Paint Spray Building (or Paint Storage Building) and Associated Waste Storage Area (AOI G.3)

A former Paint Spray Building (or Paint Storage Building) was located southeast of the former Nodular Iron Plant main building. Available information indicates that the former Paint Spray Building housed a 30 foot by 45 foot paint storage room. Paint was stored in the room in 55 gallon drums and was either mixed for spray application in a paint hood/small spray booth located inside the room or transferred to smaller containers for

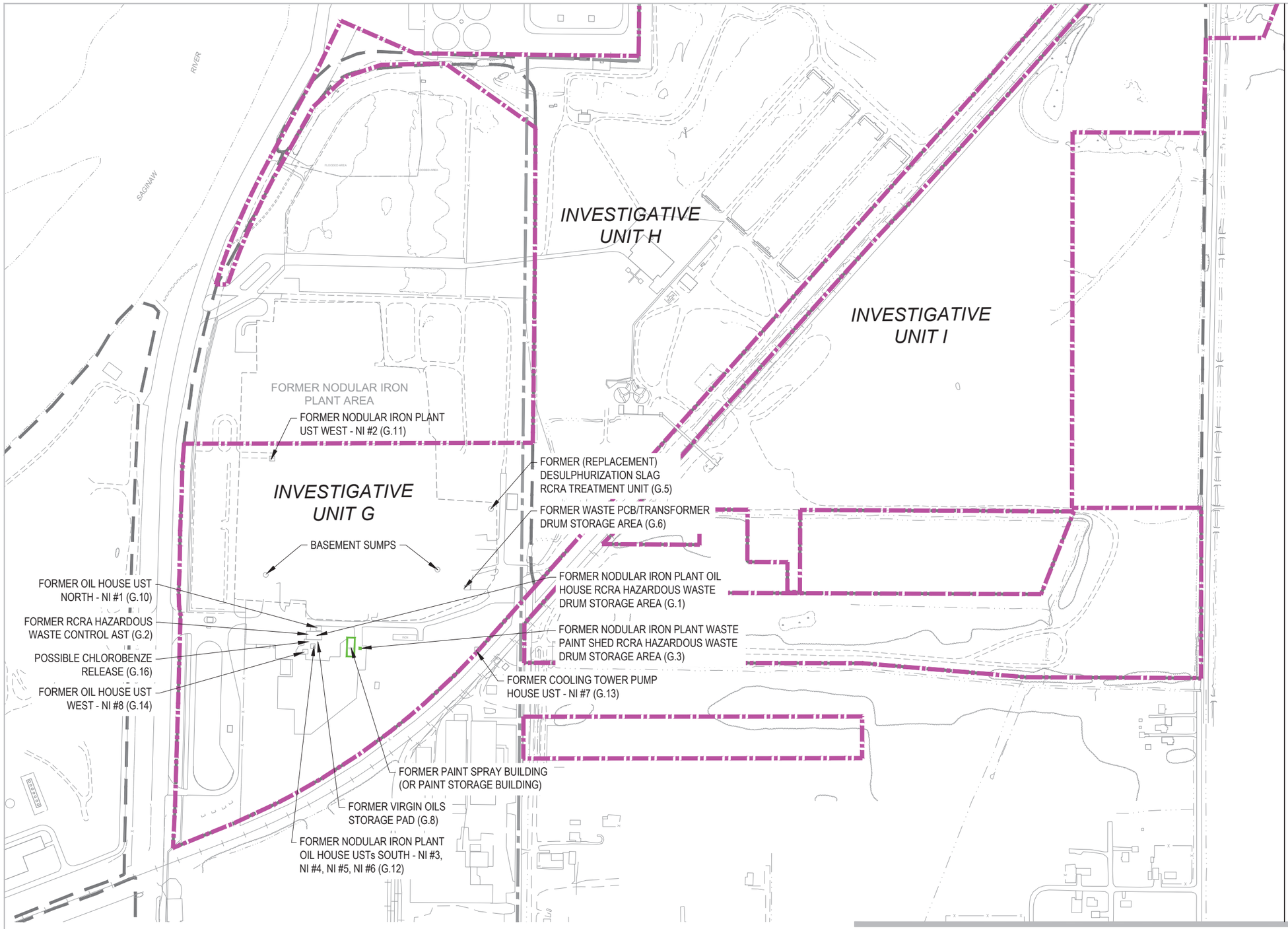
use within the plant. A manhole was located in the center of the room that was sealed and welded shut. Safety data sheets associated with paint waste at the Former Nodular Waste Paint Shed RCRA Hazardous Drum Storage Area (AOI G.3) indicate that the waste paint consisted of specialty lacquers and does not identify PFAS-related components. This paint was most likely used for batch-painting engines or other auto components for aesthetic purposes similar to painting operations that occurred at other buildings located at the Larger Facility. Based on GHD's review, engine paint must be able to able to withstand extremely high temperatures; therefore, ceramic or enamel paints were primarily used. PFAS was not identified in similar aesthetic paints used for these applications (e.g., Chevrolet Orange and Chevrolet Blue). Given that the paint was also used within the former Nodular Iron Plant, the former Paint Spray Building may also have been used for the storage of maintenance paints that are primarily used for the general upkeep of the building and/or property (e.g., walls, buildings), which would not need to be anti-corrosive or wipeable and thus would not be assumed to contain PFAS. The former Paint Spray Building was not identified as an AOI in the DOCC because there was no indication of a known or potential release to the environment. In addition, there is no known or documented indication of historical fires in these areas that could have been extinguished using PFAS-containing foam.

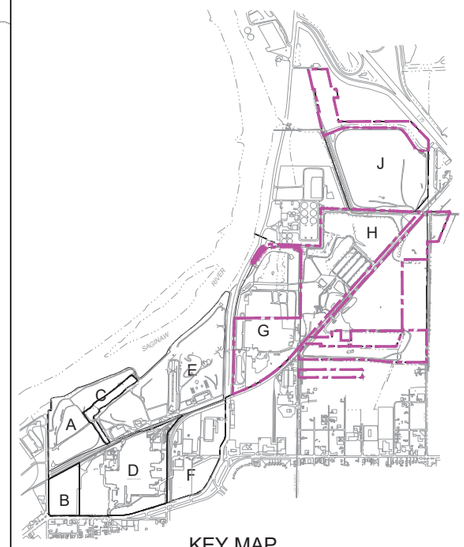
As part of the Resource Conservation and Recovery Act Facility Investigation (RFI) soil boreholes were advanced and several groundwater monitoring wells were installed in the general vicinity of the former Paint Spray Building (or Paint Storage Building) and the former Paint Shed Hazardous Waste Drum Storage Area (AOI G.3). Volatile organic compounds (VOCs) or other analytes were not detected in soil and groundwater or were detected at concentrations below the applicable screening levels, except for several metals in groundwater. No exceedances of inorganics in groundwater were identified following the initiation of low-flow sampling in 2005. Soil samples collected as part of closure for the former Paint Shed Hazardous Waste Drum Storage areas indicated that none of the soil results for metals were above the 99% prediction limits based on background sampling. Therefore, the soil and groundwater data collected from the general vicinity of the former Paint Spray Building (or Paint Storage Building) and the former Paint Shed Hazardous Waste Drum Storage Area (AOI G.3) did not identify any releases to soil or groundwater.

Therefore, there is no known use, let alone release, of PFAS at the RACER property.

4. Discussion

As described above, the anecdotal potential use of PFAS at the Site was associated with the former Paint Spray Building (or Paint Storage Building), which contained a small paint spray booth. Based on painting operations identified at other locations at the Larger Facility, it is believed that the former Paint Spray Building was used for painting auto components for aesthetic purposes and/or for the storage of paints used for building/property maintenance purposes. Paints that typically do not contain PFAS were used for these applications. Therefore, since there was minimal potential for release to the environment from the former Paint Spray Building and therefore the former Paint Shed Hazardous Waste Drum Storage Area; there is no known or suspected release of PFAS containing material to the environment that warrants investigation as part of RCRA Corrective Action.





KEY MAP

LEGEND

- INVESTIGATIVE UNIT BOUNDARY
- RACER TRUST PROPERTY
- ASSUMED POTENTIAL PFAS USAGE

0 100 200 400 ft

N

GHD

RACER TRUST
SAGINAW, MICHIGAN

SITE PLAN AND AOI
INVESTIGATIVE LOCATION MAP FOR
THE FORMER NODULAR IRON PLANT - (IU G)

Project No. **11208041**
Date **April 2021**

FIGURE 1

Data Source:

Filename: N:\CA\Waterloo\Projects\662\11208041\Digital_Design\Figures\MEMO-004\11208041-GHD-0000-MEM-EN-0101_WA-004.dwg
Plot Date: 23 April 2021 3:06 PM

Attachment B

Sampling Procedures

PFAS Sampling Checklist

Date: _____ Site: _____
(mm/dd/yy)

Weather Conditions: _____

Field Clothing and PPE:

☐ No clothing or boots containing Gore-Tex™

☐ All safety boots made from polyurethane and PVC

☐ No materials containing Tyvek®

☐ Field crew has not used cosmetics, moisturizers, hand cream, or other related products this morning

☐ Field crew has not applied unauthorized sunscreen or insect repellant

Field Equipment:

☐ No Teflon® or LDPE containing materials on-site

☐ All sample materials made from stainless steel, HDPE, acetate, silicon, or polypropylene

☐ No water proof field books on-site

☐ No plastic clipboards, binders, or spiral hard cover notebooks on-site

☐ No adhesives (Post-It Notes) on-site

☐ Coolers filled with regular ice only. No chemical (blue) ice packs in possession.

Sample Containers:

☐ All sample containers made of HDPE or polypropylene

☐ Caps are unlined and made of HDPE or polypropylene

Wet Weather (as applicable):

☐ Wet weather gear made of polyurethane and PVC only

Equipment Decontamination:

☐ "PFAS" free water on-site for decontamination of sample equipment. No other water sources to be used.

☐ Alconox and Liquinox to be used as decontamination materials

Food Consideration:

☐ No food or drink on-site with exception of bottled water and/or hydration drinks (i.e., Gatorade and Powerade) that is available for consumption only in the staging area

If any applicable boxes cannot be checked, the Field Lead shall describe the noncompliance issues below and work with field personnel to address noncompliance issues prior to commencement of that day's work. Corrective action shall include removal of noncompliance items from the site or removal of worker offsite until in compliance.

Describe the noncompliance issues (include personnel not in compliance) and action/outcome of noncompliance:

_____

_____

_____

Field Lead Name: _____

Field Lead Signature: _____ Time: _____

GHD Field Training Manual

Addendum to Section 7.0 for PFAS Considerations Water Sampling Standard Operating Procedures

(T120)

200010 | Report No 1 | Revision 0 | July 24, 2019

Please Adhere to the Following Quality System Training Requirements:

- Employees who are required to conduct a specific field activity must be properly certified to do the work.
- This involves reviewing the SOP and completing the online training course and exam.
- Employees must also conduct this field work under supervised conditions on at least three occasions, and must be certified by a qualified mentor. Only then can an employee conduct a specific field activity on their own. This is documented on a Field Method Training Record (QSF-021).
- Complete the QSF-021 and forward it to trainingrecords-northamerica@ghd.com.

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7. PFAS Water Sampling Guidelines and Considerations (Groundwater, Residential, and Surface Water) Standard Operating Procedures

7.1 Introduction

Sampling for per- and polyfluoroalkyl substances (PFAS) in drinking water, groundwater, and surface water can be a challenge if proper quality assurance and project planning (QAPP) are not developed and followed. The following sections provide guidelines and considerations for successful PFAS sampling events. These guidelines are meant to be used as a general addendum to Section 7.0 in regards to sample collections for laboratory analysis. Due to the changing nature in PFAS regulations and available best practices, this addendum will be updated as more research is performed on sampling materials and techniques.

7.2 General Field Procedures

Field team members must wash their hands with certified PFAS-free water (i.e., provided by certified laboratory or potable water that has been tested and confirmed to be free of detectable PFAS) and Alconox® or Liquinox® prior to handling any sampling equipment or bottles, touching the sampling ports, or putting on nitrile gloves. If liquids or food are consumed during the course of sampling, which is not to occur in the area of sampling activities at the Site, the field team member must wash their hands with PFAS-free water and Alconox® or Liquinox® afterwards, before putting on a new pair of nitrile gloves. It has been noted that Alconox® and/or Liquinox® may cause irritation to the skin. An acceptable alternative for hand washing is Seventh Generation® hand soap.

During sampling, never place caps from sample containers on the ground or in a pocket. The protocol is to hold the sample container in one hand and the sample container cap in the other. It is important to not touch the inside of the sample container cap or the inside of the sample container to any ports or with your hands. Don new disposable nitrile gloves at each sampling location and following contact with a potential contaminant source. If the sampler's gloves are comprised at any point during the individual sample collection, the sampler must change to a new pair of gloves so that the sample is representative of the Site conditions. Nitrile gloves must be changed prior to and after collecting samples for volatile organic compounds (VOC) analysis in order to circumvent potential contamination issues due to the Teflon®-lined caps of the VOC vials. Nitrile gloves also need to be worn when labelling bottles and preparing coolers for shipment in order to avoid contact with adhesives that are necessary to perform these procedures, which could lead to contamination of the PFAS samples.

The following represents a general PFAS sampling approach:

1. Sampler must wash hands with certified PFAS free water before wearing initial pair of nitrile gloves to limit contamination during sampling from activities undertaken prior to arrival at the site.

2. Samples for PFAS analysis should be collected in laboratory supplied polypropylene or high-density polyethylene (HDPE) bottles with unlined non-Teflon polypropylene or HDPE screw cap.

3. PFAS samples should be filled to the bottom of the neck of the bottle and not to the very top. Preservative (such as Trizma®) is required for drinking water samples to serve as a buffering agent and chlorine scavenger.

4. After filling and securely capping, the bottles should be inverted at least 5 times to distribute the preservative (only for drinking water samples) and must be placed on ice in coolers (samples cannot exceed 10°C).

5. Samples should be placed in zip-lock bags that will be packed in a cooler using regular ice doubled-bagged in zip-lock bags.

6. PFAS Field Blanks should be collected by opening the bottle of certified PFAS-free water (provided by the lab) and leaving it exposed to air in the vicinity of the sampling port.

7. Following the collection of all PFAS samples, the laboratory provided certified PFAS-free water will be poured from the container into the preserved bottles from the lab.

8. The now empty Field Blank water bottle used should be returned to the lab with the filled sample containers.

Field quality control samples will be used as a means of assessing quality from the point of sample collection. Such quality control samples will include field reagent blanks, equipment rinse blanks, and sample duplicates. The collection and analysis of QC samples are imperative for PFAS analyses due to low detection limits and widespread commercial use of PFAS containing products. The frequency of quality control sample collection will be defined in the project specific QAPP, in consideration of the data quality objectives, and factors such as program requirements and risk of cross contamination, .In general, for every 15 PFAS water samples, one field blank, one equipment blank, one matrix spike/matrix spike duplicate, and two field duplicates will be acquired.

7.3 Decontamination Procedures

Field sampling equipment used at each sample location will require cleaning between uses. The Safety Data Sheets (SDSs) of commonly used detergents or soaps used in decontamination procedures will be reviewed to assure that fluoro-containing chemicals are not in the ingredients list.

Alconox®, Liquinox®, PFAS-free water from an analytical laboratory or certified vendor are all acceptable products that can be used to decontaminate equipment. Laboratory-certified PFAS-free water will be used for the final rinse during decontamination of sampling equipment. Decontaminate larger equipment (for example, drill rigs and large downhole drilling and sampling equipment) with potable water using a high-pressure washer or steam. To the extent practical, rinse parts of equipment coming in direct contact with samples with PFAS-free water. Heavy equipment is best cleaned within a decontamination facility or other means of containment (for example, a bermed, lined pad and sump, or a portable, self-contained decontamination booth). Potable water sources should be analyzed in advance for PFAS. Wherever possible, rinse equipment with PFAS-free water immediately before use.

7.4 Sampling Precautions

Standard sampling procedures can be used at most PFAS sites with exceptions and additional considerations related to the chemical nature PFAS and issues associated with potential use of PFAS-containing or adsorbing sampling equipment and supplies. The following are sampling precautions for various environmental matrices that are defined in the Interstate Technology and Regulatory Council's Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods PFAS fact sheet:

7.4.1 Groundwater

The most inert material (for example, stainless steel, silicone, and HDPE), with respect to known or anticipated contaminants in wells should be used whenever possible. Dedicated sampling equipment installed in existing wells prior to investigation should be thoroughly checked to ensure that the equipment is PFAS-free. High density polyethylene (HDPE) and silicone tubing are both acceptable items for use in groundwater sampling. A list of acceptable field sampling items is summarized in Section 7.5. For long-term investigations, samples may be collected in duplicate with and without existing dedicated equipment. If PFAS analyses show that the equipment does not affect results, the equipment may be kept and used long term. This determination depends on project-specific requirements, however, and should only be used by a project team with full disclosure to all stakeholders.

7.4.2 Surface water

To avoid cross-contamination from sampling materials to sample media, the outside of all capped sample containers should be rinsed multiple times with the surface water being sampled before filling the containers. When site conditions require, remote sampling into sample containers can be accomplished by clamping the container onto the end of a clean extension rod. The extension rod must be made of PFAS-free material and have been decontaminated. Within the context of sample collection objectives, the sample location in the water column should consider the potential stratification of PFAS in solution and their tendency to accumulate at the air/water interface.

7.4.3 Pore water

Peristaltic pumps with silicone and HDPE tubing are typically used for porewater sample collection, along with push point samplers, porewater observation devices (PODs), or drive point piezometers. Push point samples and drive point piezometers are made of stainless steel, while PODs consist of slotted PVC pipe and silicone tubing. These samplers should be dedicated and not reused across a site or multiple sites.

7.4.4 Pit water (e.g. excavation dewatering pit)

Peristaltic pumps with silicone and HDPE tubing, bailers, and buckets are typically used for pit water sample collection. Tubing and bailers should be dedicated and not reused across a site or multiple sites. Buckets should be purchased new for each sampling event and deconned with Aloconox® or Liquinox ® and water, followed by a rinse with certified PFAS free water from the laboratory. Decon should occur immediately prior to collecting the sample of interest.

7.4.5 Influent/Effluent water (e.g. treatment system)

Water treatment system influent/effluent water is typically sampled using small buckets, beakers, or other containers, or collected directly into a laboratory supplied sample container. Sampling supplies should be purchased new for each sampling event and deconned with Aloconox® or Liquinox ® and water, followed by a rinse with certified PFAS free water from the laboratory. Decon should occur immediately prior to collecting the sample of interest.

Many gaskets, piping, hosing, pumps, and filters used in water treatment have the potential to contain Teflon or other sources of PFAS; therefore, the influent samples should be taken before they reach gaskets, piping, pumps, or filters in the water treatment assembly. Effluent samples should be taken at the point of discharge from the treatment system (after water is in contact with all gaskets, pumps, filter, etc.). To ensure the collection of a representative sample through treatment, the system should be purged or flushed to limit potential leaching from equipment materials.

7.5 Equipment/Materials

At this point, many materials used during the course of common environmental investigations can potentially contain PFAS. Due to the limited available published literature and/or guidance on which materials affect sampling results, a conservative approach will be used to rigorously exclude

materials known to contain PFAS. Safety Data Sheets (SDSs) will be reviewed prior to use during PFAS sampling.

Materials to avoid during sampling events include:

- Teflon, polytetrafluoroethylene (PTFE)
- Waterproof coatings containing PFAS
- Food containers
- Chemicals with “fluoro” on the SDS
- Fluorinated ethylene propylene (FEP)
- Ethylene tetrafluoroethylene (ETFE)
- Low density polyethylene (LDPE), polyvinylidene fluoride (PVDF)

The following represents a list of prohibited items vs. acceptable items for PFAS sampling:

Prohibited Items	Acceptable Items
Field Equipment	
Teflon® containing materials (caps, o-rings, tubing)	High-density polyethylene (HDPE) materials
Low density polyethylene (LDPE) materials	Acetate Liners
	Silicon Tubing
Waterproof field books	Loose paper (non-waterproof)
Plastic clipboards, binders, or spiral hard cover notebooks	Metal field clipboards or with Masonite
Post-It Notes®	Fine Point Sharpies®, pens
Chemical (blue) ice packs	Regular ice
Field Clothing and PPE	
New cotton clothing or synthetic water resistant, waterproof, or stain-treated clothing, clothing containing Gore-Tex™	Well-laundered clothing made of natural fibers (preferable cotton) washed at least 6 times since purchased
Clothing laundered using fabric softener	No fabric softener
Boots containing Gore-Tex™	Boots made with polyurethane and PVC and leather steel-toe safety boots
Tyvek®	Powder-free nitrile gloves
No cosmetics, moisturizers, hand cream, or other related products as part of personal cleaning/showering routine on the morning of sampling	**Sunscreens** - Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, Baby sunscreens that are “free” or “natural” **Insect Repellents** - Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellant, Herbal Armor, California Baby Natural Bug Spray, BabyGanics **Sunscreen and insect repellant -** Avon Skin So Soft Bug Guard Plus – SPF 30 Lotion
Sample Containers	
LDPE or glass containers	HDPE or polypropylene
Teflon-lined caps	Unlined polypropylene caps

Rain Events	
Waterproof or water-resistant rain gear	Gazebo tent that is only touched or moved prior to and following sampling activities and not touched with sampling gloves
Equipment Decontamination	
Decon 90®	Alconox® and/or Liquinox®
Water from an on-site well or any other potable water supply system	Deionized and deionized demonstrated PFAS-free water from an analytical laboratory or certified vendor
Food Considerations	
All food and drink, with exceptions noted on right	Bottled water and hydration fluids (i.e, Gatorade® and Powerade®) to be brought and consumed only in the staging areas and not touched with sampling gloves

In certain cases, it may be impossible to eliminate materials that affect PFAS results in samples. Safety is paramount and if otherwise 'prohibited' materials are needed to mitigate risks to safety, then these materials may be used; however, this must be documented. An example would be specific personal protective equipment (PPE) that are needed at hazardous sites where PFAS are the secondary or co-contaminant and the primary contaminant requires specific materials for proper sampling. In this case, increasing the equipment rinse blank samples will more thoroughly document PFAS concentrations.

7.6 References

For additional information pertaining to PFAS sampling activities the user of this manual may reference the following:

Government of Western Australia Department of Environmental Regulation. 2016. Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS), Contaminated Sites Guidelines. February 2016. Accessed November 22, 2017: https://www.der.wa.gov.au/images/documents/your-environment/contaminated-sites/guidelines/Guideline-on-Assessment-and-Management-of-PFAS-.pdf

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

Shoemaker, J. A., P. E. Grimmett, and B. K. Boutin. 2009. Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS). USEPA Method 537, Rev 1.1, EPA 600-R-08-092, 50 pp.

USDOD EDQW (Environmental Data Quality Workgroup). 2017a. "Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories," Version 5.1, 2017. http://www.denix.osd.mil/edqw/home/

USDOD EDQW. 2017b. “Bottle Selection and other Sampling Considerations When Sampling for Per- and Poly-Fluoroalkyl Substances (PFASs),” Revision 1.2, July.
http://www.denix.osd.mil/edqw/home/

GHD Field Training Manual

Section 7.0
Water Sampling Standard Operating Procedures
A. Groundwater
B. Residential
C. Surface Water

(T104)

July 2015

Please Adhere to the Following Quality System Training Requirements:

- Employees who are required to conduct a specific field activity must be properly certified to do the work.
- This involves reviewing the SOP and completing the online training course and exam.
- Employees must also conduct this field work under supervised conditions on at least three occasions, and must be certified by a qualified mentor. Only then can an employee conduct a specific field activity on their own. This is documented on a Field Method Training Record (QSF-021).
- Complete the QSF-021 and forward it to trainingrecords-northamerica@ghd.com.
- Please note that three topics are discussed in this SOP. A separate QSF-021 is required for each topic:
  - Groundwater Sampling
  - Residential Water Sampling
  - Surface Water Sampling

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7. Water Sampling (Groundwater, Residential, and Surface Water) Standard Operating Procedures

7.1 Introduction

Groundwater, residential, and surface water sampling are conducted in order to characterize the groundwater and surface water quality at a site. Standard Operating Procedures (SOPs) are presented herein for the collection of groundwater and surface water samples from:

- Monitoring wells
- Residential wells
- Surface water bodies

This guideline is not intended to provide the basis for designing a groundwater or surface water monitoring program, but instead assumes that a groundwater and/or surface water monitoring program has already been designed. It is also assumed that a site-specific Work Plan has been established and that a GHD representative is preparing to mobilize to the site.

Groundwater and surface water sampling procedures vary from project to project due to:

- Different chemicals of concern.
- Different guidance provided by local, provincial/state, and/or federal regulatory agencies with jurisdiction at the site.
- The specific objectives of the project.

It is essential that all groundwater, residential, and surface water sampling activities conform to local, state/provincial, and federal regulations. Therefore, it is essential that the GHD representative carefully reviews the Work Plan requirements. The primary goal of groundwater, residential, and surface water sampling is the collection of samples representative of the hydrostratigraphic unit and/or surface water body. It is necessary to use appropriate sampling techniques to collect representative samples that provide reliable and reproducible results in accordance with the Work Plan and all relevant regulations.

The remainder of this section is organized as follows:

- Section 7.2 Background
- Section 7.3 Planning and Preparation
- Section 7.4 Safety and Health
- Section 7.5 Quality Assurance/Quality Control
- Section 7.6 Equipment Decontamination
- Section 7.7 Field Procedures for Groundwater Sampling

- Section 7.8 Field Procedures for Residential Sampling
- Section 7.9 Field Procedures for Surface Water Sampling
- Section 7.10 Follow-Up Activities
- Section 7.11 References

7.2 Background

The objective of a groundwater and residential monitoring program is to obtain samples that are representative of existing groundwater conditions, or samples that retain the physical and chemical properties of groundwater in the hydrostratigraphic unit. Surface water sampling is performed to collect samples that are representative of physical and chemical properties of surface water bodies. Improper sampling and transport practices will cause compounds of interest to be removed or added to a sample prior to analysis. The importance of proper and consistent field sampling methods cannot be over emphasized. It is equally important that proper documentation occurs throughout the sampling program.

The most important aspect of groundwater sampling is the collection of groundwater samples that are free of suspended silt, sediment, or other fine-grained material. Fine-grained material has a variety of chemical compounds sorbed to the particles or has the ability to sorb chemicals from the aqueous phase. This causes a bias in the subsequent analytical results. Reproducible and reliable analytical data are invaluable to a groundwater monitoring program. GHD frequently criticizes the sampling activities completed by others due to the collection and analyses of turbid samples. This SOP discusses sampling protocols that typically achieve sediment-free samples.

When sampling for monitored natural attenuation (MNA) parameters, more stringent protocols are followed to ensure sediment-free samples that are representative of the total mobile load (i.e., dissolved and naturally suspended particles). Low-flow purging (LFP) techniques are strongly recommended, if not mandated, when collecting groundwater samples for MNA parameters. The LFP techniques detailed in Section 7.7.5.3 are in accordance with United States Environmental Protection Agency (USEPA) LFP procedures (Puls and Barcelona, 1996).

Groundwater sampling is required for various reasons, including:

- Investigating potable or industrial water supplies
- Tracking contaminant plumes
- Investigating a site with suspected groundwater contamination

Groundwater is usually sampled from in-place wells, installed either temporarily or permanently. Municipal, industrial, or residential wells may also be sampled during an investigation. When completing residential well sampling it is important that representative samples are collected. Poor or incorrect sampling techniques will result in erroneous results. Incorrect results disclosed to the public will create a false impression, making it difficult to change the perception when correct results are reported.

Groundwater and residential sample collection are performed from non-impacted to most impacted locations. This eliminates the potential for cross-contamination. A review of all historical analytical data is performed to ensure the exact sampling sequence.

Surface water sampling locations are selected based on many factors including:

- The study objectives
- The location of point source discharges
- The location of no-point source discharges and tributaries
- The presence of structures (e.g., bridges, dams)
- Accessibility

Surface water sampling should be performed from downstream to upstream locations. This ensures that surface water sampling activities do not cause suspended sediments to bias samples collected downstream.

7.3 Planning and Preparation

Prior to groundwater, residential, and surface water sampling:

1. Review the Work Plan, project documents, and Site-Specific Health and Safety Plan (HASP) with the Project Manager/Coordinator.
2. Review the Quality Assurance Project Plan (QAPP) with the Project Coordinator and Project Chemist to determine Quality Assurance/Quality Control (QA/QC) and decontamination requirements.
3. Complete a Field Equipment Requisition Form (QSF-014). Assemble all sampling equipment and supplies required per the Groundwater Sampling Equipment and Supply Checklist (Form SP-05). The Project Planning, Completion, and Follow-Up Checklist (Form SP-02) should be used for guidance throughout the project.
4. Assemble the site plan, well logs, and previous sampling/purging data required for the sampling event. Determine the exact number and locations of wells to be sampled.
5. Obtain all forms to record purging and sampling activities (Forms SP-06, SP-08, and SP-09).
6. Confirm with the Project Manager/Coordinator that a Property Access/Utility Clearance Data Sheet (QSF-019) has been completed. For residential sampling, ensure that homeowners have been notified of the intended sampling event. Confirm the presence of any dogs on site, modify the site-specific Job Safety Analysis, if there is a dog.
7. Arrange access to the site. Obtain all well and site keys. Consider site access conditions (e.g., snow).
8. For surface water sampling consider if hazards exist due to deep/fast moving water, difficult access, and if additional GHD personnel are required for safety and health reasons.

9. For residential sampling contact homeowners to make arrangements for a site visit, arrange for site dog to be removed from all areas where a GHD employee will be working. The client of another party may be responsible for making arrangements.

10. Complete a Vendor Evaluation Form (QSF-012) and file in the Project file for any Vendors that do not have full approval status or are not listed on the Approved Vendor List (QSL-004). Completion of a Safety and Health Schedule (QSF-030 for Canadian work; QSF-031 for U.S. work) is necessary for all Vendors who complete field services. Prior to mobilization on site, the Vendor must submit the form to the Regional Safety and Health Manager for review and approval (if not already posted on QSL-004).

11. Contact the GHD Chemistry group to arrange:

    - SSOW (Simplified Scope of Work)
    - Laboratory
    - Sample containers delivery
    - Preservatives if required
    - Filtration information if required
    - Coolers
    - Shipping details
    - Sample starting date
    - Expected duration of sampling program

12. If several sampling events are planned, evaluate with the client the benefit of purchasing and installing dedicated sampling equipment. Dedicated purging and sampling equipment reduces potential cross-contamination and reduces decontamination requirements. At a minimum, sample tubing is dedicated to each well and is left secured in the well for future use. For LFP it is recommended that each well is dedicated with a bladder pump and tubing to eliminate well disturbance.

13. Evaluate sample notification needs with the Project Coordinator. Have the regulatory groups, client, landowner, GHD personnel, and laboratory been notified of the sampling activities?

14. Evaluate containment and disposal requirements for purge waters.

15. Plan sampling activities to ensure that wells that historically go dry or have poor recharge fit into the sampling program. This will reduce the time required for sample collection.

16. Plan the sequence of sampling activities to reduce the potential for cross-contamination. For groundwater sampling, start with clean wells and progress to impacted wells. For surface water sampling, start downstream and progress upstream.

7.4 Safety and Health

GHD is committed to conducting field activities in accordance with sound safety and health practices. GHD adheres to high safety standards to protect the safety and health of all employees,

subcontractors, customers, and communities in which they work. The safety and health of our employees takes precedence over cost and schedule implications.

Field personnel are required to implement the Safety Means Responsibility Awareness Teamwork (SMART) program as follows:

- Assure the HASP is specific to the job and approved by a Regional Safety & Health Manager.
- Confirm that all HASP elements have been implemented for the job.
- A Job Safety Analysis (JSA) for each task has been reviewed, modified for the specific site conditions, and communicated to all appropriate site personnel. The JSAs are a component of the HASP.
- Incorporate Stop Work Authority; Stop, Think, Act, Review (STAR) process; Safe Task Evaluation Process (STEP); Observations process; Near Loss and Incident Management process in the day-to-day operations of the job.
- Review and implement applicable sections of the GHD Safety & Health Policy Manual.
- Confirm that all site personnel have the required training and medical surveillance as defined in the HASP.
- Be prepared for emergency situations, locating safety showers, fire protection equipment, evacuation route, rally point, and first aid equipment before you begin working, and make sure that the equipment is in good working order.
- Maintain all required Personal Protective Equipment (PPE), safety equipment, and instrumentation necessary to perform the work effectively, efficiently, and safely.
- Be prepared to call the GHD Incident Hotline at 1-866-529-4886 for all involving injury/illness, property damage, vehicle incident, and/or significant Near Loss.

It is the responsibility of the Project Manager to:

- Ensure that all GHD field personnel have received the appropriate health and safety and field training and are qualified to complete the work.
- Provide subcontractors with a Job Hazard Analysis to enable them to develop their own HASP.
- Ensure that all subcontractors meet GHD's (and the Client's) safety requirements.

7.5 Quality Assurance/Quality Control

A well-designed QA/QC program will:

- Ensure that data of sufficient quality are obtained, for proper site management decisions or remediation design.
- Allow for monitoring of staff and contractor performance.
- Verify the quality of the data for the regulatory agency.

It is important to note that a QA/QC program should be developed on a site-specific basis. QA/QC requirements are discussed in Section 3.9.

7.6 Equipment Decontamination

Equipment decontamination procedures for a groundwater, residential, or surface water monitoring program will be described in detail in the site-specific Work Plan or in the QAPP.

Equipment is decontaminated between sampling locations and prior to leaving the site. Upon completion of the sampling program, all equipment is decontaminated at the site and then returned clean to the appropriate field equipment manager.

For most groundwater, residential, and surface water sampling programs, sampling equipment (e.g., pumps, bailers, water level indicators) is typically cleaned as follows:

1. Wash with clean potable water and laboratory detergent, using a brush as necessary to remove particulates.
2. Rinse with tap water.
3. Rinse with deionized water.
4. Air dry for as long as possible.

If required, the following steps may be added when sampling for Volatile Organic Compounds (VOCs) and metals:

1. Rinse with 10 percent nitric acid (only if samples are to be analyzed for metals).
2. Rinse with deionized water.
3. Rinse with appropriate solvent (pesticide grade isopropanol, methanol, acetone, hexane, if required).
4. Rinse again with deionized water.
5. Air dry for as long as possible.
6. Wrap samplers in aluminum foil to prevent contamination.

> Caution: Check the QAPP to confirm the cleaning protocol. Use of incorrect cleaning protocol could invalidate chemical data.

7.6.1 Purge Water and Decontamination Fluid Disposal

Project-specific disposal methods for purged groundwater and decontamination fluids are determined by the Project Manager during the sampling program's planning and preparation stage (see Section 7.3), but may include:

1. Off-site treatment at private treatment/disposal facility or publicly owned treatment facilities (sanitary sewer).
2. On-site treatment at a client-operated facility.
3. Direct discharge to the surrounding ground surface, allowing infiltration to the underlying subsurface.
4. Direct discharge to an impervious pavement surface allowing for evaporation.

Options 3 and 4 are permitted only after careful review of these practices and the anticipated site conditions. Under no circumstances shall GHD personnel aggravate an existing condition or spread contamination into clean areas.

Decontamination fluids (specifically cleaning solvents/acids) are segregated and collected separately from wash water and purge water. Often small volumes of solvents used during the course of a groundwater, residential, or surface water sampling program will evaporate if left in an open pail. If evaporation is not possible, off-site disposal need to be arranged.

7.7 Field Procedures for Groundwater Sampling

The typical series of events that takes place for a groundwater sampling program is:

1. Well identification and inspection
2. Air monitoring
3. Water level monitoring
4. Well depth sounding
5. Well volume calculation
6. Purging and sampling equipment installation
7. Well purging and stabilization monitoring
8. Sample collection, sample preparation, completion of chain-of-custody, (COC) sample packaging
9. Final water level monitoring (if required), purging, sampling equipment removal, secure the well
10. Equipment decontamination
11. Field note completion and review
12. Sample shipment and COC distribution
13. Purged groundwater and decontamination fluid disposal
14. Sample record documentation, equipment return
15. Completion and distribution of appropriate forms

It is recommended that new plastic sheeting be placed on the ground around the well to prevent contamination of purging and sampling equipment and accessories (e.g., pumps, hoses, rope.).

7.7.1 Well Identification and Inspection

At sites with numerous wells or wells nests, misidentification of wells has occurred. The GHD representative must be alert to the possibility of potential cap switching, mislabeled wells, or unlabeled well locations.

Determine proper well location and identification by comparing the well log details to the measured well depths (i.e., total well depth, casing diameter, casing stick-up, or stick-down distances), field tie-ins, and site plan.

Once well identification has been established, complete a thorough well inspection:

1. Determine if the well cap and lock are secure, and check for vandalism.
2. If no lock is present, dedicate a new lock to the well location.
3. Examine the integrity of the surface seal.
4. Check for cracks, evidence of frost heave, or subsidence in the vicinity of the well.
5. Examine the integrity of the protective casing. Ensure that the casing can be closed and locked.
6. If required, re-label the well to assist in future identification.
7. If the well is installed with dedicated sampling equipment, check for cracks or leaks in tubing, and worn or frayed rope.
8. Record all the well inspection details in the field book to document well conditions and suitability for groundwater sampling activities.
9. Forward the well inspection results to the Project Coordinator, especially if repairs are required.

7.7.2 Air Monitoring

Prior to removing a well cap, measure the breathing space above the well with a photoionization detector (PID) to establish background of undifferentiated organic vapor levels. Repeat this process once the well cap has been removed. If either of the PID levels exceed the air quality criteria established in the HASP, air-purifying respiratory (APR) protection or a supplied air system is required. Also take a PID reading inside the riser pipe. This PID reading is a good indication of elevated chemical or non-aqueous phase liquids (NAPL) presence. Report all elevated PID levels to the Project Coordinator immediately to determine if additional health and safety and personnel protective equipment is required. The HASP will provide the required action levels and PPE.

7.7.3 Water Level Monitoring/Well Depth Sounding

Prior to commencing well purging and groundwater sampling, the water level is measured for hydraulic monitoring and to determine the well volume. Typically, a complete round of water level measurements is taken at a site to establish groundwater conditions prior to initiating well purging or groundwater sampling activities.

A watertight cap provides an airtight seal on the casing and the water level positioned in the casing area. The cap creates a vacuum or pressurized condition in the casing section which can support or depress the water column in the well casing. This can produce an artificially high or low water level in the well casing. This effect can cause a few inches or feet of error in the static water level. Once the cap is removed, allow the pressure to stabilize for about a half hour. Measure the water level

frequently to ensure that stabilization of the water level has occurred. Once the water level has stabilized (i.e., is static) the correct water level may be measured.

A number of instruments are available to measure groundwater levels. GHD typically uses:

- Battery-operated water level indicators (i.e., audible and/or visual identification of water level)
- Battery-operated oil/water interface probes (i.e., audible and/or visual identification of water levels and presence of NAPL)
- Electronic transducers (numerous manufacturers) and recording devices for long-term hydraulic monitoring
- Stevens™ recorders (both float and electronic instrumentation) for long-term hydraulic monitoring

Section 8.0 describes in detail the equipment and monitoring techniques for water level measurements.

Well depth sounding is often required to confirm well identification, evaluate the accumulation of sediment in the well bottom, or assist in determining the standing well volume. Sounding is performed using a water level indicator or a measuring tape with a weighted end. The water level indicator or weighted tape is lowered to the bottom of the well and a comparison is made of the installed well depth versus the measured well depth. The presence of excessive sediment or drill cuttings may warrant redevelopment of the well prior to well purging and groundwater sampling activities.

The total well depth is compared to the original installed total well depth. If the well screen is more than 50 percent blocked by accumulated sediment, the well is redeveloped prior to the next groundwater sampling event. Report all wells requiring redevelopment to the Project Coordinator. Well depth sounding is performed on an annual or biannual basis if the well is equipped with a dedicated pump.

For LFP, well depth measurement is performed to ensure proper pump intake placement. The used of a wide-based probe, such as a weighted tape, is necessary to minimize penetration and disturbance of accumulated sediment. The measuring device is lowered slowly through the water column to the well bottom to minimize mixing of the stagnant well casing water and disturbance of sediment.

Note: Don't forget that decontamination procedures apply to the water level monitoring equipment as well as the groundwater sampling equipment. If well sounding is performed, the entire measuring device must be thoroughly decontaminated prior to re-use. Measuring the well depth with certain water level indicators may damage the probe seal. Therefore, a tape with a weighted end should be used to measure well depth.

7.7.4 Well Volume Calculation

Prior to commencing well purging, the volume of water in the well must be known to determine the volume of groundwater to be removed. A well volume is defined as the volume of water contained in

the well screen and casing (and in the case of an open bedrock hole, the volume of water in the open corehole and possibly in the well casing). To determine the standing water volume in a well:

1. Calculate the distance from the bottom of the well to the static water level.
2. Measure the inside diameter of the well or casing. Obtain the volume of standing water in the well using the following formula:

   V = $\pi r^2 h$ (7.48 U.S. gallons/cubic feet) (1 liter/1,000 cubic centimeters)

   Where:

   V = volume of water in gallons or liters

   π = 3.142

   r = radius of well casing (feet or meters)

   h = depth of water column in the well (feet or meters)

Typical 1 - Foot Casing Volumes	
Diameter (inches)	Gallons (U.S.) of Water Per Foot of Casing
1.5	0.09
2	0.16
3	0.37
4	0.65
6	1.47

Typical 1 Meter Casing Volumes		
Diameter		Litres per Meter of Casing
(inches)	6 (cm)	
1.5	3	1.14
2	5	2.02
3	8	4.56
4	10	8.11
6	15	18.24

7.7.5 Well Purging and Stabilization Monitoring

7.7.5.1 Typical Method

Prior to initiating groundwater sample collection, the wells is purged of the standing stagnant groundwater volume. This volume is not representative of the groundwater in the hydrostratigraphic unit. Purging is performed until the water in the well is representative of the actual conditions in the hydrostratigraphic unit. Stabilization is usually achieved by the removal of three to five times the volume of standing water in the well (USEPA convention). Purging is considered complete once purged groundwater is free of sediment and field parameters including specific conductance, temperature, and turbidity are stable. Stabilization is achieved when field measurements for specific conductance and temperature are within a range of plus or minus 10 percent of the average for the

last three readings. Field measurement for pH should be within a range of plus or minus 0.1 pH unit of the average for the last three readings, and groundwater turbidity values should be less than 5 nephelometric turbidity units (NTU) (guidance value only). Once the number of well volumes required to achieve stabilization is established, the volume required to reach stabilization for future sampling events is reduced or eliminated. Extended purging of a well will generally result in achieving sediment-free groundwater conditions.

During purging, if stabilization has not occurred after removal of five well volumes, purging is continued until ten well volumes have been removed. If stabilization still has not been achieved, stabilization may be dropped as a pre-condition to groundwater sampling. The Project Coordinator should be notified that stabilization has not occurred after the removal of ten well volumes.

At high yielding wells, removing three to five well volumes is usually sufficient prior to initiating groundwater sampling. For low yield wells (i.e., wells that pump dry after one well volume) it is necessary to purge the well dry on three successive days, unless the well recovers to full static conditions in a shorter time. If the recharge is relatively high, groundwater sampling will be initiated once the well has fully recovered to static groundwater conditions, or to a level that is sufficient to collect the necessary groundwater sample volume.

> Note: Purging of dry wells should be scheduled to begin on Monday or Tuesday, to reduce weekend requirements.

Turbidity of purged groundwater is evaluated by a visual examination for sediment/silt presence or by using a nephelometer which physically measures groundwater turbidity in NTUs. Generally, a turbidity value of 50 NTU or less is acceptable, although some regulatory agencies have established lower criteria (i.e., less than 5 NTU). If 50 NTU is not achieved, filtration of samples may be required. LFP can generally result in turbidity values less than 5 NTU.

> Note: Agitation of the water column within the well will increase turbidity. Therefore, bailers and inertia pumps (Waterra™) are of limited use for collecting sediment-free samples. The tubing of peristaltic pumps must be secured to prevent movement of the tubing within the water column which would disturb sediment. The best method to reduce sediment disturbance is low-volume non-agitation pumping (i.e., bladder pump).

Well purging is accomplished using dedicated equipment or by using either peristaltic, bladder, or other approved purging methods. Purging and sampling equipment are dependent on the total well depth. Bailing can be used for well purging but this method stirs up sediment and increases the purging effort required before stabilization is achieved. Equipment available for well purging is discussed in Section 7.7.7. Monitoring equipment used during well purging includes a water level indicator, pH meter, thermometer, conductivity meter, and turbidity meter.

7.7.5.2 Purging Entire Water Column

The purging equipment is lowered into the top of the standing water column. Well purging is completed from as close to the top of the water column as possible, not from the well bottom, unless poor well recovery occurs. Purging from the top of the water column moves water from the formation through the well screen of the well and into the well casing. This allows for the entire

static volume to be removed. Purging at depth in the water column does not remove water above the pump intake and results in the collection of unrepresentative samples.

If required, the pump intake can be adjusted. If the recovery rate is greater than the pumping rate, the pump should remain suspended until the required purged volume has been removed. If the recovery rate is less than the pumping rate, the pump should be lowered to ensure the removal of the required well volume.

7.7.5.3 Low-Flow Purging (LFP) Technique

LFP purging results in minimal drawdown during well purging, so less purging is required before formation water is removed. The volume required for purging using LFP is significantly reduced. LFP results in less agitation and mobilization of sediments compared to traditional sampling techniques.

A pre-cleaned stainless steel bladder pump equipped with a Teflon™ bladder is strongly recommended for LFP. The discharge line should be polyethylene or Teflon™ lined tubing with an inside diameter of 1/4 or 3/8 inch (6 or 10 mm). Check the Work Plan or QAPP to ascertain the proper bladder and discharge tubing. Smaller discharge tubing ensures that the tubing remains filled with water and reduces air bubbles at low purging rates. The airline to the pump is generally 1/4-inch (6 mm) inside diameter polyethylene tubing. The pump is secured to nylon rope and positioned in the well so that the pump intake is set at the mid-point of the well screen, or a minimum of 2 feet (0.6 m) above the bottom of the well or accumulated sediment level. It is important that the rope, airline, and discharge tubing are measured prior to installation in the well. The bladder pump and tubing are lowered very slowly through the water column to minimize mixing of the stagnant well casing water and to minimize the agitation of sediment into suspension, which would increase the purging time. It is recommended, and in some instances regulated, that pump installation occurs at least 24 hours prior to initiating LFP. It is recommended that a bladder pump be dedicated to the well for regular monitoring events.

During LFP, the pumping rate should be between 100 and 500 milliliters per minute (mL/min). It is recommended that initial pumping be conducted at a lower rate to limit drawdown in the well. During purging, groundwater levels are measured to maintain a maximum 0.4 foot (0.1 m) of drawdown. The pumping rate can be gradually increased during LFP. Pumping rate increases will be dependent on the drawdown and the stabilization of field parameters discussed below. Pumping rate adjustments should occur in the first 15 minutes of purging. After this time the pumping rate should remain constant and flow rate adjustments should be avoided. During purging, the pumping rate and groundwater level should be measured at least every 10 minutes. It is recommended that water level measurements occur at 5-minute intervals.

During LFP, stabilization of the purged groundwater is required to ensure the collection of representative groundwater samples from the formation and not from the stagnant water in the well casing. Field parameters including pH, temperature, specific conductance, oxidation-reduction potential (ORP), dissolved oxygen (DO), and turbidity should monitored during LFP. The measurement of these field parameters is used to evaluate if stabilization of the purged groundwater has occurred prior to the collection of groundwater samples. The field measurements should be measured and recorded at 5-minute intervals. Groundwater stabilization is considered

achieved when three consecutive readings for each of the field parameters, taken at 5-minute intervals, are within the following limits:

pH	±0.1 pH units of the average value of the three readings
Temperature	±3 percent of the average value of the three readings
Conductivity	±0.005 milliSiemen per centimeter (mS/cm) of the average value of the three readings for conductivity <1 mS/cm and ±0.01 mS/cm of the average value of the three readings for conductivity >1 mS/cm
ORP	±10 millivolts (mV) of the average value of the three readings
DO	±10 percent of the average value of the three readings
Turbidity	±10 percent of the average value of the three readings, or a final value of less than 5 NTU

During LFP, field parameters are measured using a flow-through cell apparatus. At the start of LFP the purge water is visually inspected for clarity prior to connecting to the flow-through cell. If the purge water is turbid, LFP continues until the purge water is visually less turbid prior to connecting to the flow-through cell. Field parameters may be obtained using individual meters or a multiple meter unit; however, the use of a flow-through cell is highly recommended. All meters must be calibrated daily in accordance with the manufacturer's and GHD's calibration instructions, and a calibration record maintained in a standard GHD field book.

During LFP the meter readings are monitored for evidence of meter malfunction. The following are common indicators of meter malfunctions:

- DO above solubility (e.g., oxygen solubility is approximately 11 milligrams per liter (mg/L) at 10°C) may indicate a DO meter malfunction.
- Negative ORP and DO less than 1 to 2 mg/L may indicate either an ORP or a DO meter malfunction (i.e., should have positive ORP and DO less than 1 to 2 mg/L under oxidizing conditions).
- Positive ORP and DO less than 1 mg/L may indicate either an ORP or a DO meter malfunction (i.e., should have a negative ORP and DO less than 1 mg/L under reducing conditions).

Meter calibration fluids should be available for meter recalibration in the field. Spare meters should also be available for meter replacement if necessary.

Note: DO levels exceeding the solubility of oxygen in water are erroneous and are indicative of meter malfunction or poor sampling techniques causing turbulence and aeration. DO concentrations cannot exceed:

9 mg/L at 20°C 10 mg/L at 15°C 11 mg/L at 10°C 14 mg/L at 1°C

Stabilization will be considered complete when the field parameters have stabilized as indicated in the above table. Purging will continue if stabilization does not occur, until a maximum of 20 screen volumes has been removed. LFP causes groundwater to be drawn from a significant distance above or below the pump intake. Therefore, the screen volume is based on a 5-foot (1.5 m) screen length. After the removal of 20 screen volumes, purging will continue if the purged water remains

visually turbid and appears to be clearing. Also purging will continue if the field parameters vary only slightly outside of the stabilization criteria and appear to be approaching stabilization.

If the recharge to the well is insufficient to conduct LFP, the well should be pumped dry and allowed to recharge sufficiently for the collection of the groundwater sample volume. Wells purged dry are required to meet the stabilization criteria detailed above.

7.7.5.4 Sampling Techniques

Upon completion of purging, with groundwater stabilization and clarity meeting the applicable protocol described above, groundwater sample collection can proceed. Generally the field parameters of pH, temperature, and specific conductance are monitored first, then any other required field measurements.

Samples are collected directly from the purging pump, when possible, or an alternate device (i.e., pump or bailer) may be installed or used. If new sampling equipment is installed, the first few bails or discharge volumes should be discarded to allow acclimation of the sampling equipment with the groundwater.

Samples are typically collected from the pump or bailer with the discharged groundwater collected directly in the appropriate sample containers. The interior of the bottle or cap must not be touched or handled in anyway. New gloves (i.e., disposable nitrile gloves or equivalent) should be worn for the collection of each sample. Caps from sample bottles must not be placed on the ground or in pockets to eliminate the possibility of cross-contamination.

Descriptions of the various equipment and sampling methods for the collection of groundwater samples are contained in Section 7.7.7.

The following describes the main activities involved in the collection of groundwater samples.

7.7.5.5 Order of Sample Collection

Groundwater samples are collected and containerized in the order following volatilization sensitivity:

1. VOCs
2. Semi-volatile organic compounds (SVOCs)
3. Total organic carbon
4. Total organic halides
5. Extractable organics
6. Total metals
7. Dissolved metals
8. Phenols
9. Cyanide
10. Sulfate and chloride

11. Nitrate and ammonia
12. Microbiological parameters
13. Radionuclides

QA/QC requirements for groundwater sampling are described in detail in Section 3.9.

7.7.6 Sample Acquisition and Transfer

If groundwater sample collection is performed using a pump, the flow rate must not exceed 100 mL/min during the collection of groundwater samples for VOCs. The low flow rate will reduce the possibility of degassing samples. During the collection of groundwater into the sample container or filtration device, minimize agitation and aeration of the sample. Groundwater samples are transferred directly into the sample container for submittal to the laboratory. Groundwater samples should not be collected in larger containers and subsequently transferred to smaller sample containers; however, on occasion this will be required for filtration or sample composting. During VOC sample collection, samples must not be collected, handled, or containerized near or in the vicinity of a running motor or exhaust which may contaminate the samples.

Groundwater samples for VOCs are collected in laboratory supplied 40 mL glass vials. The vials are filled to the top until a meniscus is formed, then topped with a Teflon™-lined cap. To prevent the loss of volatiles, it is important that no air bubbles or headspace are present in the sample container. Inverting and tapping the vial will check for the presence of air bubbles. If air bubbles are present, the sample should be topped off again and resealed. This process may only be performed a maximum of twice, at which time the sample must be discarded and the sample retaken. If preservatives were present in the bottle from the laboratory, a new sample vial must be used.

> Note: Gas bubbles that appear in VOC containers after sample collection may be a result of degassing or reaction with preservative. If this occurs, note this occurrence on the chain-of-custody. Re-sampling is not required in most cases.

During sample collection ensure groundwater samples are preserved according to laboratory requirements. If required and supplied by the laboratory, preserve the samples in accordance with the QAPP. Some laboratories pre-preserve bottles so that once the groundwater sample is added the preservation is completed. In either case, it is advisable to check sample preservation using litmus paper. Using litmus paper ensures that groundwater sample preservation has been completed to the proper pH as required by the QAPP. If preservation of a sample does not meet the requirements of the QAPP, it may be necessary to add additional preservative, or note on the chain-of-custody that incomplete sample preservation has occurred.

Once sample collection is complete, samples are placed in a cooler on ice to maintain a sample temperature no more than 4°C.

7.7.6.1 Sample Labels/Sample Identification

Label all groundwater samples with the following, written in indelible ink:

1. A unique sample number (see Section 3.9 for guidance)
2. Date and time
3. Parameters to be analyzed
4. Job number
5. Sampler's initial

Secure the label to the bottle. It is recommended that bottle labels be covered with wide clear tape to protect the label during sample packing and shipment. Pack glassware in appropriate packing material to deter breakage during sample packing and shipment. Sample labels can be prepared in advance in GHD offices that have label-generating programs.

An example of a groundwater sample log entry is provided on Figure 3.8.

Section 3.9 details sample labeling requirements for environmental sampling programs. Section 3.9 also details COC requirements and sample shipment requirements.

7.7.7 Purging/Sampling Equipment

GHD maintains a wide variety of purging and sampling equipment for well purging and groundwater sample collection. The groundwater sampler should be familiar with purging and sampling equipment and understand equipment limitations and proper use. Some equipment is very useful for well purging (i.e., high flow rates) but is not permissible for LFP or for sampling sensitive parameters (e.g., VOCs cannot be collected with a submersible (turbine) or suction pump). If the groundwater sampler understands the various equipment operation and limitations, the proper selection of purging and sampling equipment is made, which will minimize the purging and sampling duration and maximize productivity.

> Caution: Gas powered equipment requires special attention to ensure that staff hauling these units do not cause equipment or sample contamination. Frequent changes of disposable glove as well strict separation of sampling crew tasks (i.e., those handling pumps and hoses do not contact generator or are involved in any refueling activities) are required.

The following subsections describe the equipment available for groundwater sampling, the equipment use, approximate flow rates, and advantages and disadvantages of the equipment.

7.7.7.1 Peristaltic Pumps

A peristaltic pump is acceptable for purging wells and for most groundwater sample analytes. The groundwater sampler must ensure that a peristaltic pump is acceptable to regulatory agencies with local jurisdiction for VOC and SVOC sample collection. The QAPP will provide sampling requirements.

A peristaltic pump is capable of lifting water from a maximum depth of 25 feet (7.6 m) below ground surface or the pump, whichever is greater. A peristaltic pump is a self-priming, low volume, suction pump which consists of a rotor with ball bearing rollers. Flexible silicon tubing is inserted around or in the pump rotor and squeezed in place by the heads as they revolve in a circular pattern. The section of silicon tubing must not exceed 3 feet (0.9 m) in length. Additional rigid polyethylene or Teflon™ tubing is attached to the flexible tubing and placed in the well. Another piece of rigid tubing is attached to the discharge end of the flexible silicon tubing to facilitate sample collection. The entire length of rigid and flexible silicon tubing is dedicated to the well for future use. The tubing is typically tied and suspended in the well. The flexible or rigid tubing is not reused in other wells because cross-contamination will occur.

Note: Often a length of tubing is accidentally dropped into a well and can be difficult to retrieve. Retrieval can be accomplished by sending another piece of tubing down the well overlapping the lost section of tubing. Once in place, rotate the tubing, essentially wrapping or corkscrewing the lost tubing and new tubing together. After a number of turns are completed pull the tubing, hopefully with the lost section wound around the new piece. Repeat the procedure until successful.

Liquid is pulled into the tubing by the peristaltic pump through the creation of a vacuum as the rotor head turns. An advantage of using a peristaltic pump is that no pump parts come in direct contact with the sample. A peristaltic pump is capable of providing low flow sampling rates (i.e., typically less than 500 mL/min) with less agitation than other suction pumps. However, it is important that the tubing is secured during pumping to prevent the tubing from moving and causing agitation. A peristaltic pump also allows for regulation of the flow rate by increasing or decreasing the rotor head speed.

Peristaltic pumps are small and easily mobilized to remote sample locations. They require minimal setup, and do not require decontamination between sample locations. The disadvantages of a peristaltic pump are its limited lift and flow capabilities and the limited ability to collect VOC and SVOC samples. If VOC or SVOC sampling, check the QAPP to see if sampling with a peristaltic pump is allowed. Also check with regulatory agencies with local jurisdiction to see if the use of a peristaltic pump for collection of VOC and SVOC samples is acceptable. If using a peristaltic pump for purging, and the collection of VOCs and SVOC samples with the peristaltic pump is not acceptable, it is common to collect the initial VOC and SVOC analytes with a stainless steel bottom loading bailer. The peristaltic pump can then be used to collect the remaining sample analytes.

Peristaltic pumps are becoming more popular for LFP. However, it should be noted that a peristaltic pump may cause degassing, pH modification, and possible VOC loss.

7.7.7.2 Suction Pumps

A number of suction pumps (e.g., centrifugal) exist that can be used for purging applications only. A suction pump draws water through a suction line by creating a vacuum in the suction line or hose. Once drawn into the pump, the groundwater comes into direct contact with the pump rotor/pumping chamber area and it is therefore undesirable for groundwater sampling due to high groundwater agitation. Decontamination of suction pumps is extremely difficult. As with peristaltic pumps, most suction pumps have a limited lift capability of about 25 feet (7.6 m). Larger suction pumps, like

2-inch (5 cm) trash pumps, can achieve high flow rates under low hydraulic head. Flow rates of 15 to 20 U.S. gallons per minute (USgpm) (57 to 76 liters per minute [L/min]) can be achieved. This high flow rate minimizes purging time. New or dedicated suction line should be used at each well if a suction pump is used for purging.

Large suction pumps are also useful for well development, in conjunction with agitation and surging.

Large suction pumps are not suited for LFP due to degassing, pH modifications, VOC loss, and lack of flow adjustment.

Caution: The groundwater sampler must prevent the siphoning of purged water from a bulk container back into the well. For example, the following scenario is possible: Joe Sampler has completed purging well 'xyz' and has turned off the 2-inch trash pump. The trash pump discharge line is inserted into a wastewater tank and is submerged below the tank water level. As Joe prepares his glassware and sample pump, the wastewater tank contents are siphoned back into the well. This can result in cross contamination with water from other sites/wells which have been purged either:

- into the tank
- through the pump
- through the discharge line

All discharge lines/groundwater purge pumps must be provided with a check valve to prevent this situation.

Drilling rig pumps including Moyno, progressive cavity, bean, and mud pumps can be used for well purging and well development.

Suction pumps are a useful tool for high rate purging and well development. They require no additional equipment other than a suction line and discharge line for each well. They are mobile and easily transported around and between sites. Suction pumps are limited to use in wells with less than 25 feet (7.6 m) of lift, are difficult to decontaminate, and are unsuitable for sample collection. Large suction pumps are not suitable for LFP.

7.7.7.3 Submersible Pumps

A submersible pump generally provides high discharge rates for purging at depths beyond the capabilities of a suction pump. Based on its size, a submersible pump can pump water from substantial depths at very high pumping rates and can provide higher groundwater extraction rates than other methods. At high pumping rates, a submersible pump can cause agitation and aeration. This results in some submersible pumps not being suitable for the collection of groundwater samples for VOC and SVOC analysis.

Adjustable rate submersible pumps, constructed of stainless steel or Teflon™, are suitable and approved for LFP provided low flow rates are maintained.

The submersible pump, including the electrical cable and lowering cable, must be decontaminated between wells in accordance with the Work Plan or QAPP.

A submersible pump installed in bedrock or in a deep well should be attached to rigid piping (i.e., 3/4-inch (1.9 cm) steel) to allow for pulling or pushing of the pump. The pump may need to be pushed or pulled to the appropriate installation depth, past tight spots in the well, and when affixing the electrical cable and lowering the cable/safety line. Even when rigid piping is used, a safety line must be attached to the pump in case the piping becomes unthreaded or the pump connection is lost.

Submersible pumps can provide high flow rates that are useful for deep well or large diameter well purging activities. They tend to be labor intensive because of decontamination problems, power supply, and discharge piping size. Some submersible pumps are not suitable for some sample analytes. Small submersible pumps (i.e., 2-inch (5 cm) Grundfos™) have the proper construction and have adjustable flow rates, making them suitable for LFP.

7.7.7.4 Air Lift Pumps

An air lift pump operates using compressed air or nitrogen. The compressed air or nitrogen comes into direct contact with the groundwater and forces groundwater from the pump chamber through a series of check balls into the discharge line. An air lift pump operates on alternate pump discharge and pump recharge cycles. The pump and recharge cycles are controlled using a control box at ground surface. Air lifting is possible from deep depths with moderate to low flow rates (2 to 3 USgpm [7.6 to 11.5 L/min]) depending on the pump installation depth, static head, discharge tubing diameter, and air supply pressure.

Since the air or nitrogen comes in direct contact with the groundwater, an air lift pump should not be used for the collection of groundwater samples for VOC and SVOC analysis.

An air lift pump is a good tool for deep well purging and development. If an air lift pump is used for purging, an alternate sampling method will be required (e.g., bailers or bladder pump) for the collection of VOC and SVOC groundwater samples.

7.7.7.5 Bladder Pumps

Bladder pumps, as with air lift pumps, are driven by compressed air or nitrogen but the air or nitrogen does not come in contact with the groundwater. The contact between the air or nitrogen and the groundwater is eliminated by the presence of a Teflon™, polyethylene, or natural rubber bladder. The pump operation, as with the air lift pump, is cyclic and is controlled using a control box at ground surface. The control box controls the pump filling and discharge time. Because the air or nitrogen does not come in direct contact with the groundwater, and there is limited groundwater agitation and degassing, a bladder pump is the best sampling equipment for the collection of groundwater samples for VOC and SVOC analysis.

Bladder pump operation is very quiescent, causing little formation and well disturbance. By using a bladder pump, collecting a sediment-free groundwater sample is easily achieved. An adjustable rate bladder pump should be used for LFP. Bladder pumps generally are only able to achieve a maximum pumping rate of 1.5 USgpm (5.7 L/min). It is important to note that flow rates should be reduced in deep well applications.

Well purging and sampling can be performed using a bladder pump. Once sampling is completed, the pump should be disassembled and decontaminated in accordance with the Work Plan or QAPP prior to use in other wells. The sample tubing is generally 1/4- or 3/8-inch (6 or 10 mm) diameter polyethylene or Teflon™ lined polyethylene tubing. The air line is generally 1/4-inch (6 mm) polyethylene tubing. The sample and air line tubing are typically suspended in the well for future use (dedicated). At some sites a complete sampling system (bladder pump, discharge tubing, and air line) is dedicated to each well.

Bladder pumps provide excellent sample quality and are useful in deeper sampling applications. There are no analyte restrictions. Bladder pumps are strongly recommended for LFP applications.

Bladder pumps require additional equipment including control box, compressed air or nitrogen, and tubing. The setup of a bladder pump is quite labor intensive unless a dedicated system is in place. Decontamination of a bladder pump requires pump disassembly and re-assembly. Finally, bladder pumps are not capable of high flow rates, thus purging times tend to be increased slightly.

7.7.7.6 Inertia Pumps

An Inertia pump or Waterra™ pump is a manually operated or mechanically driven pump which uses only a foot valve on the sample/purge tubing. "Jerking" the sample/purge tubing with the attached foot valve removes groundwater from the well. The rapid lifting and lowering action of the tubing imparts an inertia to the water column within the sample/purge tubing. This causes the water column to rise to ground surface and discharge from the end of the sample/purge tubing. The foot valve holds the water column in the tubing during the lifting process and allows groundwater to enter the sample/purge tubing during the lowering, or down stroke.

GHD owns both manual and mechanical gas-powered inertia systems. Flow rates with inertia pumps are variable and are dependent on cycle speed, tubing size, foot valve size, well depth, and depth to groundwater. The inertia pump is a useful method for purging and for collection of most groundwater sample analytes. Acceptability of VOC and SVOC sampling with inertia pumps is gaining approval in selected areas. Prior to using an inertia pump as a sampling device, check the sampling requirements in the QAPP, or obtain approval from the Project Coordinator.

Inertia pumps are useful for the extraction of dense non-aqueous phase liquids (DNAPL). The only equipment that is exposed to the gross contamination is the foot valve and a small section of the sample/purge tubing. On most projects, the foot valve and sample/purge tubing are dedicated to the well.

Inertia pumps tend to cause extensive disturbance to the water column. The vigorous lifting and lowering of the inertia pump tends to make it difficult to collect sediment-free groundwater samples. Therefore, inertia pumps are not suitable for LFP.

7.7.7.7 Bailers

A bailer is a manual sampling device consisting generally of a hollow tube (e.g., Teflon™, PVC, or stainless steel) with a lower check ball that permits water entry and prevents water loss. The bailer is lowered slowly into the well. This allows water to enter the bailer through the bottom, and the weight of the water inside the bailer closes the check ball when the bailer is retrieved from the well.

A rope or cable is affixed to the bailer to allow the lowering and retrieval of the bailer from the well. Bailing tends to be disruptive to the water column and formation. Obtaining sediment-free groundwater samples using a bailer tends to be difficult, if not impossible. VOCs and SVOCs, as well as other analytes can be collected using a bailer, but it is important that these analytes be as sediment-free as possible. The compatibility of the bailer material and groundwater analytes should be reviewed and approved prior to using a bailer for the collection of groundwater samples. Generally, Teflon™ bailers are acceptable for the collection of most analytes.

Power winches with overhead tripods are available to assist in purging and sampling deep or large volume wells.

Flow rates attained using a bailer is a function of the bailer size and retrieval frequency. Retrieval frequency is dependent on well depth, water depth, and well recharge rate. Bailing is not practical for deep wells or for the removal of large well volumes.

A bailer is a useful tool for well development as the surging action from the bailer insertion and removal from the well promotes sediment suspension and subsequent removal. However, obtaining completely sediment-free samples, or samples below 50 NTU, is difficult if not impossible using a bailer.

A bailer provides representative samples once the well has been adequately developed and purged. A bailer is not suitable for LFP. Rope used for bailing must be kept off the ground and free of other contaminating material that could be introduced to the well. Rope can either be dedicated to the well for future use or discarded.

7.7.7.8 Passive Diffusion Bags

When sampling with diffusion bags the well must be fully developed using an alternate method.

A diffusion bag is a polyethylene bag that contains deionized water. The bag is attached to an appropriate length of rope or cable in order to be submerged to the appropriate depth (indicated in the Work Plan, QAPP, or as instructed by the Project Coordinator). Cable or rope used to suspend diffusion bags can be dedicated to the well for future use or discarded.

Once submerged to the appropriate depth, the diffusion bag is left in the well for an extended period of time, usually 14 days, to allow the bag to equilibrate with the water in the well. The use of diffusion bags eliminates well purging prior to sampling. Placement of multiple diffusion bags in a well allows for vertical groundwater profiling.

Diffusion bags are a low cost method for the collection of groundwater samples. Advantages include:

- No purge water to dispose of.
- No equipment decontamination between wells.
- Simple logistics and operation.
- Reduction in personnel and exposure times.
- Samples collected are representative of formation water adjacent to well.

- Allow for vertical profiling of water column.
- Appropriate for long-term monitoring programs.

The disadvantage of diffusion bags is the length of equilibrium time, generally 14 days. Currently, there are membranes available for diffusion bags suitable for the collection of groundwater samples for select SVOC, and metals analyses. However, there are no membranes currently available for polychlorinated biphenyls (PCBs).

Note: Handle diffusion bags only when wearing clean nitrile or surgical gloves.

7.7.8 Filtering of Groundwater Samples

Filtering is an important process to remove suspended particulate that affect sample results. Filtration of groundwater samples is generally limited to metals analysis.

Filtering can be completed in the field using in-line filters or a vacuum filter kit. Filtering of samples can also be completed by the laboratory, in which case the samples must not be preserved and must be at the laboratory in at least 24 hours of sample collection.

7.8 Field Procedures for Residential Sampling

7.8.1 General

When sampling potable water supply wells it is important to ensure that the samples collected are representative of the aquifer being sampled. Poor or incorrect sampling techniques will result in erroneous sample results that can be disclosed to the public. Incorrect sample results may make any changes in the public perception hard to accomplish when correct results are reported.

7.8.2 Field Procedures

The requirements of a residential well sampling program should be reviewed with the Project Coordinator prior to initiating sampling activities. While similar field procedures used in groundwater sampling (including documentation, sample identification, date, time, etc.) are required in residential well sampling, additional procedures are also required.

Prior to collection of groundwater samples from a residential well, the well must be purged to ensure that samples collected are representative of the formation. Purging removes standing water from the well casing, pipes, and pressure or holding tank. Purging of a residential well requires the removal of one well volume. If access to the well is not available to determine the well volume, purging for a period of 15 to 30 minutes is generally sufficient. Field measurements for pH, conductivity, and temperature are recorded during purging activities until the readings indicate that stabilization has occurred.

Sampling of residential wells is generally performed using the existing pumping system. However, GHD purging and sampling equipment can be used. It is important that only designated **clean** purging and sampling equipment be used for residential well sampling. The use of the existing pumping system is preferred, as this is more representative of the water quality provided to the

residence. Using the existing pumping system also minimizes the possibility of damaging the well and existing pumping system when installing additional purging and sampling equipment.

If GHD equipment is used for residential well sampling, it must be cleaned prior to and between use with a bleach and deionized water solution wash followed by a thorough deionized water rinse.

> Note: In addition to the special technical procedures noted, GHD personnel must be aware of this unique situation of conducting sampling at private residences. Special care must be taken to be polite and courteous at all times. Offer only necessary information and maintain a clean work area that is returned to pre-sampling conditions. Personnel should have proper identification available, and only remain in areas long enough to complete the required tasks.

Taps selected for residential well sampling should be located as close to the well as possible. Locate the taps before any treatment systems and, if possible, the pressure tank. It is important to note, if possible, all water treatment devices in operation at the residence including:

- Water softeners
- Filtration units
- Ultraviolet light
- Reverse osmosis
- Distillers
- Chlorinators

Leaking taps that allow water to flow from the stem of the valve handle and around the tap should not be used as sampling locations. Aerators, strainers, and hose attachments should be removed prior to sampling. Maintain a steady flow of water during sampling activities to avoid pressure fluctuations that may cause sheets of microbial growth lodged in the pipes to break loose. Open the cold water tap for a period of 15 to 30 minutes to allow for the complete purging of the pumping system. Maintain a smooth-flaring water stream at a low to moderate pressure without splashing. Do not change the flow rate. Changes in the flow could dislodge particles in the pipes or faucet.

When sampling for microbiological parameters, the end of the faucet must be flame sterilized. During residential well sample, never place caps from sample containers on the ground or in a pocket. Instead, hold the sample container in one hand and the sample container cap in the other. Be very careful not to touch the inside of the sample container cap. Wear new disposable gloves at each sampling location and following contact with a potential contaminant source. The inside of the sample bottle must not be touched with bare hands or allowed to contact the surface of the faucet.

7.8.3 Field Notes for Residential Sampling

Full documentation of each residential well is required and includes:

1. Well depth
2. Casing construction and diameter

3. Well installation date if known
4. Pumping system configuration
5. Piping system construction (e.g., copper, lead-joint, ABS)
6. Presence of treatment devices

Obtain the name and exact mailing address for all residence or well owners, as well as home and work telephone numbers. This information is required to inform the residence or well owner of the results of the sampling activities.

Document residential well sampling activities in a standard GHD field book. Figure 3.8 provides typical residential well sampling field note requirements. Note that additional documentation of well details, treatment devices, piping system, and special circumstances are required in the field book in addition to the sample log entry.

7.9 Field Procedures for Surface Water Sampling

7.9.1 General

Surface water sampling is performed to obtain samples for surface water bodies that are representative of existing surface water conditions.

Surface water sampling locations for surface water quality and groundwater interaction studies are selected based on the following:

1. Study objectives
2. Location of point surface discharges
3. Non-point source discharges and tributaries
4. Presence of structures (e.g., bridge, dam)
5. Accessibility

During surface water sampling it is important to obtain samples that are not impacted by the re-suspension of sediment produced because of improper or poor surface water sampling techniques.

7.9.2 Surface Water Sample Location Selection

Prior to conducting surface water sampling activities, the first requirement is the consideration and development of surface water sampling locations. It is important that all surface water sampling locations be selected in accordance with the Work Plan and described to and discussed with the Project Coordinator.

Bridges and piers are good locations for surface water sampling locations since they provide easy access and permit water sampling across the entire width of the surface water body. The JSA for sampling from bridges must include a traffic management plan to assure the employee has considered using a spotter, signage, cones, and flags to warn car traffic of the work adjacent to the

roadway. Wading for surface water samples increases the chances of disturbance of sediments from the floor of the surface water body.

When wading for surface water samples in lakes, ponds, streams, and slow moving rivers be aware of potential safety and health risks. A life vest and safety line must be worn at all times where footing is unstable or when sampling in fast moving or more than 3 feet (0.9 m) deep. A two-person team is required for most surface water sampling activities, a Project Manager must approve a one person sampling team. If the site conditions require the use of the life vest and safety line, the two people involved in the sampling must be competent swimmers.

Surface water samples must be collected with no suspended sediments. Surface water samples are collected commencing with the furthest downstream location to avoid sediment interference with upstream locations.

7.9.2.1 Rivers, Streams, and Creeks

Surface water samples are generally collected in areas of surface water bodies that are representative of the surface water body conditions. Representative surface water samples will usually be collected in sections of surface water bodies that have a uniform cross section and flow rate. Mixing is influenced by turbulence and water velocity, therefore the selection of surface water sampling locations immediately downstream of a riffle area (i.e., fast flow zone) will ensure good vertical mixing. These locations are also likely areas for deposition of sediment since this occurs in areas of decreased flow velocity.

Surface water sampling locations should not be established in areas near point source discharges including tributaries, industrial effluents, and municipal effluents. Surface water sampling of these source discharge points can be performed to assess the impact of these source areas on overall surface water quality.

Sample tributaries as close to the mouth as possible. It is important to select surface water sample locations considering the impact downstream, including tributary flow and sediment.

In all instances, properly document all surface water sampling locations in a standard GHD field book. Documentation may include photographs and tie-ins to known structures.

7.9.2.2 Lakes, Ponds, and Impoundments

The surface water in lakes, ponds, and impoundments has a greater tendency to be stratified than water in rivers and streams. Lack of mixing in these surface water bodies may require additional surface water sample collection. Extreme turbidity variances may occur where highly turbid surface water courses enter a lake or pond. Therefore, each layer of the stratified surface water column may need to be considered separately. Stratification is generally a result of water temperature differences, with cooler heavier water being trapped below warmer water.

Surface water sample locations for lakes, ponds, and impoundments should adequately represent the conditions of the surface water body. All intakes and outflows that may provide biased surface water representation should be identified and documented. Surface water sample locations with adjacent structures (e.g., banks, piers) may also provide biased samples, as the potential for boundary flow and eddies exists.

The number of surface water sample locations on lakes, ponds, or impoundments will vary depending on the purpose of the investigation, as well as the size and shape of the surface water body. In ponds and small impoundments a single surface water sample should be collected at the deepest point. In naturally formed ponds, the deepest point is usually near the center of the surface water body. In impoundments the point is usually near the dam.

In lakes and larger impoundments, several sub-samples should be taken to form a single composite sample. These vertical surface water sampling locations are collected along a pre-determined grid.

In irregular shaped lakes with several bays and covers that are protected from the wind, additional surface water samples are required to properly represent surface water quality at various locations in the lake. Additional surface water samples should be taken at discharges, tributaries, and other factors or sources that are suspected of affecting the surface water quality.

In all instances, properly document all surface water sampling locations in a standard GHD field book. Documentation may include photographs and tie-ins to known structures.

7.9.3 Sampling Equipment and Techniques

When collecting surface water samples, direct dipping of the sample container into the stream or water is acceptable unless the sample container contains preservatives. If preserved, a pre-cleaned unpreserved sample container should be used to collect the surface water sample. The surface water sample is then transferred to the appropriate preserved sample container. When collecting surface water samples, submerse the inverted bottle to the desired sample depth and tilt the opening of the sample container upstream to fill. During surface water sample collection, wading or movement may cause sediment deposits to be re-suspended and can result in biased samples. Wading is acceptable if the stream has a noticeable current and the samples are collected directly in the sample container when faced upstream. If the stream is too deep to wade in or if addition samples must be collected at various depths, additional sampling equipment will be required. Surface water samples should be collected about 6 inches (15 cm) below the surface, with the sample bottles being completely submerged. Taking the surface water sample at this depth eliminates the collection of floating debris in the sample container.

Surface water sample collection where the flow depth is less than 1 inch (<2.5 cm) requires the use of special equipment to eliminate sediment disturbance. Surface water sampling may be conducted with a container then transferred to the appropriate sample container, or collection may be performed using a peristaltic pump. A small excavation in the stream bed to create a sump for sample collection can also be considered but should be prepared in advance to allow all the sediment to settle prior to surface water sampling activities.

Teflon™ bailers can be used for surface water sampling if it is not necessary to collect surface water samples at specific depths. A bottom loading bailer with a check ball is sufficient. When the bailer is lowered through the water, the water is continually displaced through the bailer until the desired depth is reached. The bailer is retrieved and the check ball prohibits the release of the collected surface water sample. Bailers are not suitable in surface water bodies with strong currents, or where depth-specific sampling is required.

For discrete and specified depth surface water sampling, and the parameters to be monitored do not require a Teflon™ coated sampling device, a standard Kemmerer or Van Dorn sampler can be used. The Kemmerer sampler is a brass cylinder with rubber stoppers that leave the sampler ends open while the sampler is being lowered. The sampler is lowered in a vertical position to allow water to pass through. The Van Dorn sampler is plastic and is lowered in a horizontal position. For both samplers, a messenger is sent down a rope when the sampler has reached the required depth. The messenger causes the stopper on the sampler to close. The sampler is then retrieved and the surface water sample can be collected through a valve. DO sample bottles can be filled by allowing overflow using a rubber tube attached to the valve. During depth-specific surface water sampling, take care not to disturb bottom sediments.

Glass beakers or stainless steel cups may also be used to collect surface water samples if parameter interference does not occur. The beaker or cup must be rinsed at least three times with the surface water sample prior to sample collection.

All equipment must be thoroughly decontaminated as outlined in Section 7.6.

7.9.4 Field Notes for Surface Water Sampling

Use a standard GHD field book to record daily surface sampling activities, describe surface water sampling locations, sampling techniques, and, if applicable, provide a description of photographs taken. Visual observations are important and provide valuable information when interpreting surface water quality results. Observations include:

1. Weather conditions
2. Stream flow directions
3. Stream physical conditions (width, depth, etc.)
4. Tributaries
5. Effluent discharges
6. Impoundments
7. Bridges
8. Railway trestles
9. Oil sheens
10. Odors
11. Buried debris
12. Vegetation
13. Algae
14. Fish and other aquatic life
15. Surrounding industrial areas

The following factors should be considered for surface water sampling:

1. Predominant Surrounding Land Use: Observe the prevalent land use type in the vicinity and note any other land uses in the area which, although not dominant, may potentially affect surface water quality.
2. Local Watershed Erosion: Note the existing or potential erosion of soil in the local watershed and its movement into the stream. Erosion can be rated through visual observation of watershed stream characteristics including increases or decreases in turbidity.
3. Local Watershed Non-Point Source Pollution: This refers to problems or potential problems other than erosion and sedimentation. Nonpoint source pollution can be diffuse agricultural and urban runoff. Other factors may include feed lots, wetlands, septic systems, dams, impoundments, and mine seepage.
4. Estimated Stream Width: The estimated distance from shore at a transect representative of the stream width in the area.
5. Estimated Stream Depth: Riffle (rocky area), run (steady flow area), and pool (still area). Estimate the vertical distance from the water surface to the bottom of the surface water body at a representative depth at three locations.
6. High Water Mark: Estimate the vertical distance from the bank of the surface water body to the peak overflow level, as indicated by debris hanging in bank or flood plain vegetation, and deposition of silt. In instances where bank flow is rare, high water marks may not be evident.
7. Velocity: Record or measure the stream velocity in a representative run area.
8. Dam Present: Indicate the presence or absence of a dam upstream or downstream of the surface water sampling location. If a dam is present, include specific information detailing the alteration of the surface water flow.
9. Channelized: Indicate if the area surrounding the surface water sampling location is channelized.
10. Canopy Cover: Note the general proportion of open to shaded areas which best describes the amount of cover at the surface water sampling location.

7.10 Follow-Up Activities

The following should be performed once groundwater, residential, and surface water sampling is completed:

1. Double check the Work Plan and QAPP to ensure all samples and QA/QC samples have been collected and confirm with the Project Coordinator.
2. Decontaminate all equipment at the site then return clean to the appropriate office equipment manager.
3. Dispose of purge water and cleaning fluid as specified in the Work Plan.
4. Notify the contract laboratory when the samples should arrive. Enclose a completed chain-of-custody in each cooler.

5. Complete and file the appropriate forms and data sheets. Also file the field notes. For groundwater, residential, and surface water sampling these forms include:
    - Project Planning, Completion, and Follow-Up Checklist (Form SP-02)
    - Well Development, Purging, and Sampling Form (Form SP-06)
    - Sample Collection Data Sheet - Groundwater Sampling Program (Form SP-08)
    - Monitoring Well Record for Low-Flow Purging (if performed) (Form SP-09)
6. Return site and well keys.

7.11 References

For additional information pertaining to groundwater sampling activities the user of this manual may reference the following:

ASTM D5474 Guide for Selection of Data Elements for Groundwater Investigations

ASTM D4696 Guide for Pore-Liquid Sampling from the Vadose Zone

ASTM D5979 Guide for Conceptualization and Characterization of Groundwater Systems

ASTM D5903 Guide for Planning and Preparing for a Groundwater Sampling Event

ASTM D4448 Standard Guide for Sampling Groundwater Wells

ASTM D6001 Standard Guide for Direct-Push Water Sampling for Geoenvironmental Investigations.

For additional information pertaining to surface water sampling, the user of this manual may reference the following:

ASTM D5358 Practice for Sampling with a Dipper or Pond Sampler

ASTM D4489 Practices for Sampling of Waterborne Oils

ASTM D3325 Practice for the Preservation of Waterborne Oil Samples

ASTM D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents

ASTM D4411 Guide for Sampling Fluvial Sediment in Motion

ASTM D4823 Guide for Core-Sampling Submerged, Unconsolidated Sediments

ASTM D3213 Practice for Handling, Storing, and Preparing Soft Undisturbed Marine Soil

ASTM D3976 Practice for Preparation of Sediment Samples for Chemical Analysis

ASTM E1391 Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing

ASTM D4581 Guide for Measurement of Morphologic Characteristics of Surface Water Bodies

ASTM D5906 Guide for Measuring Horizontal Positioning During Measurements of Surface Water Depths

ASTM D5073 Practice for Depth Measurement of Surface Water

Attachment C

Laboratory Standard Operating Procedures

Title: Analytical Methods for GC/MS Semivolatile Samples by SW846 8270D, 8270E, MCP, RCP and SIM

Once printed, this document is considered an uncontrolled document.

Approvals (Signature/Date):

Robert Schick 10/27/20	Mark Nemec 10/28/20
Robert Schick — Date	Mark Nemec — Date
Department Manager	Laboratory Director
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Quality Assurance Manager	Organic Ops Manager

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

This SOP contains the procedures for the determination of extractable semi-volatile organic compounds (SVOC) by gas chromatography/mass spectrometry (GC/MS).

Procedures for analyzing via Large Volume Injection (LVI), Low Level analysis and Selective Ion Monitoring (SIM) are also included in this SOP.

Technical acceptance criteria and corrective actions for MCP and RCP analysis are also included in this SOP.

The routine matrices performed by this procedure are waters and soils. Other matrices which may be performed include wipes, leachates, and wastes.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

2.0 Summary of Method

A measured volume or weight of sample is extracted using separatory funnels (3510C, 3510C_LVI), sonication (3550C) or microwave (3546) extraction procedures. The extract is then analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the relative abundance of the characteristic masses as compared to component reference spectra determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis of the target compounds is performed by the internal standard technique using a single characteristic ion. Quantitative analysis of the SIM method is performed by Isotopic Dilution.

3.0 Definitions

MCP – Massachusetts Contingency Plan
RCP – Connecticut Reasonable Confidence Protocols
SIM – Selective Ion Monitoring
Additional definitions can be found in the Eurofins TestAmerica Buffalo Laboratory Quality Manual (QAM)

4.0 Interferences

Some of the possible interferences that arise during GCMS Semivolatile analysis include, but are not limited to:

1. Glassware contamination
2. Matrix interference
3. Aldol condensation
4. System air leaks
5. Injection port/liner contamination
6. Warped filament, and/or dirty source

Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

Phthalate contamination is commonly observed in the LVI and Low Level analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with Eurofins TestAmerica Buffalo SOP BF-GP-003, current revision, to ensure it is free from contaminants and does not contribute artifacts.

High purity reagents and solvents are used to help minimize interference problems. Acetone and methylene chloride must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program (SOP CA-Q-S-001, current revision) and Eurofins TestAmerica Buffalo SOP BF-OP-013, current revision.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Chemicals that have been classified as carcinogens or potential carcinogens in association with this method, defined by OSHA include: Acrylamide, Benzo(a)anthracene, Benzidine, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Dibenz(a,h)acridine, Dibenz(a,h)anthracene, Dibenzo(a,e)pyrene, 1,4-Dichlorobenzene, 3,3'-Dichlorobenzidine, 1,4-Dioxane, Hexachlorobenzene, Hexachloroethane, Kepone, Methyl Methanesulfonate, Methylene Chloride, Naphthalene, 1-Naphthylamine, 2-Naphthylamine Nitrobenzene, n-Nitrosodimethylamine, n-Nitrosodiethylamine, n-Nitrosodi-n-butylamine, n-Nitrosodi-n-propylamine, n-Nitrosopiperidine, n-Nitrosopyrrolidine, Safrole, o-Toluidine and 2,4,6-Trichlorophenol. This list can be obtained from the TestAmerica Corporate Safety Manual CW-E-M-001, Appendix XII (current revision). Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

Analysts are expected to use caution and common sense while working in a laboratory environment. Each employee is required to read the TestAmerica Corporate Safety Manual. All of the samples to be analyzed have the potential to contain hazardous substances. Most standards also contain hazardous chemicals and many do contain known carcinogens. Employees must use protective equipment when handling standards, samples and extracts including gloves, lab coats and safety glasses. It is the analyst's responsibility to read and familiarize themselves with the SDS of each chemical and/or reagent involved in this method.

Samples, standards and/or extracts should never be opened or transferred outside of a fume hood.

Liquid waste disposal is all C waste with the exception of some acids used in the cleaning of equipment which is disposed of in AN waste.

Spills should be cleaned up promptly and waste should be disposed of as per the Chemical Hygiene Plan.

There is also the danger of burns while doing repair or maintenance on a gas chromatograph. One must use caution while working on or near the injection port or transfer line.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety data Sheets (SDS) for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Chart 1

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Micro syringes 10, 25, 50, 100, 500, 1000 microliter.

6.2 2mL amber and clear glass vials and caps.

6.3 Disposable pipets and pipet bulbs.

6.4 Volumetric flasks.

6.5 Instrumentation
Gas Chromatograph/Mass Spectrometer (GC/MS) System

6.5.1 Gas Chromatograph -
- Hewlett Packard 6890
- Carrier gas Helium UPC grade or equivalent

6.5.2 Gas Chromatography Column
- Analysis: Restek 5Sil MS with or without Integra-Guard cat# 13623 (without guard) 13623-127 (with guard) or equivalent

6.5.3 Mass Spectrometer
- HP5973 and HP5973 inert
- Tuning compound PFTBA
- Scan Range 35-500 AMU

6.5.4 Data System
- HP Chemstation
- Chrom chromatography analysis system
- TALS data analysis system

7.0 Reagents and Standards

7.1 Methylene Chloride – high purity

7.2 Stock Standards

7.2.1 Corporate approved Primary and Second Source Restek mixtures:

8270 List1/Std #1 MegaMix	8270 List2/Std #1	8270 List2/Std #5
8270 List1/Std #9	8270 List2/Std #2	8270 List2/Std #7
8270 List1/Std #10	8270 List2/Std #3	8270 Internal Standard
8270 List1/Std #11	8270 List2/Std #4	8270 Surrogate Standard

7.2.2 Equivalent vendor mixtures:

Semi-Volatile GC/MS Tuning Standard (Agilent)
Custom List 3 Mix (SPEX)
Custom List 3/Std#2 Mix (Restek)
Simazine (Absolute Standards, Inc.)
Phthalic anhydride (Absolute Standards, Inc.)
TetraEthyl Lead (Absolute Standards, Inc.)
1,4-Dioxane Stock for SIM (Restek)
1,4-Dioxane-d8 labeled analog for SIM (Restek)
Hexachlorophene (Restek)
Dibenzo(a,e)pyrene (Absolute Standards, Inc.)

All Certificates of Analysis received from the manufacturer are maintained in the laboratory's LIMS system.

7.3 Working Standards

7.3.1 Surrogate Standard Spiking Solution

Surrogate Standard spiking solution is prepared by the extractions department that contains nitrobenzene-d5, p-terphenyl1-d14, 2-fluorobiphenyl, phenol-d5, 2,4,6-tribromophenol and 2-fluorophenol at a concentration of 40µg/mL for the 3510C, 3550C and 3546 extractions and 8ug/mL for the 3510C_LVI and low level extractions. Surrogate standards are added to all QC and client samples. Additional surrogates may be added at the laboratory's discretion.

The isotopically labeled analog 1,4-Dioxane-d8 used in the SIM analysis is prepared by the extractions department at a concentration of 10 ug/mL. This is added to all QC and client samples.

7.3.2 Laboratory Control Sample and Matrix Spiking Solution

Laboratory Control Sample and Matrix spiking solution is prepared by the extractions department that contains each of the base-neutral compounds and acid compounds at 50ug/mL for the 3510C, 3550C and 3546 extractions and 8ug/mL for the 3510C_LVI and low

level extractions. SIM spiking solution is prepared that contains 1,4-Dioxane at a concentration of 1ug/mL.

7.3.3 Instrument Performance Check Solution (DFTPP)

A solution of Decafluorotriphenylphosphine (DFTPP) is prepared at a concentration of 50ug/mL in methylene chloride for soil analysis, as well as 1Liter water extractions.

For LVI, Low Level analysis and SIM, the concentration of this solution is prepared at 10ug/mL.

The instrument performance check solution contains 50ug/mL and 10ug/mL respectively of Benzidine, Pentachlorophenol and 4,4'-DDT for use in evaluating chromatographic performance.

Chart 2 – DFTPP Check Solution

DFTPP Working reagent (MB_DFTPP_WRK)	Solvent	Stock Conc. (ug/mL)	Initial Volume (uL)	Final Volume (mL)	Final Conc. (ug/mL)
1L Water/Soil	$MeCL_2$	1000	500	10	50
LVI/LL Water/SIM	$MeCL_2$	1000	100	10	10

7.3.4 Initial and Continuing Calibration Standards

Calibration standards are prepared at a minimum of five concentration levels from a working intermediate mix. For the main list of compounds, List 1, each calibration standard shall contain each compound of interest and each surrogate. A six and seventh level may be added for 2nd order quadratic curves.

Chart 3 – 8270 List 1 Working Intermediate Mix

8270 Working Intermediate Calibration Mix (MB_List1_INT)	Solvent	Stock Conc. (µg/ml)	Initial Vol. (uL)	Final Vol. (mL)	Final Conc. (µg/mL)
8270 List 1/Std #1 Mega mix	$MeCl_2$	1000	1000	5	200
8270 List 1/Std #9	$MeCl_2$	2000	500	5	200
8270 List 1/Std #10	$MeCl_2$	2000	1500	5	600
8270 List1/Std #11	$MeCl_2$	2000	500	5	200
8270 Surrogate Standard	$MeCl_2$	5000	200	5	200

Chart 4 - 1 Liter Water/Soil Calibration Levels

Calibration Level (ppm) (MB_LIST1_WRK)	Reagent Added	Solvent	Stock Conc. (µg/mL)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
2.5	MB_LIST1_INT	$MeCl_2$	200	62.5	5	2.5
	Internal Standard		2000	100		40.0
5	MB_LIST1_INT	MeCl2	200	125	5	5
	Internal Standard		2000	100		40.0
20	MB_LIST1_INT	MeCl2	200	100	1	20
	Internal Standard		2000	20		40.0
50	MB_LIST1_INT	MeCl2	200	1250	5	50
	Internal Standard		2000	100		40.0
80	MB_LIST1_INT	MeCl2	200	400	1	80
	Internal Standard		2000	20		40.0
120	MB_LIST1_INT	MeCl2	200	600	1	120.0
	Internal Standard		2000	20		40.0
160	MB_LIST1_INT	MeCl2	200	800	1	160
	Internal Standard		2000	20		40

Chart 5 – 8270 List 1 LVI Working Intermediate Mix

8270 Working Intermediate Calibration Mix (MB_L1LVI_INT)	Solvent	Stock Conc. (µg/ml)	Initial Vol. (uL)	Final Vol. (mL)	Final Conc. (µg/mL)
8270 List 1/Std #1 Mega mix	$MeCl_2$	1000	1000	10	100
8270 List 1/Std #9	$MeCl_2$	2000	1000	10	200
8270 List 1/Std #10	$MeCl_2$	2000	2500	10	500
8270 List1/Std #11	$MeCl_2$	2000	1000	10	200
8270 Surrogate Standard	$MeCl_2$	5000	200	10	100

Chart 6 - LVI/Low Level Water Calibration Levels

Calibration Level (ppm) (MB_L1LVI_WRK)	Reagent Added	Solvent	Stock Conc. (µg/mL)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
0.25	MB_L1LVI_INT	$MeCl_2$	200	25	10	0.25
	Internal Standard		2000	20		4.0
1.0	MB_L1LVI_INT	MeCl2	200	100	10	1.0
	Internal Standard		2000	20		4.0
2.0	MB_L1LVI_INT	MeCl2	200	200	10	2.0
	Internal Standard		2000	20		4.0
4.0	MB_L1LVI_INT	MeCl2	200	400	10	4.0
	Internal Standard		2000	20		4.0
8.0	MB_L1LVI_INT	MeCl2	200	800	10	8.0
	Internal Standard		2000	20		4.0
12	MB_L1LVI_INT	MeCl2	200	1200	10	12.0
	Internal Standard		2000	20		4.0
16	MB_L1LVI_INT	MeCl2	200	1600	10	16.0
	Internal Standard		2000	20		4.0

An additional calibration level is added, when required, for Low Level PAH analysis for 1L water/soil samples and LVI/Low Level water samples. For 1L/soil analysis, a 0.5ug/mL standard is prepared with 12.5uL of MB_List1_INT and 100uL of Internal Standard into 5mL of $MeCl_2$. For LVI/Low Level water samples, a 0.125ug/mL standard is prepared with 12.5uL of MB_L1LVI_INT and 20uL of Internal Standard into 10mL of $MeCl_2$.

Additional calibration standards may be analyzed to include compounds not in the main list (List 1). Surrogate analytes are not added to additional calibration mixes and are only calibrated from List 1. Additional routine calibrations are List 2 and List 3 and are prepared using Charts 7 through 12.

Chart 7 - 8270 List 2 Working Intermediate Mix

8270 Working Intermediate Calibration Mix (MB_List2_INT)	Solvent	Stock Conc. (µg/ml)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
8270 List 2/Std #1	$MeCl_2$	1000	2000	10	200
8270 List 2/Std #2	$MeCl_2$	1000	2000	10	200
8270 List 2/Std #3	$MeCl_2$	2000	1000	10	200

8270 List2/Std #4	$MeCl_2$	1000	2000	10	200
8270 List2/Std #5	$MeCl_2$	2000	1000	10	200

Chart 8 - 1 Liter Water/Soil Calibration Levels
List 2

Calibration Level (ppm) (MB_LIST2_WRK)	Reagent Added	Solvent	Stock Conc. (µg/mL)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
2.5	MB_LIST2_INT	$MeCl_2$	200	62.5	5	2.5.0
	Internal Standard		2000	100		40.0
5	MB_LIST2_INT	MeCl2	200	125	5	5.0
	Internal Standard		2000	100		40.0
20	MB_LIST2_INT	MeCl2	200	100	1	20.0
	Internal Standard		2000	100		40.0
50	MB_LIST2_INT	MeCl2	200	1250	5	50.0
	Internal Standard		2000	20		40.0
80	MB_LIST2_INT	MeCl2	200	400	1	80.0
	Internal Standard		2000	20		40.0
120	MB_LIST2_INT	MeCl2	200	600	1	120.0
	Internal Standard		2000	20		40.0
160	MB_LIST2_INT	MeCl2	200	800	1	160.0
	Internal Standard		2000	20		40.0

Chart 9 - LVI/Low Level Water Calibration Levels
List 2

Calibration Level (ppm) (MB_L2LVI_WRK)	Reagent Added	Solvent	Stock Conc. (µg/mL)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
0.25	MB_LIST2_INT	$MeCl_2$	200	12.5	10	0.25
	Internal Standard		2000	20		4.0
1.0	MB_LIST2_INT	MeCl2	200	50	10	1.0
	Internal Standard		2000	20		4.0
2.0	MB_LIST2_INT	MeCl2	200	100	10	2.0
	Internal Standard		2000	20		4.0
4.0	MB_LIST2_INT	MeCl2	200	200	10	4.0
	Internal Standard		2000	20		4.0

8.0	MB_LIST2_INT	MeCl2	200	400	10	8.0
	Internal Standard		2000	20		4.0
12	MB_LIST2_INT	MeCl2	200	600	10	12.0
	Internal Standard		2000	20		4.0
16	MB_LIST2_INT	MeCl2	200	800	10	16.0
	Internal Standard		2000	20		4.0

Chart 10 - 8270 List 3 Working Intermediate Mix

8270 Working Intermediate Calibration Mix (MB_List3_INT)	Solvent	Stock Conc. (µg/ml)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
MB_L3S2_STK	$MeCl_2$	2000	1000	10	200
MB_L2S7_STK	$MeCl_2$	2000	2000	10	400
MB_List3_STK	$MeCl_2$	2000	1000	10	200
MB_PHTHA_STK	$MeCl_2$	2000	1000	10	200
MB_TEL_STK	$MeCl_2$	2000	1000	10	200
MB_SIMAZ_STK	MeCl2	1000	2000	10	200

Chart 11 - 1 Liter Water/Soil Calibration Levels
List 3

Calibration Level (ppm) (MB_LIST3_WRK)	Reagent Added	Solvent	Stock Conc. (µg/mL)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
2.5	MB_LIST3_INT	$MeCl_2$	200	62.5	5	2.5
	Internal Standard		2000	100		40.0
5	MB_LIST3_INT	MeCl2	200	125	5	5.0
	Internal Standard		2000	20		40.0
20	MB_LIST3_INT	MeCl2	200	100	1	20.0
	Internal Standard		2000	100		40.0
50	MB_LIST3_INT	MeCl2	200	1250	5	50.0
	Internal Standard		2000	20		40.0
80	MB_LIST3_INT	MeCl2	200	400	1	80.0
	Internal Standard		2000	20		40.0
120	MB_LIST3_INT	MeCl2	200	600	1	120.0

	Internal Standard		2000	20		40.0
160	MB_LIST3_INT	MeCl2	200	800	1	160.0
	Internal Standard		2000	20		40.0

Chart 12 - LVI/Low Level Water Calibration Levels
List 3

Calibration Level (ppm) (MB_L3LVI_WRK)	Reagent Added	Solvent	Stock Conc. (µg/mL)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
0.25	MB_LIST3_INT	$MeCl_2$	200	12.5	10	0.25
	Internal Standard		2000	20		4.0
1.0	MB_LIST3_INT	MeCl2	200	50	10	1.0
	Internal Standard		2000	20		4.0
2.0	MB_LIST3_INT	MeCl2	200	100	10	2.0
	Internal Standard		2000	20		4.0
4.0	MB_LIST3_INT	MeCl2	200	200	10	4.0
	Internal Standard		2000	20		4.0
8.0	MB_LIST3_INT	MeCl2	200	400	10	8.0
	Internal Standard		2000	20		4.0
12	MB_LIST3_INT	MeCl2	200	600	10	12.0
	Internal Standard		2000	20		4.0
16	MB_LIST3_INT	MeCl2	200	800	10	16.0
	Internal Standard		2000	20		4.0

SIM calibrations are prepared using Charts 12 and 13.

Chart 13 – SIM Working Intermediate Mix

SIM Working Intermediate Calibration Mix (MB_1,4SIM_INT)	Solvent	Stock Conc.	Initial Vol.	Final Vol.	Final Conc.
1,4-Dioxane Stock	MeCl2	2000µg/ml	100 uL	10 mLs	20µg/mL
1,4-Dioxane-d8 Stock	MeCl2	2000µg/ml	1000 uL	10 mLs	200ug/mL

Chart 14 – SIM Calibration Levels

Calibration Level (ppm) (MB_LIST1_WRK)	Reagent Added	Solvent	Stock Conc. (µg/mL)	Initial Vol. (µL)	Final Vol. (mL)	Final Conc. (µg/mL)
0.2	MB_1,4SIM_INT	$MeCl_2$	20/200	10	1.0	0.2/2.0
	Internal Standard		50	20		1.0
0.4	MB_1,4SIM_INT	MeCl2	20/200	20	1.0	0.4/4.0
	Internal Standard		50	20		1.0
0.6	MB_1,4SIM_INT	MeCl2	20/200	30	1.0	0.6/6.0
	Internal Standard		50	20		1.0
0.8	MB_1,4SIM_INT	MeCl2	20/200	40	1.0	0.8/8.0
	Internal Standard		50	20		1.0
1.0	MB_1,4SIM_INT	MeCl2	20/200	50	1.0	1.0/10.0
	Internal Standard		50	20		1.0
1.2	MB_1,4SIM_INT	MeCl2	20/200	60	1.0	1.2/12.0
	Internal Standard		50	20		1.0

7.3.5 Internal Standard Solution

Internal standard used in the analysis of 1L/soil samples is from the stock standard, which contains the following compounds at a concentration of 2000ug/mL: 1,4-Dichlorobenzene-d4, Acenaphthalene-d10, Chrysene-d12, Naphthalene-d8, Perylene-d12 and Phenanthrene-d10.

Internal standard used in the analysis of LVI/LL samples is from a working standard and is prepared in accordance with Chart 14. The working standard contains the following compounds at a concentration of 200ug/mL: 1,4-Dichlorobenzene-d4, Acenaphthalene-d10, Chrysene-d12, Naphthalene-d8, Perylene-d12 and Phenanthrene-d10.

Chart 15 - LVI/Low Level Water Internal Standard Working Solution

8270 Working Internal Standard Mix (MB_LLIS_WRK)	Solvent	Stock Conc. (ug/mL)	Initial Vol. (uL)	Final Vol. (mL)	Final Conc. (ug/mL)
8270 SV Internal Standard Mix (MB_INTSTD_STK)	$MeCl_2$	2000	1000	10	200

Internal standard used in the analysis of SIM samples is from a working standard and is prepared in accordance with Chart 15. The working standard contains the following compounds at a concentration of 50ug/mL: 1,4-Dichlorobenzene-d4, Acenaphthalene-d10, Chrysene-d12, Naphthalene-d8, Perylene-d12 and Phenanthrene-d10.

Chart 16 – SIM Internal Standard Working Solution

SIM Working Internal Standard Mix (MB_SIMIS_WRK)	Solvent	Stock Conc. (ug/mL)	Initial Vol. (uL)	Final Vol. (mL)	Final Conc. (ug/mL)
8270 SV Internal Standard Mix (MB_INTSTD_STK)	$MeCl_2$	2000	250	10	50

7.4 Storage of Standards

Stock, intermediates and working standards are stored at 4ºC ± 2ºC or less in Teflon-lined crimp-cap amber bottles or vials. Standards are stored separately from sample extracts.

Preparation of standards is done in accordance with the Eurofins TestAmerica Buffalo SOP BF-GP-019. Stock and working calibration standards are prepared every twelve months or sooner, if the expiration date of any parent precedes 1 year.

The daily continuing calibration mix (CCV), DFTPP tuning mix, Reporting Limit check mix and all Internal Standard working reagents are prepared every 6 months or sooner, if the expiration date of any parent precedes 6 months.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and preservation requirements.

Water samples may be collected in 1L or 250 mL amber glass containers with Teflon lined, screw-caps.

Soil/Sediment Samples may be collected in glass containers fitted with Teflon-lined screw-caps or closed end tubes.

All samples are stored at 4ºC±2ºC from the time of collection until extraction.

Aqueous samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.

Soil samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

Sample extracts are stored at 4ºC±2ºC in the SVOA sample extract refrigerator prior to analysis.

9.0 Quality Control

9.1 Batch QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	<ReportingLimit (RL)
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the extractions group, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

Technical requirements and acceptance of batch QC for 8270D, 8270E, MCP, RCP and SIM is detailed below and summarized in Table 11 in section 18.

9.1.1 Method Blanks

A method blank is a volume of a clean reference matrix (reagent water for water samples, or purified sodium sulfate/clean sand for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the preparation and analysis of samples.

A method blank must be prepared once for the following, whichever is more frequent:

Each prep batch.
Each 20 Samples in a batch, in addition to matrix spikes/matrix spike duplicates that are of a similar matrix.
Whenever samples are extracted by the same procedure.

9.1.1.1 Preparation of the Method Blank

For semivolatile analysis, a method blank for samples consists of the following volumes/weights and spikes:

1L water analysis: 1L of reagent water is spiked with 1.0mL of the surrogate spiking solution and concentrated to 1mL.

LVI water analysis: 250mL of reagent water is spiked with 1.0mL of the LVI surrogate solution and concentrated to a final volume of 1mL.

Low Level water analysis: 1L of reagent water is spiked with 1.0mL of the LVI surrogate solution and concentrated to a final of 1mL.

SIM analysis: 1L of reagent water is spiked with 1.0mL of the SIM isotopically labeled analog solution and concentrated to a final of 1mL.

Soil/sediment samples: 30g of sodium sulfate/clean sand is spiked with 1.0mL of the surrogate spiking solution.

9.1.1.2 Technical Acceptance Criteria for Method Blank Analysis

All technical acceptance criteria for retention time, surrogate and IS recovery must be met for blank analysis. In addition, the following acceptance criterion applies.

For all target analytes, the method blank must contain less than the reporting limit (RL) of any single target compound.

If any single target compound is detected in the method blank with a concentration above the RL, samples that contain detections below the RL or samples containing detections that are 10X greater than the detection found in the blank will be flagged, noted the job narrative and reported. Final concentrations in the LIMS system are to be utilized when making this determination.

9.1.1.3 Corrective Actions for Method Blank Analyses

If the acceptance criteria for method blank analysis are not met, the analytical system may be assumed to be out of control.

Any contamination in the method must be investigated. Samples associated with the contaminated blank must be re-extracted and re-analyzed.

If surrogate recoveries in the method blank do not meet the acceptance criteria, first reanalyze the method blank. If the surrogate recoveries do not meet the acceptance criteria after reanalysis, re-extract and re-analyze the blank and all associated samples OR the samples may be reported as estimated, and noted in the case narrative.

If the method blank does not meet internal standard response requirements, check calculations, the internal standard spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the internal standard responses meet their acceptance criteria. If the internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples. If the instrument malfunctioned, correct the instrument problem and reanalyze the method blank. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the blank

9.1.2 Laboratory Control Sample/Matrix Spike/Matrix Spike Duplicate

A Laboratory Control Sample (LCS), matrix spike (MS) and matrix spike duplicate (MSD) are analyzed to evaluate the analytical system and the effects of sample matrix on the methods used for semivolatile analysis.

The LCS, matrix spike, and matrix spike duplicate are spiked with the compounds listed in table 2 (at concentrations noted in section 7.3.2).

A LCS, matrix spike and matrix spike duplicate are extracted and analyzed for every batch of 20 samples of a similar matrix. Matrix spike and matrix spike duplicates are not performed for field QC samples such as rinsates, or field/trip blanks.

If insufficient sample amount is received to perform matrix spike and matrix spike duplicate analysis, or is requested by the client, a Laboratory Control Sample Duplicate (LCSD) may be analyzed.

A LCSD is always performed for MCP analysis. A LCSD is required for RCP analysis when a site specific MS/MSD is not provided. A MS/MSD may be performed in addition for MCP when requested.

9.1.2.1 Preparation of LCS/MS/MSD Samples

For semivolatile analysis, the laboratory control sample, matrix spike and matrix spike duplicates consists of the following volumes/weights and spikes:

1L water analysis: 1L of reagent water is spiked with 1.0mL of the surrogate spiking solution and 1.0mL of the spiking solution and concentrated to 1mL.

LVI water analysis: 250mL of reagent water is spiked with 1.0mL of the LVI surrogate solution 1.0mL of the LVI spiking solution and concentrated to a final volume of 1mL.

Low Level water analysis: 1L of reagent water is spiked with 1.0mL of the LVI surrogate solution and 1.0mL of the LVI spiking solution and concentrated to a final of 1mL.

SIM analysis: 1L of reagent water is spiked with 1.0mL of the SIM isotopically labeled analog solution and 1.0mL of the SIM spiking solution and concentrated to a final of 1mL.

Soil/sediment samples: 30g of sodium sulfate/clean sand is spiked with 1.0mL of the surrogate spiking solution and 1.0mL of the spiking solution and concentrated to a final of 1mL.

9.1.2.2 Dilutions

Dilutions of MS/MSD samples are performed only if the unspiked parent sample requires a dilution in order to maintain any target compound concentration in the upper half of the calibration. Any sample diluted 20x or greater will be deemed to have too low a recovery and shall be qualified accordingly.

Preparation of dilutions are described in equation 11 in section 11.2 and Table 10 in the attachments of Section 18.

9.1.2.3 Calculations for MS/MSD

The concentrations of the spiked compounds are determined using equations 12, 13 and 14 in section 11.4.1. After determining the compound concentrations, the percent recovery is calculated using Equation 1.

Equation 1

$$\text{Matrix Spike Recovery} = \frac{\text{SSR - SR}}{\text{SA}} x100$$

Where,

SSR= Spike Sample Result

SR = Sample Result

SA = Spike Added

The relative percent difference between the matrix spike and matrix spike duplicate is calculated using Equation 2.

Equation 2

$$\text{RPD} = \frac{[\text{MSR - MSDR}]}{1/2\,(\text{MSR} + \text{MSDR})} \text{ X } 100$$

Where,

RPD = Relative Percent Difference

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

The vertical bars in the formula above indicate the absolute value of the difference; hence RPD is always expressed as a positive value.

9.1.2.4 Calculation for LCS/LCSD

The concentrations of the spiked compounds are determined using equations 12, 13 and 14 in section 11.4.1. After determining the compound concentrations, the percent recovery is calculated using Equation 3.

Equation 3

$$\text{LCS} = \frac{\text{SSR}}{\text{SA}} x100$$

Where,

SSR = Spiked Sample Result

SA = Spike Added

The relative percent difference between the laboratory control sample and the laboratory control sample duplicate is calculated using Equation 2, where the MSR and MSDR are equivalent to the LCS recovery and the LCSD recovery respectively.

9.1.2.5 Technical Acceptance Criteria for MS/MSD

The acceptance criteria for retention time and IS recovery must be met for matrix spike and matrix spike duplicate analysis.

The matrix spike recovery and RPD limits for 8270D and 8270E are based on historical data and are updated annually.

MS limits for MCP and RCP are 40-140% for base-neutral compounds and 30-130% for acid compounds. RPD limits are ≤20% for waters and ≤30% for soils.

SIM limits are 40-140%. RPD limit is ≤20%.

The matrix spike recovery limits are advisory. If the recovery limits are not met, no further corrective action will be necessary. However, frequent occurrences of this nature should be investigated.

Re-extraction and re-analysis of the matrix spike and matrix spike duplicate may be necessary if, in the technical judgment of the analyst and/or supervisors, an error was made during the extraction procedure.

Exceedances of 150% or less than 10% and/or RPD of >50% should be narrated for potential matrix interference, especially when an associated LCS meets acceptance criteria.

9.1.2.6 Technical Acceptance Criteria for LCS

The acceptance criteria for retention time, surrogate and IS recovery must be met for the LCS analysis. Any failures in the LCS are flagged automatically in the laboratories TALS LIMS system.

The Laboratory Control Sample recovery and RPD limits for 8270D and 8270E are based on historical data and are updated annually.

The laboratory defines several compounds for 8270D and 8270E as poor performers in association to this analytical method. These analytes are identified as such through current and historical performance and are listed in Table 6. Recoveries of poor performers in the laboratory control sample (and/or duplicate) that are below the lower control limit are allowed, provided that the recovery is greater than or equal to 10%, with the exception of Benzidine, which must meet 5%. Any poor performer that meets this condition described will be noted in the job narrative.

Recovery limits for MCP and RCP are 40-140% for base-neutral compounds and 30-130% for acid compounds. RPD limits are ≤20% for waters and ≤30% for soils.

MCP allows the following “difficult” analytes to be outside of criteria, provided the recovery is within 15-140%: 4-Chloroaniline, 4-Nitrophenol, Phenol and 2,4-Dinitrophenol.

For MCP, ≤10% of the target analytes may be outside acceptance limits, provided the recovery is ≥10%.

For RCP, ≤20% of the target analytes may be outside acceptance limits, provided the recovery is ≥10%.

Recovery limits for SIM are 40-140%. RPD limits are ≤20%.

Any single target compound that recovers above the upper control limit for 8270D, 8270E, MCP, RCP or SIM is to be considered high bias in all samples associated to that LCS (and/or LCSD). If the detection of that analyte in associated samples is either not detected or detected at a concentration below the reporting limit (RL), the deficiency will be noted in the job narrative and the sample(s) will be reported.

If a surrogate exceeds the upper control limit, associated samples may be reported if all target compounds associated to that surrogate class are not detected or detected at a concentration below the reporting limit (RL). The deficiency will be noted in the job narrative and the sample(s) will be reported.

9.1.2.7 Corrective Actions for Laboratory Control Sample Analysis

If the acceptance criteria for laboratory control sample/laboratory control sample duplicate analysis are not met, the analytical system may be assumed to be out of control. The following corrective actions may be taken:

If the recovery of any target analyte is above the upper control limit and associated samples contain detections for this analyte greater than the reporting limit, re-extraction and re-analysis must be performed for those samples.

If the recovery of any target analyte is below the lower control limit and is not a poor performer, or if a poor performer recovers below 10% (Benzidine less than 5%), re-analyze the laboratory control sample and/or laboratory control sample duplicate to ensure an issue with the injection did not occur. If the LCS/LCSD fails in the re-analysis, all samples associated to the LCS/LCSD that require the non-compliant compound must be re-extracted.

If surrogate recoveries in the LCS/LCSD do not meet the acceptance criteria, first reanalyze the LCS/LCSD. If the surrogate recoveries do not meet the acceptance criteria after reanalysis, re-extract and re-analyze the LCS/LCSD and all associated samples OR the samples may be reported as estimated, and noted in the job narrative.

If the LCS/LCSD does not meet internal standard response requirements, check the calculations, the internal standard spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the internal standard responses meet their acceptance criteria. If the internal standard spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/re-analyze the LCS and associated samples. If the instrument malfunctions affected the calibration, recalibrate the instrument before reanalyzing the LCS.

An exception to corrective action for LCSD-only failures may be allowed on a case by case basis, depending on client requirements.

9.2 Surrogate Recoveries

The surrogate compound concentrations are determined using equations 12, 13 and 14 in section 11.4.1. The recoveries are then determined using Equation 4.

Equation 4

$$\%\ Recovery = \frac{Concentration(\vee amount)found}{Concentration(\vee amount)spiked} \text{ X } 100$$

9.2.1 Technical Acceptance Criteria for Surrogate Recovery

Surrogate recovery limits for 8270D and 8270E are based on historical data and are updated annually.

Limits for MCP and RCP are 30-130% in a soil matrix, 30-130% for base-neutral compounds in a water matrix and 15-110% for acid compounds in a water matrix.

Up to one acid and/or one base/neutral surrogate can be outside the acceptance limits in sample analysis, provided the recovery is greater than or equal to 10%.

Multiple surrogates of the same class (acid and/or base/neutral) may recover above the upper control limit as long as sample detections are below the reporting limit or are not detected for any compound associated to that surrogate class.

Multiple surrogates of the same class may recover below the lower control limit provided the requested target analyte list does not contain any compounds in that failing surrogate class.

Surrogates failing to meet acceptance criteria related to significant and obvious matrix interference may be reported, or a dilution may be performed to reduce the amount of interference.

Surrogate recoveries in samples diluted by a factor of 20X or greater are to be considered estimated as they are below the lowest calibration level.

Any surrogate recovery outside acceptance limits will be qualified and noted in the job narrative.

9.2.2 Corrective Actions for Surrogate Recovery

Calculations, injection volumes and preparation volumes are checked to ensure an error was not made. If all calculations, volumes, etc., were correct the analyst will proceed to the next step in the corrective action process.

The sample is re-injected to verify an error was not made during the original analysis. If after re-injection, surrogate recoveries are outside of the acceptance criteria, the analysis will proceed to the next step in the corrective action process.

The sample is re-extracted. Exceptions for this are either in the case where MS/SD and parent surrogate recoveries all agree, there is significant matrix identified at the retention time of the surrogate or insufficient volume of the sample remains. In either case, the situation will be documented in the job narrative.

After re-extraction, the sample is re-injected. If after re-analysis surrogate recoveries are within criteria limits, this extract is considered the first because the original problem may have been due to a laboratory error during extraction. If, after re-analysis surrogate recoveries are not within criteria limits, a matrix effect may be assumed. If this should occur, the original analysis may be reported. The instance will be documented in the job narrative.

9.3 Internal Standard Recoveries

Internal standards are added to all initial calibration standards, initial calibration verification (ICV) and continuing calibration verification (CCV) standards, batch QC (MB/LCS/MS/MSD) and client samples. For the ICV and CCV, the internal standard responses are compared to the mid-level calibration standard. For batch QC and samples, internal standards are compared to the daily CCV. The recoveries are determined using Equation 5.

Equation 5

$$\%\text{Recovery} = \frac{\text{Area of IS in Sample}}{\text{Area of IS in Standard}} \text{ X } 100$$

9.3.1 Technical Acceptance Criteria for Internal Standard Recoveries

Internal standard recovery for instrument QC must be within 50-200% of the mid-range calibration level.

Internal standard recovery for batch QC and samples must be within 50-200% of the daily continuing calibration verification (CCV).

Retention time shifts for each Internal Standard must be within ±0.5 min between the continuing calibration verification and the mid-level standard of the most recent initial calibration.

Retention time shifts for each Internal Standard must be within ±0.5 min between the sample and the most recent continuing calibration verification.

9.3.2 Corrective Actions for Internal Standard Recoveries

Calculations, internal standard solution volumes and injected volumes are checked to ensure that an error was not made. If all calculation and volumes were correct, the analyst will proceed to the next step in the corrective action process.

The sample is re-injected to ensure that the instrument was working properly. If after re-analysis, the internal standard recoveries are within criteria limits, the second analysis will be reported. If after re-analysis the internal standard recoveries are outside of criteria limits, the following steps will be taken:

If an instrument QC standard fails internal standard recovery, the electron multipler (EM) voltage can be adjusted accordingly and the DFTPP and standards must be reanalyzed. Failure again and the reagent will be re-prepared and reanalyzed. Repeat IS failures requires initial calibration and/or instrument maintenance.

If a batch QC sample fails internal standard recovery, the entire batch will be re-extracted and re-analyzed.

If a client sample fails internal standard recovery, the sample will be re-extracted and re-analyzed.

Exception: If internal standard recoveries of a sample, MS/MSD agree (i.e., recoveries are outside of criteria limits for all three samples), it may be assumed that a matrix effect is involved and no corrective action is necessary. The instance will be documented in the job narrative.

9.4 Labeled Analog Recoveries for SIM

The isotopically labeled analog 1,4-Dioxane-d8 is added to all samples during extraction. This analyte is used for the quantitation of 1,4-Dioxane.

9.4.1 Technical Acceptance Criteria for Labeled Analog Recoveries

Recovery limits for SIM are based on historical data and are updated annually.

1,4-Dioxane-d8 may recover above the upper limit, provided the recovery of 1,4-Dioxane is below the reporting limit and/or not-detected.

Recovery failing to meet acceptance criteria due to significant and obvious matrix interference may be reported, or a dilution may be performed to reduce the amount of interference.

Recovery of 1,4-Dioxane-d8 in samples diluted below the lowest calibration level can be determined to be acceptable if the signal to noise ratio of the quantitation ion is 10:1 and the qualifier ion is 3:1.

If dilutions do not recover 1,4-Dioxane-d8, then recovery of 1,4-Dioxane cannot be calculated. In this case, lower dilutions with results over the upper calibration level may be reported for 1,4-Dioxane, with proper flagging and notation in the job narrative.

9.4.2 Corrective Actions for Labeled Analog Recovery

Calculations, injection volumes and preparation volumes are checked to ensure an error was not made. If all calculations, volumes, etc., were correct the analyst will proceed to the next step in the corrective action process.

The sample is re-injected to verify an error was not made during the original analysis. If after re-injection, analog recoveries are outside of the acceptance criteria, the analysis will proceed to the next step in the corrective action process.

The sample is re-extracted. Exceptions for this are either in the case where MS/SD and parent analog recoveries all agree, there is significant matrix identified at the retention time of the analog or insufficient volume of the sample remains. In either case, the situation will be documented in the job narrative.

After re-extraction, the sample is re-injected. If after re-analysis analog recoveries are within criteria limits, this extract is considered the first because the original problem may have been due to a laboratory error during extraction. If, after re-analysis analog recoveries are not within criteria limits, a matrix effect may be assumed. If this should occur, the original analysis may be reported. The instance will be documented in the job narrative.

10.0 Procedure

Technical requirements and acceptance of instrument QC for 8270D, 8270E, MCP, RCP and SIM is detailed below and summarized in Table 11 in section 18.

10.1 Sample preparation

For complete procedure on sample preparation, see the following Eurofins TestAmerica Buffalo SOPs:

3510C:	BF-OP-003, current revision
3510C_LVI:	BF-OP-019, current revision
3550C:	BF-OP-016, current revision
3546:	BF-OP-018, current revision

10.2 Instrument QC

Typical Instrument Operating Conditions are presented below. These may be modified as necessary to accommodate large volume injection (LVI) techniques which may utilize up to a 5uL injection.

DFTPP analyzed for SIM is through full scan and should use the LVI Suggested Parameters.

OVEN
LVI Suggested Parameters
Initial temp: 45 °C (On) Maximum temp: 340 °C
Initial time: 3.00 min Equilibration time: 0.20 min
Ramps:

#	Rate	Final temp	Final time
1	30.00	280	0.00
2	9.00	325	4.00
3	0.0(Off)		

Post temp: 70 °C

Post time: 0.00 min
Run time: 19.83 min

Note, the run time must be extended so that the instrument acquires at least 1 min after the last compound elutes off the column.

1L Suggested Parameters

Initial temp: 55 °C (On) Maximum temp: 340 °C
Initial time: 2.75 min Equilibration time: 0.20 min
Ramps:

#	Rate	Final temp	Final time
1	23.00	70	0.00
2	20.00	195	0.00
3	30.0	330	5.00
4	0.00 (off)		

Post temp: 70 °C
Post time: 0.00 min
Run time: 19.15 min

Note, the run time must be extended so that the instrument acquires at least 1 min after the last compound elutes off the column. For the analysis of Dibenzo(a,e)pyrene, the Final Time for rate #3 should be adjusted by several minutes to allow this compound to properly elute off the column.

SIM Suggested Parameters

Initial temp: 45 °C (On) Maximum temp: 340 °C
Initial time: 3.00 min Equilibration time: 0.20 min
Ramps:

#	Rate	Final temp	Final time
1	30.00	325	4.00
2	0.00 (off)		

Post temp: 45 °C
Post time: 0.00 min
Run time: 16.33 min

FRONT INLET (SPLIT/SPLITLESS)
LVI Suggested Parameters

Mode: Pulsed Splitless
Initial temp: 280 °C (On)
Pressure: 14.90 psi (On)
Pulse pressure: 30.0psi
Pulse time: 0.55 min
Purge flow: 50.0 mL/min
Purge time: 0.50 min
Total flow: 54.7 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 2.00 min
Gas type: Helium

1L Suggested Parameters
Mode: Splitless
Initial temp: 280 °C (On)
Pressure: 7.00 psi (On)
Purge Flow: 30.0 mL/min
Purge Time: 0.40 min
Total flow: 33.9 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 3.00 min
Gas type: Helium

SIM Suggested Parameters
Mode: Pulsed Splitless
Initial temp: 280 °C (On)
Pressure: 13.44 psi (On)
Pulse pressure: 30.0psi
Pulse time: 0.55 min
Purge flow: 50.0 mL/min
Purge time: 0.50 min
Total flow: 54.0 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 2.00 min
Gas type: Helium

COLUMN 1
LVI Suggested Parameters
Capillary Column
Model Number: Phenomenex ZB-Semivolatile GUARDIAN
Max temperature: 330 °C
Nominal length: 30.0 m (with integral 10m guard column)
Note, this may be removed or a column without a guard column may be installed.
Nominal diameter: 250.00 um
Nominal film thickness: 0.25 um
Note, a film thickness of 0.5 um may be utilized.
Mode: constant flow
Initial flow: 2.2 mL/min
Nominal initial pressure: 14.91 psi
Average velocity: 59 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

1L Suggested Parameters
Capillary Column
Model Number: Phenomenex ZB-Semivolatile GUARDIAN
Max temperature: 330 °C
Nominal length: 30.0 m (with integral 10m guard column)
Nominal diameter: 250.00 um

Nominal film thickness: 0.25 um
Note, a film thickness of 0.5 um may be utilized.
Mode: ramped pressure
Initial pressure: 7.00 psi
Initial time: 0.00 min

#	Rate	Final pres	Final time
1	90.00	30.00	0.10
2	99.00	12.00	2.60
3	2.40	35.00	0.00

Post pressure: 0.00 psi
Nominal initial flow: 0.7 mL/min
Average velocity: 26 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

SIM Suggested Parameters
Capillary Column
Model Number: Phenomenex ZB-Semivolatile GUARDIAN
Max temperature: 330 °C
Nominal length: 30.0 m (with integral 10m guard column)
Note, this may be removed or a column without a guard column may be installed.
Nominal diameter: 250.00 um
Nominal film thickness: 0.25 um
Note, a film thickness of 0.5 um may be utilized.
Mode: constant flow
Initial flow: 1.4 mL/min
Nominal initial pressure: 13.45 psi
Average velocity: 40 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

FRONT DETECTOR (NO DET)
LVI, 1L and SIM Suggested Parameters

SIGNAL 1	SIGNAL 2
Data rate: 20 Hz	Data rate: 20 Hz
Type: test plot	Type: test plot
Save Data: Off	Save Data: Off
Zero: 0.0 (Off)	Zero: 0.0 (Off)
Range: 0	Range: 0
Fast Peaks: Off	Fast Peaks: Off
Attenuation: 0	Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

THERMAL AUX 2
LVI and SIM Suggested Parameters
Use: MSD Transfer Line Heater
Description: MSD Transfer Line
Initial temp: 325 °C (On)
Initial time: 0.00 min
Rate Final temp Final time
1 0.0(Off)
Post Run
Post Time: 0.00 min

1L Suggested Parameters
Use: MSD Transfer Line Heater
Description: MSD Transfer Line
Initial temp: 310 °C (On)
Initial time: 0.00 min
Rate Final temp Final time
1 0.0(Off)
Post Run
Post Time: 0.00 min

GC INJECTOR
LVI and SIM Suggested Parameters
Front Injector:

Sample Washes	2
Sample Pumps	4
Injection Volume	2.00 microliters
Syringe Size	10.0 microliters
PreInj Solvent A Washes	0
PreInj Solvent B Washes	0
PostInj Solvent A Washes	4
PostInj Solvent B Washes	2
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

1L Suggested Parameters
Front Injector:

Sample Washes	1
Sample Pumps	4
Injection Volume	1.00 microliters
Syringe Size	10.0 microliters
PreInj Solvent A Washes	0
PreInj Solvent B Washes	0
PostInj Solvent A Washes	2
PostInj Solvent B Washes	2
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

MS ACQUISITION PARAMETERS
LVI and 1L Suggested Parameters

General Information
Tune File : dftpp.u
Acquisition Mode : Scan

MS Information
Solvent Delay : 2.60 min
Note, this will vary depending on the age of the column.
EM Offset : 0
Resulting EM Voltage : 976.5
Note, this will vary depending on the age of the Electron Multiplier. Once the EM Voltage is ~ 2300-2600, it may need to be replaced. 3000 is the maximum voltage of an EM.

[Scan Parameters]

Low Mass : 35.0
High Mass : 500.0
Threshold : 100
Sample # : 2
A/D Samples : 4

[MSZones]
MS Quad : 150 °C maximum 200 °C
MS Source : 230 °C maximum 250 °C

SIM Suggested Parameters

General Information
Tune File : dftpp.u
Acquisition Mode : SIM

MS Information
Solvent Delay : 1.50 min
Note, this will vary depending on the age of the column.
EM Voltage : False
EM Offset : 47
Resulting EM Voltage : 1705.9
Note, this will vary depending on the age of the Electron Multiplier. Once the EM Voltage is ~ 2300-2600, it may need to be replaced. 3000 is the maximum voltage of an EM.

[SIM Parameters]

Group 1
Group ID : 1
Resolution : Low
Plot 1 Ion : 88.0
Plot 2 Ion : 88.0

Ions / Dwell In Group	(Mass, Dwell)	(Mass, Dwell)	(Mass, Dwell)
	(43.0, 100)	(58.0, 100)	(64.0, 100)
	(88.0, 100)	(96.0, 100)	(115.0, 100)
	(152.0, 100)		

[MSZones]
MS Quad : 150 °C maximum 200 °C
MS Source : 230 °C maximum 250 °C

10.3 Instrument Performance Check

The GC/MS system is tuned using Perfluorotributylamine (PFTBA) such that an injection of 50ng (for 1L/Soils) or 10ng (for LVI/Low Level/SIM) of DFTPP will meet the abundance criteria listed in Table 3.

Prior to the analysis of standards or samples, the mass calibration and resolution of the GC/MS system is verified by the analysis of DFTPP. This analysis will verify the proper tuning of the system for 12 hours. After 12 hours, the instrument performance must be verified before standard and sample analysis may continue.

For 8270E, DFTPP tuning is only required prior to calibration. Daily DFTPP analysis is not required for samples.

The average of the apex of the DFTPP peak, the scan before and scan after the apex is used to assess ion abundances. If the criteria is not met, a single scan of the apex may be evaluated. This is performed automatically in the Chrom system.

The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. The background spectrum is selected as one scan before the start of the integrated DFTPP peak.

Breakdown of 4,4'-DDT into 4,4'-DDD and 4,4'-DDE may be used to assess GC column performance and injection port inertness and must be less than 20%.

The compounds Benzidine and Pentachlorophenol should be present and at their normal responses for this concentration. Peak tailing should not be visible (PCP tailing factor ≤2 and Benzidine ≤2). If responses are poor and excessive peak tailing is present, corrective actions for the GC/MS instrument performance check solution may be required. Benzidine and Pentachlorophenol tailing may also be verified in the CCV.

Tailing is not evaluated for SIM analysis. Breakdown is evaluated, and in cases where it exceeds 20%, the peak shape for 1,4-Dioxane is checked. As long as there are no negative effects on peak shape, analysis may proceed with narration.

All subsequent standards and samples must be acquired under the same GC/MS tuning conditions that were used for the analysis of the instrument performance check solution.

10.3.1 Technical Acceptance Criteria for the GC/MS Instrument Performance Check

DFTPP criteria is listed in Table 3.

Tailing of Pentachlorophenol and Benzidine must be ≤2.

Breakdown of 4,4’-DDT into 4,4’-DDD and 4,4’-DDE must be ≤20%.

All tune acceptance criteria applies to MCP and RCP analysis.

10.3.2 Corrective Actions for the GC/MS Instrument Performance Check

If any of the acceptance criteria are not met, the DFTPP should be re-injected to ensure that the injection made was not a cause for failure. If, after reinjection, acceptance criteria has not been met, one or more of the following corrective actions may be taken:

1. Replace the injection port liner
2. Replace the septum in the injector
3. Cut the column at the injector end
4. Re-prepare the DFTPP working standard and re-analyze
5. Clean injection port with $MeCl_2$
6. Change injection port seal
7. Retune the GC/MS
8. Replace the column
9. Clean the source; replace parts, etc.
10. An instrument service call may be placed.

10.4 Initial Calibration

After the instrument performance check criteria has been met and prior to the analysis of samples, the GC/MS system is calibrated at a minimum of five concentration levels in order to establish instrument sensitivity and linearity.

The initial calibration shall be performed when major instrument maintenance has been performed or if continuing calibration criteria cannot be met.

Major instrument maintenance may consist of source cleaning, column changing, injection port replacement or quadrupole rod adjustment. Preventative maintenance such as septum changes, injector liner changes or column cutting may not require an initial calibration to be performed.

10.4.1 Procedure for Initial Calibration

Calibration standards for common target semivolatile compounds are prepared to contain all target, internal standard and surrogate compounds. Additional calibration mixes may be prepared that contains an extra list of target compounds and internal standards only. Surrogates should not be added to additional mixes. Refer to section 7.3.4 for preparation of calibration mixes and section 7.3.5 for preparation of Internal Standard working mix.

The relative response factors (RRF) for each target and surrogate compound is determined using equation 6. The characteristic ions for a given compound are listed in Tables 5. Internal standard assignments are listed in Table 4.

Equation 6

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

Where,
A_x = Area of the quantitation ion for the compound to be measured (see Table 5)
A_{is} = Area of the quantitation ion for specific internal standard (see Table 5)
C_{is} = Amount of the internal standard injected (ng)
C_x = Amount of the compound to be measured injected (ng)

The mean relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of the RRF values for the initial calibration using the following equation:

Equation 7

$$\%RDS = \frac{StandardDeviation}{Mean} x\,100$$

Where,

$$StandardDeviation = \sqrt{\frac{\sum_{i+1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

x_i = each individual value used to calculate the mean
x = the mean of n values
n = the total number of values

For the SIM analysis, the RRF of the analog compound (1,4-Dioxane-d8) is calculated by Equation 6 using the Internal Standard 1,4-Dichlorobenzene-d4. For the analyte 1,4-Dioxane, the RRF is calculated by Equation 6 using the analog 1,4-Dioxane-d8 as the Internal Standard.

10.4.2 Technical Acceptance Criteria for Initial Calibration

The relative standard deviation (RSD) should be less than or equal to 20% for each target analyte for 8270D, 8270E, MCP, RCP and SIM. If the relative standard deviation is greater than 20%, a linear calibration fit may be used. The criterion for this is a correlation coefficient (r^2) value greater than or equal to 0.990.

If less than 10% of the calibrated compounds fail to meet the above criteria for 8270D and 8270E, analysis may proceed, given the following conditions:

A standard with a concentration at or below the reporting limit for compounds failing to meet the RSD or correlation coefficient must be analyzed with each analytical sequence, preceding the analysis of samples. Acceptance criterion for this is detection only.

Any non-detection in sample analysis associated to compounds failing to meet RSD or correlation coefficient criteria may be reported without flagging, only with the successful detection of the analyte in the reporting limit standard.

Any detection associated to compounds failing to meet RSD or correlation coefficient criteria must be must be flagged as estimated. Every effort should be made to reanalyze the sample on an instrument with a passing calibration.

If less than 10% of the compounds for MCP fail to meet the above criteria, analysis may proceed, given the following conditions:

The %RSD is <40% or the r^2 value is >0.98

If less than 20% of the compounds for RCP fail to meet the above criteria, analysis may continue.

The relative response factors (RRF) for the most common target analytes are compared to the minimum relative response factor criteria required by 8270D and 8270E, listed in Table 7.

If a compound fails to meet the minimum response factor defined in Table 7, a standard with a concentration at or below the reporting limit (RL) must be analyzed in each analytical batch, preceding sample analysis. Acceptance criterion for this is detection only.

Samples containing non-detections for these compounds may be reported without flagging only with a passing RL check.

Samples with positive detections must be flagged as estimated. Every effort should be made to reanalyze the sample on an instrument with passing minimum response factors in the initial calibration.

The relative response factors for MCP must meet the minimum response factors listed in Table 7 for the lowest concentration standard and for the average RF. All other compounds must recover a response of 0.05 or greater on the lowest calibration standard and average RF.

If the minimum response factor is not met, the non-conformity will be narrated.

For RCP analysis, all compounds must meet a minimum response factor of 0.05.

Non-conforming compounds will be narrated.

Analytes fitted with a linear calibration model must have a readback concentration within 30% of the true value at the low level of the calibration, for 8270D and MCP. Linear-fitted analytes for RCP and SIM should meet 30% readback as well. For 8270E, the low calibration point readback must be within 50%. For 8270D and 8270E, any non-detection in samples associated to a compound that fails to meet this criterion in the calibration may be reported without flagging. Detections must be flagged as estimated and noted in the job narrative.

Any compound that fails to meet the criterion for MCP must be reported as estimated in the narrative.

Identification of analytes in all calibration levels can be made only if there are 5-10 scans of the quantitation ion across the peak. All minor ions, where the expected abundance set from the mid-level standard is greater than 10%, must also be present.

For SIM, the signal to noise ratio for each level should be 10:1 for the quantitation ion and 3:1 for the qualifying ion(s) for both the analyte and isotopically labeled analog.

Internal Standard responses of each calibration level should be within 50%-200% of the mid-level standard.

Relative retention times of Internal Standards, surrogates and compounds must be within ±0.06 mins of the RT set in the mid-level point of the calibration.

Additional Initial Calibration requirements are described in TestAmerica Buffalo SOP BF-GP-012 (current revision), beginning with section 5.5: Initial Calibration Review.

10.4.3 Corrective Actions for Initial Calibration

If any of the acceptance criteria for initial calibration are not met, it may be necessary to reanalyze one or more of the calibration standards. This must be completed within the same 12 hour tune as the other calibration levels and before sample analysis. If after reanalysis, the acceptance criteria have not been met, it may be necessary to take further corrective actions.

The following corrective actions may be taken if the acceptance criteria for initial calibration cannot be met.

1. Replace the septum on the injector
2. Replace the injector liner
3. Cut column at the injector end
4. Prepare fresh standards and reanalyze the initial calibration
5. Re-tune the GC/MS system and reanalyze the instrument performance check
6. Replace the analytical column
7. Clean the source
8. An instrument service call may be placed

The acceptance criteria must be met before sample analysis may proceed.

10.4.4 Initial Calibration Verification

To verify the accuracy of the initial calibration, a standard is obtained from a source different from the calibration standards. Alternatively, if a different source is not available, a differing lot number of the standards used in the initial calibration may substitute as the second source.

Immediately following analysis of an acceptable initial calibration curve, an aliquot of the second source standard with a concentration approximating the mid point of the curve of is injected.

10.4.5 Technical Acceptance Criteria for Initial Calibration Verification

For 8270D and 8270E, recoveries of all compounds shall fall within the required 70-130% acceptance limit with the exception of the “Poor performers”, whose criteria are listed Table 6.

Relative response factors (RRF) must meet the minimum response factor criteria listed in Table 7 for the most common semivolatile target analytes.

If any analyte recovery exceeds the upper control limit, data analysis may continue. Non-detections in sample analysis associated to a failing analyte may be reported without flagging. Any detection in sample analysis must be flagged as estimated. Every effort should be made to re-analysis samples with detections on an instrument which has a passing initial calibration and initial calibration verification.

If any analyte recovery exceeds the lower control limit, including limits set for poor performing compounds, the calibration is deemed to be out of control and corrective action must be taken prior to sample analysis.

For MCP, recoveries of all compounds shall fall within the required 70-130% acceptance limit with the exception of the “difficult” analytes, listed below, whose recovery must be within 40-160%. 10% of the total compounds may fail the criteria. If any analyte is outside acceptance limits, report the non-conforming compound in the narrative.

> MCP list of “difficult” analytes includes 4-Chloroaniline, 4-Nitrophenol, Phenol and 2,4-Dinitrophenol.

For RCP, recoveries of all compounds shall fall within 80-120%. 20% of the total compounds may fail the criteria, as long as recovery is within 65-135%. If any analyte is outside acceptance limits, report the non-conforming compound in the narrative.

Internal Standard retention times and responses are evaluated after acquisition of the initial calibration verification. If the retention time of any internal standard shifts by more than 30 seconds from that in the mid-point standard level of the initial calibration or the response of any internal standard is outside of the 50% to 200%

range compared to the mid-point standard level of the initial calibration, the system shall be inspected and corrected as needed. The ICV will be reanalyzed after inspection. If the problem is not resolved, a new initial calibration must be performed.

10.4.6 Corrective Actions for Initial Calibration Verification

If the Technical Acceptance Criteria for Initial Calibration Verification is not met, the following corrective action steps should be taken.

Re-inject the ICV to verify there was not an error made during the original analysis.

Re-prepare the ICV to verify an error was not made during the original preparation.

Perform instrument maintenance and re-calibrate.

Re-prepare initial calibration standards and re-calibrate.

Prepare the ICV and/or initial calibration reagents from different lot numbers to verify degradation hasn't occurred.

Re-order either initial calibration or ICV reagents.

10.4.7 Continuing Calibration

If there is no time left in the 12-hour time period after initial calibration, the instrument performance is verified by the injection of a mid level standard.

The continuing calibration check must be analyzed once every 12-hour time period of operation. This check must be analyzed prior to the analysis of samples for a given 12-hour time period.

10.4.8 Procedure for Continuing Calibration

A mid-level calibration standard is used for the continuing calibration verification (CCV). The relative response factor is calculated using Equation 6 in section 10.4.1. The relative response factor is compared to the minimum relative response factors required by 8270D, listed in Table 7.

If quantitation is performed using average response factor, calculate the percent difference between the mean relative response factor from the most recent initial calibration and the continuing calibration relative response factor for each semivolatile target and surrogate compound using Equation 8.

Equation 8

$$\%\ Difference_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} x\ 100$$

Where,

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria
RRF_c = Relative response factor from continuing calibration standard

If quantitation is performed using a linear regression or a non-linear model, calculate the concentration using equations 12, 13 and 14 in section 11.4.1 of this SOP. Calculate the percent drift using Equation 9.

Equation 9:

$$\%Drift = \frac{Conc_E - Conc_A}{Conc_E} x100$$

Where:

$Conc_E$ = Expected Concentration
$Conc_A$ = Actual Concentration

10.4.9 Acceptance Criteria for Continuing Calibration

The relative response factor (RRF) for the most common semivolatile compounds must be greater than or equal to the minimum response factors listed in Table 7 for 8270D and MCP.

For 8270E, there is no minimum RRF requirement for the continuing calibration verification.

RCP and compounds excluded from Table 7 for MCP must have a minimum response factor greater or equal to 0.05.

The percent difference or percent drift (%D) should be less than or equal to ±20% for all compounds, for 8270D, 8270E, MCP, RCP and SIM.

For 8270D and 8270E, 10% of the total calibrated analyte list is allowed to have %D limits outside ±20%, provided that the limit is set to a maximum of ±50%. These analytes are defined as poor perfomers by the laboratory. Poor performers and their limits are listed in Table 6.

For 8270D and 8270E, up to 20% of the total compounds analyzed between CCVs in a batch are allowed to be outside of the ±20%D criterion, or outside of the limits set for poor performing compounds. The total number allowed to fail based on the CCVs analyzed in any given batch, are listed in Table 9. Note: this is based on the laboratory's main list of calibrated analytes. Additional analytes/CCVs may be analyzed and the total number may be adjusted accordingly.

For 8270D and 8270E, if a compound fails to meet the %D criteria or minimum response factor criteria, a standard at or below the reporting limit must be analyzed following the CCV and prior to sample analysis. Acceptance criteria for this is detection only.

Any non-detection for analytes failing to meet the %D or minimum response factor criteria in samples and only with a passing reporting limit check may be reported with notation in the job narrative only.

Any detection for analytes failing to meet the %D or minimum response factor criteria in samples must be flagged as estimated. Every effort should be made to reanalyze the sample on an instrument with a passing CCV.

For MCP, 20% of the total compound list is allowed to exceed criteria, as long as the %D is <40%.

If %D and/or minimum response factors are not met, the non-conformance will be narrated.

For RCP, 10% of the total compound list is allowed to exceed criteria. Any failures will be narrated.

Internal Standard retention times and responses are evaluated after acquisition of the continuing calibration check. If the retention time of any internal standard shifts by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration or the response of any internal standard is outside of the 50% to 200% range compared to the mid-point standard level of the most recent initial calibration, the system shall be inspected and corrected as needed. The CCV will be reanalyzed after inspection. If the problem is not resolved, a new initial calibration must be performed.

10.4.10 Corrective Actions for Continuing Calibration

If any of the technical acceptance criteria for continuing calibration are not met, it may be necessary to reanalyze the continuing calibration standard. If after reanalysis the acceptance criteria cannot be met, further corrective actions may be required.

The following corrective actions may be taken if the acceptance criteria for continuing calibration cannot be met.

1. Replace the septum on the injector
2. Replace the injector liner
3. Replace injection port seal
4. Cut the column at the injector end
5. Retune the GC/MS system and reanalyze the instrument performance check
6. Prepare fresh standards
7. Reanalyze the initial calibration

11.0 Sample Analysis

11.1 Procedure

Sample extracts shall be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, second source calibration verification and continuing calibration requirements. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

Internal standard solution is added to each sample extract. 20µL of internal standard solution is added to each accurately measured 1.0mL of sample extract so that the expected concentration for 1L and soil samples is 40 ng/uL, 4 ng/uL for LVI and LL samples and 1 ng/uL for SIM. The amount of internal standard needs to be adjusted according to how much extract volume was present in the extract vial. The exact volume of extract is measured using a syringe. The amount of Internal Standard solution to be added is then adjusted accordingly. The calculation to determine the amount of IS to add is provided below:

Equation 10

$$\frac{\text{Vol. Extract (ul)} \times 20\ \text{uL}}{1000} = \text{FV of IS (uL)}$$

Necessary dilutions are made prior to adding internal standard solution.

11.2 Dilutions

Dilutions of sample extracts are required if any target compound exceeds the initial calibration range.

The dilution chosen should keep the response of the largest target compound within the upper portion calibration range.

Dilutions of sample extracts may be performed due to the matrix of the sample. Any coating of the vial by the sample will be diluted appropriately to the level of viscosity observed.

Dilutions are prepared according to equation 11:

Equation 11:

$$\text{Dilution Factor} = \frac{\text{Final Volume}}{\text{Sample extract volume added}}$$

Dilutions are performed by adding a volume of sample extract and bringing to a final volume of 1mL with $MeCl_2$. Internal standards are added after and are not included in the calculation for final volume.

The final volume may be adjusted accordingly for cases where the sample extract volume received after extraction is not enough to perform a dilution to reach a 1mL final volume.

Dilutions that are greater than 100X must be performed by serial dilution.

For routine dilutions, see Table 10 for volumes utilized in performing these dilutions.

Dilutions above 20x will be deemed to have to low a surrogate recovery and shall be qualified accordingly.

For dilutions associated with SIM analysis, Internal Standard 1,4-Dichlorobenzene should be added accordingly. Analog 1,4-Dioxane-d8 should not be added to the dilution.

11.3 Qualitative Identification

11.3.1 Target Compounds

Target compound identification is done by comparing the sample mass spectrum to that of the standard. The following criteria must be satisfied in order to verify identifications.

Elution of the sample analyte within GC relative retention time unit window established from the 12-hour calibration standard.

To establish correspondence of the GC relative retention time (RRT), the sample component RRT must compare with ±0.06 RRT units of the mid level calibration. If samples are analyzed within the same 12-hour period as the initial calibration, the 50ng standard is used to verify relative retention times.

Correspondence of the sample analyte and calibration standard component mass spectra.

To establish correspondence of the sample component mass spectra to that of the standard, the following criteria must be met:

All ions present in the standard mass spectrum at a relative intensity greater than 10.0 percent (most abundant ion in the spectrum equals 100.0 percent) must be present in the sample spectrum.

The relative intensities of ions specified in the paragraph above must agree within ±20.0 percent between the standard and sample spectrum. (Example: For an ion with an abundance of 50.0 percent in the standard spectrum, the corresponding sample ion abundance must be between 30.0 and 70.0 percent).

Ions greater than 10.0 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. When target compounds are above the method detection limit (MDL) but are below the reporting limit (RL) but the spectrum meets the identification criteria, report the concentration with a "J".

If a compound does not meet all of the above criteria, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, the compound will be identified

For SIM, the quantitation ions of 88 for 1,4-Dioxane and 96 for 1,4-Dioxane-d8 must demonstrate a signal to noise ratio of at least 10:1. For the qualifying ions of 58 (1,4-Dioxane) and 64 (1,4-Dioxane-d8), the signal to noise ratio must be at least than 3:1.

11.3.2 Non-Target Compounds

A library search may be executed for non-target sample components for the purpose of tentative identification. For this purpose, the NIST/EPA/NIH mass spectral library is used to identify non-target compounds of greatest apparent concentration by a forward search of the library. A background subtraction method may be employed to better match a peak's spectrum to the library. TIC processing is performed only on client requested samples and the Method Blank (MB) associated to those samples. The following compounds will not be identified by a library search routine:

Internal standard compounds
Surrogate compounds
Methylene Chloride

11.3.3 Guidelines for Making Tentative Identifications

After samples have been processed for Target compounds, any unidentified peak in a sample which has an area count of 10% or greater of the closest Internal Standard will be eligible for TIC identification.

A start and end retention time should be set to 0, which allows the entire chromatogram to be searched.

Note, if the solvent delay is not set appropriately during sample acquisition, the solvent may be collected. This should not be reported as a TIC.

Major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within $\pm$20 percent. Molecular ions present in reference spectrum should be present in sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

Sample spectra are compared to the NIST/EPA/NIH library for tentative identification. A criterion of 85% or greater confidence is used in determining IDs.

These settings are entered into the data processing software (Chrom). For routine work, these settings perform the bulk of TIC identification. Manual review of all TIC matches are not part of the standard review, except in the following situations:

CO_2 should be removed as a TIC.

Methylene Chloride should be removed as a TIC.

Internal Standards/surrogates not required by the client should be removed as a TIC.

Any aldol condensation product should be reported as “Aldol Condensation Products”. These include the following compounds: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one and 5,5-dimethyl-2(5H)-furanone.

Siloxanes should be reported as “Column Bleed”.

Multiple peaks may result in the same ID from the library. In this case, every effort should be made to identify the peak with the greatest confidence for that ID. The other shall be re-identified with the next ID listed, or re-identified as unknown.

If, in the technical judgment of the mass spectral interpretation specialist, no tentative identification can be made the compound will be reported as unknown. Further identification may be possible, such as molecular weights or classifications (i.e., unknown hydrocarbon, unknown acid, etc.)

Further information on TICs is documented in Eurofins TestAmerica Corporate Quality Policy Memorandum No. CA-Q-QM-001.

TIC processing is not performed on SIM analysis.

10.3.4 Targeted TICs

Targeted Tentatively Identified Compounds may be requested and reported on occasion. Unlike TICS, Targeted TICs are searched and reported even if they are not detected.

These are included in the client requested compound list, but are not calibrated.

Identification is made using the NIST/EPA/NIH spectral library to compare all peaks in a chromatogram that are not identified as part of the client target analyte list.

An Internal Standard is a pre-determined based on the proximity of a detected peak to the closest internal standard.

A match threshold of 50% is used for identification of the sample spectrum versus the reference spectrum assigned for that compound.

A response factor of 1 is assumed for quantitation.

Any detection of a Targeted TIC is flagged as estimated in the data system (TALS).

11.4 Quantitative Identification

11.4.1 Target Compounds

Target compounds identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (see Table 4). The EICP area of primary characteristic ions of analytes listed in Tables 5 are used for quantitation.

The calculation of analyte on-column (raw) concentration is based on the equations 12, 13 and 14. In each equation, the concentration is designated as "x".

Average calibration fit:

Equation 12:

$$x = \frac{A_c \times C_I}{A_I \times RF}$$

Where:

Ac = Area of the compound
C_I = Expected concentration of the Internal Standard
A_I = Area of the Internal Standard
RF = Response Factor from the initial calibration

Linear calibration fit:

Equation 13:

$$y = mx + b$$

Where:

m = slope of the line
b = y-intercept
y = response factor as determined from equation 14.

Equation 14:

$$y = \frac{A_c \times C_I}{A_I}$$

Where:

A_c, C_I and A_I are given above.

The quantification of 1,4-Dioxane-d8 in the SIM analysis is based off the area and expected concentration of Internal Standard 1,4-Dichlorobenzene. The quantification of 1,4-Dioxane is based off the area and expected concentration of analog 1,4-Dioxane-d8.
In instances where manual integration is necessary due to co-elution, baseline noise or matrix interferences, all instances will be initialed and dated by the analyst. The quantitation report is documented as such by a "m" next to the compound that has been edited. In all instances of manual integration, a hardcopy of the EICP for that compound will be supplied with the raw data. This applies to all target compounds, internal standards and surrogate compounds. Manual Integrations are completed in accordance with TestAmerica Buffalo SOP BF-GP-013.

11.4.2 Water Samples

The following Equation (Eq. 15) is used to determine the final concentration of target compounds identified in water samples:

Equation 15

$$Concentration\ \mu g/L = \frac{(A_x)(I_s)(V_c)(Df)}{(A_{is})(RRFi)(V_o)(V_i)}$$

Where,

A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the characteristic ion for the internal standard
I_s = Amount of internal standard injected in nanograms (ng)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (µL)
Note: A value of 1uL should be assumed. LVI injections of 2uL or greater are accounted for in the initial calibration and are consistent through the calculation of the on-column (raw) concentrations.
V_c = Volume of the concentrated extract in microliters (µL)
RRFi= Relative response factor determined from the initial calibration
Df = Dilution factor. The dilution factor for analysis of water samples for semivolatiles by this method is defined in equation 11.

If no dilution is performed, Df = 1.0

11.4.3 Soil/Sediment Samples

The following Equation (Eq. 16) is used to determine the concentration of target compounds in soil/sediment samples:

Equation 16

$$Concentration\ \mu g/Kg\ (Dry\ weight\ basis) = \frac{(A_x)(I_s)(V_c)(Df)}{(A_{is})(RRF_i)(V_i)(W_s)(D)}$$

Where,
A_x, I_s, A_{is} are as given for water, above.
V_c = Volume of the concentrated extract in microliters (µL)
V_i = Volume of the extract injected in microliters (µL)
D = $\frac{100 - \%\ moisture}{100}$

W_s = Weight of sample extracted in grams (g)
RRFi= Relative response factor determined from the initial calibration.
Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for semivolatile by this method is defined in equation 11.

11.4.4 Tentatively Identified Compounds

Non-Target Compounds
An estimated concentration for non-target tentatively identified compounds is quantitated by the internal standard method. For quantitation, the nearest internal standard free of interferences is to be used. The equations for calculating concentrations are the same as equations 15 and 16. Total area counts from the total ion chromatograms are used for both the compounds to be measured and the internal standard. A relative response factor (RRF) of one (1) is assumed. The resulting concentration is to be qualified as "J" (estimated, due to lack of a compound specific response factor), and "N" (Presumptive evidence of presence), indicating the quantitative and qualitative uncertainties is calculated for all tentatively identified compounds as well as those identified as unknowns.

11.5 Technical Acceptance Criteria For Sample Analysis

The samples must be analyzed on a GC/MS system meeting the DFTPP, initial calibration and continuing calibration criteria.

The sample must be extracted and analyzed within specified holding times.

The sample must have an associated method blank meeting the technical acceptance criteria for a MB, defined in section 9.1.1.2.

The sample must have an associated laboratory control sample meeting the technical acceptance criteria for a LCS, defined in section 9.1.2.6.

A matrix spike/matrix spike duplicate should be prepared with samples. If insufficient volume for a MS/SD, a laboratory control sample duplicate must be analyzed and meet the technical acceptance criteria for a LCS, defined in section 9.1.2.6.

All surrogates must meet the technical acceptance criteria for Surrogate Recoveries, defined in section 9.2.1.

The relative retention time of each compound must be within $\pm$0.06 RRT units of its relative retention time in the continuing calibration standard.

The instrumental response (EICP area) for each of the internal standards must meet the technical acceptance criteria for Internal Standard recoveries, defined in section 9.3.1.

Excluding those ions in the solvent front, no ion may saturate the detector. No target compound concentration may exceed the upper limit of the initial calibration range unless a more dilute aliquot of the sample extract is also analyzed.

Exception: For SIM, the concentration of 1,4-Dioxane is adjusted based on the recovery of isotopically labeled analog 1,4-Dioxane-d8. In situations where the area count for 1,4-

Dioxane is within the calibration range, but the concentration is not, the results may be reported with narration.

11.6 Corrective Actions for Sample Analysis

The technical acceptance criteria must be met before data are reported. If any of the criteria listed above are not met, either re-analyze the sample on an instrument meeting all technical criteria, refer to corrective actions defined throughout sections 9.0 and 10.0, or re-extract and re-analyze the sample.

If the technical acceptance criteria for the relative retention times of the internal standard, surrogate or target compounds are not met, the following corrective actions are taken in the given order:

Carrier gas, zone temperatures and instrument temperature programs are checked to ensure that an error was not made or that the gas tank was not dry or clogged. If no errors are found the analyst will proceed to the next step in the corrective action process.

The sample is re-analyzed to ensure that an error was not made during the first injection. If, after reanalysis, the relative retention times are not within the technical acceptance criteria, it may be assumed that a matrix effect was involved. Both analyses will be reported and the instance will be documented in the job narrative. If, after re-analysis, the relative retention times are within the technical acceptance criteria, the second analysis will be reported only.

Exception: If the relative retention times of a sample, MS/MSD agree (i.e., relative retention times are outside of criteria limits for the sample, MS and MSD), it may be assumed that a matrix effect was involved and further corrective action is not necessary.

12.0 Documentation

12.1 Instrument Logbook

A logbook must be maintained to track major maintenance as well as daily maintenance to an instrument. The logbook must contain the date of the maintenance, the initials of the analyst performing the work, the reason why maintenance was performed and the maintenance completed. If any parts are replaced, catalog and lot numbers must be recorded. If maintenance either resolves the issue or further maintenance is required, this should be notated as well.

12.2 Reagents

All standards must be entered into LIMS. Each ampule will receive a LIMS ID# for traceability.

The certificate of analysis (COA) for each standard is initialized, dated and given the corresponding LIMS ID#. It is then scanned and attached to the reagent in LIMS.

When intermediates or working mixes are created, they are to be logged into LIMS and will be assigned a unique LIMS reference number.

12.3 Sample Logbook

Prior to the start of the analysis, QC and samples are logged into a unique LIMS worklist which serves as an electronic run log. This is accomplished with either a barcode scanner or the prep batch import function in Chrom, which uses the unique sample ID supplied directly from TALS via the prep batch.

Run Logs must contain the following information:

Date, time, and analyst initials
File number, sample ID, vial #, and job #
Injection volume, final volume, initial volume and dilution factor
References for the standards, tune mix, IS mix

All samples injected must be added to a LIMS worklist. If injections are not used, they are labelled accordingly in the worklist. Files must not remain in the Missing Samples list in Chrom and must not be deleted from this list. These must be entered into the worklist, properly linked and processed.

12.4 Checklists

Calibration checklist CA-Q-WI-046 (current revision) is to be completed by first and second level review. This is scanned and attached to the batch in LIMS.

Data Review checklist CA-Q-WI-045 (current revision) is to be completed by first and second level review. This is scanned and attached to the batch in LIMS.

An electronic checklist for an initial calibration as well as sample batch is also completed by first and second level review in LIMS.

Reagent Review Checklists for Unopened, Opened and Intermediate reagents (current revisions) are used for first and second level review of all LIMs reagents. These are scanned and attached with the COA to the reagent in LIMs.

13.0 Data Review

Technical data review of initial calibrations, instrument/batch QC and client data criteria is listed in Eurofins TestAmerica Buffalo SOP BF-GP-012 (current revision).

13.1 Method Detection Limit Study (MDL)

The Method Detection Limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the spiked analyte is present and distinguishable from method blank results. This value is calculated as the MDL_s. Method Blanks are also used to calculate a MDL (MDL_b), which calculates the 99% confidence level that the MDL is derived from the sample and not from contamination or noise. The laboratory's working MDL is then the higher of the two values (MDL_s vs MDL_b). The MDL reflects a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. Further detail of the MDL procedure can be found in Eurofins TestAmerica SOP CA-Q-S-006, current revision.

Ref. EPA Code of Federal Regulations, 40 CFR Part 136, Appendix B

13.2 Demonstration of Capabilities

Initial Demonstration of Capability (IDOC): The initial demonstration with each sample preparation technique and analytical method combination utilized must be performed by generating data of acceptable accuracy and precision for target analytes in a clean matrix. This is also done for new staff or when significant changes in instrumentation are made. Demonstration of Capability (DOC) will be performed annually for those analysts whom have passing IDOCs.

14.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to section 13 of the Corporate Safety Manual. The following waste streams are produced when this method is carried out.

There are two types of aqueous waste generated in the lab:

A-Waste: All non-nitric acid and alkaline aqueous waste.
AN-Waste: All aqueous waste containing nitric acid.

These types of waste are to be disposed of into appropriately marked plastic containers.

The following lists other types of non-aqueous lab waste and where to dispose of:

C-Waste: all solvent waste gets dumped into appropriately marked metal cans. These cans need to be grounded whenever they are emptied to reduce explosion hazards. Discarded standards (except PCBs) will also be dumped into C-waste cans.

Solid Waste: all contaminated paper, solid sample waste, sodium sulfate and all other non-glass material that has been contaminated is to be wrapped in foil and gathered to be dumped into 55 gallon drums.

Glass: contaminated glass needs to be rinsed off with methylene chloride and disposed of with all other glass in glass specific containers with special extra thick polypropylene liners. These containers are for glass only.

Extract Vials: extract vials are to be archived for 6 months after they have been analyzed. After the archival period, vials are to be crushed into a 55 gallon drum.

16.0 References / Cross-References

16.1 US EPA Methods for Evaluating Solid Waste; SW-846, Third Edition, Update IV, Method 8270D, 07/14.

16.2 US EPA Methods for Evaluating Solid Waste; SW-846, Third Edition, Update IV, Method 8000D, 7/14.

16.3 MassDEP MCP WSC-CAM, section II-B, revision 1, 7/10.

16.4 Connecticut DEP RCP, version 2.0, 7/06.

16.5 Method 8270E; Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry; SW-826 Update VI, 6/18.

17.0 Method Modifications

Eurofins TestAmerica Corporate Quality Policy Memorandum NO. CA-Q-QM-009

18.0 Attachments

18.1 Table 1: Semi-volatile Target Compound List and Reporting Limits
18.2 Table 2: LCS/MS/MSD Spike Analytes
18.3 Table 3: DFTPP Ion Abundance Criteria
18.4 Table 4: Internal Standards, Surrogates and Corresponding Target Compounds Assigned for Quantitation.
18.5 Table 5: Characteristic Ions for Target Compounds, Surrogates and Internal Standards
18.6 Table 6: Poor Performing Compounds
18.7 Table 7: Update 4 Minimum Response Factors for selected compounds
18.8 Table 8: Surrogate Recovery Limits
18.9 Table 9: Allowable CCV Failures
18.10 Table 10: Sample Dilutions
18.11 Table 11: 8270D/8270E/MCP/RCP/SIM QC Requirements and Acceptance Criterion Summary.

19.0 Revision History

- Revision 6, Dated 1 November 2020
  - Added 8270E criteria description to all appropriate sections
  - Added 8270E criteria table to Section 18

- Revision 5, Dated 6 February 2020
  - Updated Dept. Manager, Lab Director, signatures added
  - Edited reagents listed in section 7.2.2
  - Added Chart 5 to section 7.3.4
  - Edited Charts in section 7.3.4 to match current calibration standards and renumber charts.
  - Added information on SIM breakdown criteria in section 10.3
  - Added information on SIM calibration range criteria in section 10.5

- Revision 4, Dated 18 December 2017
  - Updated QA Manager, signatures added
  - Added SIM, MCP and RCP to the scope and application section. Removed tissue from matrices performed.
  - Added definitions of MCP, RCP and SIM to section 3.0
  - Updated OSHA's list of known carcinogens in section 5.1
  - Added SIM reagents to section 7.2
  - Added preparation of SIM extraction reagents to section 7.3
  - Added preparation information for List2 and 3 calibration levels in charts 6 through 11.
  - Added SIM calibration preparation information in charts 12, 13 and 15.
  - Added SIM, MCP and RCP QC requirements throughout section 9.0.
  - Included SIM instrument parameters in section 10.2.
  - Section 10.3 – added how the DFTPP spectrum is evaluated.
  - Section 10.4 – added initial calibration, initial calibration verification and continuing calibration verification requirements for SIM, MCP and RCP.
  - Section 11.2 – added SIM dilution information.
  - Section 11.3.1 – included Signal to Noise requirement for SIM.
  - Section 11.3.2 – added Methylene Chloride as a compound to be removed as a reportable TIC.
  - Added reagent review checklists to section 12.4.
  - Section 16 – added references to 8000D, MADEP CAM and Connecticut DEP RCP.
  - Updated Tables 1,4,5 and 8 to include SIM compound information.
  - Updated limits in Table 8 to match current LIMS limits.
  - Added Table 11 – Acceptance criterion summary for 8270D, SIM, MCP and RCP.

- Revision 3, Dated 23 September 2016
  - Updated Department Manager, Laboratory Director, QA Manager, signatures added.
  - Added Organic Ops Manager, signature added
  - Added 8270D_LL method techniques and requirements to multiple sections.
  - Reformatted multiple sections, primarily section titles and numbers.
  - Renumbered charts, equations and tables.
  - Added SOP and Corporate Policy numbers when applicable.
  - Replaced all references to MSDS with SDS.
  - Updated Reagents to include Corporate approved Restek Standards.
  - Included preparation tables of working calibration standards in Charts 3, 4 and 5.
  - Added Internal Standard preparation table for LVI/LL in Chart 6.
  - Section 9.1.2: Added preparation, calculation information for LCS/MS/MSD samples. Added criteria to Technical Acceptance and Corrective actions sections.
  - Section 9.1.2: Removed Marginal Exceedance
  - Section 9.2.1/9.2.2: Added Technical Acceptance Criteria and Corrective Actions for Surrogates
  - Added section 9.3: Calculation, Technical Acceptance Criteria and Corrective Actions for Internal Standards

- Added preparation SOP numbers to section 10.1
- Section 10.2: Updated parameters to match current LVI instrument parameters. Added parameters for 1L acquisition parameters.
- Updated Initial Calibration and Continuing Calibration Verification Technical Acceptance Criteria to follow Corporate Quality Policy Memorandum CA-Q-QM-009.
- Added Technical Acceptance Criteria and Corrective Actions for Initial Calibration Verification.
- Section 11.4.1: Added equations for calculating concentrations based on an average and linear calibration model.
- Removed references to GPC.
- Replaced contract required quantitation limits (CRQL) with reporting limits (RL).
- Updated TIC qualification procedures in accordance with Corporate Quality Policy Memorandum CA-Q-QM-001.
- Added Targeted TIC procedures.
- Section 11.2: Added dilution calculation equation.
- Added section 12 to include Instrument, Reagent and Sample Logbooks.
- Section 13: Replaced Method Performance with Data Review.
- Updated Table 1 to include all routinely calibrated compounds and RLs.
- Added Table 2 – LCS/MS/MSD Spike Analytes
- Table 3: Updated mass 441 to be compared to mass 442; previously 443.
- Updated Table 4 to include all routinely calibrated compounds and current IS assignments.
- Updated Table 5 to include all routinely calibrated compounds and current quantitation/qualifying ions.
- Table 6: Updated the poor performer list of analytes. Added alternative %D criteria for ICV and CCV recoveries.
- Reformatted Table 7.
- Added Table 8: Surrogate Recoveries
- Added Table 9: Allowable CCV Failures
- Added Table 10: Sample Dilutions
- Removed Attachment A: SOP Procedure Summary

- Revision 2, Dated 11 March 2015
  - Added LVI into sampling and preparation sections
  - Added 8270 % drift requirements
  - Updated instrument operation parameters
  - Changed Department Manager, signature added
  - Changed Lab Director, signature added
- Revision 1, Dated 11 March 2011
  - QA Manager updated, signature added

18.1 TABLE 1
Semivolatiles Target Compound List and Reporting Limits

CAS #	Analytes	Water Limits (1L/LVI) ug/L	Soil Limits (3550C/3546) ug/kg	Water Limits (LL) ug/L	Water Limits (LL_PAH) ug/L	Soil Limits (LL_PAH) ug/kg	SIM Limits ug/L
92-52-4	1,1'-Biphenyl	5	170	5			
95-94-3	1,2,4,5-Tetrachlorobenzene	5	170	5			
120-82-1	1,2,4-Trichlorobenzene	10	330	0.5			
95-50-1	1,2-Dichlorobenzene	10	330	0.5			
122-66-7	1,2-Diphenylhydrazine	10	330	5			
99-35-4	1,3,5-Trinitrobenzene	10	330				
541-73-1	1,3-Dichlorobenzene	10	330				
99-65-0	1,3-Dinitrobenzene	20	330				
106-46-7	1,4-Dichlorobenzene	10	330	0.5			
81-64-1	1,4-Dihydroxyanthraquinone	40	660				
100-25-4	1,4-Dinitrobenzene	10	330				
123-91-1	1,4-Dioxane	10	200				0.2
130-15-4	1,4-Naphthoquinone	10	330				
90-13-1	1-Chloronaphthalene	10	330	0.5			
129-43-1	1-Hydroxyanthraquinone	20	660				
90-12-0	1-Methylnaphthalene	5	330	5			
134-32-7	1-Naphthylamine	10	330				
108-60-1	2,2'-oxybis[1-chloropropane]	5	170	5			
58-90-2	2,3,4,6-Tetrachlorophenol	5	170	5			
935-95-5	2,3,5,6-Tetrachlorophenol	20	660				
95-95-4	2,4,5-Trichlorophenol	5	170	5			
88-06-2	2,4,6-Trichlorophenol	5	170	5			
120-83-2	2,4-Dichlorophenol	5	170	0.5			
105-67-9	2,4-Dimethylphenol	5	170	1			
51-28-5	2,4-Dinitrophenol	10	1660	5			
121-14-2	2,4-Dinitrotoluene	5	170	5			
87-65-0	2,6-Dichlorophenol	10	330				
606-20-2	2,6-Dinitrotoluene	5	170	5			
53-96-3	2-Acetylaminofluorene	10	330	0.5			
95-51-2	2-Chloroaniline	10	330				
91-58-7	2-Chloronaphthalene	5	170	0.5			
95-57-8	2-Chlorophenol	5	170	5			
91-57-6	2-Methylnaphthalene	5	170	0.5	0.5	17	
95-48-7	2-Methylphenol	5	170	1			
91-59-8	2-Naphthylamine	10	330				
88-74-4	2-Nitroaniline	10	330	5			

88-75-5	2-Nitrophenol	5	170	5			
109-06-8	2-Picoline	80	330				
95-53-4	2-Toluidine	10	330				
15831-10-4	3 & 4 Methylphenol	10	330	1			
91-94-1	3,3'-Dichlorobenzidine	5	330	5			
119-93-7	3,3'-Dimethylbenzidine	40	660				
56-49-5	3-Methylcholanthrene	10	330				
99-09-2	3-Nitroaniline	10	330				
101-14-4	4,4'-Methylene bis(2-chloroaniline)	10	330				
534-52-1	4,6-Dinitro-2-methylphenol	10	330	5			
92-67-1	4-Aminobiphenyl	10	330				
101-55-3	4-Bromophenyl phenyl ether	5	170	5			
59-50-7	4-Chloro-3-methylphenol	5	170	5			
106-47-8	4-Chloroaniline	5	170	5			
7005-72-3	4-Chlorophenyl phenyl ether	5	170	5			
106-49-0	4-Methylbenzenamine	10	330				
106-44-5	4-Methylphenol	10	330	1			
100-01-6	4-Nitroaniline	10	330	5			
100-02-7	4-Nitrophenol	10	330	5			
56-57-5	4-Nitroquinoline-1-oxide	10	660				
1705-85-7	6-Methylchrysene	10	330	0.5			
57-97-6	7,12-Dimethylbenz(a)anthracene	10	330				
301-02-0	9-Octadecenamide	100	3300				
83-32-9	Acenaphthene	5	170	0.5	0.5	17	
208-96-8	Acenaphthylene	5	170	0.3	0.5	17	
98-86-2	Acetophenone	5	170	5			
79-06-1	Acrylamide	5	330				
15972-60-8	Alachlor	10	330	1.5			
122-09-8	alpha,alpha-Dimethyl phenethylamine	100	330				
98-55-5	Alpha-Terpineol	10	330				
62-53-3	Aniline	10	330	0.5			
120-12-7	Anthracene	5	170	0.5	0.5	17	
84-65-1	Anthraquinone	10	330				
140-57-8	Aramite, Total	20	330				
1912-24-9	Atrazine	5	170	2			
103-33-3	Azobenzene	10	330	0.5			
100-52-7	Benzaldehyde	5	170	5			
92-87-5	Benzidine	80	5000	5			
56-55-3	Benzo[a]anthracene	5	170	0.3	0.5	17	
50-32-8	Benzo[a]pyrene	5	170	0.18	0.5	17	
205-99-2	Benzo[b]fluoranthene	5	170	0.3	0.5	17	

191-24-2	Benzo[g,h,i]perylene	5	170	0.5	0.5	17	
207-08-9	Benzo[k]fluoranthene	5	170	0.3	0.5	17	
65-85-0	Benzoic acid	150	4800	5			
100-51-6	Benzyl alcohol	20	330	5			
111-91-1	Bis(2-chloroethoxy)methane	5	170	5			
111-44-4	Bis(2-chloroethyl)ether	5	170	5			
117-81-7	Bis(2-ethylhexyl) phthalate	5	170	5			
85-68-7	Butyl benzyl phthalate	5	170	3			
105-60-2	Caprolactam	5	170	5			
86-74-8	Carbazole	5	170	5			
510-15-6	Chlorobenzilate	20	330	0.5			
218-01-9	Chrysene	5	170	0.5			
2303-16-4	Diallate	10	330				
53-70-3	Dibenz(a,h)anthracene	5	170	0.5	0.5	17	
226-36-8	Dibenz[a,h]acridine	10	330	0.5			
192-65-4	Dibenzo[a,e]pyrene	10	330				
132-64-9	Dibenzofuran	10	170	5	0.5	17	
101-83-7	Dicyclohexylamine	10	3000				
84-66-2	Diethyl phthalate	5	170	0.5			
60-51-5	Dimethoate	10	330				
131-11-3	Dimethyl phthalate	5	170	0.5			
84-74-2	Di-n-butyl phthalate	5	170	2			
117-84-0	Di-n-octyl phthalate	5	170	5			
88-85-7	Dinoseb	10	330				
122-39-4	Diphenylamine	10	330	5			
298-04-4	Disulfoton	10	330				
62-50-0	Ethyl methanesulfonate	10	330				
56-38-2	Ethyl Parathion	10	330	1			
52-85-7	Famphur	40	660				
206-44-0	Fluoranthene	5	170	0.5	0.5	17	
86-73-7	Fluorene	5	170	0.5	0.5	17	
118-74-1	Hexachlorobenzene	5	170	0.5			
87-68-3	Hexachlorobutadiene	5	170	1.0			
77-47-4	Hexachlorocyclopentadiene	5	170	1			
67-72-1	Hexachloroethane	5	170	5			
70-30-4	Hexachlorophene	310	5000				
1888-71-7	Hexachloropropene	10	330				
544-76-3	Hexadecane	10	330	0.5			
95-13-6	Indene	60	3000	5			
193-39-5	Indeno[1,2,3-cd]pyrene	5	170	0.5	0.5	17	
465-73-6	Isodrin	10	330				

78-59-1	Isophorone	5	170				
120-58-1	Isosafrole	10	330	0.5			
143-50-0	Kepone	50	660				
91-80-5	Methapyrilene	50	1500				
66-27-3	Methyl methanesulfonate	10	330				
298-00-0	Methyl parathion	10	330				
91-20-3	Naphthalene	5	170	1	0.5	17	
124-18-5	n-Decane	5	330				
98-95-3	Nitrobenzene	5	170	0.5			
99-55-8	N-Nitro-o-toluidine	10	330				
55-18-5	N-Nitrosodiethylamine	10	330				
62-75-9	N-Nitrosodimethylamine	10	330	5			
924-16-3	N-Nitrosodi-n-butylamine	10	330				
621-64-7	N-Nitrosodi-n-propylamine	5	170	5			
86-30-6	N-Nitrosodiphenylamine	5	170	5			
10595-95-6	N-Nitrosomethylethylamine	10	330				
59-89-2	N-Nitrosomorpholine	10	330				
100-75-4	N-Nitrosopiperidine	10	330				
930-55-2	N-Nitrosopyrrolidine	10	330				
593-45-3	n-Octadecane	5	330				
126-68-1	o,o',o''-Triethylphosphorothioate	10	330				
60-11-7	p-Dimethylamino azobenzene	10	330				
608-93-5	Pentachlorobenzene	10	330				
76-01-7	Pentachloroethane	10	330				
82-68-8	Pentachloronitrobenzene	10	330				
87-86-5	Pentachlorophenol	10	330	1			
62-44-2	Phenacetin	10	330				
85-01-8	Phenanthrene	5	170	0.2	0.5	17	
108-95-2	Phenol	5	170	1			
298-02-2	Phorate	10	330				
85-44-9	Phthalic anhydride	500	10000				
106-50-3	p-Phenylene diamine	800	800				
23950-58-5	Pronamide	10	330				
129-00-0	Pyrene	5	170	0.5	0.5	17	
110-86-1	Pyridine	25	330				
91-22-5	Quinoline	10	330				
94-59-7	Safrole, Total	10	330				
122-34-9	Simazine	10	330	0.5			
3689-24-5	Sulfotepp	10	330				
78-00-2	Tetraethyl lead	10	1000				
297-97-2	Thionazin	10	330				

126-73-8	Tributyl phosphate	10	330				

Note, the most current reporting limits are maintained in the laboratory's LIMS system. These may be updated in LIMS as MDL studies are performed.

18.2 TABLE 2
LCS/MS/MSD Spike Analytes

1,1'-Biphenyl	4-Bromophenyl phenyl ether	Chrysene
1,2,4,5-Tetrachlorobenzene	4-Chloro-3-methylphenol	Dibenz(a,h)anthracene
1,2,4-Trichlorobenzene	4-Chloroaniline	Dibenzofuran
1,2-Dichlorobenzene	4-Chlorophenyl phenyl ether	Diethyl phthalate
1,2-Diphenylhydrazine	4-Methylphenol	Dimethyl phthalate
1,3-Dichlorobenzene	4-Nitroaniline	Di-n-butyl phthalate
1,4-Dichlorobenzene	4-Nitrophenol	Di-n-octyl phthalate
1,4-Dioxane	Acenaphthene	Diphenylamine
1-Methylnaphthalene	Acenaphthylene	Fluoranthene
2,2'-oxybis[1-chloropropane]	Acetophenone	Fluorene
2,3,4,6-Tetrachlorophenol	Aniline	Hexachlorobenzene
2,3-Dimethylphenol	Anthracene	Hexachlorobutadiene
2,4,5-Trichlorophenol	Atrazine	Hexachlorocyclopentadiene
2,4,6-Trichlorophenol	Azobenzene	Hexachloroethane
2,4-Dichlorophenol	Benzaldehyde	Hexadecane
2,4-Dimethylphenol	Benzidine	Indene
2,4-Dinitrophenol	Benzo[a]anthracene	Indeno[1,2,3-cd]pyrene
2,4-Dinitrotoluene	Benzo[a]pyrene	Isophorone
2,6-Dinitrotoluene	Benzo[b]fluoranthene	Naphthalene
2-Chloronaphthalene	Benzo[g,h,i]perylene	Nitrobenzene
2-Chlorophenol	Benzo[k]fluoranthene	N-Nitrosodimethylamine
2-Methylnaphthalene	Benzoic acid	N-Nitrosodi-n-propylamine
2-Methylphenol	Benzyl alcohol	N-Nitrosodiphenylamine
2-Nitroaniline	Bis(2-chloroethoxy)methane	Pentachlorophenol
2-Nitrophenol	Bis(2-chloroethyl)ether	Phenanthrene
3,3'-Dichlorobenzidine	Bis(2-ethylhexyl) phthalate	Phenol
3-Methylphenol	Butyl benzyl phthalate	Pyrene
3-Nitroaniline	Caprolactam	Pyridine
4,6-Dinitro-2-methylphenol	Carbazole	

18.3 TABLE 3
DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10.0 – 80.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	0-100 percent of mass 198

70	Less than 2.0 percent of mass 69
127	10.0 – 80.0 percent of mass 198
197	Less than 2.0 percent of mass 198
198	Base peak, or greater than 50 percent of mass 442
199	5.0-9.0 percent of mass 198
275	10.0-60.0 percent of mass 198
365	Greater than 1.0 percent of mass 198
441	Present but less than 24 percent of mass 442
442	Base Peak, or greater than50 percent of mass 198
443	15.0 – 24.0 percent of mass 442

18.4 TABLE 4
Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

1,4-Dichlorobenzene-d4	**Naphthalene-d8**	**Acenaphthene-d10**	**Phenanthrene-d10**	**Chrysene-d12**	**Perylene-d12**
1,2,3,4-Tetrachlorobenzene	1,2,4-Trichlorobenzene	1,1'-Biphenyl	1,2-Diphenylhydrazine	2-Methylanthracene	3-Methylcholanthrene
1,2-Dichlorobenzene	1,3-Dinitrobenzene	1,2,4,5-Tetrachlorobenzene	1,4-Dihydroxyanthraquinone	3,3'-Dichlorobenzidine	Benzo[a]pyrene
1,3,5-Trichlorobenzene	1,4-Dinitrobenzene	1,3,5-Trinitrobenzene	1-Hydroxyanthraquinone	4,4'-Methylene bis(2-chloroaniline)	Benzo[b]fluoranthene
1,3-Dichlorobenzene	1-Methylnaphthalene	1,4-Naphthoquinone	2,3,5,6-Tetrachlorophenol	6-Methylchrysene	Benzo[g,h,i]perylene
1,4-Dichlorobenzene	2,4-Dichlorophenol	1-Chloronapthalene	2,4,6-Tribromophenol (surr)	7,12-Dimethylbenz(a)anthracene	Benzo[k]fluoranthene
1,4-Dioxane	2,4-Dimethylphenol	1-Naphthylamine	2-Acetylaminofluorene	Benzidine	Dibenz[a,h]acridine
2,2'-oxybis-(1-Chloropropane)	2,6-Dichlorophenol	2,3,4,6-Tetrachlorophenol	3.3'-Dimethylbenzidine	Benzo[a]anthracene	Dibenzo(a,h)anthracene
2-chloroaniline	2-Methylnaphthalene	2,4,5-Trichlorophenol	4,6-Dinitro-2-methylphenol	bis(2-ethylhexyl) phthalate	Dibenzo[a,e]pyrene
2-Chlorophenol	2-Nitrophenol	2,4,6-Trichlorophenol	4-Aminobiphenyl	Butyl benzyl phthalate	Indeno[1,2,3-cd]pyrene
2-Fluorophenol(surr)	4-Chloro-3-methylphenol	2,4-Dinitrophenol	4-Bromophenyl phenyl ether	Chrysene	
2-Methylphenol	4-Chloroaniline	2,4-Dinitrotoluene	4-Nitroquinoline-1-oxide	Di-n-octyl phthalate	
2-Picoline	alpha,alpha-Dimethyl phenethylamine	2,6-Dinitrotoluene	9-Octadecenamide	Hexachlorophene	
2-Toluidine	Alpha-Terpineol	2-Chloroaphthalene	Alachlor	p-Dimethylamino azobenzene	
4-Methylbenzenamine	Benzoic acid	2-Fluorobiphenyl (surr)	Anthracene	Pyrene	
4-Methylphenol	bis(2-Chloroethoxy)methane	2-Naphthylamine	Anthraquinone	p-Terphenyl-d14 (surr)	

Acetophenone	Caprolactam	2-Nitroaniline	Aramite, Total		
Acrylamide	Hexachlorobutadiene	3-Nitroaniline	Atrazine		
Aniline	Hexachloropropene	4-Chlorophenyl phenyl ether	Azobenzene		
Benzaldehyde	Isophorone	4-Nitroaniline	Carbazole		
Benzyl Alcohol	Naphthalene	4-Nitrophenol	Chlorobenzilate		
bis(2-Chloroethyl)ether	Nitrobenzene	Acenaphthene	Di-n-butyl phthalate		
Ethyl methanesulfonate	Nitrobenzene-d5 (surr)	Acenaphthylene	Dinoseb		
Hexachloroethane	N-Nitrosodi-n-butylamine	Diallate	Diphenylamine		
Indene	N-Nitrosopiperidine	Dibenzofuran	Disulfoton		
Methyl methanesulfonate	Phthalic Anhydride	Dicyclohexylamine	Ethyl parathion		
n-Decane	Quinoline	Diethyl phthalate	Famphur		
N-nitrosodiethylamine	Safrole, Total	Dimethoate	Fluoranthene		
N-nitrosodimethylamine	TetraEthyl Lead	Dimethyl phthalate	Hexachlorobenzene		
N-Nitrosodi-n-propylamine		Fluorene	Isodrin		
N-Nitrosomethylethylamine		Hexachlorocyclopentadiene	Kepone		
N-Nitrosomorpholine		Hexadecane	Methyapyrilene		
N-nitrosopyrrolidine		Isosafrole	Methyl parathion		
o,o',o"-Triethylphosphorothioate		N-Nitro-o-toluidine	N-nitrosodiphenylamine		
Pentachloroethane		Pentachlorobenzene	n-Octadecane		
Phenol		Phorate	Pentachloronitrobenzene		
Phenol-d5 (surr)		Simazine	Pentachlorophenol		
p-Phenylene diamine		Sulfotepp	Phenacetin		
Pyridine		Thionazin	Phenanthrene		
1,4-Dioxane-d8 (analog)		Tributyl phosphate	Pronamide		

Note: Internal Standard assignments are by suggestion only. Assignments may vary slightly between instrument methods.

18.5 Table 5
Characteristic Ions for Semivolatile
Target Compounds, Surrogates and Internal Standards

Parameters	Primary Quantitation Ion	Secondary Ion(s)	Parameters	Primary Quantitation Ion	Secondary Ion(s)	Parameters	Primary Quantitation Ion	Secondary Ion(s)
1,1'-Biphenyl	154	153, 152	4-Methylphenol	108	107	Famphur	218	125, 93
1,2,4,5-Tetrachlorobenzene	216	214, 179	4-Nitroaniline	138	92, 108	Fluoranthene	202	101, 203
1,2,4-Trichlorobenzene	180	182, 145	4-Nitrophenol	109	139, 64	Fluorene	166	165, 167
1,2-Dichlorobenzene	146	111, 75	4-Nitroquinoline-1-oxide	190	160, 89	Hexachlorobenzene	284	142, 249
1,2-Diphenylhydrazine	77	182, 51	6-Methylchrysene	242	239, 119	Hexachlorobutadiene	225	223, 227
1,3,5-Trichlorobenzene	180	182, 184	7,12-Dimethylbenz(a)anthracene	256	241, 239	Hexachlorocyclopentadiene	237	235, 272
1,3,5-Trinitrobenzene	213	75, 74	9-Otadecenamide	59	72, 55	Hexachloroethane	117	201, 199
1,3-Dichlorobenzene	146	111, 75	a.a.-Dimethyl phenethylamine	58	91, 134	Hexachlorophene	196	198, 209
1,3-Dinitrobenzene	168	50, 76	Acenaphthene	153	152, 154	Hexachloropropene	213	215, 117
1,4-Dichlorobenzene	146	111, 75	Acenaphthylene	152	151, 153	Hexadecane	57	43, 71
1,4-Dihydroxyanthraquinone	240	239, 128	Acetophenone	105	77, 51	Indene	115	116, 89
1,4-Dinitrobenzene	168	75, 50	Acrylamide	71	55, 44	Indeno(1,2,3-cd)pyrene	276	138, 277
1,4-Dioxane	88	58	Alachlor	160	188, 146	Isodrin	193	195, 66
1,4-Naphthoquinone	158	102, 130	Aniline	93	66, 39	Isophorone	82	95, 138
1-Chloronaphthalene	162	127, 164	Anthracene	178	179, 176	Isosafrole	162	104, 131
1-Hydroxyanthraquinone	224	139, 168	Anthraquinone	180	208, 152	Kepone	272	237, 357
1-Methylnaphthalene	142	141, 115	Aramite, Total	185	63, 135	Methapyrilene	58	97, 191
1-Naphthylamine	143	115, 116	A-Terpineol	59	93, 121	Methyl parathion	109	125, 263
2,2'-oxybis[1-chloropropane]	45	77, 79	Atrazine	200	215, 202	Naphthalene	128	129, 127

2,3,4,6-Tetrachlorophenol	232	230, 131	Azobenzene	77	182, 51	n-Decane	57	43, 41
2,3,5,6-Tetrachlorophenol	232	230, 234	Benzaldehyde	77	105, 106	Nitrobenzene	77	123, 65
2,4,5-Trichlorophenol	196	198, 200	Benzidine	184	92, 156	N-Nitro-o-toluidine	152	106, 77
2,4,6-Trichlorophenol	196	198, 200	Benzo[a]anthracene	228	229, 226	N-Nitrosodiethylamine	102	42, 44
2,4-Dichlorophenol	162	164, 98	Benzo[a]pyrene	252	253, 125	N-Nitrosodimethylamine	42	74, 43
2,4-Dimethylphenol	107	121, 122	Benzo[b]fluoranthene	252	253, 125	N-Nitrosodi-n-butylamine	84	57, 116
2,4-Dinitrophenol	184	63, 154	Benzo[g,h,i]perylene	276	138,277	N-Nitrosodi-n-propylamine	70	42, 130
2,4-Dinitrotoluene	165	63, 182	Benzo[k]fluoranthene	252	253, 125	N-Nitrosodiphenylamine	169	168, 167
2,6-Dichlorophenol	162	164, 166	Benzoic Acid	105	122, 77	N-Nitrosomethylethylamine	88	42, 43
2,6-Dinitrotoluene	165	89, 121	Benzyl Alcohol	108	79, 77	N-Nitrosomorpholine	56	86, 116
2-Acetylaminofluorene	181	180, 223	bis(2-Chloroethoxy) methane	93	95, 123	N-Nitrosopiperidine	114	55, 42
2-Chloroaniline	127	129, 65	bis(2-Chloroethyl)ether	93	63, 95	N-Nitrosopyrrolidine	100	41, 42
2-Chloronaphthalene	162	164, 127	bis(2-Ethylhexyl) phthalate	149	167, 279	n-Octadecane	57	43, 71
2-Chlorophenol	128	64, 130	Butyl benzyl phthalate	149	91, 206	o,o'o"-Triethylphosphorothioate	198	121, 97
2-Methylanthracene	192	191, 193	Caprolactam	113	85, 84	p-Dimethylamino azobenzene	120	225, 77
2-Methylnaphthalene	142	141, 115	Carbazole	167	139, 166	Pentachlorobenzene	250	252, 254
2-Methylphenol	108	107, 77	Chlorobenzilate	251	139, 111	Pentachloroethane	167	165, 169
2-Naphthylamine	143	115, 116	Chrysene	228	226, 229	Pentachloronitrobenzene	237	214, 295
2-Nitroaniline	65	92, 138	Diallate	43	234, 236	Pentachlorophenol	266	264, 268
2-Nitrophenol	139	65, 109	Dibenz[a,h]acridine	279	139, 125	Phenacetin	108	179, 137
2-Picoline	93	66, 39	Dibenzo(a,h)anthracene	278	139, 279	Phenanthrene	178	179, 176

2-Toluidine	106	107, 77	Dibenzo[a,e]pyrene	302	151, 150	Phenol	94	65, 66
3,3'-Dichlorobenzidine	252	254, 154	Dibenzofuran	168	139, 169	Phorate	75	121, 97
3,3'-Dimethylbenzidine	212	106, 196	Dicyclohexylamine	138	56, 55	Phthalic Anhydride	104	76, 148
3-Methylcholanthrene	268	252, 126	Diethyl phthalate	149	177, 150	p-Phenylene diamine	108	107, 80
3-Nitroaniline	138	92, 65	Dimethoate	87	125, 93	Pronamide	173	175, 177
4,4'-Methylene bis(2-chloroaniline	231	266, 268	Dimethyl phthalate	163	194, 164	Pyrene	202	101, 100
4,6-Dinitro-2-methylphenol	198	121, 105	Di-n-butylphthalate	149	150, 104	Pyridine	52	79, 51
4-Aminobiphenyl	169	168, 170	Di-n-octyl phthalate	149	150, 167	Quinoline	129	128, 102
4-Bromophenyl-phenylether	248	250, 141	Dinoseb	211	163, 147	Safrole, Total	162	131, 104
4-Chloro-3-methylphenol	107	144, 142	Diphenylamine	169	168, 167	Simazine	201	186, 173
4-Chloroaniline	127	129, 65	Disulfoton	88	97, 61	Sulfotepp	322	202, 97
4-Chlorophenyl phenyl ether	204	206, 141	Ethyl methanesulfonate	79	109, 97	TetraEthyl Lead	237	295, 208
4-Methylbenzenamine	106	107, 77	Ethyl parathion	97	109, 291	Thionazin	97	107, 143
SURROGATES			**INTERNAL STANDARDS**			Tributyl phosphate	99	155, 211
Phenol-d5	99	42, 71	1,4-Dichlorobenzene-d4	152	115, 150	1,4-Dioxane-d8 (analog)	96	64
2-Fluorophenol	112	64, 92	Naphthalene-d8	136	68, 108			
2,4,6-Tribormophenol	330	332, 141	Acenaphthene-d10	164	162, 160			
Nitrobenzene-d5	82	128, 54	Phenanthrene-d10	188	94, 80			
2-Fluorobiphenyl	172	171, 170	Chrysene-d12	240	120, 236			
Terphenyl-d14	244	122, 212	Perylene-d12	264	260, 265			

Note: Quantitation and/or secondary qualifying ions are by suggestion only. Assignments may vary slightly between instrument methods.

18.6 Table 6
Poor Performing Compounds

Poor Performers	ICV %D Limit	CCV %D Limit	LCS %R Limit
3,3'-Dichlorobenzidine	± 50%	± 50%	10
9-Octadecenamide	± 50%	± 50%	10
a,a-Dimethyl phenethylamine	± 50%	± 50%	10
Acrylamide	± 50%	± 50%	10
Benzaldehyde	± 50%	± 50%	10
Benzidine	± 50%	± 50%	5
Benzoic Acid	± 50%	± 50%	10
Caprolactam	± 50%	± 50%	10
Isosafrole	± 50%	± 50%	10
Kepone	± 50%	± 50%	10
Methapyrilene	± 50%	± 50%	10
n-Nitrosodimethylamine	± 50%	± 50%	10
p-Phenylene diamine	± 50%	± 50%	10
Phthalic Anhydride	± 50%	± 50%	10
Pyridine	± 50%	± 50%	10
Safrole, Total	± 50%	± 50%	10

The laboratory’s GC/MS semi-volatiles group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data as well as calibration data to validate each compound as a “poor performer”.

18.7 Table 7
Minimum Response Factors for common target compounds

Semivolatile Compounds	Minimum Response Factor (RF)	Semivolatile Compounds	Minimum Response Factor (RF)
1,2,4-Trichlorobenzene	0.010	bis(2-Chloroethoxy)methane	0.300
1,2-Dichlorobenzene	0.010	bis(2-Chloroethyl)ether	0.700
1,3-Dichlorobenzene	0.010	Bis(2-chloroisopropyl)ether	0.010
1,4-Dichlorobenzene	0.010	bis(2-Ethylhexyl)phthalate	0.010
2,4,5-Trichlorophenol	0.200	Butylbenzylphthalate	0.010
2,4,6-Trichlorophenol	0.200	Chrysene	0.700
2,4-Dichlorophenol	0.200	Dibenzo(a,h)anthracene	0.400
2,4-Dimethylphenol	0.200	Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200	Diethyl phthalate	0.010
2,6-Dinitrotoluene	0.200	Dimethyl phthalate	0.010
2-Chloronaphthalene	0.800	Di-n-butyl phthalate	0.010
2-Chlorophenol	0.800	Di-n-octyl phthalate	0.010
2-Methylnaphthalene	0.400	Fluoranthene	0.600
2-Methylphenol	0.700	Fluorene	0.900
2-Nitrophenol	0.100	Hexachlorobenzene	0.100
4-Bromophenyl-phenylether	0.100	Hexachlorobutadiene	0.010
4-Chloro-3-methylphenol	0.200	Hexachlorocyclopentadiene	0.050
4-Chloroaniline	0.010	Hexachloroethane	0.300
4-Chlorophenyl-phenylether	0.400	Indeno(1,2,3-cd)pyrene	0.500
4-Methylphenol	0.600	Isophorone	0.400
Acenaphthene	0.900	Naphthalene	0.700
Acenaphthylene	0.900	Nitrobenzene	0.200
Anthracene	0.700	N-Nitroso-di-n-propylamine	0.500
Benzo(a)anthracene	0.800	N-Nitrosodiphenylamine	0.010
Benzo(a)pyrene	0.700	Pentachlorophenol	0.050
Benzo(b)fluoranthene	0.700	Phenanthrene	0.700
Benzo(g,h,i)perylene	0.500	Phenol	0.800
Benzo(k)fluoranthene	0.700	Pyrene	0.600
Benzyl Alcohol	0.010		

18.8 TABLE 8
Surrogate Recovery Limits[1]

Surrogate	% Recovery Limit (1L, LVI)	% Recovery Limit (3350C/3546)	% Recovery Limit (LL)	% Recovery Limit (LL_PAH)	% Recovery Limit (SIM)
2,4,6-Tribromophenol	41-120	54-120	24-146	---	---
2-Fluorobiphenyl	48-120	60-120	37-120	37-120	---
2-Fluorophenol	35-120	52-120	10-120	---	---
Nitrobenzene-d5	46-120	53-120	26-120	34-132	---
Phenol-d5	22-120	54-120	11-120	---	---
p-Terphenyl-d14	59-136	65-121	64-127	58-147	---
1,4-Dioxane-d8 (analog)	---	---	---	---	15-110

[1] Limits are updated and entered annually into LIMS

18.9 TABLE 9
Allowable CCV Failures

Total CCVs Analyzed in a Batch	Total # of Analytes	# Allowed out between CCVs (20%)
List 1	94	18
List 1, List 2	148	29
List 1, List 3	109	21
List 1, List 2, List 3	163	32

Note: this is based on the laboratory's main list of calibrated analytes.
Additional analytes/CCVs may be analyzed and the total number may be adjusted accordingly.

18.10 TABLE 10
Sample Dilutions

Dilution Factor	uL of Sample Extract	uL of $MeCl_2$	Total Volume (uL)	uL of IS
2	500	500	1000	20
4	250	750	1000	20
5	200	800	1000	20
10	100	900	1000	20
20	50	950	1000	20
25	40	960	1000	20
40	25	975	1000	20
50	20	980	1000	20
100	10	990	1000	20

Dilutions greater than 100X must be performed by serial dilution.

18.11 TABLE 11
8270D/E/MCP/RCP/SIM QC Requirements and Acceptance Criteria Summary

8270D			
QA Parameter	**Technical Acceptance**	**Allowable Failures**	**Narration Required**
DFTPP	1. Criteria listed in Table 3. 2. Tailing for Benzidine and PCP ≤2. 3. Breakdown of 4,4'-DDT ≤20 %	None	NA
ICAL	1. 20% RSD or r^2 ≥0.990 2. Minimum RF for all levels must meet criteria in Table 7. 3. ≤30% Readback on low point.	10% allowed out with passing RL check.	Detection only
ICV	1. 70-130%. 2. Meets min RF criteria in Table 7.	Recovery high - sample analysis ND.	Detection only
CCV	1. 20%D. 2. Meets min RF criteria in Table 7.	20% allowed out with passing RL check.	Yes
MB	Less than RL.	1. >RL with ND sample. 2. >RL with detection in sample >10X the MB.	Yes
LCS/LCSD	Historical limits for %R and %RPD.	1. Poor performers ≥10% recovery, ≥5% for benzidine. 2. High recovery, sample analysis ND.	Yes
MS/MSD	Historical limits for %R and %RPD.	Recoveries within limits in the LCS.	Recoveries ≤10% or ≥150% and/or RPD ≥50%.
Surrogates	Historical limits.	1. 1 acid and/or 1 B/N out, provided recovery is ≥10%. 2. Recovery high, samples ND. 3. Recovery low for 1 class, target analyte list requires compounds from other class only. 4. Obvious matrix interference. 5. Sample diluted 20X or greater.	Yes
Internal Standards	1. 50-200% of CCV to mid-level ICAL standard. 2. ≤30 sec shift of CCV to mid-level standard. 3. 50-200% recovery of samples to daily CCV. 4. ≤ 30 sec shift of samples to daily CCV.	None	NA

8270E			
QA Parameter	**Technical Acceptance**	**Allowable Failures**	**Narration Required**
DFTPP	1. Criteria listed in Table 3. 2. Tailing for Benzidine and PCP ≤2. 3. Breakdown of 4,4'-DDT ≤20 % 4. DFTPP tuning only required prior to ICAL	None	NA
ICAL	1. 20% RSD or r^2 ≥0.990 2. Minimum RF for all levels must meet criteria in Table 7. 3. ≤50% Readback on low point.	10% allowed out with passing RL check.	Detection only
ICV	1. 70-130%. 2. Meets min RF criteria in Table 7.	Recovery high - sample analysis ND.	Detection only
CCV	1. 20%D. 2. No min RF requirement.	20% allowed out with passing RL check.	Yes
MB	Less than RL.	1. >RL with ND sample. 2. >RL with detection in sample >10X the MB.	Yes
LCS/LCSD	Historical limits for %R and %RPD.	1. Poor performers ≥10% recovery, ≥5% for benzidine. 2. High recovery, sample analysis ND.	Yes
MS/MSD	Historical limits for %R and %RPD.	Recoveries within limits in the LCS.	Recoveries ≤10% or ≥150% and/or RPD ≥50%.
Surrogates	Historical limits.	1. 1 acid and/or 1 B/N out, provided recovery is ≥10%. 2. Recovery high, samples ND. 3. Recovery low for 1 class, target analyte list requires compounds from other class only. 4. Obvious matrix interference. 5. Sample diluted 20X or greater.	Yes
Internal Standards	1. 50-200% of CCV to mid-level ICAL standard. 2. ≤30 sec shift of CCV to mid-level standard. 3. 50-200% recovery of samples to daily CCV. 4. ≤ 30 sec shift of samples to daily CCV.	None	NA

MCP			
QA Parameter	**Technical Acceptance**	**Allowable Failures**	**Narration Required**
DFTPP	1. Criteria listed in Table 3. 2. Tailing for Benzidine and PCP ≤2. 3. Breakdown of 4,4'-DDT ≤20 %	None	NA
ICAL	1. 20% RSD or r^2 ≥0.990 2. Minimum RF must meet criteria in Table 7. 3. 0.05 min RF for other compounds on the low pt and average RF only. 4. ≤30% Readback on low point.	10% allowed out as long as %RSD is ≤40% or r^2 ≥0.980	Yes - RSD, r^2 and RF failures
ICV	1. 70-130% except "difficult" analytes**, which are 60-140%.	10% allowed out.	Yes
CCV	1. 20%D. 2. Meets min RF criteria in Table 7. 3. 0.05 min RF for remaining compounds.	20% allowed out, provided recovery is ≤40%.	Yes
MB	Less than RL.	1. >RL with ND sample. 2. >RL with detection in sample >10X the MB.	Yes
LCS/LCSD	1. 40-140% for base-neutral, 30-130% for acids, except "difficult" analytes**, which are 15-140%. 2. RPD ≤20% for waters, ≤30% for soils.	1. "Difficult" analytes 15-140%. 2. ≤10% allowed to fail provided recovery is ≥10%.	Yes
MS/MSD	1.40-140% for base-neutral, 30-130% for acids. 2. RPD ≤20% for waters, ≤30% for soils.	Recoveries within limits in the LCS.	Recoveries ≤10% or ≥150% and/or RPD ≥50%.
Surrogates	30-130% for soils, 30-130% for base-neutrals in water, 15-110% for acids in water.	1. 1 acid and/or 1 B/N out, provided recovery is ≥10%. 2. Recovery high, samples ND. 3. Recovery low for 1 class, target analyte list requires compounds from other class only. 4. Obvious matrix interference. 5. Sample diluted 20X or greater.	Yes
Internal Standards	1. 50-200% of CCV to mid-level ICAL standard. 2. ≤30 sec shift of CCV to mid-level standard. 3. 50-200% recovery of samples to daily CCV. 4. ≤ 30 sec shift of samples to daily CCV.	None	NA

** "Difficult" analytes are 4-Chloroaniline, 4-Nitrophenol, Phenol and 2,4-Dinitrophenol.

RCP			
QA Parameter	**Technical Acceptance**	**Allowable Failures**	**Narration Required**
DFTPP	1. Criteria listed in Table 3. 2. Tailing for Benzidine and PCP ≤2. 3. Breakdown of 4,4'-DDT ≤20 %	None	NA
ICAL	1. 20% RSD or r^2 ≥0.990 2. Minimum RF must meet 0.05 for all compounds.	20% allowed out.	Yes - RSD, r^2 and RF failures
ICV	80-120%.	20% allowed out as long as recovery within 65-135%.	Yes
CCV	20%D.	10% allowed out.	Yes
MB	Less than RL.	1. >RL with ND sample. 2. >RL with detection in sample >10X the MB.	Yes
LCS/LCSD	1. 40-140% for base-neutral, 30-130% for acids. 2. RPD ≤20% for waters, ≤30% for soils.	≤20% allowed to fail provided recovery is ≥10%.	Yes
MS/MSD	1. 40-140% for base-neutral, 30-130% for acids. 2. RPD ≤20% for waters, ≤30% for soils.	Recoveries within limits in the LCS.	Recoveries ≤10% or ≥150% and/or RPD ≥50%.
Surrogates	30-130% for soils, 30-130% for base-neutrals in water, 15-110% for acids in water.	1. 1 acid and/or 1 B/N out, provided recovery is ≥10%. 2. Recovery high, samples ND. 3. Recovery low for 1 class, target analyte list requires compounds from other class only. 4. Obvious matrix interference. 5. Sample diluted 20X or greater.	Yes
Internal Standards	1. 50-200% of CCV to mid-level ICAL standard. 2. ≤30 sec shift of CCV to mid-level standard. 3. 50-200% recovery of samples to daily CCV. 4. ≤ 30 sec shift of samples to daily CCV.	None	NA

SIM			
QA Parameter	**Technical Acceptance**	**Allowable Failures**	**Narration Required**
DFTPP	1. Criteria listed in Table 3.	None	NA
ICAL	1. 20% RSD or $r^2 \geq 0.990$ 2. ≤30% Readback on low point.	None	NA
ICV	80-120%.	Recovery high - sample analysis ND.	Detection only
CCV	20%D.	Recovery high - sample analysis ND.	Yes
MB	Less than RL.	1. >RL with ND sample. 2. >RL with detection in sample >10X the MB.	Yes
LCS/LCSD	1. 40-140%. 2. RPD ≤20%.	High recovery, sample analysis ND.	Yes
MS/MSD	1. 40-140%. 2. RPD ≤20%.	Recoveries within limits in the LCS.	Recoveries ≤10% or ≥150% and/or RPD ≥50%.
Analog	Historical limits.	1. Recovery high, samples ND. 2. Obvious matrix interference.	Yes
Internal Standards	1. 50-200% of CCV to mid-level ICAL standard. 2. ≤30 sec shift of CCV to mid-level standard. 3. 50-200% recovery of samples to daily CCV. 4. ≤ 30 sec shift of samples to daily CCV.	None	NA

Title: Per- and Polyfluorinated Alkyl Substances (PFAS) in Water, Soils, Sediments and Tissue

[Method 537 (Modified), Method PFAS by LCMSMS Compliant with QSM Table B-15, Revision 5.3 and higher]

Approvals (Signature/Date):

01/27/2021	01/27/2021
Robert Hrabak Date	Joe Schairer Date
Technical Manager	Health & Safety Manager / Coordinator
01/27/2021	01/27/2021
Lisa Stafford Date	Chris Williams Date
Quality Assurance Manager	Laboratory Manager

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water, soil, sediment, and tissue samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

Table 1.1
PFAS Supported

Compound Name	Abbreviations	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
Perfluoro-n-hexanoic acid	PFHxA	307-24-4
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluoro-n-decanoic acid	PFDA	335-76-2
Perfluoro-n-undecanoic acid	PFUdA, PFUnA	2058-94-8
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
Perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8
Perfluoro-n-tetradecanoic acid	PFTeDA, PFTA	376-06-7
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-pentanesulfonic acid	PFPeS	2706-91-4
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1
Perfluoro-nonanesulfonic acid	PFNS	68259-12-1
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3
Perfluoro-1-dodecansulfonic acid	PFDoS	79780-39-5
Perfluorinated sulfonamides (FOSA)		
Perfluoro-1-octanesulfonamide	PFOSA, FOSA	754-91-6
N-ethylperfluoro-1-octanesulfonamide	Et-FOSA, N-Et-FOSA	4151-50-2
N-methylperfluoro-1-octanesulfonamide	Me-FOSA, N-Me-FOSA	31506-32-8
Perfluorinated sulfonamide ethanols (FOSE)		
2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	Et-FOSE, N-Et-FOSE	1691-99-2
2-(N-methylperfluoro-1-octanesulfonamido) ethanol	Me-FOSE, N-Me-FOSE	24448-09-7
Perfluorinated sulfonamidoacetic acids (FOSAA)		
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6

Table 1.1 PFAS Supported		
Compound Name	**Abbreviations**	**CAS #**
	N-EtFOSAA	
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA, N-MeFOSAA	2355-31-9
Fluorotelomer sulfonates (FTS)		
1H,1H,2H,2H-perfluorohexane sulfonic acid *(4:2)*	4:2 FTS	757124-72-4
1H,1H,2H,2H-perfluorooctane sulfonic acid *(6:2)*	6:2 FTS	27619-97-2
1H,1H,2H,2H-perfluorodecane sulfonic acid *(8:2)*	8:2 FTS	39108-34-4
1H,1H,2H,2H-perfluorododecane sulfonic acid *(10:2)*	10:2 FTS	120226-60-0
Fluorinated Replacement Chemicals		
4,8-dioxa-3H-perfluorononanoic acid	DONA, ADONA(1)	919005-14-4
Perfluoro(2-propoxypropanoic) acid or Hexafluoropropylene oxide dimer acid	HFPO-DA, GenX	13252-13-6
F53B (reported as the summation of the following)	F53B	NA
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	F53B major, 9Cl-PF3ONS	756426-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	F5B minor, 11Cl-PF3OUdS	763051-92-9

***Note**: Abbreviations in parenthesis are the abbreviations listed in Method 537/537.1, where they differ from the abbreviation used by the laboratory's LIMS.*

(1) In some literature, the acronym ADONA refers to the ammonium salt, CAS 958445-44-8, and DONA refers to the parent acid. In Method 537.1, ADONA refers to the parent acid. DONA is the acronym present on the laboratory raw data.

1.2. Additional analytes supported by this method: The following analytes can be supported by this method under special request.

Table 1.2 Additional Compounds		
Compound Name	**Abbreviation**	**CAS #**
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-hexadecanoic acid	PFHxDA	67905-19-5
Perfluoro-n-octadecanoic acid	PFODA	16517-11-6
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-4-ethylcyclohexanesulfonic acid	PFECHS	133201-07-7
Perfluoropropanesulfonic acid	PFPrS	423-41-6
Fluorotelomer carboxylic acids (FTCA)		
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
2-Perfluorohexylethanoic acid	6:2 FTCA	53826-12-3
2-Perfluorooctylethanoic acid	8:2 FTCA, FOEA	27854-31-5

Table 1.2 Additional Compounds		
Compound Name	**Abbreviation**	**CAS #**
2-Perfluorodecylethanoic acid	10:2 FTCA	53826-13-4
Fluorotelomer unsaturated carboxylic acids (FTUCA)		
2H-Perfluoro-2-octenoic acid	6:2 FTUCA	70887-88-6
2H-Perfluoro-2-decenoic acid	8:2 FTUCA	70887-84-2
2H-Perfluoro-2-dodecenoic acid	10:2 FTUCA	70887-94-4
Short Chain		
Perfluoropropionic acid (PPF Acid)	PFPrA, PPF Acid	422-64-0
Perfluoro-3-methoxypropanoic acid (PFMPA)	PFECA F, PFMPA, PFMOPrA	377-73-1
Perfluoro-4-methoxybutanoic acid (PFMBA)	PFECA A, PFMBA, PFMOBA	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	PFECA B, NFDHA	151772-58-6
Perfluoro(2-ethoxyethane) sulfonic acid (PFEESA)	PES, PFEESA	113507-82-7
Difluoro(perfluoromethoxy)acetic acid	PFMOAA	674-13-5
Perfluoro-4-isopropoxybutanoic acid	PFECA G, PFPE-1	801212-59-9
Perfluoro-3,5,7,9-butaoxadecanoic acid	PFO4DA	39492-90-5
Perfluoro-3,5,7-trioxaoctanoic acid	PFO3OA	39492-89-2
Perfluoro-3,5-dioxahexanoic acid	PFO2HxA	39492-88-1
Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid	PFESA BP 1 PS Acid	29311-67-9
Perfluoro-2-{[perfluoro-3-(perfluoroethoxy)-2-propanyl]oxy}ethanesulfonic acid	PFESA BP 2 Hydro-PS Acid	749836-20-2
Perfluoro-3,5,7,9,11-pentaoxadodecanoic acid	PFO5DA, PFO5DoA, TAF	39492-91-6
Perfluoro-2-(perfluoromethoxy)propanoic acid	PMPA	13140-29-9
2,3,3,3-Tetrafluoro-2-(pentafluoroethoxy)propanoic acid	PEPA	267239-61-2
3-(Methoxy)tetrafluoropropionic acid	MTP	93449-21-9
4-(2-carboxy-1,1,2,2-tetrafluoroethoxy)-2,2,3,3,4,5,5,5-octafluoro-pentanoic acid	R-EVE	2416366-22-6
2,2,3,3-Tetrafluoro-3-{[1,1,1,2,3,3-hexafluoro-3-(1,2,2-trifluoroethenoxy)propan-2-yl]oxy}propanoic acid	EVE Acid	69087-46-3
1,1,2,2,4,5,5,5-heptafluoro-3-oxapentanesulfonic acid	NVHOS	1132933-86-8(2)
2,2,3,3-Tetrafluoro-3-{[1,1,1,2,3,3-hexafluoro-3-(1,2,2,2-tetrafluoroethoxy)propan-2-yl]oxy}propanoic acid	Hydro-EVE Acid	773804-62-9
Ethanesulfonic acid, 1,1,2,2-tetrafluoro-2-[1,2,2,3,3-pentafluoro-1-(trifluoromethyl)propoxy]-	Byproduct 6, R-PDSCA	2416366-21-5

(2) The CAS Number listed for NVHOS is for the sodium salt. As of this writing, there isn't a CAS number for the parent acid. The laboratory performs analysis for the sulfonic acid.

1.3. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Table 1.3
Reporting Limits and Working Range

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	250 mL	2.0 ng/L – 20 ng/L	2.0 ng/L - 800 ng/L
Soil/Sediment	5 g	0.2 µg/kg – 1.0 µg/kg	0.2 µg/kg - 50 µg/kg
Tissue	1 g	0.4 µg/kg – 2.0 µg/kg	0.4 µg/kg – 100 µg/kg

1.4. This procedure also includes direction for preparing samples to determine "Total Oxidizable Precursors", which may assist in improving understanding of potential PFAS environmental risk.

1.5. When undertaking projects for the Department of Defense (DoD) and/or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements" must be checked and incorporated.

1.6. Many jurisdictions have additional requirements for PFAS analysis, such as different holding times, preservation, or calibration criteria. These are detailed in the document WS-WI-0066, "Agency Specific Criteria for PFAS in Matrices Other Than Drinking Water".

2. SUMMARY OF METHOD

2.1. Water samples are extracted using a solid phase extraction (SPE) cartridge. PFAS are eluted from the cartridge with an [redacted] solution.

2.2. Soil/sediment/tissue samples are extracted with a KOH/methanol solution using sonication for 1 hour. The mixture is centrifuged and the solvent filtered.

2.3. The final 80:20 methanol:water extracts are analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using [redacted] The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFAS.

2.4. An isotope dilution technique is employed with this method for the compounds of interest. The isotope dilution analytes (IDA) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs, or deuterated analogs of the compounds of interest, and they are fortified into the samples at the time of extraction. This technique allows for the correction for analytical bias encountered when analyzing more chemically complex environmental samples. The isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related

interferences to the same extent as the compounds of concern. Compounds that do not have an identically labeled analog are quantitated by the IDA method using a closely related labeled analog.

2.5. Quantitation by the internal standard method is employed for the IDA analytes/recoveries. Peak response is measured as the area of the peak.

2.6. Samples for the "Total Oxidizable Precursor" assay (TOP) are analyzed in two phases – an aliquot is prepared and analyzed as a normal sample, and a second aliquot is subjected to [redacted] prior to solid phase extraction and analysis. The total perfluorocarboxylic acid value is determined for each aliquot, and the difference calculated.

3. DEFINITIONS

3.1. PFCAs: Perfluorocarboxylic acids

3.2. PFSAs: Perfluorinated sulfonic acids

3.3. FOSA: Perfluorinated sulfonamide

3.4. PFOA: Perfluorooctanoic acid

3.5. PFOS: Perfluorooctane sulfonic acid

3.6. PTFE: Polytetrafluoroethylene (e.g. Teflon®)

3.7. SPE: Solid phase extraction

3.8. PP: Polypropylene

3.9. PE: Polyethylene

3.10. HDPE: High density polyethylene

3.11. AFFF: Aqueous Film Forming Foam

3.12. IDA: Isotope dilution analyte

3.13. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.

4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

4.3. PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFOA contamination.

4.3.1. Standards and samples are injected from polypropylene autosampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

4.3.2. Random evaporation losses have been observed with the polypropylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.

4.3.3. Teflon-lined screw caps have detected PFAS at low concentrations. Repeated injection from the same Teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene screw caps.

4.4. Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

4.5. Both branched and linear PFAS isomers can potentially be found in the environment. Linear and branched isomers are known to exist for PFOS, PFOA, PFHxS, PFBS, Et-FOSAA, and Me-FOSAA based upon the scientific literature. If multiple isomers are present for one of these PFAS they might be adjacent peaks that completely resolve or not, but usually with a deflection point resolved during peak integration. The later of these peaks matches the retention time of its labeled linear analog. In general, earlier peaks are the branched isomers and are not the result of peak splitting.

As of this writing, only PFOS, PFOA, PFHxS, Et-FOSAA and Me-FOSAA are commercially available as technical mixtures. These reference standards of the technical mixtures for these specific PFAS are used to ensure that all appropriate peaks are included during peak integration.

4.6. In an attempt to reduce PFOS bias, it is required that m/z 499>80 transition be used as the quantitation transition.

4.7. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.15 ng/L or 0.0075 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

4.8. Aluminum foil should not be used for this analysis due to the potential interferences from the PFAS used as release agents.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns

5.1.1. Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS and PFAS samples must be handled in the laboratory as hazardous and toxic chemicals.

5.1.2. Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. Eye protection that satisfies ANSI Z87.1 (as per the Eurofins TestAmerica Safety Manual), laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.5. Perfluorocarboxylic acids are acids and are not compatible with strong bases.

5.1.6. The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed, or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.7. Glass containers are not to be used for "tumbling" soil samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material[1]	**Hazards**	**Exposure Limit[2]**	**Signs and Symptoms of Exposure**
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
[redacted] (3-1-0)	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent damage, including blindness. Brief exposure to 5000 PPM can be fatal.
Hexane (3-3-1)	Flammable Irritant	50 ppm PEL	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Hydrochloric Acid (3-0-1)	Corrosive Poison	5 ppm (Ceiling)	Can cause pain and severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause deep ulcerations to skin, permanent eye damage, circulatory failure and swallowing may be fatal.
Methanol (2-3-0)	Flammable Poison Irritant	200 ppm PEL 250 ppm STEL	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Potassium Hydroxide (3-0-1)	Corrosive Poison	2 mg/m^3 (Ceiling)	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
[redacted] (2-0-1-OX)	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Sodium Hydroxide (3-0-1)	Corrosive Poison	2 mg/m^3 (Ceiling)	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6. EQUIPMENT AND SUPPLIES

Due to the ubiquitous nature of PFAS, all disposable equipment (including, but not limited to vials, pipet tips, and SPE manifold parts) that directly contacts a sample or extract is subject to QC checks on a by-lot basis prior to use. At a minimum, the QC checks include either a rinse with DI water or an extraction with basic methanol to mimic the usage encountered during sample preparation. QC check data is kept on file for reference as needed

6.1. 15 mL polypropylene test tubes with polypropylene screw caps.

6.2. 50 mL graduated plastic centrifuge tubes.

6.3. 125 mL HDPE bottles with HDPE screw caps.

6.4. 250 mL HDPE bottles with HDPE screw caps. The average weight of the HDPE bottles with HDPE screw caps are calibrated once per year. The calibration is

performed by weighing 10 bottles with caps and dividing by 10 to get the average weight. The average weight is used in section (11.3.5.1 Step 4).

6.5. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.

6.6. Extract concentrator or nitrogen manifold with water bath heating to 65°C.

6.7. Syringe filter, Millipore Millex-HV 0.45 um, or equivalent. Do not use PTFE type filters.

6.8. 300 µL autosampler vials, polypropylene, with polypropylene screw caps, Waters PN 1860004112, or equivalent.

6.9. SPE columns

6.9.1. [redacted] or equivalent for the TOP assay.

6.9.2. [redacted] or equivalent. This cartridge incorporates a graphitized carbon.

6.10. Graphitized carbon (Envi-Carb™ or equivalent).

6.11. Vacuum manifold for Solid Phase Extraction (SPE).

6.12. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.

6.13. Water bath: Heated with concentric ring cover capable of temperature control (±5°C) up to 95°C. The bath must be used in a fume hood.

6.14. Plastic tub for an ice bath, AKRO-N.S.T. part No. 35-180 or equivalent.

6.15. pH indicator paper, wide range.

6.16. Bottle rotating apparatus for soil extractions.

6.17. Glass fiber filter, Whatman GF/F, catalog number 1825 090 or equivalent. Filters, if used, are QC checked by lot by extraction with basic methanol. The filters must be clean to less than ½ RL before they can be used, and the data kept on file.

6.18. Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) –The instrument described below, or equivalent, may be used for this method. The HPLC is equipped

with a refrigerated autosampler, an injection valve, and a pump capable of variable flow rate. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak Review, version 2.3 or equivalent. The MS/MS is capable of running in the NI-ESI mode at the recommended flow rate with a minimum of 10 scans per peak.

6.18.1. [redacted] LC/MS/MS

This system consists of a Shimadzu HPLC interfaced with a [redacted] Triple Quad MS, or equivalent. The instrument control and data acquisition software is [redacted] version 1.6.3 or equivalent.

6.18.1.1. [redacted] HPLC equipped with [redacted] pumps and one DGU-20 degassing unit or equivalent.

6.18.1.2. [redacted]

6.18.1.3. PFAS Isolator column, [redacted] This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.19. Preventive and routine maintenance is described in the table below

Table 6.19
HPLC/MS/MS Preventative Maintenance

As Needed:	**Daily (When in use)**
Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes. Create all eluents in Reagent module, label eluent containers with TALS label and place 2nd label into maintenance log when put into use.	Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.
Semi-Annually	**Annually**

Table 6.19 HPLC/MS/MS Preventative Maintenance	
Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable	Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1. Acetic acid, glacial

7.1.2. [redacted] This solution has volatile components, thus it should be replaced every 7 days or sooner.

7.1.3. Ammonium hydroxide (NH4OH), 0.3% in methanol: Prepared by diluting 12 mL of ammonium hydroxide into 4L of methanol.

7.1.4. Hexane

7.1.5. Hydrochloric acid (HCl), 2.0 M solution in water

7.1.6. Hydrochloric acid (HCl), concentrated, reagent grade

7.1.7. Methanol

7.1.8. [redacted]

7.1.9. [redacted], reagent grade

7.1.10. Ottawa Sand (blank matrix for solid samples)

7.1.11. Vegetable Oil, Crisco® brand (blank matrix for tissue samples) – replace within one year of opening.

7.1.12. Sodium hydroxide (NaOH), 0.1 N, in water: Prepared by diluting 400 mL of 1N NaOH into 3.6L of water for a total volume of 4 L.

7.1.13. Sodium hydroxide (NaOH), 10 N, reagent grade

7.1.14. Water, Nanopure or Millipore, must be free of interference and target analytes.

7.1.15. Nitrogen, Ultra High Purity, used for the ESI interface, collision cell, and concentration of extracts.

7.1.16. Air, Ultra-Pure, used for vacuum and source gas.

7.1.17. 30:70 methanol:water (v/v), prepared by diluting 30 mL methanol with 70 mL HPLC reagent water or equivalent volume in respect to the ratio.

7.2. Standards

7.2.1. PFAS are purchased as high purity solids (96% or greater) or as certified solutions. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stock material is stored at room temperature or as specified by the manufacturer or vendor.

7.2.1.1. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.15 ng/L or 0.0075 µg/kg of perfluorohexadecanoic acid expected in all samples and blanks.

7.2.2. As of this writing, only PFOS, PFOA, PFHxS, Et-FOSAA and Me-FOSAA are commercially available as technical mixtures. These reference standards of the technical mixtures for these specific PFAS are used to ensure that all appropriate peaks are included during peak integration.

7.2.3. If solid material is used for preparing a standard, stock standard solutions are prepared from the solids and are stored at 0 - 6°C. Stock standard solutions should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.

7.2.4. PFBS, PFHxS, PFHpS, PFOS, PFDS, and many other PFAS are not available in the acid form, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the equation below.

$$Mass_{acid} = Measured\ Mass_{salt} \times MW_{acid} / MW_{salt}$$

Where: MW_{acid} is the molecular weight of PFAA

MW_{salt} is the molecular weight of the purchased salt.

For example, the molecular weight of PFOS is 500.1295 and the molecular weight of NaPFOS is 523.1193. Therefore, the amount of NaPFOS used must be adjusted by a factor of 0.956.

7.2.5. For the primary source calibration solutions, individual solutions for each PFAS (both native and isotopically labelled) are purchased from Wellington Laboratories, or other reputable vendors, and are predominantly at a concentration of 50 ug/mL in basic methanol. In the case of the sulfonic compounds, the concentration is 50ug/mL of the alkali (potassium or sodium) salt. The laboratory uses the concentration of the acid form when determining the concentration of individual sulfonic acids in solution (See Section 7.2.4 above).

7.2.6. While PFAS standards commercially purchased are supplied in glass ampoules, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene or HDPE containers. Vortex all standard solutions prior to removing aliquots.

7.3. PFC IM/LCS (LCS/Matrix PFC Spike Solution), 20 ng/mL

250 ml of a mixed stock solution at a nominal concentration of 0.02 ug/mL in methanol (see note above) is prepared from the individual solutions, using 100 uL of each individual solution. This mixed stock is used as the spiking solution during sample preparation, as well an intermediate for the calibration curve, using the recipe below:

Table 7.3
PFC IM/LCS Solution Recipe
The solutions below are combined and diluted to 250 mL in methanol

Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	PFC IM/LCS Conc. (µg/mL)	Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	PFC IM/LCS Conc. (µg/mL)
PFBA	50	0.1	0.02	EtFOSAA	50	0.1	0.02
PFPeA	50	0.1	0.02	MeFOSAA	50	0.1	0.02
PFHxA	50	0.1	0.02	4:2 FTS	46.7	0.1	0.01868
PFHpA	50	0.1	0.02	6:2 FTS	47.4	0.1	0.01896
PFOA	50	0.1	0.02	8:2 FTS	47.9	0.1	0.01916
PFNA	50	0.1	0.02	10:2 FTS	48.2	0.1	0.01928
PFDA	50	0.1	0.02	HFPO-DA	50	0.1	0.02
PFUdA	50	0.1	0.02	9CI-PF3ONS	46.6	0.1	0.01864
PFDoA	50	0.1	0.02	11CI-PF3OUdS	47.1	0.1	0.01884
PFTrDA	50	0.1	0.02	4,8-dioxa-3H-PFNA (DONA)	47.1	0.1	0.01884

Table 7.3
PFC IM/LCS Solution Recipe
The solutions below are combined and diluted to 250 mL in methanol

Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	PFC IM/LCS Conc. (µg/mL)	Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	PFC IM/LCS Conc. (µg/mL)
PFTeDA	50	0.1	0.02	3:3 FTCA	50	0.1	0.02
PFHxDA	50	0.1	0.02	5:3 FTCA	50	0.1	0.02
PFODA	50	0.1	0.02	7:3 FTCA	50	0.1	0.02
PFBS	44.2	0.1	0.01768	6:2 FTCA	50	0.1	0.02
PFPeS	46.9	0.1	0.01876	8:2 FTCA	50	0.1	0.02
PFHxS	45.5	0.1	0.0182	10:2 FTCA	50	0.1	0.02
PFHpS	47.6	0.1	0.01904	6:2 FTUCA	50	0.1	0.02
PFOS	46.2	0.1	0.01856	8:2 FTUCA	50	0.1	0.02
PFNS	48	0.1	0.0192	10:2 FTUCA	50	0.1	0.02
PFDS	48.2	0.1	0.01928	PFECHS	46.1	0.1	0.01844
PFDoS	48.4	0.1	0.01936	PFPrA	48.5	0.1	0.0194
FOSA	50	0.1	0.02	PFPrS	45.8	0.1	0.01832
Et-FOSA	50	0.1	0.02	PFECA F	50	0.1	0.02
Me-FOSA	50	0.1	0.02	PFECA A	50	0.1	0.02
Et-FOSE	50	0.1	0.02	PFECA B	50	0.1	0.02
Me-FOSE	50	0.1	0.02	PES	44.5	0.1	0.0178

7.4. PFC Expanded Analyte Intermediate (PFC3SP IM)

250 ml of a mixed stock solution at a nominal concentration of 0.5 ug/mL in methanol (see note above) is prepared from the individual solutions, using 50 uL of each individual solution, as denoted in the recipe below. This mixed stock is used as an intermediate for the calibration curve and for the PFC Expanded Analyte LCS (Section 7.4.1).

Table 7.4
PFC Expanded Analyte IM Solution Recipe
The solutions below are combined and diluted to 250 mL in methanol

Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	PFC3SP IM Conc. (µg/mL)	Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	PFC3SP IM Conc. (µg/mL)
Hydro-EVE Acid	1000	0.05	0.5	PFO3OA	1000	0.05	0.5
Hydro-PS Acid	1000	0.05	0.5	PFO4DA	1000	0.05	0.5
MTP	1000	0.05	0.5	PMPA	1000	0.05	0.5
NVHOS	1000	0.05	0.5	PS Acid	1000	0.05	0.5
PEPA	1000	0.05	0.5	R-EVE	1000	0.05	0.5
PFECA G	1000	0.05	0.5	R-PSDCA	1000	0.05	0.5

Table 7.4
PFC Expanded Analyte IM Solution Recipe
The solutions below are combined and diluted to 250 mL in methanol

Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	PFC3SP IM Conc. (µg/mL)	Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	PFC3SP IM Conc. (µg/mL)
PFMOAA	1000	0.05	0.5	TAF (PFO5DoA)	1000	0.05	0.5
PFO2HxA	1000	0.05	0.5	EVE Acid	1000	0.05	0.5

7.4.1. PFC Expanded Analyte IM/LCS Solution (PFC3SP)

The expanded analyte spike solution is made by diluting 10 mL of the Expanded Analyte Intermediate (Section 7.4, above) to 250 mL in methanol:

Table 7.4.1
PFC Expanded Analyte Spiking Solution Composition
(following dilution of 10 mL IM to 250 mL MeOH)

Analyte	PFC3SP (ug/mL)	Analyte	PFC3SP (ug/mL)
Hydro-EVE Acid	0.02	PFO3OA	0.02
Hydro-PS Acid	0.02	PFO4DA	0.02
MTP	0.02	PMPA	0.02
NVHOS	0.02	PS Acid	0.02
PEPA	0.02	R-EVE	0.02
PFECA G	0.02	R-PSDCA	0.02
PFMOAA	0.02	TAF (PFO5DoA)	0.02
PFO2HxA	0.02	EVE Acid	0.02

7.5. PFC Isotope Dilution Analyte Solution (Extracted Internal Standards), 25 ng/mL

The PFC-IDA solution is added to all samples prior to extraction and used as an intermediate solution for preparation of the instrument calibration standards. 200 mL of the solution at a nominal concentration of 0.025 ug/mL (25 ng/mL) is prepared from the individual solutions described in Section 7.2.5. using the recipe below:

Table 7.5
PFC-IDA Recipe
The solutions below are combined and diluted to 200 mL with Methanol.

IDA	Stock Conc. (µg/mL)	Aliquot (mL)	IDA Mix Conc. (µg/mL)	IDA	Stock Conc. (µg/mL)	Aliquot (mL)	IDA Mix Conc. (µg/mL)
13C4-PFBA	50	0.10	0.025	d3-MeFOSAA	50	0.10	0.025
13C5-PFPeA	50	0.10	0.025	M2-4:2FTS	46.7	0.10	0.02335
13C2-PFHxA	50	0.10	0.025	M2-6:2FTS	47.5	0.10	0.02375
13C4-PFHpA	50	0.10	0.025	M2-8:2FTS	47.9	0.10	0.02395

Table 7.5
PFC-IDA Recipe
The solutions below are combined and diluted to 200 mL with Methanol.

IDA	Stock Conc. (µg/mL)	Aliquot (mL)	IDA Mix Conc. (µg/mL)	IDA	Stock Conc. (µg/mL)	Aliquot (mL)	IDA Mix Conc. (µg/mL)
13C4-PFOA	50	0.10	0.025	M2 10:2 FTS	47.36	0.10	0.02368
13C5-PFNA	50	0.10	0.025	d5-EtFOSA	50	0.10	0.025
13C2-PFDA	50	0.10	0.025	d3-MeFOSA	50	0.10	0.025
13C2-PFUdA	50	0.10	0.025	d9-Et-FOSE	50	0.10	0.025
13C2-PFDoA	50	0.10	0.025	d7-Me-FOSE	50	0.10	0.025
18O2-PFHxS	47.3	0.10	0.02365	13C3-HFPO-DA	50	0.10	0.025
13C4-PFOS	47.8	0.10	0.0239	13C-6:2 FTCA	50	0.10	0.025
13C3-PFBS	46.5	0.10	0.02325	13C-8:2 FTCA	50	0.10	0.025
13C2-PFTeDA	50	0.10	0.025	13C-10:2 FTCA	50	0.10	0.025
13C2-PFHxDA	50	0.10	0.025	13C-6:2 FTUCA	50	0.10	0.025
13C8-FOSA	50	0.10	0.025	13C-8:2 FTUCA	50	0.10	0.025
d5-EtFOSAA	50	0.10	0.025	13C-10:2 FTUCA	50	0.10	0.025

7.6. Internal Standard Solution, 25 ng/mL

The PFC_IS solution is added to all extracts prior to analysis and used as an intermediate solution for preparation of the instrument calibration standards. This solution is prepared by diluting 100 uL of a 50 ug/mL solution containing 13C2-PFOA to 200 mL in methanol, for a final concentration of 0.025 ug/L (25 ng/mL).

7.7. Calibration Standards

Calibration solutions are prepared from the standards described in Sections 7.3, 7.4.1, 7.5, and 7.6, above. For each level, a 100 mL volumetric flask is filled with 20 mL of water, and methanol added. The appropriate amount (see table below) of the solutions are added, and then the flask is filled to the mark with methanol to achieve the ratio of 80% methanol to 20% water, v/v.

Table 7.7
Calibration Solution Recipe

PFAS Standards	Volume (mL) to add in 100 mL FV						
	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
PFC IM/LCS (0.02 µg/mL)	0.125	0.25	1.25	5	12.5	25	50
PFC3SP (0.02 ug/mL)	0.125	0.25	1.25	5	12.5	25	50
IDA Mix (0.025µg/mL)	5.0	5.0	5.0	5.0	5.0	5.0	5.0
IS Mix (0.025 µg/mL)	5.0	5.0	5.0	5.0	5.0	5.0	5.0

7.7.1. Initial Calibration (ICAL) Levels (ng/mL)

Table 7.7.1
Initial Calibration Solution Concentrations (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
PFBA	0.025	0.05	0.25	1	2.5	5	10
PFPeA	0.025	0.05	0.25	1	2.5	5	10
PFHxA	0.025	0.05	0.25	1	2.5	5	10
PFHpA	0.025	0.05	0.25	1	2.5	5	10
PFOA	0.025	0.05	0.25	1	2.5	5	10
PFNA	0.025	0.05	0.25	1	2.5	5	10
PFDA	0.025	0.05	0.25	1	2.5	5	10
PFUdA	0.025	0.05	0.25	1	2.5	5	10
PFDoA	0.025	0.05	0.25	1	2.5	5	10
PFTrDA	0.025	0.05	0.25	1	2.5	5	10
PFTeDA	0.025	0.05	0.25	1	2.5	5	10
PFHxDA	0.025	0.05	0.25	1	2.5	5	10
PFODA	0.025	0.05	0.25	1	2.5	5	10
PFBS	0.0221	0.0442	0.221	0.0884	2.21	4.42	0.884
PFPeS	0.02345	0.0469	0.2345	0.0938	2.345	4.69	0.938
PFHxS*	0.02275	0.0455	0.2275	0.91	2.275	4.55	9.1
PFHpS	0.0238	0.0476	0.238	0.952	2.38	4.76	9.52
PFOS*	0.0232	0.0464	0.232	0.928	2.32	4.64	9.28
PFNS	0.024	0.048	0.24	0.96	2.4	4.8	9.6
PFDS	0.0241	0.0482	0.241	0.0964	2.41	4.82	0.964
PFDoS	0.0242	0.0484	0.242	0.0968	2.42	4.84	0.968
FOSA	0.025	0.05	0.25	1	2.5	5	10
Et-FOSA	0.025	0.05	0.25	1	2.5	5	10
Me-FOSA	0.025	0.05	0.25	1	2.5	5	10
Et-FOSE	0.025	0.05	0.25	1	2.5	5	10
Me-FOSE	0.025	0.05	0.25	1	2.5	5	10
EtFOSAA*	0.025	0.05	0.25	1	2.5	5	10
MeFOSAA*	0.025	0.05	0.25	1	2.5	5	10
4:2 FTS	0.02335	0.0467	0.2335	0.934	2.335	4.67	9.34
6:2 FTS	0.0237	0.0474	0.237	0.948	2.37	4.74	9.48
8:2 FTS	0.02395	0.0479	0.2395	0.958	2.395	4.79	9.58
10:2 FTS	0.0241	0.0482	0.241	0.964	2.41	4.82	9.64
HFPO-DA	0.025	0.05	0.25	1	2.5	5	10
9Cl-PF3ONS	0.0233	0.0466	0.233	0.932	2.33	4.66	9.32
11Cl-PF3OUdS	0.02355	0.0471	0.2355	0.0942	2.355	4.71	9.42
DONA	0.02355	0.0471	0.2355	0.0942	2.355	4.71	0.942
Hydro-EVE Acid	0.025	0.05	0.25	1	2.5	5	10
Hydro-PS Acid	0.025	0.05	0.25	1	2.5	5	10
MTP	0.025	0.05	0.25	1	2.5	5	10
NVHOS	0.025	0.05	0.25	1	2.5	5	10
PEPA	0.025	0.05	0.25	1	2.5	5	10
PFECA G	0.025	0.05	0.25	1	2.5	5	10
PFMOAA	0.025	0.05	0.25	1	2.5	5	10
PFO2HxA	0.025	0.05	0.25	1	2.5	5	10
PFO3OA	0.025	0.05	0.25	1	2.5	5	10
PFO4DA	0.025	0.05	0.25	1	2.5	5	10
PMPA	0.025	0.05	0.25	1	2.5	5	10

Table 7.7.1
Initial Calibration Solution Concentrations (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
PS Acid	0.025	0.05	0.25	1	2.5	5	10
EVE Acid	0.025	0.05	0.25	1	2.5	5	10
R-EVE	0.025	0.05	0.25	1	2.5	5	10
R-PSDCA	0.025	0.05	0.25	1	2.5	5	10
TAF	0.025	0.05	0.25	1	2.5	5	10
3:3 FTCA	0.025	0.05	0.25	1	2.5	5	10
5:3 FTCA	0.025	0.05	0.25	1	2.5	5	10
7:3 FTCA	0.025	0.05	0.25	1	2.5	5	10
6:2 FTCA	0.025	0.05	0.25	1	2.5	5	10
8:2 FTCA	0.025	0.05	0.25	1	2.5	5	10
10:2 FTCA	0.025	0.05	0.25	1	2.5	5	10
6:2 FTUCA	0.025	0.05	0.25	1	2.5	5	10
8:2 FTUCA	0.025	0.05	0.25	1	2.5	5	10
10:2 FTUCA	0.025	0.05	0.25	1	2.5	5	10
PFECHS	0.02305	0.0461	0.2305	0.922	2.305	4.61	9.22
PFPrA	0.02425	0.0485	0.2425	0.97	2.425	4.85	9.7
PFPrS	0.0229	0.0458	0.229	0.916	2.29	4.58	9.16
PFECA F	0.025	0.05	0.25	1	2.5	5	10
PFECA A	0.025	0.05	0.25	1	2.5	5	10
PFECA B	0.025	0.05	0.25	1	2.5	5	10
PES	0.0215	0.043	0.215	0.86	2.15	4.3	8.9
Labeled Isotope Dilution Analytes (IDA)							
13C4-PFBA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C5-PFPeA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFHxA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C4-PFHpA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C4-PFOA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C5-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFUdA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
18O2-PFHxS	1.1825	1.1825	1.1825	1.1825	1.1825	1.1825	1.1825
13C4-PFOS	1.195	1.195	1.195	1.195	1.195	1.195	1.195
13C3-PFBS	1.1625	1.1625	1.1625	1.1625	1.1625	1.1625	1.1625
13C2-PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFHxDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C8-FOSA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
d5-EtFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
d3-MeFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
M2-4:2FTS ǂ	1.1675	1.1675	1.1675	1.1675	1.1675	1.1675	1.1675
M2-6:2FTS	1.1875	1.1875	1.1875	1.1875	1.1875	1.1875	1.1875
M2-8:2FTS	1.1975	1.1975	1.1975	1.1975	1.1975	1.1975	1.1975
M2 10:2 FTS	1.184	1.184	1.184	1.184	1.184	1.184	1.184
d5-EtFOSA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
d3-MeFOSA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
d9-Et-FOSE	1.25	1.25	1.25	1.25	1.25	1.25	1.25

Table 7.7.1
Initial Calibration Solution Concentrations (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
d7-Me-FOSE	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C3-HFPO-DA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C-6:2 FTCA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C-8:2 FTCA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C-10:2 FTCA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C-6:2 FTUCA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C-8:2 FTUCA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C-10:2 FTUCA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
Internal Standard (IS)							
13C2-PFOA	1.25	1.25	1.25	1.25	1.25	1.25	1.25

** Both branched and linear isomers are used.*

ǂ - This compound is used as a reverse surrogate for the TOP analysis.

***Note**: Sample extracts are in 80% MeOH/H_2O.*

***Note**: The above calibration limits are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.*

7.7.2. A technical (qualitative) grade PFOA standard which contains both linear and branched isomers is used as a retention time (RT) marker. This is used to integrate the total response for both linear and branched isomers of PFOA in environmental samples while relying on the initial calibration with the linear isomer quantitative standard. This technical (qualitative) grade PFOA standard is analyzed initially, after every initial calibration or when significant changes are made to the HPLC parameters.

7.7.2.1. Attach this document to the ICV from the associated ICAL by scanning the document and associating it to the file as a document type of High Res MS Tune in TALS. Use the following naming convention: "_TFOA_Instrument_Date." Example: _TFOA_A10_15Mar2019.

7.8. Initial Calibration Verification Standard (ICV)

7.8.1. The ICV is prepared from commercially available mixed solutions (the PFC-MXB mixture from Wellington) augmented by individual stock solutions for those components not present in the commercial mixture. When available, individual stock solutions are purchased from a vendor other than Wellington laboratories. If not available, a second lot from Wellington is sourced, and if that is not available, a second laboratory chemist will prepare the intermediate mixed solution for the ICV. Currently, the commercially available mixture contains the following

compounds at the listed concentrations in methanol:

Table 7.8.1
PFC-MXB composition

Analyte	Stock Conc. (µg/mL)	Analyte	Stock Conc. (µg/mL)
PFHxA	2	PFBS	2
PFHpA	2	PFHxS	2
PFOA	2	PFOS	2
PFNA	2	EtFOSAA	2
PFDA	2	MeFOSAA	2
PFUdA	2	HFPO-DA	2
PFDoA	2	9CI-PF3ONS	2
PFTrDA	2	11CI-PF3OUdS	2
PFTeDA	2	4,8-dioxa-3H- PFNA (DONA)	2

7.8.2. ICV-IM: 10 mL of a combined stock for the analytes listed below is created, using the recipe below, and methanol as the final solvent:

Table 7.8.2
ICV-IM Recipe

Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	ICV-IM Conc. (µg/mL)	Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	ICV-IM Conc. (µg/mL)
PFBA	50	0.1	0.5	FOSA	50	0.1	0.5
PFPeA	50	0.1	0.5	Et-FOSA	50	0.1	0.5
PFHxDA	50	0.1	0.5	Me-FOSA	50	0.1	0.5
PFODA	50	0.1	0.5	Et-FOSE	50	0.1	0.5
PFPeS	46.9	0.1	0.469	Me-FOSE	50	0.1	0.5
PFHpS	47.6	0.1	0.476	4:2 FTS	46.7	0.1	0.467
PFNS	48	0.1	0.480	6:2 FTS	47.4	0.1	0.474
PFDS	48.2	0.1	0.482	8:2 FTS	47.9	0.1	0.479
PFDoS	48.4	0.1	0.484	10:2 FTS	48.2	0.1	0.482

7.8.3. ICV-IM2: 10 mL of a combined stock for the analytes listed below is created, using the recipe below, and methanol as the final solvent:

Table 7.8.3
ICV-IM2 Recipe

Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	ICV-IM Conc. (µg/mL)	Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	ICV-IM Conc. (µg/mL)
3:3 FTCA	50	0.1	0.5	10:2 FTUCA	50	0.1	0.5
5:3 FTCA	50	0.1	0.5	PES	44.5	0.1	0.445
7:3 FTCA	50	0.1	0.5	PFECA F	50	0.1	0.5
6:2 FTCA	50	0.1	0.5	PFECA A	50	0.1	0.5
8:2 FTCA	50	0.1	0.5	PFECA B	50	0.1	0.5
10:2 FTCA	50	0.1	0.5	PFECHS	46.1	0.1	0.461
6:2 FTUCA	50	0.1	0.5	PFPrS	45.8	0.1	0.458
8:2 FTUCA	50	0.1	0.5	PFPrA (PPF Acid)	97	0.05	0.458

7.8.4. ICV-IM3: 10 mL of a combined stock for the analytes listed below is created, using the recipe below, and methanol as the final solvent:

Table 7.8.4
ICV-IM3 Recipe

Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	ICV-IM Conc. (µg/mL)	Analyte	Stock Conc. (µg/mL)	Aliquot (mL)	ICV-IM Conc. (µg/mL)
DFSA	1000	0.025	0.05	PFMOAA	1000	0.025	0.05
EVE Acid	1000	0.025	0.05	PFO2HxA	1000	0.025	0.05
Hydro-EVE Acid	1000	0.025	0.05	PFO3OA	1000	0.025	0.05
Hydrolyzed PSDA	1000	0.025	0.05	PFO4DA	1000	0.025	0.05
Hydro-PS Acid	1000	0.025	0.05	PMPA	1000	0.025	0.05
MMF	1000	0.025	0.05	PS Acid	1000	0.025	0.05
MTP	1000	0.025	0.05	R-EVE	1000	0.025	0.05
NVHOS	1000	0.025	0.05	R-PSDA	1000	0.025	0.05
PEPA	1000	0.025	0.05	R-PSDCA	1000	0.025	0.05
PFECA G	1000	0.025	0.05	TAF	1000	0.025	0.05

7.8.5. Finally, the ICV solution is created, at a nominal concentration of 2.5 ng/mL for target analytes (sulfonic acids slightly less), and the same concentrations as the calibration solutions for IS and IDA, by filling a 100 mL flask with 20 mL of water, then adding methanol. After adding the solutions below, the contents are diluted to the mark with methanol:

Table 7.8.5
ICV Recipe

PFAS Standards	Volume (mL) to add in 100 mL FV
Commercial PFAS Mix	0.125
Combined ICV IM Mix (0.5 ug/mL)	0.50
Combined ICV IM2 Mix (0.5 ug/mL)	0.50
Combined ICV IM3 Mix (0.5 ug/mL)	0.50
IDA Mix (0.05 µg/mL)	5.0
IS Mix (0.05 µg/mL)	5.0

7.9. TOP-Surr, 1000 ng/mL: The reverse surrogate solution used for samples subjected to the oxidation process to monitor the efficiency of the oxidation process. This solution is prepared by diluting 2 mL of a 50 ug/mL solution containing M2-4:2 FTS to 100 mL in water, for a final concentration of 1000 ng/mL.

7.10. TOP-IDA, 25ng/mL

The TOP-IDA solution is used for those samples subjected to the oxidation process. It omits M2-4:2 FTS, as that compound is used as a reverse surrogate for demonstrating the efficiency of the oxidation step. 200 mL of the solution at a nominal concentration of 0.025 ug/mL (25 ng/mL) is prepared from the individual solutions described in Section 7.2.5 using the recipe below:

Table 7.10
TOP-IDA Recipe
All solutions below are diluted to 200 mL with Methanol.

IDA	Stock Conc. (µg/mL)	Aliquot (mL) to	IDA Mix Conc. (µg/mL)	IDA	Stock Conc. (µg/mL)	Aliquot (mL) to	IDA Mix Conc. (µg/mL)
13C4-PFBA	50	0.10	0.025	d3-MeFOSAA	50	0.1	0.025
13C5-PFPeA	50	0.10	0.025	M2-6:2FTS	47.5	0.1	0.02375
13C2-PFHxA	50	0.10	0.025	M2-8:2FTS	47.9	0.1	0.02395
13C4-PFHpA	50	0.10	0.025	M2 10:2 FTS	47.36	0.1	0.02368
13C4-PFOA	50	0.10	0.025	d5-EtFOSA	50	0.1	0.025
13C5-PFNA	50	0.10	0.025	d3-MeFOSA	50	0.1	0.025
13C2-PFDA	50	0.10	0.025	d9-Et-FOSE	50	0.1	0.025
13C2-PFUdA	50	0.10	0.025	d7-Me-FOSE	50	0.1	0.025
13C2-PFDoA	50	0.10	0.025	13C3-HFPO-DA	50	0.1	0.025
18O2-PFHxS	47.3	0.10	0.02365	13C-6:2 FTCA	50	0.1	0.025
13C4-PFOS	47.8	0.10	0.0239	13C-8:2 FTCA	50	0.1	0.025
13C3-PFBS	46.5	0.10	0.02325	13C-10:2 FTCA	50	0.1	0.025

13C2-PFTeDA	50	0.10	0.025	13C-6:2 FTUCA	50	0.1	0.025
13C2-PFHxDA	50	0.10	0.025	13C-8:2 FTUCA	50	0.1	0.025
13C8-FOSA	50	0.10	0.025	13C-10:2 FTUCA	50	0.1	0.025
d5-EtFOSAA	50	0.10	0.025				

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Laboratory default requirements for sample containers, sample size, preservation and holding time are detailed in the table below.

Table 8
Sample Collection, Preservation, and Storage Requirements

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]
Water	250 mL HDPE Bottle	250 mL	0-6°C *(if from a known chlorinated source add Trizma (5g/L))*	14 days
Soil/Sediment	4 oz. HDPE wide-mouth container	100 g	0-6°C	14 days
Tissue	4 oz. HDPE wide-mouth container	50 g	≤ -10 °C	1 year (365 days)

Extraction holding time is calculated from date of collection. Analytical holding time is determined from date of extraction.

8.1. Extracts are stored at 0 - 6°C and must be analyzed within 40 days of extraction.

8.2. Many jurisdictions have additional requirements for PFAS analysis, including different holding times and preservation requirements. These are detailed in the document WS-WI-0066, "Agency Specific Criteria for PFAS in Matrices Other Than Drinking Water".

8.3. Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

Note: *As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. Eurofins TestAmerica Sacramento has conducted time stability studies that support a 14 day holding time for aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6°C. The 14/40 day holding times given above are based on the stability study and general EPA convention for the holding time of extractable organic compounds in water and soil.*

8.4. Biphasic samples

8.4.1. Samples denoted as aqueous (groundwaters, surface waters, and waste waters) are prepared and handled as a liquid sample (Section 11.2) regardless of solids content unless otherwise instructed or agreed upon with the client. Detailed descriptions of such deviations from the procedure must be documented in the LIMS NCM program.

8.4.2. Samples considered solids (including biosolids, sediments, and soils) are prepared and handled as solid samples following appropriate homogenization as per Section 11.7. Correction for moisture content is provided through the LIMS when required by the client.

8.4.3. In the event that results are required individually for the solid and aqueous phases of a sample, the phases are separated via centrifugation, and extracted separately using the appropriate preparation (Section 11.2 for the aqueous phase and Section 11.7 for the solid phase). The extracts are analyzed, and results reported for each phase separately.

9. QUALITY CONTROL

Please note: Many states and regulatory programs have their own specifications which differ from the laboratory's default program for QC, calibration, sample preparation and data evaluation. Please refer to WS-WI-0066 for state/client specific programs that have differing criteria from those listed in Sections 9 through 12.

9.1. Initial Demonstration of Capability (IDOC)

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.2. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.

9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/matrix spike duplicate (MS/MSD), a laboratory control sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD, an LCSD may be substituted if batch

precision is required by the program or client. In the event that multiple MS/MSDs are run with a batch due to client requirements, the additional MS/MSDs do not count toward the maximum 20 samples in a batch.

9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. For solid samples, the method blank is an aliquot of Ottawa sand. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, and then implemented when target analytes are detected in the method blank above the reporting limit or when IDA recoveries are outside of the control limits. Re-extraction of the blank, other batch QC and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria.

9.3.1. If the MB produces a peak within the retention time window of any of the analytes, determine the source of the contamination and eliminate the interference before processing samples.

9.3.2. The method blank must not contain any analyte at or above the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.4. Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.

9.3.6. Projects performed under the auspices of the DOD/DOE must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit/LOQ for each analyte, or less than 1/10 of the regulatory limit, or less than 1/10 of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. Reprepare and reanalyze all field and QC samples associated with the contaminated method blank.

9.3.7. The position of the method blank in the SPE manifold during SPE extraction

is rotated across batches.

9.4. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples and Ottawa sand for solids) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits. Re-extraction of the blank, other batch QC, and all associated samples are required if the LCS is deemed unacceptable. See WS-PQA-0003 for specific acceptance criteria. The control limits for the LCS are stored in TALS.

9.5. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a nonconformance memo, and then implemented when recoveries of any spiked analyte are outside of the control limits provided by TALS or by the client.

9.6. A duplicate control sample (LCSD or DCS) may be added when insufficient sample volume is provided to process an MS/MSD pair, or is requested by the client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 for specific acceptance criteria.

9.7. Initial calibration verification (ICV) –A second source standard is analyzed with the initial calibration curve. The concentration should be at the mid-range of the curve.

Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.
- Rerun the initial calibration.

9.8. Isotope Dilution Analytes

9.8.1. The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 11. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.

9.8.2. IDA recoveries are flagged if they are outside of the acceptance limits (25–150%). Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-nose ratio is greater than 10:1.

9.8.2.1. Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.

9.8.2.2. If IDA recovery is >150%, check for laboratory error and correct if identified. If no laboratory error is identified proceed as follows:

Condition:	**Corrective Action:**
Field samples are ND for associated native target analytes.	Report the data with appropriate qualifiers and narrative comments.
Field samples are positive for the associated native target analytes and IDA recovery is <200%	Report the data with appropriate qualifiers and narrative comments.
Field samples are positive for the associated native analytes and IDA recovery >200%	RI at an appropriate dilution then report both sets of data with appropriate qualifiers and narrative comments.

9.8.2.3. If IDA recovery is <25% (10% for Me/Et-FOSE), check laboratory error and correct if identified, if no laboratory error is identified, proceed as follows:

<table>
<tr><th>Condition:</th><th colspan="2">Corrective Action:</th></tr>
<tr><td>Field samples are positive for the associated native target analyte and IDA recovery is >10% (>5% for Me/Et-FOSE)</td><td colspan="2">Report the data with appropriate qualifiers and narrative comments</td></tr>
<tr><td rowspan="3">Field samples are ND for associated native target analytes and IDA recovery is >10% (>%5 for Me/Et-FOSE)</td><td colspan="2">Evaluate the S/N of the associated Native analytes in the most recent RL (CCVL) standard or L1 if an ICAL is ran that day:</td></tr>
<tr><td>S/N X the IDA recovery is >10</td><td>Report the data with appropriate qualifiers and narrative comments</td></tr>
<tr><td>S/N X the IDA recovery is <10</td><td>Report an elevated RL if project DQO allows. Qualify and narrate, otherwise RX or RI at an appropriate dilution</td></tr>
<tr><td colspan="3">Example: If the CCVL has 50 S/N X 0.25 (25% IDA) = 12.5. 12.5 > 10 and RL is supported.

If the CCVL has 50 S/N X 0.15 (15% IDA) = 7.5. 7.5 < 10, therefor the RL must be elevated.

Note: if the RL is to be elevated add a comment into the worksheet about such for the 2nd level reviewer.</td></tr>
</table>

9.8.2.4. Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10:1 or if the IDA recoveries fall below 10% (<5% for Me/Et-FOSE).

9.8.2.4.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

9.8.2.5. For samples analyzed in accordance with version 5.1 or higher of the DoD/DOE QSM, the IDA recovery criteria is 50-150%. If QC or field samples do not meet these criteria then see sections 9.8.2.1, 9.8.2.2, and 9.8.2.3for actions.

9.9. Internal Standard

9.9.1. The Internal Standard (IS) is added to each field and QC samples prior to analysis. The CCV IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

9.9.2. Sample IS response (peak area) must be within ±50% of the response (peak area) in the most recent CCV.

9.9.3. If the IS does not meet criteria, re-analyze the extract, at dilution if needed. If the IS meets criteria in the second analysis, report that analysis. If the IS does not meet criteria in the second analysis, report the first analysis with narration.

9.10. TOP Oxidation Efficiency

9.10.1. If the field sample data indicates incomplete oxidation (i.e. the Post-TOP M2-4:2 FTS recovery is greater than 10% or the Post-TOP precursor concentration is greater than 10% of the Pre-TOP concentration), but the laboratory QA are in control, report the data with narration and/or contact the client to prepare a second billable aliquot (10 mL or a 0.1g equivalent) to be processed.

9.10.2. A reduced sample size may be used initially if sample history or other information indicates the sample is grossly contaminated.

9.11. Ion Ratio

9.11.1. Compare the quantifier/qualifier SRM transition ratio in the sample to the SRM transition ratio in the standard.

Equation 1 $$Ion\ Ratio = \frac{Area\ Quantitation\ Ion\ (1°\ Transition)}{Area\ Qualitative\ Ion\ (2°\ Transition)}$$

9.11.2. The quantifier/qualifier SRM ion ratio should be within ±50% of the average of the quantifier/qualifier SRM ion ratios calculated from the midlevel ICAL point or from the CCV, if an ICAL is not run.

9.11.3. At this time the ion ratio evaluation is a quantitative identification tool. Analyst judgement should be used if the ratio does not meet criteria. Data should be qualified "I" if the ratio is not met.

9.11.4. If the ion ration >2X the target, then do not report the analyte. It should be either ND at the RL or elevate the RL as needed (G flag).

9.11.5. For samples analyzed in accordance with the DoD/DOE QSM version 5.3; if the quantitation ion peak does not meet the maximization criteria the peak shall be included in the summed integration. The result should be flagged "estimated, high bias". As there is not a default qualifier for this in the TALS formatter, use the "see case narrative" flag and NCM the issue.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-P-003 "Calibration Curves and Selection of Calibration Points".

10.2. Routine instrument operating conditions are listed in the table in Section 11.17.

10.3. Instrument Tuning & Mass Calibration

10.3.1. Mass Calibration is performed by instrument manufacturer service representatives in accordance with the manufacturer's procedures during installation, and annually thereafter. Mass calibration is performed such that all precursor and product ions for primary and confirmation transitions are bracketed.

10.3.2. Instrument tuning is done initially when the method is first developed and thereafter as needed during troubleshooting. Tuning is done by infusing each individual compound (native and/or IDA) into the mobile phase using a tee fitting at a point just before the entrance to the electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and updated as needed. The mass assignments must be within ± 0.5 amu of the values shown in the table in Section 11.17.

10.3.3. Once the optimal mass assignments (within ±0.5 amu of true) are made immediately following the initial tune, the lowest level standard from the initial calibration curve is assessed to ensure that a signal to noise ratio greater than 10 to 1 (S/N > 10:1) is achieved for each PFAS analyte. The first level standard from the initial calibration curve is used to evaluate the tune stability on an ongoing basis. The instrument mass windows are set initially at ± 0.5 amu of the true value; therefore, continued detection of the analyte transition with S/N > 10:1 serves as verification that the assigned mass remains within ± 0.5 amu of the true value, which meets the DoD/DOE QSM 5.1 tune criterion. For QSM 5.1 or higher work, the instrument sensitivity check (section 10.12.4) is also evaluated to ensure that the signal to noise criteria is met.

10.3.3.1. For samples run in accordance with the DoD/DOE QSM version 5.3, the instrument must have a valid mass calibration prior to sample analysis. This is verified through the acquisition of a full scan continuum mass spectrum of a PFAS stock standard. All masses must be verified to be within ± 0.5 amu of true value.

10.4. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to, new columns or pump seals. A new calibration is not required after minor maintenance.

10.5. With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Average Response Factor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points.

10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.

10.8. Some jurisdictions may have additional requirements for PFAS in initial and continuing calibrations. These are detailed in the document WS-WI-0066, “Agency Specific Criteria for PFAS in Matrices Other Than Drinking Water”.

10.9. Retention Times

10.9.1. Retention time windows of at least ± 0.25 minutes are set within the data system to facilitate peak identification.

10.9.2. The retention times of the target and reference compounds are initially set in the data system using the mid-range standard from the initial calibration. They are updated using the first CCV on days when an initial calibration is not performed.

10.10. Initial Calibration

10.10.1. A number of analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.

10.10.1.1. A minimum of five analytical standards is used when using average response factor and/or linear calibration fits.

10.10.1.2. A minimum of six analytical standards is used when a quadratic fit is used to generate the curve.

10.10.2. Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.

10.10.2.1. For average response factor (RFa), the relative standard deviation (RSD) for all compounds except those identified as poor performers must be < 30% for the curve to be valid.

10.10.2.2. Poor performing analytes are: 6:2 FTS, PFHxDA and PFODA.

10.10.2.3. For average response factor (RFa), the relative standard deviation (RSD) for poor performing compounds must be < 50% for the curve to be valid.

10.10.2.4. For linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).

10.10.2.5. For quadratic fits, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid.

10.10.2.6. The Internal Standard (IS) response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

10.10.2.7. Criteria for samples analyzed in accordance with QSM 5.3 or higher:

- The %RSD of the RFs for all analytes must be <20%.
- Linear or non-linear calibrations must have r^2>0.99 for each analyte.
- Analytes must be within 70-130% of their true value for each calibration standard.

10.11. Calibration Curve Fits

10.11.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.11.2. The Chrom data system is programmed to complement the calibration evaluation guidelines in policy CA-Q-P-003 by evaluating calibration curve fits in the order listed below. An optimal fit is recommended to the analyst, who may override based on evaluation of the residuals for each calibration level, as per policy CA-Q-P-003.

- Average Response Factor
- Linear, 1/concentration^2 weighting
- Linear, 1/concentration weighting, forced through zero
- Quadratic, 1/concentration^2 weighting

10.11.3. The linear curve uses the following function:

Equation 2

$$y = bx + c$$

Where:

y = $\frac{Area\ (Analyte)}{Area\ (IDA)} \times Concentration(IDA)$

x = concentration

b = slope

c = intercept

10.11.4. The quadratic curve uses the following function:

Equation 3

$$y = ax^2 + bx + c$$

Where y, x, b, and c are the same as above, and a = curvature.

10.11.5. Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.

- The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.
- There should be no carryover at or above 1/2 MRL after a high CAL standard.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

10.11.6. Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points. 1/concentration or 1/x weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.12. Initial Calibration Blank (ICB)

10.12.1. Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 80:20 methanol:water blank containing both IDA and IS.

10.12.2. The result for the calibration blank must be less than the reporting limit.

10.12.3. If the ICB is greater than the reporting limit then the source of contamination must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.12.4. Criteria for samples analyzed in accordance with QSM 5.3 or higher:

- Instrument blanks are required immediately following the highest standard analyzed and *daily prior to sample analysis*.
- The instrument blank must be < ½ the LOQ.

10.13. Initial Calibration Verification (ICV)

10.13.1. Following the ICAL and the ICB, an ICV standard obtained from a different source or vendor than the ICAL standards is analyzed. This ICV standard is a mid-range standard.

10.13.2. The recovery for the ICV must meet the appropriate following criteria:

10.13.2.1. The native analyte must be within or equal to 70-130% for all native analytes. The native analyte must be within or equal to 50-150% for all poor performing analytes. The IDA must be within or equal to 50-150%.

10.13.3. Criteria for samples analyzed in accordance with QSM 5.3 or higher: Analyte concentrations must be within ±30% of their true values for all analytes, IDA and target.

10.13.4. See Section 9.7 for corrective actions in the event that the ICV does not meet the criteria above.

10.14. Continuing Calibration Verification (CCV)

Analyze a CCV at the beginning of a run, the end of a run, and after every 10 samples to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are usually at the mid-level range of the curve and should vary throughout the run from low level (LOQ/RL) to mid-level. The curve and ICV do not need to be run every day. To start an analytical on days when an ICAL is not performed, a CCV and CCVL (low standard at or below the RL and at or above the lowest levels used in the ICAL) are analyzed and if they meet acceptance criteria a run can be started.

10.14.1. The recovery for the CCV standards must be equal to or within 70-130% for all natives and equal to or within 50% to 150% for all poor performers. The recovery for the IDA must be within or equal to 50-150%.

10.14.2. The recovery for the CCVL (low-level CCV at or below the RL) standards must be equal to or within 50-150% for all natives and IDA.

10.14.3. The Internal Standard (IS) response (peak area) must be within ± 50% from the response (peak area) from the midpoint of the initial calibration.

10.14.3.1. Sample IS response (peak area) must be within ± 50% of the response (peak area) in the most recent CCV.

10.14.4. If this is not achieved, the instrument has drifted outside the calibration limits. The instrument must be recalibrated.

10.14.5. Criteria for samples analyzed in accordance with QSM 5.3 or higher:

- All analyte concentrations must be within ± 30% of their true value.
- Additionally, prior to analysis and at least once every 12 hours an instrument sensitivity check (ISC/CCVL) must be analyzed. The analyte

concentrations must be at LOQ and the concentrations must be within ± 30% of their true value. This can be used as a CCV.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a non-conformance memo (NCM). The NCM process is described in more detail in SOP WS-QA-0023. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.2. Water Sample Preparation

11.2.1. Visually inspect samples for the presence of settled and/or suspended sediment/particulates. If present or if the sample is biphasic add IDA prior to any sample decanting or centrifugation. If the sample requires decanting or centrifugation contact the client for guidance prior to such action. Decanting or filtering of the sample can lead to a low bias. Filtration is discouraged.

11.2.2. If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

11.2.2.1. Filters are rinsed with 4 mL of 0.3% NH_4OH/methanol when the bottle is rinsed (Section 11.5.1).

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.2.3. Weigh the sample container prior to extraction and then weigh the sample container after extraction to determine the initial volume. Unless otherwise directed by client, use the entire sample volume, and spike directly into the sample container.

11.2.4. Prepare additional aliquots of a field sample for the MS/MSD, if requested.

11.2.5. Prepare two 250 mL aliquots of HPLC-grade water for the method blank

and LCS.

11.2.6. Vortex the LCS/Matrix PFC Spike and IDA PFC solutions prior to use.

11.2.7. Spike the LCS and MS/MSD (if requested) with 0.5 mL of the LCS/Matrix PFC Spike solution (Section 7.3). This will result in a sample concentration of 40 ng/L.

11.2.8. Add 0.5 mL of the IDA PFC solution (Section 7.4) into each sample and QC sample, for a fixed concentration of 1.25-2.5 ng/mL in the final sample vial. Allow the spikes to equilibrate with the samples for at least 10 minutes before loading onto the SPE cartridge (Section 11.3.5).

11.3. Solid Phase Extraction (SPE) of Aqueous Samples

The automated Zymark Auto-Trace Workstation can be used as long as the program follows these conditions and passes the background check.

11.3.1. Condition the SPE cartridges (Section 6.9.2, [redacted] or equivalent) by passing the following without drying the column.

Note: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.3.2. Wash with 5.0 mL of [redacted]

11.3.3. Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 200 uL remains on top to keep column wet. After this step, the columns cannot go dry until the completion of loading and rinsing samples.

11.3.4. Appropriately label the columns and add the reservoir to the column. Be certain to rotate method blank samples through each sample port on the SPE manifold, such that each new batch uses a different port for the MB.

11.3.5. Add samples to the columns and with vacuum, pull the entire 250 mL aliquot of the sample through the cartridge at a rate of approximately 2 to 5 drops per second.

11.3.5.1. If the SPE column should plug (flow rate <1 drop per minute) prior to the entire content of the sample container passing through the column do the following:

1. Stop adding sample to the reservoir.
2. Return any remaining sample volume back to the original container.
3. Weigh the original container and record this weight into the worksheet notes field within the TALS extraction batch.
4. Determine the full volume of sample fortified by using the "Gross Weight" – (remaining sample volume – default tare weight of a sample container (26.1 g)).
5. Enter this value into the "Initial Amount" field in the TALS extraction batch.
6. Proceed to Section 11.4, noting that additional vacuum or pressure might be needed to elute the SPE column.

11.3.6. After the entire sample has been loaded onto the column, rinse the sample bottle with two 5 mL aliquots of reagent water and pour onto the column reservoir.

11.3.7. After the final loading of the sample but before completely passed through the column, rinse the SPE column with 1 mL of water.

11.3.8. After the sample and water rinse have completely passed through the cartridge, allow the column to dry well with vacuum for 15 minutes.

11.4. SPE Column Wash of Aqueous Samples with 30:70 methanol:water.

11.4.1. Load the first 5 mL of 30:70 methanol:water wash and let soak for five minutes and then elute to waste.

11.4.2. Load the second 5 mL of 30:70 methanol:water wash and elute to waste (without a soaking period).

11.4.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.

11.5. SPE Elution of Aqueous Samples – using 15 mL polypropylene test tubes as receiving tubes in the SPE manifold.

11.5.1. Rinse sample bottles with 4 mL [redacted] and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.5.2. Repeat sample bottle to column reservoir rinse and cartridge elution with a second 4 mL aliquot [redacted] The total collection should be approximately 8 mL.

11.5.3. Proceed to Section 11.6 for final volume.

11.6. Final volume for extract

11.6.1. Vortex the IS solution prior to use.

11.6.2. Add 0.5 mL of IS (Section 7.6) at 25 ng/mL concentration and 2 mL of water to the extract, for a final volume of 10 mL. Verify that the volume 10 mL using the graduations on the tube. This will create an extract with a final solvent composition of 80:20 methanol:water.

11.6.2.1. Seal the test tube tightly. Invert container several times and then vortex. Allow extract to settle for 10 minutes prior to moving to the next step. This permits particulates (SPE resin and/or residual carbon) to settle to the bottom of the tube so that they are not transferred to the autosampler vial.

11.6.3. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.6.4. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps cannot be used due to detection of low level concentration of PFAS.

11.7. Soil, Sediment and Tissue Sample Preparation and Extraction

11.7.1. Visually inspect soil samples. Homogenize the entire sample in accordance with SOP WS-QA-0018. If the sample cannot be mixed in the container, pour into a larger QC'd PFAS-free container and mix thoroughly. Transfer the sample label to the new container.

11.7.2. Weigh a representative 5 g aliquot of sample (1g for tissues) into a 50 mL centrifuge tube. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested.

11.7.3. For the method blank and LCS matrix, use 5 g each of Ottawa sand (solids) or 0.02 g of vegetable oil (tissues).

11.7.4. Vortex the LCS/Matrix PFC Spike and IDA PFC solutions prior to use.

11.7.5. Spike the LCS and MS/MSD (if requested) with 0.5 mL of the LCS/Matrix PFC Spike solution (Section 7.3).

11.7.6. Add 0.5 mL of the IDA PFC solution (Section 7.4) into each sample and QC sample, for a fixed concentration of 1.25 ng/mL in the final sample vial.

11.7.7. Cap the tubes and allow the spike to settle into the sample matrix. Gently shake the tubes to mix the spike into the matrix.

11.7.8. Add 10 mL of [redacted] to each sample. Cap and vortex.

11.7.9. Extract the samples in an ultrasonic water bath for 1 hour.

11.7.10. After the completion of extraction, centrifuge each sample at 3500 rpm for 5 minutes.

11.7.11. Collect and decant the [redacted] into a new container.

11.7.12. Add another 4 mL of [redacted] solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 5 minutes.

11.7.13. Combine the rinsate to the first corresponding containers.

11.7.14. To the final [redacted] extract, bring the volume up to 125 mL with HPLC-grade water.

11.7.15. Neutralize with glacial acetic acid and/or sodium hydroxide, and mix the contents well with vortex mixer. Check the pH to ensure pH is between 6 and 8.

11.8. Solid Extract Cleanup by SPE

Set up SPE cartridges (Section 6.9.2, [redacted] or equivalent) for sample cleanup using vacuum manifold.

11.8.1. Condition the SPE cartridges by passing the following without drying the column.

Note: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.8.2. Wash with 5.0 mL of [redacted]

11.8.3. Wash with 5 mL of 0.1 N NaOH/water. Close valve when ~ 500 µL remains on top of column to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*

11.8.4. Be certain to rotate method blank samples through each sample port on the

SPE manifold, such that each new batch uses a different port for the MB.

11.8.5. Add extracts to the columns and with vacuum, pull the entire extracts through the cartridge at rate of approximately 3 to 5 drops per second.

11.8.6. Rinse the sample tube with 5 mL of water and add to the SPE column.

11.8.7. Dry the columns with vacuum for 15 minutes.

11.9. SPE Column Wash of Solid Extracts with 30:70 methanol:water.

11.9.1. Load the first 5 mL of the 30:70 methanol:water wash to soak for five minutes, and elute to waste.

11.9.2. Load the second 5 mL of the 30:70 methanol:water wash and elute to waste (without a soaking period).

11.9.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.

11.10. SPE Elution of Solid Extracts – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.

11.10.1. Rinse extraction bottles with 4 mL of [redacted] and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.10.2. Repeat extract bottle to column reservoir rinse and cartridge elution with a second 4 mL aliquot of [redacted] The total collection should be approximately 8 mL.

11.10.3. Proceed to Section 11.6 for final volume.

11.11. Product/Waste Samples

Note: Please see WS-WI-0065 for the preparation of methanol extracts and dispersion samples.

Vortex all spike solutions immediately prior to use.

11.11.1. Check the solubility of the material in both methanol and water

11.11.1.1. If the material is soluble in water, dilute 0.5 mL of sample into 250 mL of DI water and proceed to Section 11.3 (follow water extraction procedures). Fortify sample appropriately with IDA or PFC spike solution, see Section 11.2.

11.11.1.2. If the material is soluble in methanol, dilute 1 g (if solid) or 1 mL (if liquid) of material into 10 mL of methanol (MeOH).

- If the material does not completely dissolve, contact your immediate supervisor.

11.11.2. Take 100 µL of the 10 mL solution and dilute it to 10 mL in MeOH.

11.11.3. Take a 1 mL aliquot of this solution (effective dilution of 1000x (1 mg for solid or 0.001 mL for liquid)) and fortify with 0.5 mL of labeled IDA solution (Section 7.4).

11.11.4. Prepare two 1.0 mL/g aliquots of HPLC-grade water for the method blank and LCS.

11.11.5. Spike the LCS and MS/MSD (if requested) with 0.5 mL of the LCS/Matrix PFC Spike solution (Section 7.3).

11.11.6. Add 0.5 mL of the IDA PFC solution (Section 7.4) into each QC sample.

11.11.7. DO NOT PASS EXTRACT THROUGH SPE CARTIRIDGE (omit steps 11.9 – 11.11).

11.11.8. Proceed to Section 11.6 for final volume.

11.12. TOP (Total Oxidizable Precursor) Assay for Aqueous Samples

11.12.1. Prepare 3-250 mL HDPE containers with HPLC grade water to create the needed QC Samples (MB, LCS/LCSD).

11.12.2. Prepare enough 125 mL HDPE containers as needed for all “Pre” and “Post” samples, including QC. Label each appropriately.

11.12.3. Vortex reverse surrogate and LCS spike solutions immediately prior to use.

11.12.4. Spike the “Pre” and “Post” MB 125 mL containers with 25 µL of the reverse surrogate solution of M2-4:2 FTS (Section 7.8).

11.12.5. Spike the “Pre” and “Post” LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution (Section 7.6), and 25 uL of the reverse surrogate solution (Section 7.8).

11.12.6. Add 2g of [redacted] and 1.9 mL of [redacted] to each “Post” sample container.

11.12.7. Subsample 100 mL aliquots of water from each field sample and QC from the 250 mL containers into each of the corresponding 125 mL containers for both the "Pre" and "Post" samples. Spike all "Pre" and "Post" samples with 25uL of the reverse surrogate solution (Section 7.9).

11.12.8. Set aside all "Pre" sample containers.

11.12.9. Cap each "Post" sample container, invert 2-3 times prior to placing container into water bath.

11.12.10. Add 2 g of [redacted] and 1.9 mL of [redacted] to each "Post" sample container.

11.12.11. Heat each "Post" sample container in a water bath (KD) at 85°C for 6 hours.

11.12.12. After digestion for 6 hours, place the "Post" sample containers in an ice bath for 30 minutes.

11.12.13. Adjust the pH of "Post" samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.12.14. Spike both "Pre" and "Post" samples and their associated QC samples with 0.5 mL of TOP IDA solution (Section 7.10). Vortex the IDA solution prior to use.

11.12.15. Proceed to Section 11.13.26 SPE for TOP Assay for both "Pre" and "Post" aliquots.

11.13. TOP (Total Oxidizable Precursor) Assay for Soil Samples

11.13.1. Weigh representative 1 g aliquots of soil for each "Pre" and "Post" sample into a 50 mL centrifuge tube.

11.13.2. For the method blank and LCS matrix, use 1 g each of Ottawa sand for each "Pre" and "Post" QC sample.

11.13.3. Add 20 mL of [redacted] to each sample.

11.13.4. Extract the samples in an ultrasonic water bath for 1 hour.

11.13.5. After the completion of extraction, centrifuge each sample at 3500 rpm for 5 minutes.

11.13.6. Collect and decant the [redacted] extract to a new 50 mL centrifuge tube.

11.13.7. Add another 2 mL of [redacted] solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.13.8. Combine the rinsate to the first corresponding tubes.

11.13.9. Proceed to Section 11.15.2 (Envi-carb clean up), following steps 11.15.2.1. through 11.15.2.4.

11.13.10. To the final [redacted] extract, add 0.5 mL of water to each.

11.13.11. Concentrate the [redacted] extract under nitrogen to less than 0.25 mL.

11.13.12. Dilute extract up to 50 mL with water in the centrifuge tube and vortex.

11.13.13. Prepare enough 125 mL HDPE containers as needed for all “Pre” and “Post” samples, including QC. Label each appropriately.

11.13.14. Vortex reverse surrogate and LCS spike solutions immediately prior to use.

11.13.15. Spike the “Pre” and “Post” MB 125 mL containers with 25 µL of the reverse surrogate solution of M2-4:2 FTS (Section 7.9).

11.13.16. Spike the “Pre” and “Post” LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution and 25 µL of the reverse surrogate solution (Sections 7.3 and 7.9).

11.13.17. Add 2 g of [redacted] and 1.9 mL of [redacted] to each “Post” sample container.

11.13.18. Transfer extract from the centrifuge tube to the appropriate 125 mL container.

11.13.19. Rinse the centrifuge container with an additional 50 mL of water and transfer to the appropriate 125 mL container.

11.13.20. Set aside all “Pre” sample containers.

11.13.21. Cap each “Post” sample container, invert 2-3 times prior to placing container into water bath.

11.13.22. Heat each “Post” sample container in a water bath (KD) at 85°C for 6 hours.

11.13.23. After digestion for 6 hours, place the “Post” sample containers in an ice bath for 30 minutes.

11.13.24. Adjust the pH of both "Pre" and "Post" samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.13.25. Spike both "Pre" and "Post" samples and their associated QC samples with 0.5 mL of TOP IDA solution (Section 7.10). Vortex the IDA solution prior to use.

11.13.26. Proceed to Section 11.13.26 SPE for TOP Assay for both "Pre" and "Post" aliquots.

11.14. SPE Extraction for TOP Assay

Use the following SPE procedure for both "Pre" and "Post" samples for both solid and aqueous samples:

11.14.1. Set up [redacted] SPE columns for sample extraction using a vacuum manifold.

11.14.2. Establish a sample loading flow rate of 3-5 drops per second for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.14.3. Wash/condition the SPE column with 5 mL of [redacted] then 5 mL water.

11.14.4. Load 100 mL of sample onto the SPE cartridge at a flow rate of 3-5 drops per second.

11.14.5. Add 5 mL rinse water

11.14.6. After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.14.7. Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.14.8. Allow the column to dry well using vacuum for 5 minutes. Columns must be dry before continuing.

11.14.9. Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 4 mL of [redacted], and add to the SPE cartridge as eluent.

11.14.10. Repeat with another 4 mL of [redacted].

11.14.11. Collect the eluent and proceed to Section 11.6 for final volume.

11.15. Other Types of Sample Cleanup

11.15.1. Freezing technique to remove lipids.

If samples contain lipids then freeze the methanolic extract and QC extracts at -20^{o}C for at least 1 hour. Collect the solvent layer.

11.15.2. Additional cleanup with graphitized carbon can be applied to those samples with severe matrix impacts that can benefit from an additional treatment of carbon.

11.15.2.1. Add 100 mg of graphitized carbon to each sample extract and QC extracts.

11.15.2.2. Shake vigorously and then let sit for 10 minutes.

11.15.2.3. Centrifuge each sample for 2 minutes at 1000 rpm.

11.15.2.4. Decant the solvent layer.

11.15.2.5. Proceed to Section 11.6.

11.16. AFFF Sample Preparation

11.16.1. QC for AFFF samples consists of a method blank, a laboratory control sample and a sample or matrix duplicate only. No matrix spike or matrix spike duplicate is needed.

11.16.2. Perform a 1,000,000 X serial dilution of the AFFF sample. Dilute 1 mL of AFFF sample to 1 L with laboratory supplied water. Then dilute 1mL of this dilution to 1 L with laboratory supplied water.

11.16.2.1. Be sure to retain all dilutions should the initial analysis warrant re-analysis at higher concentration.

11.16.3. Subsample 2.0 mL of this dilution and fortify with 0.5 mL IDA solution and 0.5 mL of IS (1.25-2.5 ng/mL) solution: then add 7.0 mL of methanol. Vortex the IDA solution prior to use.

11.16.4. Vortex the subsample, then transfer a portion of the sample to a 300 µL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the sample for re-injection or dilution.

11.17. Instrument Analysis

Suggested operating conditions are listed in Tables 1-4 for the SCIEX LCMS systems:

Table 11.17 - 1 **Recommended Instrument Operating Conditions**				
HPLC Conditions ([redacted])				
Column (Column temp = [redacted]°C)	[redacted]			
Mobile Phase Composition	A = [redacted] B = [redacted]			
Gradient Program	**Time**	**%A**	**%B**	**Flow Rate - mL/min**
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	Maximum pressure limit = 5,000 psi			
Injection Size	[redacted] (fixed amount throughout the sequence).			
Run Time	~[redacted]			
Mass Spectrometer Interface Settings ([redacted])				
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.			
Ion Spray Voltage (kV)	[redacted]			
Entrance Potential (V)	[redacted]			
Declustering Potential (V)	[redacted]			
Desolvation Temp	[redacted]°C			
Curtain Gas	[redacted]			
Collision Gas	[redacted]			

Table 11.17 - 2 **Recommended Instrument Operating Conditions**								
Mass Spectrometer Scan Settings ([redacted])								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFPrA	Native Analyte	162.95>119	0.011	[redacted]				
MTP	Native Analyte	175>97	0.011	[redacted]				
PFMOAA	Native Analyte	179>84.9	0.011	[redacted]				
PFBA	Native Analyte	212.9>169	0.011	[redacted]				
13C4_PFBA	IDA	217>172	0.011	[redacted]				
PFECA F	Native Analyte	229>85	0.011	[redacted]				
PMPA	Native Analyte	229>185	0.011	[redacted]				
3:3 FTCA	Native Analyte	241>177.1	0.011	[redacted]				
3:3 FTCA_2	Native Analyte	241>116.9	0.011	[redacted]				
PFO2HxA	Native Analyte	245>85	0.011	[redacted]				
PFPrS	Native Analyte	249.1>80	0.011	[redacted]				

Table 11.17 - 2
Recommended Instrument Operating Conditions
Mass Spectrometer Scan Settings ([redacted])

Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFPeA	Native Analyte	262.9>219	0.011	[redacted]				
13C5_PFPeA	IDA	267.1>223	0.011					
PEPA	Native Analyte	278.9>234.9	0.011					
PFECA A	Native Analyte	278.95>84.9	0.011					
HFPO-DA	Native Analyte	285>169	0.011					
HFPO-DA_2	Native Analyte	285>185	0.011					
13C3_HFPO-DA	IDA	287>169	0.011					
PFECA B	Native Analyte	295.1>201	0.011					
NVHOS	Native Analyte	297>135	0.011					
PFBS	Native Analyte	298.9>80	0.011					
PFBS_2	Native Analyte	298.9>99	0.011					
M3-PFBS	IDA	301.9>80	0.011					
PFO3OA	Native Analyte	311.1>85.2	0.011					
PFHxA	Native Analyte	313>269	0.011					
PFHxA_2	Native Analyte	313>119	0.011					
PES	Native Analyte	314.8>135	0.011					
13C2_PFHxA	IDA	315>270	0.011					
4:2 FTS	Native Analyte	327>307	0.011					
4:2FTS_2	Native Analyte	327>80	0.011					
M2-4:2FTS	IDA	329>81	0.011					
5:3 FTCA	Native Analyte	340.88>236.9	0.011					
5:3 FTCA_2	Native Analyte	340.88>216.9	0.011					
PFPeS	Native Analyte	349>80	0.011					
PFPeS_2	Native Analyte	349>99	0.011					
6:2 FTUCA	Native Analyte	356.86>292.9	0.011					
6:2 FTUCA_2	Native Analyte	356.86>243	0.011					
6:2 FTUCA_3	Native Analyte	356.95>93	0.011					
13C-6:2 FTUCA	IDA	358.86>293.9	0.011					
PFHpA	Native Analyte	363>319	0.011					
PFHpA_2	Native Analyte	363>169	0.011					
13C4_PFHpA	IDA	367>322	0.011					
PFO4DA	Native Analyte	376.9>85	0.011					
DONA	Native Analyte	377>251	0.011					
DONA_2	Native Analyte	377>85	0.011					
6:2 FTCA	Native Analyte	377.1>313.1	0.011					
6:2 FTCA_2	Native Analyte	377.1>63	0.011					
13C-6:2 FTCA	IDA	378.88>293.9	0.011					
PFECA G	Native Analyte	378.9>184.9	0.011					
R-PSDCA	Native Analyte	397>217	0.011					
PFHxS	Native Analyte	399>80	0.011					
PFHxS_2	Native Analyte	399>99	0.011					
18O2_PFHxS	IDA	403>84	0.011					
R-EVE	Native Analyte	405>217	0.011					
PFOA	Native Analyte	413>369	0.011					
PFOA_2	Native Analyte	413>169	0.011					
13C2_PFOA	IDA	415>370	0.011					
13C4_PFOA	IDA	417>372	0.011					

Table 11.17 - 2
Recommended Instrument Operating Conditions

Mass Spectrometer Scan Settings ([redacted])

Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
13C8_PFOA	IDA	421>376	0.011	[redacted]				
6:2 FTS	Native Analyte	427>407	0.011					
Hydro-EVE Acid	Native Analyte	427>282.9	0.011					
6:2 FTS_2	Native Analyte	427>79.96	0.011					
M2-6:2FTS	IDA	429>81	0.011					
7:3 FTCA	Native Analyte	441>337	0.011					
7:3 FTCA_2	Native Analyte	441>317	0.011					
PS Acid	Native Analyte	442.8>146.8	0.011					
PFO5DoA (TAF)	Native Analyte	442.9>85	0.011					
PFHpS	Native Analyte	449>80	0.011					
PFHpS_2	Native Analyte	449>99	0.011					
8:2 FTUCA	Native Analyte	456.86>392.9	0.011					
8:2 FTUCA_2	Native Analyte	456.86>343	0.011					
13C-8:2 FTUCA	IDA	458.86>393.6	0.011					
EVE Acid	Native Analyte	407 > 262.9	0.011					
PFECHS_2	Native Analyte	460.8>98.9	0.011					
PFECHS	Native Analyte	460.8>380.9	0.011					
PFNA	Native Analyte	463>419	0.011					
PFNA_2	Native Analyte	463>169	0.011					
Hydro-PS Acid	Native Analyte	463>263	0.011					
13C5_PFNA	IDA	468>423	0.011					
8:2 FTCA	Native Analyte	477>393.1	0.011					
8:2 FTCA_2	Native Analyte	477>63.2	0.011					
13C-8:2 FTCA	IDA	478.85>393.9	0.011					
PFOSA	Native Analyte	498>78	0.011					
PFOS	Native Analyte	499>80	0.011					
PFOS_2	Native Analyte	499>99	0.011					
13C4_PFOS	IDA	503>80	0.011					
13C8_PFOSA	IDA	506>78	0.011					
13C8_PFOS	IDA	507>99	0.011					
MeFOSA	Native Analyte	512>169	0.011					
MeFOSA_2	Native Analyte	512>218.99	0.011					
PFDA	Native Analyte	513>469	0.011					
PFDA_2	Native Analyte	513>169	0.011					
13C2_PFDA	IDA	515>470	0.011					
d3MeFOSA	IDA	515>169	0.011					
EtFOSA	Native Analyte	526>169	0.011					
EtFOSA_2	Native Analyte	526>218.99	0.011					
8:2 FTS	Native Analyte	527>507	0.011					
8:2 FTS_2	Native Analyte	528.97>79.96	0.011					
M2-8:2FTS	IDA	529>81	0.011					
d5EtFOSA	IDA	531>169	0.011					
9Cl-PF3ONS	Native Analyte	531>351	0.011					
9Cl-PF3ONS_2	Native Analyte	531>79.96	0.011					
PFNS	Native Analyte	549>80	0.011					
PFNS_2	Native Analyte	549>99	0.011					
10:2 FTUCA	Native Analyte	556.86>492.9	0.011					

Table 11.17 - 2
Recommended Instrument Operating Conditions
Mass Spectrometer Scan Settings ([redacted])

Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
10:2 FTUCA_2	Native Analyte	556.97>472.99	0.011	[redacted]				
13C-10:2 FTUCA	IDA	558.86>493.9	0.011					
PFUdA	Native Analyte	563>519	0.011					
PFUdA_2	Native Analyte	563>169	0.011					
13C2_PFUdA	IDA	565>520	0.011					
N-MeFOSAA	Native Analyte	570>419	0.011					
N-MeFOSAA_2	Native Analyte	570>483	0.011					
d3-MeFOSAA	IDA	573>419	0.011					
10:2 FTCA	Native Analyte	576.8>493	0.011					
10:2 FTCA_2	Native Analyte	576.8>63.1	0.011					
13C-10:2 FTCA_3	IDA	578.8>493.9	0.011					
N-EtFOSAA	Native Analyte	584>419	0.011					
N-EtFOSAA_2	Native Analyte	584>526.1	0.011					
d5-EtFOSAA	IDA	589>419	0.011					
PFDS	Native Analyte	599>80	0.011					
PFDS_2	Native Analyte	599>99	0.011					
PFDoA	Native Analyte	613>569	0.011					
PFDoA_2	Native Analyte	613>169	0.011					
13C2_PFDoA	IDA	615>570	0.011					
N-MeFOSE	Native Analyte	616>59	0.011					
d7N-MeFOSE	IDA	623>59	0.011					
10:2 FTS	Native Analyte	627>607	0.011					
10:2 FTS_2	Native Analyte	627>79.96	0.011					
N-EtFOSE	Native Analyte	630>59	0.011					
11Cl-PF3OUdS	Native Analyte	631>451	0.011					
11Cl-PF3OUdS_2	Native Analyte	631>79.96	0.011					
M2-10:2FTS	IDA	634.21>612	0.011					
d9N-EtFOSE	IDA	639>59	0.011					
PFTrDA	Native Analyte	663>619	0.011					
PFTrDA_2	Native Analyte	663>169	0.011					
PFDoS	Native Analyte	699>80	0.011					
PFDoS_2	Native Analyte	699>99	0.011					
PFTeDA	Native Analyte	713>169	0.011					
PFTeDA_2	Native Analyte	713>219	0.011					
13C2_PFTeDA	IDA	715>670	0.011					
PFHxDA	Native Analyte	813>769	0.011					
PFHxDA_2	Native Analyte	813>169	0.011					
13C2_PFHxDA	IDA	815>770	0.011					
PFODA	Native Analyte	913>869	0.011					
PFODA_2	Native Analyte	913>169	0.011					

Table 11.17 – 3
Retention Times & Quantitation

Native Compounds	Typical Native RT (minutes)	IDA analog	Typical IDA RT (minutes)	Quantitation Method
PFPrA	[redacted]	13C4_PFBA	[redacted]	Isotope Dilution
PFMOAA		13C4_PFBA		Isotope Dilution
R-EVE		13C4_PFBA		Isotope Dilution
PFBA		13C4_PFBA		Isotope Dilution
PMPA		13C4_PFBA		Isotope Dilution
PFPrS		13C3-PFBS		Isotope Dilution
NVHOS		13C4_PFBA		Isotope Dilution
PFECA F		13C5_PFPeA		Isotope Dilution
PFO2HxA		13C5_PFPeA		Isotope Dilution
PEPA		13C5_PFPeA		Isotope Dilution
3:3 FTCA		13C3-PFBS		Isotope Dilution
PFPeA		13C5_PFPeA		Isotope Dilution
PFBS		M3-PFBS		Isotope Dilution
PFECA A		13C5_PFPeA		Isotope Dilution
PES		13C3-PFBS		Isotope Dilution
PFECA B		13C2_PFHxA		Isotope Dilution
4:2 FTS		M2-4:2FTS		Isotope Dilution
PFO3OA		13C2_PFHxA		Isotope Dilution
PFHxA		13C2_PFHxA		Isotope Dilution
PFPeS		13C3-PFBS		Isotope Dilution
HFPO-DA		13C3_HFPO-DA		Isotope Dilution
R-PSDCA		13C4_PFHpA		Isotope Dilution
Hydro-EVE Acid		13C4_PFHpA		Isotope Dilution
5:3 FTCA		13C-6:2 FTCA		Isotope Dilution
PFO4DA		13C4_PFHpA		Isotope Dilution
PFECA_G		13C-6:2 FTCA		Isotope Dilution
PFHpA		13C4_PFHpA		Isotope Dilution
PFHxS		18O2_PFHxS		Isotope Dilution
6:2 FTUCA		13C-6:2 FTUCA		Isotope Dilution
6:2 FTCA		13C-6:2 FTCA		Isotope Dilution
DONA		13C4_PFOS		Isotope Dilution
PS Acid		13C4_PFOA		Isotope Dilution
EVE Acid		13C4_PFOA		Isotope Dilution
PFECHS		13C4_PFOA		Isotope Dilution
6:2 FTS		M2-6:2FTS		Isotope Dilution
PFOA		13C4_PFOA		Isotope Dilution
PFHpS		13C4_PFOS		Isotope Dilution
Hydro-PS Acid		13C4_PFHpA		Isotope Dilution
PFO5DoA (TAF		13C4_PFOA		Isotope Dilution
7:3 FTCA		13C-8:2 FTCA		Isotope Dilution

Table 11.17 – 3
Retention Times & Quantitation

Native Compounds	Typical Native RT (minutes)	IDA analog	Typical IDA RT (minutes)	Quantitation Method
8:2 FTUCA	[redacted]	13C-8:2 FTUCA	[redacted]	Isotope Dilution
PFOS		13C4_PFOS		Isotope Dilution
PFNA		13C5_PFNA		Isotope Dilution
8:2 FTCA		13C-8:2 FTCA		Isotope Dilution
9Cl-PF3ONS		13C4_PFOS		Isotope Dilution
PFOSA		13C8_PFOSA		Isotope Dilution
PFNS		13C4_PFOS		Isotope Dilution
PFDA		13C2_PFDA		Isotope Dilution
8:2 FTS		M2-8:2FTS		Isotope Dilution
N-MeFOSAA		d3-MeFOSAA		Isotope Dilution
PFDS		13C4_PFOS		Isotope Dilution
10:2 FTUCA		13C-10:2 FTUCA		Isotope Dilution
10:2 FTCA		13C-10:2 FTCA		Isotope Dilution
PFUdA		13C2_PFUdA		Isotope Dilution
N-EtFOSAA		d5-EtFOSAA		Isotope Dilution
N-MeFOSE		d7N-MeFOSE		Isotope Dilution
MeFOSA		d3MeFOSA		Isotope Dilution
11Cl-PF3OUdS		13C4_PFOS		Isotope Dilution
N-EtFOSE		d9N-EtFOSE		Isotope Dilution
EtFOSA		d5EtFOSA		Isotope Dilution
PFDoA		13C2_PFDoA		Isotope Dilution
10:2 FTS		M2-10:2FTS		Isotope Dilution
PFDoS		13C4_PFOS		Isotope Dilution
PFTrDA		13C2_PFDoA		Isotope Dilution
PFTeDA		13C2_PFTeDA		Isotope Dilution
PFHxDA		13C2_PFHxDA		Isotope Dilution
PFODA		13C2_PFHxDA		Isotope Dilution

11.17.1. Post Spike Sample Analysis for AFFF samples

11.17.1.1. This section only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of <LOQ (RL) for any analyte.

11.17.1.2. Spike aliquots of the sample at the final dilution reported for the sample with all analytes that have reported of <LOQ in the final dilution. The spike must be at the LOQ concentration to be reported with the sample (the < LOQ value).

11.17.1.3. When analyte concentrations are calculated as <LOQ, the spike must recover within 70-130% of its true value.

11.17.1.4. It the recovery does not meet this criteria, the sample, sample duplicate and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.

11.17.2. Tune and calibrate the instrument as described in Section 10.

11.17.3. A typical run sequence is as follows:

- Rinse Blank (RB, not linked to anything)
- Start ICAL with CCVL but called IC in TALS (starts the 12 hour clock or time 0:00)
- Rest of ICAL
- ICB: link to midpoint of ICAL and samples
- ICV: link to midpoint of ICAL and samples (If ICAL good)
- CCB: link to midpoint of ICAL and samples
- PFOA RT marker
- Rinse Blank (RB, not linked to anything)
- 10 samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- 10 more samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- Etc.
- CCVL (within 12 hours from CCVL in ICAL, can be the ending CCV and starts 12 hours all over again): if this occurs link to the midpoint of the ICAL/toggle it as opening/closing CCV.
- CCV: link to midpoint of ICAL
- 10 samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- If no ICAL run that day
- CCB: link to CCVIS
- CCVL (starts 12 hour clock): link to CCVIS
- CCVIS: link to midpoint of ICAL
- 10 samples: link to CCVIS
- CCV: link to CCVIS
- 10 samples: link to CCVIS
- CCV: link to CCVIS
- Etc.

- If going over 12 hours in the sequence: CCVL (within 12 hours from CCVL at item 2 above, can be the ending CCV and starts 12 hours all over again): if this occurs link to the CCVIS /toggle as opening and closing CCV.
- CCV: link to CCVIS
- 10 samples: link to CCVIS
- CCV: link to CCVIS

11.18. Vortex all sample aliquots and standards prior to placing on the autosampler.

11.19. Samples analyzed subsequent to any sample with results at or above the upper calibration limit must be evaluated for potential carryover, and corrective actions taken, as detailed below.

11.19.1. If carryover is suspected, those samples are to be re-analyzed from a fresh extract aliquot (i.e. go the archive of the extract).

11.19.2. Should there be instrument contamination, as evident by sample carryover, any sample >5X the UCL or instrument blanks with detections > RL:

- Analyze 20 blanks alternating between 1% formic acid/methanol and 1% formic acid/water.
- Then analyze 3 methanol only blanks.
- If the system is clean resume analyses. Proceed to 11.19.4. If not clean, proceed as directed below.

11.19.3. If the system is still contaminated the following items might need to be cleaned or replaced:

- Reverse flush the analytical column
- Reverse flush the isolation column
- Replace the column (isolation, analytical or both)
- Clean the cones/entry port
- Replace the PEEK tubing in the sample pathway
- Then, repeat 11.19.2.

11.19.4. Should a high level sample be analyzed that triggers these steps then detections for those analytes over the next 2-3 days require additional evaluation (are all instrument blanks from the sequence < ½ RL) and possible re-analysis. If sample results replicate and the associated instrument blanks from the sequences are <1/2 RL then one can assume the system is under control and confirmation of positive detections can stop.

12. CALCULATIONS / DATA REDUCTION

12.1. If the concentration of the analyte ions exceeds the working range as defined by the calibration standards, then the sample might require to be diluted and reanalyzed, based upon client need. It may be necessary to dilute samples due to matrix.

12.2. Extracts can be diluted up to 100X without diluting out the IDA and thus preserving quantitation via isotope dilution. Dilutions greater than 100X can be performed but additional IDA must be added. The quantitation will now be via internal standard as a result and will have a low bias as extraction losses will no longer be taken into account. Consult the client for authorization of such a dilution.

12.3. Results less than the reporting limit are flagged in the client report as estimated. Generally, the "J" flag is used to denote ≥ MDL and ≤ RL, but the specific flag may change based on client requirements.

12.4. Qualitative Identification

12.4.1. The retention times of PFAS with labeled standards should be the same as that of the labeled IDA's to within 0.1 min. For PFAS with no labeled standards, the RT must be within ± 0.3 minutes of the ICV if analyzed immediately following the ICAL or the most recent CCV standard.

Note: The IDA RT and native RT may be offset by 0.02 to 0.04 minutes.

12.4.1.1. Criteria for samples analyzed in accordance with QSM 5.3: The peak RT must be within 0.4 mins of the CCV or midpoint of the ICAL.

12.4.2. PFBS, PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions specified in the method due to the linear and branch isomers of these compounds. Most PFAS compounds are produced by one of two processes. One gives rise to linear PFAS only while the other process produces both linear and branched isomers. Both branched and linear PFAS compounds can potentially be found in the environment. For the aforementioned compounds that give rise to more than one peak, all chromatographic peaks observed in the standard must be integrated and the areas totaled. Chromatographic peaks in the sample must be integrated in the same way as the calibration standard and concentrations reported as a total for each of these analytes.

12.4.3. The expected retention times (RT) are established in the Chrom data processing module during the processing of the ICAL by selecting Edit>Method>Update RT. Once the retention times are established Chrom

will look for a peak within ± 0.25 minutes of the RT. The analyst confirms that the branched isomers present in the quantitative calibration standards for PFOS, PFHxS, NEtFOSAA and NMeFOSAA are within the ± 0.25 minute window. If they are not, an adjustment to the RT window is made. The analyst confirms the presence of the branched isomers in the technical (qualitative) PFOA standard as well, and adjusts the RT window for PFOA if they are not present within the ± 0.25 minute window.

12.4.3.1. If a peak is detected within this window of ±0.25 minutes, Chrom will assign the absolute retention time at the apex of the peak. Chrom assigns the RT to the most predominant peak within this window. As the linear peak is the predominant peak in calibration solutions for those PFAS that are calibrated with the combination of both branched and linear isomers, those PFAS require additional evaluation in the event that the branched isomer is the predominant peak in a field sample and Chrom has not positively identified the peak due to the RT shift, as the apex may now be the branched isomer.

12.4.3.2. Additional evaluation is required if the field samples contain branched isomers not present in the quantitative or qualitative standards. The analyst confirms that only the peaks present in the calibration standards are included in the peak integration, or adjusts the peak integration to assure that only the peaks present in the standards are identified and quantitated.

12.4.3.3. RT are updated as needed based upon evaluation of the daily CCV.

12.4.4. The signal to noise ratio for both quantitative and qualitative ions/transitions must be ≥ 3:1 for a baseline deflection to be considered a peak. If this criterion is not met, the analyte is not considered and reported as "non-detect".

12.5. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.6. Extract concentrations are calculated as below. The first equation applies Average Response Factor model, the second to a linear fit, and the third to the quadratic line fit.

Equation 4 $$Concentration\ (ng/mL) = \frac{y}{RRF}$$

Equation 5 $$Concentration\ (ng/mL) = \frac{y-c}{b}$$

Equation 6 $$Concentration\ (ng/mL) = \frac{-b \pm \sqrt{b^2 - 4a(c-y)}}{2a}$$

Where:

y = $\frac{Area_{Target}}{Area_{IDA}} \times Concentration(IDA)$

RRF = Relative Response Factor

x = concentration

a = curvature

b = slope

c = intercept

12.7. Water Sample Result Calculation:

Equation 7 $$Concentration\ (ng/L) = \frac{C_{ex}V_t}{V_0}$$

Where:

C_{ex} = Concentration measured in sample extract (ng/mL)

V_t = Volume of total extract (mL)

V_o = Volume of water extracted (L), i.e. total volume fortified with IDA

12.8. Soil Sample Result Calculation:

Equation 8 $$Concentration\ (ng/g) = \frac{C_{ex}V_t}{W_s D}$$

Where ng/g = µg/kg and:

C_{ex} = Concentration measured in sample extract (ng/mL)

V_t = Volume of total extract (mL)

W_s = Weight of sample extracted (g)

D = Fraction of dry solids, which is calculated as follows:

$\frac{100 - \%\ moisture\ in\ sample}{100}$ (for dry weight result)

12.9. IDA Recovery Calculation:

Equation 9 $$\%\ Recovery = \frac{A_{IDA}Q_{IS}}{A_{IS}Q_{IDA}RRF_{IDA}} \times 100$$

Where:

RRF_{IDA} = Response Factor for IDA compound

A_{IDA} = Area response for IDA compound

A_{IS} = Area Response for IS compound

Q_{IS} = Amount of IS added

Q_{IDA} = Amount of IDA added

12.10. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See the Data Review Policy (WS-PQA-0012). These reviews are documented in TALS.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006 and policy WS-PQA-003. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration of Capability (IDOC)

Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

14. POLLUTION PREVENTION

14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.

14.2. Solid phase extraction used for water samples greatly reduces the amount of solvent used compared to liquid-liquid extraction.

14.3. Standards and reagents are purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.4. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

14.5. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

14.6. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out:

15.1. Assorted test tubes, autovials, syringes, filter discs and cartridges. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the hazardous waste – landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Extracted soil samples, used sodium sulfate, paper funnel filters, glass wool, thimbles, and extracted solids saturated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the incineration steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Waste Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When the drum is full to between four and six inches of the top, or after no more than 75 days, move the steel flammable solvent drum to the waste collection area for shipment.

15.4. Mixed water/methanol waste from soil extraction. Collect the waste in the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between four and six inches of the top or after no more than 75 days, move it to the waste collection area for shipment.

15.5. Aqueous acidic waste from the LCMS instrument contaminated with methanol. This is collected in a 1-gallon carboy at the instrument. When the carboy is full, or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between four and six inches of the top or after no more than 75 days, move it to the waste collection area for shipment.

15.6. Autovials contaminated with methanol. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methanol and other contaminants to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES

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16.2. John Giesy et al., "Accumulation of Perfluorooctane Sulfonate in Marine Mammals", Environmental Science & Technology, 2001 Vol. 35, No. 8, pages 1593-1598.

16.3. U.S. EPA, "Residue Chemistry Test Guidelines, OPPTS 860.1340, Residue Analytical Method", EPA 712-C-95-174, August 1995.

16.4. STL Denver White Paper DEN-W-LC-002, "Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, September 5, 2003.

16.5. STL Denver White Paper DEN-W-LC-003, "Addendum A to Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, August 6, 2003.

16.6. STL Denver White Paper DEN-W-LC-004, "Method Validation Study for Analysis of Perfluorooctanoic Acid in Waters by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, January 26, 2005.

16.7. Waters application note; "Acquity UPLC System for Quantifying Trace Levels of Perfluorinated Compounds with an Acquity PFC Analysis Kit", Peter J. Lee, Evan T. Bernier, Gordon T. Fujimoto, Jeremy Shia, Michael S. Young, and Alice J. Di Gloia, Waters Corporation, Milford, MA. USA.

16.8. US EPA, "Method 537 - Determination of Selected Perfluorinated alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometery (LC/MS/MS)", Version 1.1, September 2009, J.A. Shoemaker, P.E. Grimmett, B.K. Boutin, EPA Document #: EPA/600/R-08/092

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16.10. Erika F. Houtz and David L. Sedlak, "Oxidative Conversion as a Means of Detecting Precursors to Perfluoroalkyl Acids in Urban Runoff," Environmental Science and Technology 46, no. 17 (2012): 9342-49.

16.11. U.S. Department of Denfense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1.1, dated 2017.

16.12. U.S. Department of Denfense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 dated 2019.

17. METHOD MODIFICATIONS

17.1. Modifications from Method 537 are detailed below:

17.1.1. Target analyte results are quantitated via isotope dilution.

17.1.2. Two ion transitions (precursor to quant ion and precursor to confirmation ion) are monitored for those analytes that have two transitions. Ion ratios are monitored as well for these analytes.

17.1.3. Water sample containers are not preserved with Trizma.

17.1.4. The method has been modified to address soil/solid matrices. The extraction holding time is set at 14 days.

17.1.5. The analyte list has been expanded. The number of labeled analytes has been expanded as well to improve quantitation.

17.1.6. The reporting limits differ as they are all set at one consistent value whenever possible.

17.1.7. Calibration levels differ from the referenced method.

17.1.8. More labeled analytes are fortified into the samples prior to the extraction process. Most target analytes are quantitated against a labeled analyte.

17.1.9. There is no symmetry requirement.

17.1.10. Calibration, both initial and continuing, has different acceptance criteria due to the longer list of analytes, and the use of isotope dilution quantitation.

17.1.11. The eluents and HPLC configuration differs. As a result the final extract is in 80:20 methanol:water.

17.1.12. The LCS and MS/MSD are spiked at one concentration and do not rotate between a low to high levels.

17.1.13. Samples are not checked for residual chlorine or pH.

17.1.14. A different SPE cartridge [redacted] is used for the extraction process. As a result solvents and elution procedures are different.

18. ATTACHMENTS

There are no attachments to this SOP.

19. REVISION HISTORY

Revisions prior to 9/1/2020 have been removed and are available in previous versions of this SOP.

19.1. Comparison of WS-LC-0025 Revision 3.6 to WS-LC-0025 Revision 4.0, Effective 01/27/2021

19.1.1. This SOP has undergone substantial revision to incorporate many new analytes. The addition of parameters affects Sections 1, 7.4 through 7.9, and 11.16.

19.1.2. Removed all references to Waters LCMS systems – this affects sections 6 and 11.

19.1.3. Removed all references and procedures for concentrating extracts.

19.1.4. All references to the DOD/DOE QSM have been updated to refer to Version 5.3.

19.1.5. The soil preparation has been revised to use a shorter extraction process. Table 1.3, Section 2.2 and Section 11.7 have been updated to reflect these changes.

19.1.6. Agency-specific criteria for calibration, holding times, QC acceptance, etc., have been codified in SOP WS-WI-0066. With the exception of DOD/DOE requirements, they are not included in this document.

19.1.7. Specific instruction for preparation of standard solutions (intermediate and working) has been included in Sections 7.4 through 7.9. This includes information formerly present in WS-LC-0025 Revision 3.6 Attachment 2. The tables have been updated to reflect changed concentration values for specific IDA. The IDA that were at 5x the others are now at the same concentration.

19.1.8. The information present in Attachment 1 of Revision 3.6 (describing an in-

line SPE process for analysis of water samples) has been moved to SOP WS-LC-0025 Att1.

19.1.9. The SPE cartridges (Section 6.9) used for water extraction and solid extract cleanup have been changed. The new cartridges incorporate graphitized carbon, which is now applied to all sample extracts. The solvent systems used with this cartridges have been updated as needed in Section 11.

19.1.10. Columns and SPE cartridges no longer used have been removed from Section 6.

19.1.11. Section 4.5 (discussion of branched and linear isomers) has been revised to include Et-FOSAA and Me-FOSAA.

19.1.12. Section 7.15 (Ammonium Acetate preparation) has been revised to remove the filtration prior to use, and to correct the weight of the salt to 1.54g.

19.1.13. Section 8 (Sample Collection, Preservation, and Holding Times) has been reformatted with a table to clarify containers, preservation, and holding times. Guidance for bi-phasic samples has been incorporated into this section.

19.1.14. Corrective actions for IDA have been added to Section 9.8

19.1.15. Updated the guidance and criteria for the TOP Assay in the event of incomplete oxidation of field samples in Section 9.10.

19.1.16. Guidance and criteria for ion ratios has been added as Section 9.11.

19.1.17. Section 10.3 updated to clarify mass calibration process and frequency for the LCMS.

19.1.18. Section 11.7, Tables 2 and 3 have been combined. The transitions monitored for HFPO-DA and 13C-HFPO-DA have been updated to match those detailed in Method 537.1.

19.1.19. Added guidance in Section 12 regarding the maximum dilution before IDA are diluted out.

19.1.20. Added a note in Section 12 regarding flagging of results less than the reporting limit.

19.1.21. Section 16 has been updated to include reference to QSM version 5.1.1 and 5.3, as well as Method 537.1

19.1.22. Updated Section 17 to include mention of isotope dilution, monitoring two ion transitions, and ion ratios.

19.1.23. Editorial changes

19.2. Comparison of WS-LC-0025 Revision 3.8 to WS-LC-0025 Revision 4.0, Effective 09/30/2020

19.2.1. This SOP has undergone substantial revision to incorporate many new analytes. The addition of parameters affects Sections 1, 7.4 through 7.9, and 11.16.

19.2.2. All references to the DOD/DOE QSM have been updated to refer to Version 5.3.

19.2.3. The soil preparation has been revised to use a different aliquot size, and a shorter extraction process. Table 1.3, Section 2.2 and Section 11.7 have been updated to reflect these changes.

19.2.4. References to agency-specific criteria for calibration, holding times, QC acceptance, as codified in SOP WS-WI-0066 have been added.

19.2.5. The SPE cartridges (Section 6.9) used for water extraction and solid extract cleanup have been changed. The new cartridges incorporate graphitized carbon, which is now applied to all sample extracts. The solvent systems used with this cartridges have been updated as needed in Section 11.

19.2.6. Section 4.5 (discussion of branched and linear isomers) has been revised to include Et-FOSAA and Me-FOSAA.

19.2.7. Section 7.1.2 (Ammonium Acetate preparation) has been revised to remove the filtration prior to use, and to correct the weight of the salt to 1.54g.

19.2.8. Section 7.10 now directs the user to make the TOP reverse surrogate solution in water, rather than Methanol.

19.2.9. Section 8 (Sample Collection, Preservation, and Holding Times) has been reformatted with a table to clarify containers, preservation, and holding times. Guidance for bi-phasic samples has been incorporated into this section.

19.2.10. Expanded the guidance and corrective actions for IDAs in Section 9.8. Also updated the criteria for the FOSE IDA.

19.2.11. Updated the guidance and criteria for the TOP Assay in the event of incomplete oxidation of field samples in Section 9.10.

19.2.12. Updated the guidance and criteria for ion ratios has been added in Section 9.11.

19.2.13. Section 11.7, Tables 2 and 3 have been combined. The transitions monitored for HFPO-DA and 13C-HFPO-DA have been updated to match those detailed in Method 537.1.

19.2.14. Section 12.4 has been expanded to include more detail regarding retention times and qualitative identification.

19.2.15. Editorial changes.

Attachment D

Scope of Work Approval Form

USEPA ID Number: MID 005 356 621
Revision Number **0**
February 2020
Prepared by: **GHD**
Prepared for: **RACER Trust Site**

Approved By: _____ Date: _____
Project Manager - GHD
John-Eric Pardys

Approved By: _____ Date: _____
QA Officer - GHD
Ruth Mickle

Approved By: _____ Date: _____
Deputy Cleanup Manager – RACER
Dave Favero

Approved By: _____ Date: _____
Project Manager – Eurofins TestAmerica North Canton, Ohio
Denise Heckler

Approved By: _____ Date: _____
USEPA Region 5 Project Manager
Michael Beedle

Appendix B
Analytical Reports

eurofins | Environment Testing

ANALYTICAL REPORT

PREPARED FOR

Attn: Ms. Ruth Mickle
GHD Services Inc.
26850 Haggerty Rd.
Farmington Hills, Michigan 48331

Generated 8/15/2023 3:34:26 PM Revision 2

JOB DESCRIPTION

11208041, RACER Nodular

JOB NUMBER

240-188340-1

Eurofins Cleveland
180 S. Van Buren Avenue
Barberton OH 44203

See page two for job notes and contact information.

Job Notes

This report may not be reproduced except in full, and with written approval from the laboratory. The results relate only to the samples tested. For questions please contact the Project Manager at the e-mail address or telephone number listed on this page.

The test results in this report relate only to the samples as received by the laboratory and will meet all requirements of the methodology, with any exceptions noted. This report shall not be reproduced except in full, without the express written approval of the laboratory. All questions should be directed to the Eurofins Environment Testing North Central, LLC Project Manager.

Authorization

Generated
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Revision 2

Authorized for release by
Denise Heckler, Project Manager II
Denise.Heckler@et.eurofinsus.com
(330)966-9477

Table of Contents

Job ID: 240-188340-1

Laboratory: Eurofins Cleveland

Narrative

Job Narrative
240-188340-1

Comments
A revised report was provided on August 3, 2023. At the request of GHD, the project name was changed to RACER Nodular.

A revised report was provided on August 15, 2023. An incorrect comment was removed from the narrative.

Receipt
The samples were received on 7/12/2023 9:45 AM. Unless otherwise noted below, the samples arrived in good condition, and where required, properly preserved and on ice. The temperature of the cooler at receipt was 1.1° C.

GC/MS Semi VOA
No analytical or quality issues were noted, other than those described in the Definitions/Glossary page.

LCMS
Method 537 (modified): The continuing calibration verification (CCV) associated with batch 320-692595 recovered above the upper control limit for 6:2 FTS. The samples associated with this CCV were non-detect for the affected analyte; therefore, the data have been reported. The associated samples are impacted: GW-11208041-071023-BW-001 (240-188340-1), GW-11208041-071023-BW-002 (240-188340-2), GW-11208041-071023-BW-003 (240-188340-3), GW-11208041-071123-BW-004 (240-188340-4), GW-11208041-071123-BW-005 (240-188340-5) and (CCV 320-692595/26).

Method 537 (modified): The laboratory control sample duplicate (LCSD) for preparation batch 320-690595 and analytical batch 320-692595 recovered outside control limits for the following analyte: F-53B Minor. This analyte was biased high in the LCSD and was not detected in the associated samples; therefore, the data have been reported: GW-11208041-071023-BW-001 (240-188340-1), GW-11208041-071023-BW-002 (240-188340-2), GW-11208041-071023-BW-003 (240-188340-3), GW-11208041-071123-BW-004 (240-188340-4), GW-11208041-071123-BW-005 (240-188340-5), GW-11208041-071123-BW-006 (240-188340-6) and (LCSD 320-690595/3-A).

Method 537 (modified): The following sample has chromatographic interferences that could adversely impact the identification and quantitation of target analyte: Perfluoropentanoic acid (PFPeA). This interference could cause false positive results: GW-11208041-071123-BW-004 (240-188340-4) and GW-11208041-071123-BW-005 (240-188340-5).

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

Organic Prep
Method 3535: During the solid phase extraction process, the following sample contained non-settleable particulates which clogged the solid phase extraction column: GW-11208041-071123-BW-005 (240-188340-5).

Method 3535: The following samples in preparation batch 320-690595 were observed to have a thin layer of sediment present in the bottom of the bottle prior to extraction. GW-11208041-071123-BW-004 (240-188340-4) and GW-11208041-071123-BW-005 (240-188340-5)

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

Definitions/Glossary

Client: GHD Services Inc.
Job ID: 240-188340-1
Project/Site: 11208041, RACER Nodular

Qualifiers

GC/MS Semi VOA

Qualifier	Qualifier Description
U	Indicates the analyte was analyzed for but not detected.

LCMS

Qualifier	Qualifier Description
*+	LCS and/or LCSD is outside acceptance limits, high biased.
CI	The peak identified by the data system exhibited chromatographic interference that could not be resolved. There is reason to suspect there may be a high bias.
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.
U	Indicates the analyte was analyzed for but not detected.

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
¤	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CFU	Colony Forming Unit
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MCL	EPA recommended "Maximum Contaminant Level"
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
MPN	Most Probable Number
MQL	Method Quantitation Limit
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
NEG	Negative / Absent
POS	Positive / Present
PQL	Practical Quantitation Limit
PRES	Presumptive
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)
TNTC	Too Numerous To Count

Sample Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Lab Sample ID	Client Sample ID	Matrix	Collected	Received
240-188340-1	GW-11208041-071023-BW-001	Water	07/10/23 14:57	07/12/23 09:45
240-188340-2	GW-11208041-071023-BW-002	Water	07/10/23 15:07	07/12/23 09:45
240-188340-3	GW-11208041-071023-BW-003	Water	07/10/23 15:46	07/12/23 09:45
240-188340-4	GW-11208041-071123-BW-004	Water	07/11/23 09:21	07/12/23 09:45
240-188340-5	GW-11208041-071123-BW-005	Water	07/11/23 10:40	07/12/23 09:45
240-188340-6	GW-11208041-071123-BW-006	Water	07/11/23 12:05	07/12/23 09:45

Detection Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Client Sample ID: GW-11208041-071023-BW-001 — Lab Sample ID: 240-188340-1

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
1,4-Dioxane	3.9		0.20	0.10	ug/L	1		8270D SIM ID	Total/NA
Perfluorobutanoic acid (PFBA)	11		4.8	2.3	ng/L	1		537 (modified)	Total/NA
Perfluorohexanoic acid (PFHxA)	1.6	J	1.9	0.55	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	1.1	J	1.9	0.24	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	5.2		1.9	0.81	ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	0.69	J	1.9	0.19	ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	1.3	J	1.9	0.54	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanesulfonic acid (PFHpS)	0.27	J	1.9	0.18	ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	13		1.9	0.52	ng/L	1		537 (modified)	Total/NA

Client Sample ID: GW-11208041-071023-BW-002 — Lab Sample ID: 240-188340-2

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
1,4-Dioxane	4.1		0.20	0.10	ug/L	1		8270D SIM ID	Total/NA
Perfluorobutanoic acid (PFBA)	10		4.9	2.4	ng/L	1		537 (modified)	Total/NA
Perfluoropentanoic acid (PFPeA)	1.2	J	2.0	0.48	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	1.1	J	2.0	0.25	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	4.9		2.0	0.84	ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	0.84	J	2.0	0.20	ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	1.2	J	2.0	0.56	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanesulfonic acid (PFHpS)	0.28	J	2.0	0.19	ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	13		2.0	0.53	ng/L	1		537 (modified)	Total/NA

Client Sample ID: GW-11208041-071023-BW-003 — Lab Sample ID: 240-188340-3

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
1,4-Dioxane	5.2		0.20	0.10	ug/L	1		8270D SIM ID	Total/NA
Perfluorobutanoic acid (PFBA)	8.7		4.7	2.3	ng/L	1		537 (modified)	Total/NA
Perfluorohexanoic acid (PFHxA)	1.7	J	1.9	0.55	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	1.3	J	1.9	0.24	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	4.8		1.9	0.81	ng/L	1		537 (modified)	Total/NA
Perfluorononanoic acid (PFNA)	0.86	J	1.9	0.26	ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	1.4	J	1.9	0.19	ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	1.4	J	1.9	0.54	ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	8.2		1.9	0.51	ng/L	1		537 (modified)	Total/NA

Client Sample ID: GW-11208041-071123-BW-004 — Lab Sample ID: 240-188340-4

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorobutanoic acid (PFBA)	29		4.8	2.3	ng/L	1		537 (modified)	Total/NA
Perfluoropentanoic acid (PFPeA)	2.7	CI	1.9	0.47	ng/L	1		537 (modified)	Total/NA
Perfluorohexanoic acid (PFHxA)	3.2		1.9	0.56	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	3.4		1.9	0.24	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	5.0		1.9	0.82	ng/L	1		537 (modified)	Total/NA
Perfluorononanoic acid (PFNA)	0.41	J	1.9	0.26	ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	4.3		1.9	0.19	ng/L	1		537 (modified)	Total/NA

Client Sample ID: GW-11208041-071123-BW-005 — Lab Sample ID: 240-188340-5

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
1,4-Dioxane	4.1		0.20	0.10	ug/L	1		8270D SIM ID	Total/NA

This Detection Summary does not include radiochemical test results.

Eurofins Cleveland

Client Sample ID: GW-11208041-071123-BW-005 (Continued)

Lab Sample ID: 240-188340-5

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Perfluorobutanoic acid (PFBA)	26		4.8	2.3	ng/L	1		537 (modified)	Total/NA
Perfluoropentanoic acid (PFPeA)	4.2	CI	1.9	0.47	ng/L	1		537 (modified)	Total/NA
Perfluorohexanoic acid (PFHxA)	4.8		1.9	0.56	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	2.5		1.9	0.24	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	6.0		1.9	0.82	ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	1.2	J	1.9	0.19	ng/L	1		537 (modified)	Total/NA
Perfluoropentanesulfonic acid (PFPeS)	0.63	J	1.9	0.29	ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	1.9		1.9	0.55	ng/L	1		537 (modified)	Total/NA

Client Sample ID: GW-11208041-071123-BW-006

Lab Sample ID: 240-188340-6

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
1,4-Dioxane	0.52		0.21	0.10	ug/L	1		8270D SIM ID	Total/NA
Perfluorobutanoic acid (PFBA)	22		5.1	2.4	ng/L	1		537 (modified)	Total/NA
Perfluorohexanoic acid (PFHxA)	3.2		2.0	0.59	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	2.5		2.0	0.25	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	4.4		2.0	0.86	ng/L	1		537 (modified)	Total/NA
Perfluorononanoic acid (PFNA)	0.60	J	2.0	0.27	ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	3.0		2.0	0.20	ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	1.3	J	2.0	0.58	ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	6.1		2.0	0.55	ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonamide (FOSA)	1.1	J	2.0	1.0	ng/L	1		537 (modified)	Total/NA

This Detection Summary does not include radiochemical test results.

Method Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method	Method Description	Protocol	Laboratory
8270D SIM ID	Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)	SW846	EET BUF
537 (modified)	Fluorinated Alkyl Substances	EPA	EET SAC
3510C	Liquid-Liquid Extraction (Separatory Funnel)	SW846	EET BUF
3535	Solid-Phase Extraction (SPE)	SW846	EET SAC

Protocol References:

EPA = US Environmental Protection Agency

SW846 = "Test Methods For Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 And Its Updates.

Laboratory References:

EET BUF = Eurofins Buffalo, 10 Hazelwood Drive, Amherst, NY 14228-2298, TEL (716)691-2600

EET SAC = Eurofins Sacramento, 880 Riverside Parkway, West Sacramento, CA 95605, TEL (916)373-5600

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-071023-BW-001
Date Collected: 07/10/23 14:57
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-1
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	3.9		0.20	0.10	ug/L		07/14/23 14:31	07/17/23 21:30	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*34*		*15 - 110*	*07/14/23 14:31*	*07/17/23 21:30*	*1*

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-071023-BW-002
Date Collected: 07/10/23 15:07
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-2
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	4.1		0.20	0.10	ug/L		07/14/23 14:31	07/17/23 21:47	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*35*		*15 - 110*	*07/14/23 14:31*	*07/17/23 21:47*	*1*

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-071023-BW-003
Date Collected: 07/10/23 15:46
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-3
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	5.2		0.20	0.10	ug/L		07/14/23 14:31	07/17/23 22:04	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*28*		*15 - 110*	*07/14/23 14:31*	*07/17/23 22:04*	*1*

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-071123-BW-004
Date Collected: 07/11/23 09:21
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-4
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	0.20	U	0.20	0.10	ug/L		07/14/23 14:31	07/17/23 22:21	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*35*		*15 - 110*	*07/14/23 14:31*	*07/17/23 22:21*	*1*

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-071123-BW-005
Date Collected: 07/11/23 10:40
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-5
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	4.1		0.20	0.10	ug/L		07/14/23 14:31	07/17/23 22:38	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*36*		*15 - 110*	*07/14/23 14:31*	*07/17/23 22:38*	*1*

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-071123-BW-006
Date Collected: 07/11/23 12:05
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-6
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	0.52		0.21	0.10	ug/L		07/14/23 14:31	07/17/23 22:55	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*40*		*15 - 110*	*07/14/23 14:31*	*07/17/23 22:55*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-071023-BW-001
Date Collected: 07/10/23 14:57
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-1
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**11**		4.8	2.3	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluoropentanoic acid (PFPeA)	1.9	U	1.9	0.47	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorohexanoic acid (PFHxA)	**1.6**	**J**	1.9	0.55	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluoroheptanoic acid (PFHpA)	**1.1**	**J**	1.9	0.24	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorooctanoic acid (PFOA)	**5.2**		1.9	0.81	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorononanoic acid (PFNA)	1.9	U	1.9	0.26	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.30	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.1	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.53	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.2	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.70	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorobutanesulfonic acid (PFBS)	**0.69**	**J**	1.9	0.19	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	1.9	0.29	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorohexanesulfonic acid (PFHxS)	**1.3**	**J**	1.9	0.54	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluoroheptanesulfonic acid (PFHpS)	**0.27**	**J**	1.9	0.18	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorooctanesulfonic acid (PFOS)	**13**		1.9	0.52	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.35	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.31	ng/L		07/14/23 11:39	07/20/23 01:52	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.94	ng/L		07/14/23 11:39	07/20/23 01:52	1
NMeFOSAA	4.8	U	4.8	1.1	ng/L		07/14/23 11:39	07/20/23 01:52	1
NEtFOSAA	4.8	U	4.8	1.2	ng/L		07/14/23 11:39	07/20/23 01:52	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		07/14/23 11:39	07/20/23 01:52	1
6:2 FTS	4.8	U	4.8	2.4	ng/L		07/14/23 11:39	07/20/23 01:52	1
8:2 FTS	1.9	U	1.9	0.44	ng/L		07/14/23 11:39	07/20/23 01:52	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.38	ng/L		07/14/23 11:39	07/20/23 01:52	1
HFPO-DA (GenX)	3.8	U	3.8	1.4	ng/L		07/14/23 11:39	07/20/23 01:52	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		07/14/23 11:39	07/20/23 01:52	1
11Cl-PF3OUdS	1.9	U *+	1.9	0.31	ng/L		07/14/23 11:39	07/20/23 01:52	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*76*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C5 PFPeA	*103*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C2 PFHxA	*94*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C4 PFHpA	*97*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C4 PFOA	*98*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C5 PFNA	*108*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C2 PFDA	*111*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C2 PFUnA	*99*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C2 PFDoA	*90*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C2 PFTeDA	*76*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C3 PFBS	*118*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
18O2 PFHxS	*96*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C4 PFOS	*101*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
13C8 FOSA	*110*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
d3-NMeFOSAA	*98*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*
d5-NEtFOSAA	*98*		*25 - 150*	*07/14/23 11:39*	*07/20/23 01:52*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-071023-BW-001
Date Collected: 07/10/23 14:57
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-1
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-6:2 FTS	97		25 - 150	07/14/23 11:39	07/20/23 01:52	1
M2-8:2 FTS	109		25 - 150	07/14/23 11:39	07/20/23 01:52	1
M2-4:2 FTS	92		25 - 150	07/14/23 11:39	07/20/23 01:52	1
13C3 HFPO-DA	106		25 - 150	07/14/23 11:39	07/20/23 01:52	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-071023-BW-002
Date Collected: 07/10/23 15:07
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-2
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**10**		4.9	2.4	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluoropentanoic acid (PFPeA)	**1.2**	**J**	2.0	0.48	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorohexanoic acid (PFHxA)	2.0	U	2.0	0.57	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluoroheptanoic acid (PFHpA)	**1.1**	**J**	2.0	0.25	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorooctanoic acid (PFOA)	**4.9**		2.0	0.84	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorononanoic acid (PFNA)	2.0	U	2.0	0.27	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorodecanoic acid (PFDA)	2.0	U	2.0	0.31	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluoroundecanoic acid (PFUnA)	2.0	U	2.0	1.1	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorododecanoic acid (PFDoA)	2.0	U	2.0	0.54	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorotridecanoic acid (PFTrDA)	2.0	U	2.0	1.3	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorotetradecanoic acid (PFTeA)	2.0	U	2.0	0.72	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorobutanesulfonic acid (PFBS)	**0.84**	**J**	2.0	0.20	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluoropentanesulfonic acid (PFPeS)	2.0	U	2.0	0.30	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorohexanesulfonic acid (PFHxS)	**1.2**	**J**	2.0	0.56	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluoroheptanesulfonic acid (PFHpS)	**0.28**	**J**	2.0	0.19	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorooctanesulfonic acid (PFOS)	**13**		2.0	0.53	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorononanesulfonic acid (PFNS)	2.0	U	2.0	0.37	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorodecanesulfonic acid (PFDS)	2.0	U	2.0	0.32	ng/L		07/14/23 11:39	07/20/23 02:03	1
Perfluorooctanesulfonamide (FOSA)	2.0	U	2.0	0.97	ng/L		07/14/23 11:39	07/20/23 02:03	1
NMeFOSAA	4.9	U	4.9	1.2	ng/L		07/14/23 11:39	07/20/23 02:03	1
NEtFOSAA	4.9	U	4.9	1.3	ng/L		07/14/23 11:39	07/20/23 02:03	1
4:2 FTS	2.0	U	2.0	0.24	ng/L		07/14/23 11:39	07/20/23 02:03	1
6:2 FTS	4.9	U	4.9	2.5	ng/L		07/14/23 11:39	07/20/23 02:03	1
8:2 FTS	2.0	U	2.0	0.45	ng/L		07/14/23 11:39	07/20/23 02:03	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	2.0	U	2.0	0.39	ng/L		07/14/23 11:39	07/20/23 02:03	1
HFPO-DA (GenX)	3.9	U	3.9	1.5	ng/L		07/14/23 11:39	07/20/23 02:03	1
9Cl-PF3ONS	2.0	U	2.0	0.24	ng/L		07/14/23 11:39	07/20/23 02:03	1
11Cl-PF3OUdS	2.0	U *+	2.0	0.32	ng/L		07/14/23 11:39	07/20/23 02:03	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*81*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C5 PFPeA	*109*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C2 PFHxA	*100*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C4 PFHpA	*98*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C4 PFOA	*100*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C5 PFNA	*107*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C2 PFDA	*108*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C2 PFUnA	*97*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C2 PFDoA	*98*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C2 PFTeDA	*80*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C3 PFBS	*116*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
18O2 PFHxS	*99*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C4 PFOS	*101*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
13C8 FOSA	*116*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
d3-NMeFOSAA	*95*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*
d5-NEtFOSAA	*100*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:03*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-071023-BW-002
Date Collected: 07/10/23 15:07
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-2
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-6:2 FTS	98		25 - 150	07/14/23 11:39	07/20/23 02:03	1
M2-8:2 FTS	107		25 - 150	07/14/23 11:39	07/20/23 02:03	1
M2-4:2 FTS	96		25 - 150	07/14/23 11:39	07/20/23 02:03	1
13C3 HFPO-DA	106		25 - 150	07/14/23 11:39	07/20/23 02:03	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-071023-BW-003
Date Collected: 07/10/23 15:46
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-3
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**8.7**		4.7	2.3	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluoropentanoic acid (PFPeA)	1.9	U	1.9	0.47	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorohexanoic acid (PFHxA)	**1.7**	**J**	1.9	0.55	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluoroheptanoic acid (PFHpA)	**1.3**	**J**	1.9	0.24	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorooctanoic acid (PFOA)	**4.8**		1.9	0.81	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorononanoic acid (PFNA)	**0.86**	**J**	1.9	0.26	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.29	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.0	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.52	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.2	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.69	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorobutanesulfonic acid (PFBS)	**1.4**	**J**	1.9	0.19	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	1.9	0.28	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorohexanesulfonic acid (PFHxS)	**1.4**	**J**	1.9	0.54	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	1.9	0.18	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorooctanesulfonic acid (PFOS)	**8.2**		1.9	0.51	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.35	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.30	ng/L		07/14/23 11:39	07/20/23 02:15	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.93	ng/L		07/14/23 11:39	07/20/23 02:15	1
NMeFOSAA	4.7	U	4.7	1.1	ng/L		07/14/23 11:39	07/20/23 02:15	1
NEtFOSAA	4.7	U	4.7	1.2	ng/L		07/14/23 11:39	07/20/23 02:15	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		07/14/23 11:39	07/20/23 02:15	1
6:2 FTS	4.7	U	4.7	2.4	ng/L		07/14/23 11:39	07/20/23 02:15	1
8:2 FTS	1.9	U	1.9	0.44	ng/L		07/14/23 11:39	07/20/23 02:15	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.38	ng/L		07/14/23 11:39	07/20/23 02:15	1
HFPO-DA (GenX)	3.8	U	3.8	1.4	ng/L		07/14/23 11:39	07/20/23 02:15	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		07/14/23 11:39	07/20/23 02:15	1
11Cl-PF3OUdS	1.9	U *+	1.9	0.30	ng/L		07/14/23 11:39	07/20/23 02:15	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*31*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C5 PFPeA	*106*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C2 PFHxA	*94*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C4 PFHpA	*95*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C4 PFOA	*102*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C5 PFNA	*106*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C2 PFDA	*116*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C2 PFUnA	*98*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C2 PFDoA	*89*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C2 PFTeDA	*72*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C3 PFBS	*116*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
18O2 PFHxS	*98*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C4 PFOS	*107*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
13C8 FOSA	*114*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
d3-NMeFOSAA	*94*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*
d5-NEtFOSAA	*105*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:15*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-071023-BW-003
Date Collected: 07/10/23 15:46
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-3
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-6:2 FTS	97		25 - 150	07/14/23 11:39	07/20/23 02:15	1
M2-8:2 FTS	107		25 - 150	07/14/23 11:39	07/20/23 02:15	1
M2-4:2 FTS	88		25 - 150	07/14/23 11:39	07/20/23 02:15	1
13C3 HFPO-DA	117		25 - 150	07/14/23 11:39	07/20/23 02:15	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-071123-BW-004
Date Collected: 07/11/23 09:21
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-4
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**29**		4.8	2.3	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluoropentanoic acid (PFPeA)	**2.7**	**CI**	1.9	0.47	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorohexanoic acid (PFHxA)	**3.2**		1.9	0.56	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluoroheptanoic acid (PFHpA)	**3.4**		1.9	0.24	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorooctanoic acid (PFOA)	**5.0**		1.9	0.82	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorononanoic acid (PFNA)	**0.41**	**J**	1.9	0.26	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.30	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.1	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.53	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.3	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.70	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorobutanesulfonic acid (PFBS)	**4.3**		1.9	0.19	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	1.9	0.29	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorohexanesulfonic acid (PFHxS)	1.9	U	1.9	0.55	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	1.9	0.18	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorooctanesulfonic acid (PFOS)	1.9	U	1.9	0.52	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.36	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.31	ng/L		07/14/23 11:39	07/20/23 02:26	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.95	ng/L		07/14/23 11:39	07/20/23 02:26	1
NMeFOSAA	4.8	U	4.8	1.2	ng/L		07/14/23 11:39	07/20/23 02:26	1
NEtFOSAA	4.8	U	4.8	1.3	ng/L		07/14/23 11:39	07/20/23 02:26	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		07/14/23 11:39	07/20/23 02:26	1
6:2 FTS	4.8	U	4.8	2.4	ng/L		07/14/23 11:39	07/20/23 02:26	1
8:2 FTS	1.9	U	1.9	0.44	ng/L		07/14/23 11:39	07/20/23 02:26	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.39	ng/L		07/14/23 11:39	07/20/23 02:26	1
HFPO-DA (GenX)	3.9	U	3.9	1.4	ng/L		07/14/23 11:39	07/20/23 02:26	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		07/14/23 11:39	07/20/23 02:26	1
11Cl-PF3OUdS	1.9	U *+	1.9	0.31	ng/L		07/14/23 11:39	07/20/23 02:26	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*86*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C5 PFPeA	*96*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C2 PFHxA	*95*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C4 PFHpA	*93*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C4 PFOA	*96*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C5 PFNA	*109*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C2 PFDA	*100*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C2 PFUnA	*95*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C2 PFDoA	*90*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C2 PFTeDA	*83*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C3 PFBS	*102*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
18O2 PFHxS	*92*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C4 PFOS	*96*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
13C8 FOSA	*104*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
d3-NMeFOSAA	*88*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
d5-NEtFOSAA	*94*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*
M2-6:2 FTS	*91*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:26*	*1*

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-071123-BW-004
Date Collected: 07/11/23 09:21
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-4
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-8:2 FTS	106		25 - 150	07/14/23 11:39	07/20/23 02:26	1
M2-4:2 FTS	80		25 - 150	07/14/23 11:39	07/20/23 02:26	1
13C3 HFPO-DA	120		25 - 150	07/14/23 11:39	07/20/23 02:26	1

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-071123-BW-005
Date Collected: 07/11/23 10:40
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-5
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**26**		4.8	2.3	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluoropentanoic acid (PFPeA)	**4.2**	**CI**	1.9	0.47	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorohexanoic acid (PFHxA)	**4.8**		1.9	0.56	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluoroheptanoic acid (PFHpA)	**2.5**		1.9	0.24	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorooctanoic acid (PFOA)	**6.0**		1.9	0.82	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorononanoic acid (PFNA)	1.9	U	1.9	0.26	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.30	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.1	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.53	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.3	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.70	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorobutanesulfonic acid (PFBS)	**1.2**	**J**	1.9	0.19	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluoropentanesulfonic acid (PFPeS)	**0.63**	**J**	1.9	0.29	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorohexanesulfonic acid (PFHxS)	**1.9**		1.9	0.55	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	1.9	0.18	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorooctanesulfonic acid (PFOS)	1.9	U	1.9	0.52	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.36	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.31	ng/L		07/14/23 11:39	07/20/23 02:37	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.94	ng/L		07/14/23 11:39	07/20/23 02:37	1
NMeFOSAA	4.8	U	4.8	1.2	ng/L		07/14/23 11:39	07/20/23 02:37	1
NEtFOSAA	4.8	U	4.8	1.3	ng/L		07/14/23 11:39	07/20/23 02:37	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		07/14/23 11:39	07/20/23 02:37	1
6:2 FTS	4.8	U	4.8	2.4	ng/L		07/14/23 11:39	07/20/23 02:37	1
8:2 FTS	1.9	U	1.9	0.44	ng/L		07/14/23 11:39	07/20/23 02:37	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.38	ng/L		07/14/23 11:39	07/20/23 02:37	1
HFPO-DA (GenX)	3.8	U	3.8	1.4	ng/L		07/14/23 11:39	07/20/23 02:37	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		07/14/23 11:39	07/20/23 02:37	1
11Cl-PF3OUdS	1.9	U *+	1.9	0.31	ng/L		07/14/23 11:39	07/20/23 02:37	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*61*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C5 PFPeA	*65*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C2 PFHxA	*67*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C4 PFHpA	*68*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C4 PFOA	*70*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C5 PFNA	*76*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C2 PFDA	*82*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C2 PFUnA	*70*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C2 PFDoA	*65*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C2 PFTeDA	*50*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C3 PFBS	*79*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
18O2 PFHxS	*66*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C4 PFOS	*74*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
13C8 FOSA	*75*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
d3-NMeFOSAA	*63*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*
d5-NEtFOSAA	*68*		*25 - 150*	*07/14/23 11:39*	*07/20/23 02:37*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-071123-BW-005
Date Collected: 07/11/23 10:40
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-5
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-6:2 FTS	66		25 - 150	07/14/23 11:39	07/20/23 02:37	1
M2-8:2 FTS	82		25 - 150	07/14/23 11:39	07/20/23 02:37	1
M2-4:2 FTS	63		25 - 150	07/14/23 11:39	07/20/23 02:37	1
13C3 HFPO-DA	83		25 - 150	07/14/23 11:39	07/20/23 02:37	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-071123-BW-006
Date Collected: 07/11/23 12:05
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-6
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**22**		5.1	2.4	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluoropentanoic acid (PFPeA)	2.0	U	2.0	0.50	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorohexanoic acid (PFHxA)	**3.2**		2.0	0.59	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluoroheptanoic acid (PFHpA)	**2.5**		2.0	0.25	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorooctanoic acid (PFOA)	**4.4**		2.0	0.86	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorononanoic acid (PFNA)	**0.60**	**J**	2.0	0.27	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorodecanoic acid (PFDA)	2.0	U	2.0	0.32	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluoroundecanoic acid (PFUnA)	2.0	U	2.0	1.1	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorododecanoic acid (PFDoA)	2.0	U	2.0	0.56	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorotridecanoic acid (PFTrDA)	2.0	U	2.0	1.3	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorotetradecanoic acid (PFTeA)	2.0	U	2.0	0.74	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorobutanesulfonic acid (PFBS)	**3.0**		2.0	0.20	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluoropentanesulfonic acid (PFPeS)	2.0	U	2.0	0.30	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorohexanesulfonic acid (PFHxS)	**1.3**	**J**	2.0	0.58	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluoroheptanesulfonic acid (PFHpS)	2.0	U	2.0	0.19	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorooctanesulfonic acid (PFOS)	**6.1**		2.0	0.55	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorononanesulfonic acid (PFNS)	2.0	U	2.0	0.38	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorodecanesulfonic acid (PFDS)	2.0	U	2.0	0.33	ng/L		07/14/23 11:39	07/20/23 00:56	1
Perfluorooctanesulfonamide (FOSA)	**1.1**	**J**	2.0	1.0	ng/L		07/14/23 11:39	07/20/23 00:56	1
NMeFOSAA	5.1	U	5.1	1.2	ng/L		07/14/23 11:39	07/20/23 00:56	1
NEtFOSAA	5.1	U	5.1	1.3	ng/L		07/14/23 11:39	07/20/23 00:56	1
4:2 FTS	2.0	U	2.0	0.24	ng/L		07/14/23 11:39	07/20/23 00:56	1
6:2 FTS	5.1	U	5.1	2.5	ng/L		07/14/23 11:39	07/20/23 00:56	1
8:2 FTS	2.0	U	2.0	0.47	ng/L		07/14/23 11:39	07/20/23 00:56	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	2.0	U	2.0	0.41	ng/L		07/14/23 11:39	07/20/23 00:56	1
HFPO-DA (GenX)	4.1	U	4.1	1.5	ng/L		07/14/23 11:39	07/20/23 00:56	1
9Cl-PF3ONS	2.0	U	2.0	0.24	ng/L		07/14/23 11:39	07/20/23 00:56	1
11Cl-PF3OUdS	2.0	U *+	2.0	0.33	ng/L		07/14/23 11:39	07/20/23 00:56	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*26*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C5 PFPeA	*105*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C2 PFHxA	*97*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C4 PFHpA	*97*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C4 PFOA	*97*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C5 PFNA	*108*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C2 PFDA	*110*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C2 PFUnA	*88*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C2 PFDoA	*91*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C2 PFTeDA	*78*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C3 PFBS	*112*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
18O2 PFHxS	*101*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C4 PFOS	*94*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
13C8 FOSA	*112*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*
d3-NMeFOSAA	*83*		*25 - 150*	*07/14/23 11:39*	*07/20/23 00:56*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-071123-BW-006
Date Collected: 07/11/23 12:05
Date Received: 07/12/23 09:45

Lab Sample ID: 240-188340-6
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
d5-NEtFOSAA	90		25 - 150	07/14/23 11:39	07/20/23 00:56	1
M2-6:2 FTS	93		25 - 150	07/14/23 11:39	07/20/23 00:56	1
M2-8:2 FTS	99		25 - 150	07/14/23 11:39	07/20/23 00:56	1
M2-4:2 FTS	92		25 - 150	07/14/23 11:39	07/20/23 00:56	1
13C3 HFPO-DA	111		25 - 150	07/14/23 11:39	07/20/23 00:56	1

QC Association Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

GC/MS Semi VOA

Prep Batch: 676417

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-188340-1	GW-11208041-071023-BW-001	Total/NA	Water	3510C	
240-188340-2	GW-11208041-071023-BW-002	Total/NA	Water	3510C	
240-188340-3	GW-11208041-071023-BW-003	Total/NA	Water	3510C	
240-188340-4	GW-11208041-071123-BW-004	Total/NA	Water	3510C	
240-188340-5	GW-11208041-071123-BW-005	Total/NA	Water	3510C	
240-188340-6	GW-11208041-071123-BW-006	Total/NA	Water	3510C	
MB 480-676417/1-A	Method Blank	Total/NA	Water	3510C	
LCS 480-676417/2-A	Lab Control Sample	Total/NA	Water	3510C	

Analysis Batch: 676522

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-188340-1	GW-11208041-071023-BW-001	Total/NA	Water	8270D SIM ID	676417
240-188340-2	GW-11208041-071023-BW-002	Total/NA	Water	8270D SIM ID	676417
240-188340-3	GW-11208041-071023-BW-003	Total/NA	Water	8270D SIM ID	676417
240-188340-4	GW-11208041-071123-BW-004	Total/NA	Water	8270D SIM ID	676417
240-188340-5	GW-11208041-071123-BW-005	Total/NA	Water	8270D SIM ID	676417
240-188340-6	GW-11208041-071123-BW-006	Total/NA	Water	8270D SIM ID	676417
MB 480-676417/1-A	Method Blank	Total/NA	Water	8270D SIM ID	676417
LCS 480-676417/2-A	Lab Control Sample	Total/NA	Water	8270D SIM ID	676417

LCMS

Prep Batch: 690595

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-188340-1	GW-11208041-071023-BW-001	Total/NA	Water	3535	
240-188340-2	GW-11208041-071023-BW-002	Total/NA	Water	3535	
240-188340-3	GW-11208041-071023-BW-003	Total/NA	Water	3535	
240-188340-4	GW-11208041-071123-BW-004	Total/NA	Water	3535	
240-188340-5	GW-11208041-071123-BW-005	Total/NA	Water	3535	
240-188340-6	GW-11208041-071123-BW-006	Total/NA	Water	3535	
MB 320-690595/1-A	Method Blank	Total/NA	Water	3535	
LCS 320-690595/2-A	Lab Control Sample	Total/NA	Water	3535	
LCSD 320-690595/3-A	Lab Control Sample Dup	Total/NA	Water	3535	

Analysis Batch: 692595

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-188340-1	GW-11208041-071023-BW-001	Total/NA	Water	537 (modified)	690595
240-188340-2	GW-11208041-071023-BW-002	Total/NA	Water	537 (modified)	690595
240-188340-3	GW-11208041-071023-BW-003	Total/NA	Water	537 (modified)	690595
240-188340-4	GW-11208041-071123-BW-004	Total/NA	Water	537 (modified)	690595
240-188340-5	GW-11208041-071123-BW-005	Total/NA	Water	537 (modified)	690595
240-188340-6	GW-11208041-071123-BW-006	Total/NA	Water	537 (modified)	690595
MB 320-690595/1-A	Method Blank	Total/NA	Water	537 (modified)	690595
LCS 320-690595/2-A	Lab Control Sample	Total/NA	Water	537 (modified)	690595
LCSD 320-690595/3-A	Lab Control Sample Dup	Total/NA	Water	537 (modified)	690595

Method: 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Lab Sample ID: MB 480-676417/1-A
Matrix: Water
Analysis Batch: 676522

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 676417

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	0.20	U	0.20	0.10	ug/L		07/14/23 14:31	07/17/23 18:04	1

Isotope Dilution	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*38*		*15 - 110*	*07/14/23 14:31*	*07/17/23 18:04*	*1*

Lab Sample ID: LCS 480-676417/2-A
Matrix: Water
Analysis Batch: 676522

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 676417

Analyte			Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec Limits		
1,4-Dioxane			2.00	2.17		ug/L		108	40 - 140		

Isotope Dilution	*LCS %Recovery*	*LCS Qualifier*	*Limits*
1,4-Dioxane-d8	*36*		*15 - 110*

Method: 537 (modified) - Fluorinated Alkyl Substances

Lab Sample ID: MB 320-690595/1-A
Matrix: Water
Analysis Batch: 692595

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 690595

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	5.0	U	5.0	2.4	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluoropentanoic acid (PFPeA)	2.0	U	2.0	0.49	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorohexanoic acid (PFHxA)	2.0	U	2.0	0.58	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluoroheptanoic acid (PFHpA)	2.0	U	2.0	0.25	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorooctanoic acid (PFOA)	2.0	U	2.0	0.85	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorononanoic acid (PFNA)	2.0	U	2.0	0.27	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorodecanoic acid (PFDA)	2.0	U	2.0	0.31	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluoroundecanoic acid (PFUnA)	2.0	U	2.0	1.1	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorododecanoic acid (PFDoA)	2.0	U	2.0	0.55	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorotridecanoic acid (PFTrDA)	2.0	U	2.0	1.3	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorotetradecanoic acid (PFTeA)	2.0	U	2.0	0.73	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorobutanesulfonic acid (PFBS)	2.0	U	2.0	0.20	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluoropentanesulfonic acid (PFPeS)	2.0	U	2.0	0.30	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorohexanesulfonic acid (PFHxS)	2.0	U	2.0	0.57	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluoroheptanesulfonic acid (PFHpS)	2.0	U	2.0	0.19	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorooctanesulfonic acid (PFOS)	2.0	U	2.0	0.54	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorononanesulfonic acid (PFNS)	2.0	U	2.0	0.37	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorodecanesulfonic acid (PFDS)	2.0	U	2.0	0.32	ng/L		07/14/23 11:39	07/19/23 23:26	1
Perfluorooctanesulfonamide (FOSA)	2.0	U	2.0	0.98	ng/L		07/14/23 11:39	07/19/23 23:26	1
NMeFOSAA	5.0	U	5.0	1.2	ng/L		07/14/23 11:39	07/19/23 23:26	1
NEtFOSAA	5.0	U	5.0	1.3	ng/L		07/14/23 11:39	07/19/23 23:26	1
4:2 FTS	2.0	U	2.0	0.24	ng/L		07/14/23 11:39	07/19/23 23:26	1
6:2 FTS	5.0	U	5.0	2.5	ng/L		07/14/23 11:39	07/19/23 23:26	1
8:2 FTS	2.0	U	2.0	0.46	ng/L		07/14/23 11:39	07/19/23 23:26	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	2.0	U	2.0	0.40	ng/L		07/14/23 11:39	07/19/23 23:26	1

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: MB 320-690595/1-A
Matrix: Water
Analysis Batch: 692595

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 690595

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
HFPO-DA (GenX)	4.0	U	4.0	1.5	ng/L		07/14/23 11:39	07/19/23 23:26	1
9Cl-PF3ONS	2.0	U	2.0	0.24	ng/L		07/14/23 11:39	07/19/23 23:26	1
11Cl-PF3OUdS	2.0	U	2.0	0.32	ng/L		07/14/23 11:39	07/19/23 23:26	1

Isotope Dilution	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*113*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C5 PFPeA	*113*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C2 PFHxA	*107*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C4 PFHpA	*112*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C4 PFOA	*104*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C5 PFNA	*102*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C2 PFDA	*107*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C2 PFUnA	*113*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C2 PFDoA	*105*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C2 PFTeDA	*94*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C3 PFBS	*108*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
18O2 PFHxS	*104*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C4 PFOS	*102*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C8 FOSA	*102*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
d3-NMeFOSAA	*107*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
d5-NEtFOSAA	*120*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
M2-6:2 FTS	*107*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
M2-8:2 FTS	*122*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
M2-4:2 FTS	*117*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*
13C3 HFPO-DA	*100*		*25 - 150*	*07/14/23 11:39*	*07/19/23 23:26*	*1*

Lab Sample ID: LCS 320-690595/2-A
Matrix: Water
Analysis Batch: 692595

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 690595

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec Limits
Perfluorobutanoic acid (PFBA)	40.0	39.4		ng/L		99	76 - 136
Perfluoropentanoic acid (PFPeA)	40.0	40.5		ng/L		101	71 - 131
Perfluorohexanoic acid (PFHxA)	40.0	41.3		ng/L		103	73 - 133
Perfluoroheptanoic acid (PFHpA)	40.0	42.0		ng/L		105	72 - 132
Perfluorooctanoic acid (PFOA)	40.0	43.5		ng/L		109	70 - 130
Perfluorononanoic acid (PFNA)	40.0	43.5		ng/L		109	75 - 135
Perfluorodecanoic acid (PFDA)	40.0	39.5		ng/L		99	76 - 136
Perfluoroundecanoic acid (PFUnA)	40.0	43.1		ng/L		108	68 - 128
Perfluorododecanoic acid (PFDoA)	40.0	40.7		ng/L		102	71 - 131
Perfluorotridecanoic acid (PFTrDA)	40.0	37.6		ng/L		94	71 - 131
Perfluorotetradecanoic acid (PFTeA)	40.0	48.9		ng/L		122	70 - 130
Perfluorobutanesulfonic acid (PFBS)	35.5	37.0		ng/L		104	67 - 127
Perfluoropentanesulfonic acid (PFPeS)	37.6	39.9		ng/L		106	66 - 126

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: LCS 320-690595/2-A
Matrix: Water
Analysis Batch: 692595

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 690595

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec Limits
Perfluorohexanesulfonic acid (PFHxS)	36.5	37.0		ng/L		102	59 - 119
Perfluoroheptanesulfonic acid (PFHpS)	38.2	42.8		ng/L		112	76 - 136
Perfluorooctanesulfonic acid (PFOS)	37.2	41.9		ng/L		113	70 - 130
Perfluorononanesulfonic acid (PFNS)	38.5	42.1		ng/L		109	75 - 135
Perfluorodecanesulfonic acid (PFDS)	38.6	44.7		ng/L		116	71 - 131
Perfluorooctanesulfonamide (FOSA)	40.0	39.6		ng/L		99	73 - 133
NMeFOSAA	40.0	39.5		ng/L		99	76 - 136
NEtFOSAA	40.0	41.5		ng/L		104	76 - 136
4:2 FTS	37.5	38.9		ng/L		104	79 - 139
6:2 FTS	38.1	46.7		ng/L		123	59 - 175
8:2 FTS	38.4	39.0		ng/L		102	75 - 135
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	37.8	45.5		ng/L		120	79 - 139
HFPO-DA (GenX)	40.0	42.9		ng/L		107	51 - 173
9Cl-PF3ONS	37.4	43.2		ng/L		116	75 - 135
11Cl-PF3OUdS	37.8	39.6		ng/L		105	54 - 114

Isotope Dilution	*LCS %Recovery*	*LCS Qualifier*	*Limits*
13C4 PFBA	*117*		*25 - 150*
13C5 PFPeA	*109*		*25 - 150*
13C2 PFHxA	*106*		*25 - 150*
13C4 PFHpA	*105*		*25 - 150*
13C4 PFOA	*104*		*25 - 150*
13C5 PFNA	*102*		*25 - 150*
13C2 PFDA	*113*		*25 - 150*
13C2 PFUnA	*108*		*25 - 150*
13C2 PFDoA	*97*		*25 - 150*
13C2 PFTeDA	*87*		*25 - 150*
13C3 PFBS	*105*		*25 - 150*
18O2 PFHxS	*105*		*25 - 150*
13C4 PFOS	*98*		*25 - 150*
13C8 FOSA	*104*		*25 - 150*
d3-NMeFOSAA	*106*		*25 - 150*
d5-NEtFOSAA	*105*		*25 - 150*
M2-6:2 FTS	*102*		*25 - 150*
M2-8:2 FTS	*107*		*25 - 150*
M2-4:2 FTS	*111*		*25 - 150*
13C3 HFPO-DA	*100*		*25 - 150*

Lab Sample ID: LCSD 320-690595/3-A
Matrix: Water
Analysis Batch: 692595

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 690595

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec Limits	RPD	RPD Limit
Perfluorobutanoic acid (PFBA)	40.0	41.2		ng/L		103	76 - 136	4	30

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: LCSD 320-690595/3-A
Matrix: Water
Analysis Batch: 692595

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 690595

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec Limits	RPD	RPD Limit
Perfluoropentanoic acid (PFPeA)			40.0	44.2		ng/L		111	71 - 131	9	30
Perfluorohexanoic acid (PFHxA)			40.0	44.1		ng/L		110	73 - 133	7	30
Perfluoroheptanoic acid (PFHpA)			40.0	43.4		ng/L		109	72 - 132	3	30
Perfluorooctanoic acid (PFOA)			40.0	44.4		ng/L		111	70 - 130	2	30
Perfluorononanoic acid (PFNA)			40.0	43.1		ng/L		108	75 - 135	1	30
Perfluorodecanoic acid (PFDA)			40.0	43.4		ng/L		109	76 - 136	10	30
Perfluoroundecanoic acid (PFUnA)			40.0	42.7		ng/L		107	68 - 128	1	30
Perfluorododecanoic acid (PFDoA)			40.0	40.6		ng/L		101	71 - 131	0	30
Perfluorotridecanoic acid (PFTrDA)			40.0	44.1		ng/L		110	71 - 131	16	30
Perfluorotetradecanoic acid (PFTeA)			40.0	41.5		ng/L		104	70 - 130	17	30
Perfluorobutanesulfonic acid (PFBS)			35.5	38.5		ng/L		108	67 - 127	4	30
Perfluoropentanesulfonic acid (PFPeS)			37.6	40.1		ng/L		107	66 - 126	0	30
Perfluorohexanesulfonic acid (PFHxS)			36.5	38.1		ng/L		104	59 - 119	3	30
Perfluoroheptanesulfonic acid (PFHpS)			38.2	43.5		ng/L		114	76 - 136	1	30
Perfluorooctanesulfonic acid (PFOS)			37.2	42.6		ng/L		114	70 - 130	2	30
Perfluorononanesulfonic acid (PFNS)			38.5	42.6		ng/L		111	75 - 135	1	30
Perfluorodecanesulfonic acid (PFDS)			38.6	44.4		ng/L		115	71 - 131	1	30
Perfluorooctanesulfonamide (FOSA)			40.0	43.0		ng/L		107	73 - 133	8	30
NMeFOSAA			40.0	42.7		ng/L		107	76 - 136	8	30
NEtFOSAA			40.0	41.0		ng/L		103	76 - 136	1	30
4:2 FTS			37.5	39.9		ng/L		106	79 - 139	2	30
6:2 FTS			38.1	44.1		ng/L		116	59 - 175	6	30
8:2 FTS			38.4	37.9		ng/L		99	75 - 135	3	30
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)			37.8	44.0		ng/L		116	79 - 139	3	30
HFPO-DA (GenX)			40.0	45.2		ng/L		113	51 - 173	5	30
9Cl-PF3ONS			37.4	43.8		ng/L		117	75 - 135	1	30
11Cl-PF3OUdS			37.8	44.2	*+	ng/L		117	54 - 114	11	30

Isotope Dilution	*LCSD %Recovery*	*LCSD Qualifier*	*Limits*
13C4 PFBA	*117*		*25 - 150*
13C5 PFPeA	*102*		*25 - 150*
13C2 PFHxA	*99*		*25 - 150*
13C4 PFHpA	*105*		*25 - 150*
13C4 PFOA	*101*		*25 - 150*
13C5 PFNA	*101*		*25 - 150*
13C2 PFDA	*100*		*25 - 150*
13C2 PFUnA	*105*		*25 - 150*
13C2 PFDoA	*96*		*25 - 150*
13C2 PFTeDA	*89*		*25 - 150*

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: LCSD 320-690595/3-A
Matrix: Water
Analysis Batch: 692595

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 690595

Isotope Dilution	LCSD %Recovery	LCSD Qualifier	Limits
13C3 PFBS	105		25 - 150
18O2 PFHxS	103		25 - 150
13C4 PFOS	98		25 - 150
13C8 FOSA	97		25 - 150
d3-NMeFOSAA	102		25 - 150
d5-NEtFOSAA	102		25 - 150
M2-6:2 FTS	106		25 - 150
M2-8:2 FTS	104		25 - 150
M2-4:2 FTS	108		25 - 150
13C3 HFPO-DA	97		25 - 150

Lab Chronicle

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Client Sample ID: GW-11208041-071023-BW-001
Lab Sample ID: 240-188340-1
Date Collected: 07/10/23 14:57
Matrix: Water
Date Received: 07/12/23 09:45

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			676417	LSC	EET BUF	07/14/23 14:31
Total/NA	Analysis	8270D SIM ID		1	676522	JMM	EET BUF	07/17/23 21:30
Total/NA	Prep	3535			690595	FXY	EET SAC	07/14/23 11:39
Total/NA	Analysis	537 (modified)		1	692595	C1P	EET SAC	07/20/23 01:52

Client Sample ID: GW-11208041-071023-BW-002
Lab Sample ID: 240-188340-2
Date Collected: 07/10/23 15:07
Matrix: Water
Date Received: 07/12/23 09:45

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			676417	LSC	EET BUF	07/14/23 14:31
Total/NA	Analysis	8270D SIM ID		1	676522	JMM	EET BUF	07/17/23 21:47
Total/NA	Prep	3535			690595	FXY	EET SAC	07/14/23 11:39
Total/NA	Analysis	537 (modified)		1	692595	C1P	EET SAC	07/20/23 02:03

Client Sample ID: GW-11208041-071023-BW-003
Lab Sample ID: 240-188340-3
Date Collected: 07/10/23 15:46
Matrix: Water
Date Received: 07/12/23 09:45

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			676417	LSC	EET BUF	07/14/23 14:31
Total/NA	Analysis	8270D SIM ID		1	676522	JMM	EET BUF	07/17/23 22:04
Total/NA	Prep	3535			690595	FXY	EET SAC	07/14/23 11:39
Total/NA	Analysis	537 (modified)		1	692595	C1P	EET SAC	07/20/23 02:15

Client Sample ID: GW-11208041-071123-BW-004
Lab Sample ID: 240-188340-4
Date Collected: 07/11/23 09:21
Matrix: Water
Date Received: 07/12/23 09:45

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			676417	LSC	EET BUF	07/14/23 14:31
Total/NA	Analysis	8270D SIM ID		1	676522	JMM	EET BUF	07/17/23 22:21
Total/NA	Prep	3535			690595	FXY	EET SAC	07/14/23 11:39
Total/NA	Analysis	537 (modified)		1	692595	C1P	EET SAC	07/20/23 02:26

Client Sample ID: GW-11208041-071123-BW-005
Lab Sample ID: 240-188340-5
Date Collected: 07/11/23 10:40
Matrix: Water
Date Received: 07/12/23 09:45

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			676417	LSC	EET BUF	07/14/23 14:31
Total/NA	Analysis	8270D SIM ID		1	676522	JMM	EET BUF	07/17/23 22:38
Total/NA	Prep	3535			690595	FXY	EET SAC	07/14/23 11:39
Total/NA	Analysis	537 (modified)		1	692595	C1P	EET SAC	07/20/23 02:37

Lab Chronicle

Client: GHD Services Inc.
Job ID: 240-188340-1
Project/Site: 11208041, RACER Nodular

Client Sample ID: GW-11208041-071123-BW-006
Lab Sample ID: 240-188340-6

Date Collected: 07/11/23 12:05
Matrix: Water
Date Received: 07/12/23 09:45

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			676417	LSC	EET BUF	07/14/23 14:31
Total/NA	Analysis	8270D SIM ID		1	676522	JMM	EET BUF	07/17/23 22:55
Total/NA	Prep	3535			690595	FXY	EET SAC	07/14/23 11:39
Total/NA	Analysis	537 (modified)		1	692595	C1P	EET SAC	07/20/23 00:56

Laboratory References:

EET BUF = Eurofins Buffalo, 10 Hazelwood Drive, Amherst, NY 14228-2298, TEL (716)691-2600

EET SAC = Eurofins Sacramento, 880 Riverside Parkway, West Sacramento, CA 95605, TEL (916)373-5600

Accreditation/Certification Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Laboratory: Eurofins Buffalo

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Arkansas DEQ	State	88-0686	07-06-23 *
Connecticut	State	PH-0568	03-31-24
Florida	NELAP	E87672	06-30-23 *
Georgia	State	10026 (NY)	03-31-24
Georgia	State Program	N/A	03-31-09 *
Illinois	NELAP	200003	09-30-23
Iowa	State	374	03-01-23 *
Iowa	State Program	374	03-01-09 *
Kansas	NELAP	E-10187	02-01-24
Kentucky (DW)	State	90029	01-01-24
Kentucky (UST)	State	30	04-01-23 *
Kentucky (WW)	State	KY90029	12-31-23
Louisiana	NELAP	02031	06-30-23 *
Louisiana (All)	NELAP	02031	06-30-23 *
Maine	State	NY00044	12-04-24
Maryland	State	294	06-30-24
Massachusetts	State	M-NY044	07-01-24
Michigan	State	9937	04-01-24
Michigan	State Program	9937	04-01-09 *
New Hampshire	NELAP	2973	09-11-19 *
New Hampshire	NELAP	2337	11-17-23
New Jersey	NELAP	NY455	06-30-24
New York	NELAP	10026	03-31-24
Pennsylvania	NELAP	68-00281	07-31-23
Rhode Island	State	LAO00328	12-30-23
Texas	NELAP	T104704412-18-10	07-31-23
USDA	US Federal Programs	P330-18-00039	03-25-24
Virginia	NELAP	460185	09-14-23
Washington	State	C784	02-10-23 *
Wisconsin	State	998310390	08-31-23

Laboratory: Eurofins Sacramento

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Alaska (UST)	State	17-020	02-20-24
ANAB	Dept. of Defense ELAP	L2468	01-20-24
ANAB	Dept. of Energy	L2468.01	01-20-24
ANAB	ISO/IEC 17025	L2468	01-20-24
Arizona	State	AZ0708	08-11-23
Arkansas DEQ	State	88-0691	05-18-24
California	State	2897	01-22-24
Colorado	State	CA0004	08-31-23
Florida	NELAP	E87570	06-30-24
Georgia	State	4040	01-29-24
Hawaii	State	<cert No.>	01-29-24
Illinois	NELAP	200060	03-17-24
Kansas	NELAP	E-10375	10-31-23
Louisiana (All)	NELAP	01944	06-30-24
Maine	State	CA00004	04-14-24

* Accreditation/Certification renewal pending - accreditation/certification considered valid.

Accreditation/Certification Summary

Client: GHD Services Inc.
Job ID: 240-188340-1
Project/Site: 11208041, RACER Nodular

Laboratory: Eurofins Sacramento (Continued)

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Michigan	State	9947	06-01-23 *
Nevada	State	CA00044	07-31-23
New Hampshire	NELAP	2997	04-18-24
New Jersey	NELAP	CA005	06-30-24
New York	NELAP	11666	04-01-24
Ohio	State	41252	01-29-24
Oregon	NELAP	4040	01-29-24
Texas	NELAP	T104704399-19-13	05-31-24
US Fish & Wildlife	US Federal Programs	58448	04-30-24
USDA	US Federal Programs	P330-18-00239	02-28-26
Utah	NELAP	CA000442021-12	02-28-24
Virginia	NELAP	460278	03-14-24
Washington	State	C581	05-05-24
West Virginia (DW)	State	9930C	12-31-23
Wisconsin	State	998204680	08-31-23
Wyoming	State Program	8TMS-L	01-28-19 *

* Accreditation/Certification renewal pending - accreditation/certification considered valid.

Eurofins Cleveland
180 S. Van Buren Avenue
Barberton, OH 44203
Phone: 330-497-9396 Fax: 330-497-0772

MICHIGAN 190

0.7/1.1

Chain of Custody Record

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Client Information	Sampler: Bmw	Lab PM: Heckler, Denise D	Carrier Tracking No(s):	COC No: 240-109690-39133.2
Client Contact: Ms. Ruth Mickle	Phone: 7342609863	E-Mail: Denise.Heckler@et.eurofinsus.com	State of Origin:	Page: Page ~~2 of 2~~ 1/1
Company: GHD Services Inc.	PWSID:			Job #:

Address: 26850 Haggerty Rd	Due Date Requested:
City: Farmington Hills	TAT Requested (days):
State, Zip: MI, 48331	Compliance Project: Δ Yes Δ No
Phone: 612-524-6872(Tel)	PO #: Purchase Order Requested
Email: ruth.mickle@ghd.com	WO #: 58502
Project Name: 11208041, SMCO	Project #: 24006032
Site:	SSOW#:

Analysis Requested

Sample Identification	Sample Date	Sample Time	Sample Type (C=comp, G=grab)	Matrix (W=water, S=solid, O=waste/oil, BT=Tissue, A=Air)	Field Filtered Sample (Yes or No)	Perform MS/MSD (Yes or No)	PFC_IDA - PFAS, Standard List (28 Analytes)	8270D_SIM_MS_ID - 1,4-Dioxane	Total Number of containers	Special Instructions/Note:
			Preservation Code:				N	N		
GW-11208041-071023-BW-001	7-10-23	1457	G	Water			X	X		
↓ 002	↓	1507	↓	Water			X	X		
↓ 003	↓	1546	↓	↓			X	X		
GW-11208041-071123-BW-004	7-11-23	0921	↓	↓			X	X		
↓ 005	↓	1040	↓	↓			X	X		
↓ 006	↓	1205	↓	↓			X	X		

Preservation Codes:
A - HCL
B - NaOH
C - Zn Acetate
D - Nitric Acid
E - NaHSO4
F - MeOH
G - Amchlor
H - Ascorbic Acid
I - Ice
J - DI Water
K - EDTA
L - EDA
M - Hexane
N - None
O - AsNaO2
P - Na2O4S
Q - Na2SO3
R - Na2S2O3
S - H2SO4
T - TSP Dodecahydrate
U - Acetone
V - MCAA
W - pH 4-5
Y - Trizma
Z - other (specify)
Other:

240-188340 Chain of Custody

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ■ Unknown ☐ Radiological

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ Return To Client ■ Disposal By Lab ☐ Archive For ____ Months

Deliverable Requested: I, II, III, IV, Other (specify)

Special Instructions/QC Requirements:

Empty Kit Relinquished by:		Date:	Time:		Method of Shipment:
Relinquished by: [signature]	Date/Time: 7-11	Company:	Received by: [signature]	Date/Time: 7-12-23 945	Company: EETNC
Relinquished by:	Date/Time:	Company:	Received by:	Date/Time:	Company:
Relinquished by:	Date/Time:	Company:	Received by:	Date/Time:	Company:

Custody Seals Intact: Δ Yes Δ No	Custody Seal No.:	Cooler Temperature(s) °C and Other Remarks:

Ver: 06/08/2021

Eurofins – Cleveland Sample Receipt Form/Narrative Login # :_____
Barberton Facility

Client GHD Site Name_____ Cooler unpacked by: Rachelle Haidet
Cooler Received on 7 12 23 Opened on 7 12 23
FedEx: 1st Grd (Exp) UPS FAS Waypoint Client Drop Off Eurofins Courier Other_____

Receipt After-hours: Drop-off Date/Time_____ Storage Location_____

Eurofins Cooler # EC ~~Foam Box~~ Client Cooler ~~Box~~ Other_____
Packing material used: ~~Bubble Wrap~~ Foam ~~Plastic Bag~~ None Other_____
COOLANT: ~~Wet Ice~~ Blue Ice Dry Ice Water None

1. Cooler temperature upon receipt ☐ See Multiple Cooler Form
IR GUN # 13 (CF +0.4 °C) Observed Cooler Temp. 0.7 °C Corrected Cooler Temp. 1.1 °C

2. Were tamper/custody seals on the outside of the cooler(s)? If Yes Quantity 1	(Yes)	No
-Were the seals on the outside of the cooler(s) signed & dated?	(Yes)	No NA
-Were tamper/custody seals on the bottle(s) or bottle kits (LLHg/MeHg)?	Yes	(No)
-Were tamper/custody seals intact and uncompromised?	(Yes)	No NA
3. Shippers' packing slip attached to the cooler(s)?	(Yes)	No
4. Did custody papers accompany the sample(s)?	(Yes)	No
5. Were the custody papers relinquished & signed in the appropriate place?	(Yes)	No
6. Was/were the person(s) who collected the samples clearly identified on the COC?	(Yes)	No
7. Did all bottles arrive in good condition (Unbroken)?	(Yes)	No
8. Could all bottle labels (ID/Date/Time) be reconciled with the COC?	(Yes)	No
9. For each sample, does the COC specify preservatives (Y/N), # of containers (Y/N) and sample type of grab/comp (Y/N)?		
10. Were correct bottle(s) used for the test(s) indicated?	(Yes)	No
11. Sufficient quantity received to perform indicated analyses?	(Yes)	No
12. Are these work share samples and all listed on the COC?	Yes	(No)
If yes, Questions 13-17 have been checked at the originating laboratory.		
13. Were all preserved sample(s) at the correct pH upon receipt?	Yes No	(NA) pH Strip Lot# HC312502
14. Were VOAs on the COC?	Yes	(No)
15. Were air bubbles >6 mm in any VOA vials? ● ⬅ Larger than this.	Yes No	(NA)
16. Was a VOA trip blank present in the cooler(s)? Trip Blank Lot #_____	Yes	(No)
17. Was a LL Hg or Me Hg trip blank present? _____	Yes	(No)

Tests that are not checked for pH by Receiving:
VOAs
Oil and Grease
TOC

Contacted PM _____ Date _____ by _____ via Verbal Voice Mail Other

Concerning _____

18. CHAIN OF CUSTODY & SAMPLE DISCREPANCIES ☐ additional next page | Samples processed by:

_____
_____
_____
_____

19. SAMPLE CONDITION
Sample(s) _____ were received after the recommended holding time had expired.
Sample(s) _____ were received in a broken container.
Sample(s) _____ were received with bubble >6 mm in diameter. (Notify PM)

20. SAMPLE PRESERVATION

Sample(s) _____ were further preserved in the laboratory.
Time preserved: _____ Preservative(s) added/Lot number(s):_____

VOA Sample Preservation - Date/Time VOAs Frozen: _____

Eurofins Cleveland
180 S. Van Buren Avenue
Barberton, OH 44203
Phone: 330-497-9396 Fax: 330-497-0772

Chain of Custody Record

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Client Information (Sub Contract Lab)	Sampler:	Lab PM: Heckler, Denise D	Carrier Tracking No(s):	COC No: 240-170504.1
Client Contact: Shipping/Receiving	Phone:	E-Mail: Denise.Heckler@et.eurofinsus.com	State of Origin: Michigan	Page: Page 1 of 1
Company: Eurofins Environment Testing Northeast,		Accreditations Required (See note):		Job #: 240-188340-1
Address: 10 Hazelwood Drive, ,	Due Date Requested: 7/25/2023	Analysis Requested		Preservation Codes:
City: Amherst	TAT Requested (days):			A - HCL, B - NaOH, C - Zn Acetate, D - Nitric Acid, E - NaHSO4, F - MeOH, G - Amchlor, H - Ascorbic Acid, I - Ice, J - DI Water, K - EDTA, L - EDA, M - Hexane, N - None, O - AsNaO2, P - Na2O4S, Q - Na2SO3, R - Na2S2O3, S - H2SO4, T - TSP Dodecahydrate, U - Acetone, V - MCAA, W - pH 4-5, Y - Trizma, Z - other (specify)
State, Zip: NY, 14228-2298				
Phone: 716-691-2600(Tel) 716-691-7991(Fax)	PO #:			Other:
Email:	WO #:			
Project Name: 11208041 or 58502, RACER Nodular Iron	Project #: 24006032			
Site:	SSOW#:			

Sample Identification - Client ID (Lab ID)	Sample Date	Sample Time	Sample Type (C=Comp, G=grab)	Matrix (W=water, S=solid, O=waste/oil, BT=Tissue, A=Air)	8270D_SIM_MS_ID/3510C_LVI 1,4-Dioxane	Total Number of containers	Special Instructions/Note:
GW-11208041-071023-BW-001 (240-188340-1)	7/10/23	14:57 Eastern		Water	X	2	
GW-11208041-071023-BW-002 (240-188340-2)	7/10/23	15:07 Eastern		Water	X	2	
GW-11208041-071023-BW-003 (240-188340-3)	7/10/23	15:46 Eastern		Water	X	2	
GW-11208041-071123-BW-004 (240-188340-4)	7/11/23	09:21 Eastern		Water	X	2	
GW-11208041-071123-BW-005 (240-188340-5)	7/11/23	10:40 Eastern		Water	X	2	
GW-11208041-071123-BW-006 (240-188340-6)	7/11/23	12:05 Eastern		Water	X	2	

Note: Since laboratory accreditations are subject to change, Eurofins Environment Testing North Central, LLC places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under chain-of-custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analysis/tests/matrix being analyzed, the samples must be shipped back to the Eurofins Environment Testing North Central, LLC laboratory or other instructions will be provided. Any changes to accreditation status should be brought to Eurofins Environment Testing North Central, LLC attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to Eurofins Environment Testing North Central, LLC

Possible Hazard Identification	Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
Unconfirmed	☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months
Deliverable Requested: I, II, III, IV, Other (specify) Primary Deliverable Rank: 2	Special Instructions/QC Requirements:

Empty Kit Relinquished by:		Date:	Time:		Method of Shipment:		
Relinquished by: Rochelle Reese	Date/Time: 7/12/23 1500	Company: ISC EETNC	Received by:	Date/Time: 7/13/23 1000	Company: ETNC		
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company		
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company		
Custody Seals Intact: Δ Yes Δ No	Custody Seal No.:		Cooler Temperature(s) °C and Other Remarks: 2.8 ICE				

Login Sample Receipt Checklist

Client: GHD Services Inc.

Job Number: 240-188340-1

Login Number: 188340
List Number: 3
Creator: Kolb, Chris M

List Source: Eurofins Buffalo
List Creation: 07/17/23 11:07 AM

Question	Answer	Comment
Radioactivity either was not measured or, if measured, is at or below background	True	
The cooler's custody seal, if present, is intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	2.8 ir gun #1 ice
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the sample IDs on the containers and the COC.	True	
Samples are received within Holding Time (Excluding tests with immediate HTs)..	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.	True	
If necessary, staff have been informed of any short hold time or quick TAT needs	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Sampling Company provided.	True	
Samples received within 48 hours of sampling.	True	
Samples requiring field filtration have been filtered in the field.	True	
Chlorine Residual checked.	True	

Login Sample Receipt Checklist

Client: GHD Services Inc.

Job Number: 240-188340-1

Login Number: 188340
List Number: 2
Creator: Simmons, Jason C

List Source: Eurofins Sacramento
List Creation: 07/13/23 06:16 PM

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	N/A	
Sample custody seals, if present, are intact.	N/A	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	1.2c
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	N/A	Received project as a subcontract.
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time (excluding tests with immediate HTs)	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	N/A	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

Isotope Dilution Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular

Job ID: 240-188340-1

Method: 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Matrix: Water **Prep Type: Total/NA**

		Percent Isotope Dilution Recovery (Acceptance Limits)
Lab Sample ID	Client Sample ID	DXE (15-110)
240-188340-1	GW-11208041-071023-BW-001	34
240-188340-2	GW-11208041-071023-BW-002	35
240-188340-3	GW-11208041-071023-BW-003	28
240-188340-4	GW-11208041-071123-BW-004	35
240-188340-5	GW-11208041-071123-BW-005	36
240-188340-6	GW-11208041-071123-BW-006	40
LCS 480-676417/2-A	Lab Control Sample	36
MB 480-676417/1-A	Method Blank	38

Surrogate Legend

DXE = 1,4-Dioxane-d8

Method: 537 (modified) - Fluorinated Alkyl Substances

Matrix: Water **Prep Type: Total/NA**

		Percent Isotope Dilution Recovery (Acceptance Limits)							
Lab Sample ID	Client Sample ID	PFBA (25-150)	PFPeA (25-150)	PFHxA (25-150)	C4PFHA (25-150)	PFOA (25-150)	PFNA (25-150)	PFDA (25-150)	PFUnA (25-150)
240-188340-1	GW-11208041-071023-BW-001	76	103	94	97	98	108	111	99
240-188340-2	GW-11208041-071023-BW-002	81	109	100	98	100	107	108	97
240-188340-3	GW-11208041-071023-BW-003	31	106	94	95	102	106	116	98
240-188340-4	GW-11208041-071123-BW-004	86	96	95	93	96	109	100	95
240-188340-5	GW-11208041-071123-BW-005	61	65	67	68	70	76	82	70
240-188340-6	GW-11208041-071123-BW-006	26	105	97	97	97	108	110	88
LCS 320-690595/2-A	Lab Control Sample	117	109	106	105	104	102	113	108
LCSD 320-690595/3-A	Lab Control Sample Dup	117	102	99	105	101	101	100	105
MB 320-690595/1-A	Method Blank	113	113	107	112	104	102	107	113

		Percent Isotope Dilution Recovery (Acceptance Limits)							
Lab Sample ID	Client Sample ID	PFDoA (25-150)	PFTDA (25-150)	C3PFBS (25-150)	PFHxS (25-150)	PFOS (25-150)	PFOSA (25-150)	d3NMFOS (25-150)	d5NEFOS (25-150)
240-188340-1	GW-11208041-071023-BW-001	90	76	118	96	101	110	98	98
240-188340-2	GW-11208041-071023-BW-002	98	80	116	99	101	116	95	100
240-188340-3	GW-11208041-071023-BW-003	89	72	116	98	107	114	94	105
240-188340-4	GW-11208041-071123-BW-004	90	83	102	92	96	104	88	94
240-188340-5	GW-11208041-071123-BW-005	65	50	79	66	74	75	63	68
240-188340-6	GW-11208041-071123-BW-006	91	78	112	101	94	112	83	90
LCS 320-690595/2-A	Lab Control Sample	97	87	105	105	98	104	106	105
LCSD 320-690595/3-A	Lab Control Sample Dup	96	89	105	103	98	97	102	102
MB 320-690595/1-A	Method Blank	105	94	108	104	102	102	107	120

		Percent Isotope Dilution Recovery (Acceptance Limits)			
Lab Sample ID	Client Sample ID	M262FTS (25-150)	M282FTS (25-150)	M242FTS (25-150)	HFPODA (25-150)
240-188340-1	GW-11208041-071023-BW-001	97	109	92	106
240-188340-2	GW-11208041-071023-BW-002	98	107	96	106
240-188340-3	GW-11208041-071023-BW-003	97	107	88	117
240-188340-4	GW-11208041-071123-BW-004	91	106	80	120
240-188340-5	GW-11208041-071123-BW-005	66	82	63	83
240-188340-6	GW-11208041-071123-BW-006	93	99	92	111
LCS 320-690595/2-A	Lab Control Sample	102	107	111	100
LCSD 320-690595/3-A	Lab Control Sample Dup	106	104	108	97

Isotope Dilution Summary

Client: GHD Services Inc. Job ID: 240-188340-1
Project/Site: 11208041, RACER Nodular

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Matrix: Water **Prep Type: Total/NA**

		Percent Isotope Dilution Recovery (Acceptance Limits)							
Lab Sample ID	Client Sample ID	M262FTS (25-150)	M282FTS (25-150)	M242FTS (25-150)	HFPODA (25-150)				
MB 320-690595/1-A	Method Blank	107	122	117	100				

Surrogate Legend

PFBA = 13C4 PFBA
PFPeA = 13C5 PFPeA
PFHxA = 13C2 PFHxA
C4PFHA = 13C4 PFHpA
PFOA = 13C4 PFOA
PFNA = 13C5 PFNA
PFDA = 13C2 PFDA
PFUnA = 13C2 PFUnA
PFDoA = 13C2 PFDoA
PFTDA = 13C2 PFTeDA
C3PFBS = 13C3 PFBS
PFHxS = 18O2 PFHxS
PFOS = 13C4 PFOS
PFOSA = 13C8 FOSA
d3NMFOS = d3-NMeFOSAA
d5NEFOS = d5-NEtFOSAA
M262FTS = M2-6:2 FTS
M282FTS = M2-8:2 FTS
M242FTS = M2-4:2 FTS
HFPODA = 13C3 HFPO-DA

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ANALYTICAL REPORT

PREPARED FOR

Attn: Ms. Ruth Mickle
GHD Services Inc.
26850 Haggerty Rd.
Farmington Hills, Michigan 48331

Generated 9/9/2023 3:59:48 PM

JOB DESCRIPTION

11208041, RACER Nodular Iron

JOB NUMBER

240-190457-1

Eurofins Cleveland
180 S. Van Buren Avenue
Barberton OH 44203

See page two for job notes and contact information.

Job Notes

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Authorization

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Table of Contents

Job ID: 240-190457-1

Laboratory: Eurofins Cleveland

Narrative

Job Narrative
240-190457-1

Comments
No additional comments.

Receipt
The samples were received on 8/19/2023 9:30 AM. Unless otherwise noted below, the samples arrived in good condition, and where required, properly preserved and on ice. The temperature of the cooler at receipt was 1.1º C.

GC/MS Semi VOA
No analytical or quality issues were noted, other than those described in the Definitions/Glossary page.

LCMS
Method 537 (modified): The following sample has chromatographic interferences that could adversely impact the identification and quantitation of target analytes: Perfluorobutanoic acid (PFBA) and Perfluoropentanoic acid (PFPeA). This interference could cause false positive results: GW-11208041-081823-BW-001 (240-190457-1).

Method 537 (modified): The following sample has chromatographic interferences that could adversely impact the identification and quantitation of target analytes: Perfluorobutanoic acid (PFBA), Perfluoropentanoic acid (PFPeA) and Perfluorobutanesulfonic acid (PFBS). This interference could cause false positive results: GW-11208041-081823-BW-004 (240-190457-4).

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

Organic Prep
Method 3535: The following samples in preparation batch 320-702737 were yellow in color prior to extraction: GW-11208041-081823-BW-005 (240-190457-5)

Method 3535: The following samples in preparation batch 320-702737 were orange in color prior to extraction: GW-11208041-081823-BW-001 (240-190457-1)

Method 3535: The following samples in preparation batch 320-702737 were observed to have a thin layer of sediment present in the bottle prior to extraction. GW-11208041-081823-BW-001 (240-190457-1)

Method 3535: During the solid phase extraction process, the following samples contained non-settleable particulates which clogged the solid phase extraction column: GW-11208041-081823-BW-001 (240-190457-1).

Method 3535: The following sample in preparation batch 320-702737 was yellow in color following extraction: GW-11208041-081823-BW-001 (240-190457-1)

Method 3535: The Matrix Spike (MS)/Matrix Spike Duplicate (MSD) in the prep batch 320-702737 were generated from the 2 backup bottles of the parent sample. GW-11208041-081823-BW-005 (240-190457-5[MS]) and GW-11208041-081823-BW-005 (240-190457-5[MSD])

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

Definitions/Glossary

Client: GHD Services Inc.
Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

Qualifiers

GC/MS Semi VOA

Qualifier	Qualifier Description
U	Indicates the analyte was analyzed for but not detected.

LCMS

Qualifier	Qualifier Description
CI	The peak identified by the data system exhibited chromatographic interference that could not be resolved. There is reason to suspect there may be a high bias.
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.
U	Indicates the analyte was analyzed for but not detected.

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
¤	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CFU	Colony Forming Unit
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MCL	EPA recommended "Maximum Contaminant Level"
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
MPN	Most Probable Number
MQL	Method Quantitation Limit
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
NEG	Negative / Absent
POS	Positive / Present
PQL	Practical Quantitation Limit
PRES	Presumptive
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)
TNTC	Too Numerous To Count

Sample Summary

Client: GHD Services Inc.
Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

Lab Sample ID	Client Sample ID	Matrix	Collected	Received
240-190457-1	GW-11208041-081823-BW-001	Water	08/18/23 10:37	08/19/23 09:30
240-190457-2	GW-11208041-081823-BW-002	Water	08/18/23 10:44	08/19/23 09:30
240-190457-3	GW-11208041-081823-BW-003	Water	08/18/23 10:53	08/19/23 09:30
240-190457-4	GW-11208041-081823-BW-004	Water	08/18/23 12:11	08/19/23 09:30
240-190457-5	GW-11208041-081823-BW-005	Water	08/18/23 13:30	08/19/23 09:30
240-190457-6	TRIP BLANK	Water	08/18/23 00:00	08/19/23 09:30

Detection Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular Iron

Job ID: 240-190457-1

Client Sample ID: GW-11208041-081823-BW-001 — Lab Sample ID: 240-190457-1

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
1,4-Dioxane	2.3		0.20	0.10	ug/L	1		8270D SIM ID	Total/NA
Perfluorobutanoic acid (PFBA)	8.5	CI	4.8	2.3	ng/L	1		537 (modified)	Total/NA
Perfluoropentanoic acid (PFPeA)	2.8	CI	1.9	0.47	ng/L	1		537 (modified)	Total/NA
Perfluorohexanoic acid (PFHxA)	2.9		1.9	0.56	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	1.2	J	1.9	0.82	ng/L	1		537 (modified)	Total/NA

Client Sample ID: GW-11208041-081823-BW-002 — Lab Sample ID: 240-190457-2

No Detections.

Client Sample ID: GW-11208041-081823-BW-003 — Lab Sample ID: 240-190457-3

No Detections.

Client Sample ID: GW-11208041-081823-BW-004 — Lab Sample ID: 240-190457-4

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
1,4-Dioxane	0.96		0.20	0.10	ug/L	1		8270D SIM ID	Total/NA
Perfluorobutanoic acid (PFBA)	6.8	CI	5.0	2.4	ng/L	1		537 (modified)	Total/NA
Perfluoropentanoic acid (PFPeA)	1.4	J CI	2.0	0.49	ng/L	1		537 (modified)	Total/NA
Perfluorohexanoic acid (PFHxA)	1.7	J	2.0	0.58	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	0.72	J	2.0	0.25	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	3.8		2.0	0.85	ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	0.67	J CI	2.0	0.20	ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	4.4		2.0	0.54	ng/L	1		537 (modified)	Total/NA

Client Sample ID: GW-11208041-081823-BW-005 — Lab Sample ID: 240-190457-5

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
1,4-Dioxane	2.8		0.20	0.10	ug/L	1		8270D SIM ID	Total/NA
Perfluorobutanoic acid (PFBA)	11		4.8	2.3	ng/L	1		537 (modified)	Total/NA
Perfluoropentanoic acid (PFPeA)	1.8	J	1.9	0.47	ng/L	1		537 (modified)	Total/NA
Perfluorohexanoic acid (PFHxA)	2.4		1.9	0.56	ng/L	1		537 (modified)	Total/NA
Perfluoroheptanoic acid (PFHpA)	0.93	J	1.9	0.24	ng/L	1		537 (modified)	Total/NA
Perfluorooctanoic acid (PFOA)	5.1		1.9	0.82	ng/L	1		537 (modified)	Total/NA
Perfluorobutanesulfonic acid (PFBS)	0.80	J	1.9	0.19	ng/L	1		537 (modified)	Total/NA
Perfluorohexanesulfonic acid (PFHxS)	1.2	J	1.9	0.55	ng/L	1		537 (modified)	Total/NA
Perfluorooctanesulfonic acid (PFOS)	12		1.9	0.52	ng/L	1		537 (modified)	Total/NA

Client Sample ID: TRIP BLANK — Lab Sample ID: 240-190457-6

No Detections.

This Detection Summary does not include radiochemical test results.

Method Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular Iron

Job ID: 240-190457-1

Method	Method Description	Protocol	Laboratory
8270D SIM ID	Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)	SW846	EET BUF
537 (modified)	Fluorinated Alkyl Substances	EPA	EET SAC
3510C	Liquid-Liquid Extraction (Separatory Funnel)	SW846	EET BUF
3535	Solid-Phase Extraction (SPE)	SW846	EET SAC

Protocol References:

EPA = US Environmental Protection Agency

SW846 = "Test Methods For Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 And Its Updates.

Laboratory References:

EET BUF = Eurofins Buffalo, 10 Hazelwood Drive, Amherst, NY 14228-2298, TEL (716)691-2600

EET SAC = Eurofins Sacramento, 880 Riverside Parkway, West Sacramento, CA 95605, TEL (916)373-5600

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular Iron

Job ID: 240-190457-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-081823-BW-001
Date Collected: 08/18/23 10:37
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-1
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	2.3		0.20	0.10	ug/L		08/22/23 11:43	08/23/23 15:39	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*38*		*15 - 110*	*08/22/23 11:43*	*08/23/23 15:39*	*1*

Client: GHD Services Inc.
Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-081823-BW-003
Lab Sample ID: 240-190457-3
Date Collected: 08/18/23 10:53
Matrix: Water
Date Received: 08/19/23 09:30

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	0.20	U	0.20	0.10	ug/L		08/22/23 11:43	08/23/23 15:56	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*36*		*15 - 110*	*08/22/23 11:43*	*08/23/23 15:56*	*1*

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular Iron

Job ID: 240-190457-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-081823-BW-004
Date Collected: 08/18/23 12:11
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-4
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	0.96		0.20	0.10	ug/L		08/22/23 11:43	08/23/23 16:13	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*38*		*15 - 110*	*08/22/23 11:43*	*08/23/23 16:13*	*1*

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular Iron

Job ID: 240-190457-1

Method: SW846 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Client Sample ID: GW-11208041-081823-BW-005
Date Collected: 08/18/23 13:30
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-5
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	2.8		0.20	0.10	ug/L		08/22/23 11:43	08/23/23 12:30	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*26*		*15 - 110*	*08/22/23 11:43*	*08/23/23 12:30*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-081823-BW-001
Date Collected: 08/18/23 10:37
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-1
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**8.5**	**CI**	4.8	2.3	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluoropentanoic acid (PFPeA)	**2.8**	**CI**	1.9	0.47	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorohexanoic acid (PFHxA)	**2.9**		1.9	0.56	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluoroheptanoic acid (PFHpA)	1.9	U	1.9	0.24	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorooctanoic acid (PFOA)	**1.2**	**J**	1.9	0.82	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorononanoic acid (PFNA)	1.9	U	1.9	0.26	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.30	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.1	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.53	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.3	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.70	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorobutanesulfonic acid (PFBS)	1.9	U	1.9	0.19	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	1.9	0.29	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorohexanesulfonic acid (PFHxS)	1.9	U	1.9	0.55	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	1.9	0.18	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorooctanesulfonic acid (PFOS)	1.9	U	1.9	0.52	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.36	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.31	ng/L		08/30/23 12:20	09/01/23 13:09	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.94	ng/L		08/30/23 12:20	09/01/23 13:09	1
NMeFOSAA	4.8	U	4.8	1.2	ng/L		08/30/23 12:20	09/01/23 13:09	1
NEtFOSAA	4.8	U	4.8	1.3	ng/L		08/30/23 12:20	09/01/23 13:09	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 13:09	1
6:2 FTS	4.8	U	4.8	2.4	ng/L		08/30/23 12:20	09/01/23 13:09	1
8:2 FTS	1.9	U	1.9	0.44	ng/L		08/30/23 12:20	09/01/23 13:09	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.38	ng/L		08/30/23 12:20	09/01/23 13:09	1
HFPO-DA (GenX)	3.8	U	3.8	1.4	ng/L		08/30/23 12:20	09/01/23 13:09	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 13:09	1
11Cl-PF3OUdS	1.9	U	1.9	0.31	ng/L		08/30/23 12:20	09/01/23 13:09	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*42*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C5 PFPeA	*52*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C2 PFHxA	*59*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C4 PFHpA	*59*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C4 PFOA	*59*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C5 PFNA	*61*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C2 PFDA	*58*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C2 PFUnA	*49*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C2 PFDoA	*44*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C2 PFTeDA	*41*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C3 PFBS	*60*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
18O2 PFHxS	*60*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C4 PFOS	*56*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
13C8 FOSA	*55*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
d3-NMeFOSAA	*50*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
d5-NEtFOSAA	*49*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
M2-6:2 FTS	*53*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*
M2-8:2 FTS	*57*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:09*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-081823-BW-001
Date Collected: 08/18/23 10:37
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-1
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-4:2 FTS	60		25 - 150	08/30/23 12:20	09/01/23 13:09	1
13C3 HFPO-DA	52		25 - 150	08/30/23 12:20	09/01/23 13:09	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-081823-BW-002
Date Collected: 08/18/23 10:44
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-2
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	4.7	U	4.7	2.3	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluoropentanoic acid (PFPeA)	1.9	U	1.9	0.46	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorohexanoic acid (PFHxA)	1.9	U	1.9	0.55	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluoroheptanoic acid (PFHpA)	1.9	U	1.9	0.24	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorooctanoic acid (PFOA)	1.9	U	1.9	0.80	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorononanoic acid (PFNA)	1.9	U	1.9	0.25	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.29	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.0	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.52	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.2	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.69	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorobutanesulfonic acid (PFBS)	1.9	U	1.9	0.19	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	1.9	0.28	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorohexanesulfonic acid (PFHxS)	1.9	U	1.9	0.54	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	1.9	0.18	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorooctanesulfonic acid (PFOS)	1.9	U	1.9	0.51	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.35	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.30	ng/L		08/30/23 12:20	09/01/23 13:20	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.92	ng/L		08/30/23 12:20	09/01/23 13:20	1
NMeFOSAA	4.7	U	4.7	1.1	ng/L		08/30/23 12:20	09/01/23 13:20	1
NEtFOSAA	4.7	U	4.7	1.2	ng/L		08/30/23 12:20	09/01/23 13:20	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 13:20	1
6:2 FTS	4.7	U	4.7	2.4	ng/L		08/30/23 12:20	09/01/23 13:20	1
8:2 FTS	1.9	U	1.9	0.43	ng/L		08/30/23 12:20	09/01/23 13:20	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.38	ng/L		08/30/23 12:20	09/01/23 13:20	1
HFPO-DA (GenX)	3.8	U	3.8	1.4	ng/L		08/30/23 12:20	09/01/23 13:20	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 13:20	1
11Cl-PF3OUdS	1.9	U	1.9	0.30	ng/L		08/30/23 12:20	09/01/23 13:20	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*99*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C5 PFPeA	*95*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C2 PFHxA	*92*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C4 PFHpA	*90*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C4 PFOA	*91*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C5 PFNA	*96*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C2 PFDA	*96*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C2 PFUnA	*85*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C2 PFDoA	*83*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C2 PFTeDA	*80*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C3 PFBS	*95*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
18O2 PFHxS	*90*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C4 PFOS	*90*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
13C8 FOSA	*94*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
d3-NMeFOSAA	*82*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
d5-NEtFOSAA	*89*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
M2-6:2 FTS	*89*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*
M2-8:2 FTS	*92*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:20*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-081823-BW-002
Date Collected: 08/18/23 10:44
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-2
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-4:2 FTS	92		25 - 150	08/30/23 12:20	09/01/23 13:20	1
13C3 HFPO-DA	77		25 - 150	08/30/23 12:20	09/01/23 13:20	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-081823-BW-003
Date Collected: 08/18/23 10:53
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-3
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	4.8	U	4.8	2.3	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluoropentanoic acid (PFPeA)	1.9	U	1.9	0.47	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorohexanoic acid (PFHxA)	1.9	U	1.9	0.56	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluoroheptanoic acid (PFHpA)	1.9	U	1.9	0.24	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorooctanoic acid (PFOA)	1.9	U	1.9	0.82	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorononanoic acid (PFNA)	1.9	U	1.9	0.26	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.30	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.1	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.53	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.2	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.70	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorobutanesulfonic acid (PFBS)	1.9	U	1.9	0.19	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	1.9	0.29	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorohexanesulfonic acid (PFHxS)	1.9	U	1.9	0.55	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	1.9	0.18	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorooctanesulfonic acid (PFOS)	1.9	U	1.9	0.52	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.35	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.31	ng/L		08/30/23 12:20	09/01/23 13:32	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.94	ng/L		08/30/23 12:20	09/01/23 13:32	1
NMeFOSAA	4.8	U	4.8	1.2	ng/L		08/30/23 12:20	09/01/23 13:32	1
NEtFOSAA	4.8	U	4.8	1.2	ng/L		08/30/23 12:20	09/01/23 13:32	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 13:32	1
6:2 FTS	4.8	U	4.8	2.4	ng/L		08/30/23 12:20	09/01/23 13:32	1
8:2 FTS	1.9	U	1.9	0.44	ng/L		08/30/23 12:20	09/01/23 13:32	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.38	ng/L		08/30/23 12:20	09/01/23 13:32	1
HFPO-DA (GenX)	3.8	U	3.8	1.4	ng/L		08/30/23 12:20	09/01/23 13:32	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 13:32	1
11Cl-PF3OUdS	1.9	U	1.9	0.31	ng/L		08/30/23 12:20	09/01/23 13:32	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*94*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C5 PFPeA	*91*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C2 PFHxA	*86*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C4 PFHpA	*82*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C4 PFOA	*87*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C5 PFNA	*95*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C2 PFDA	*84*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C2 PFUnA	*85*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C2 PFDoA	*75*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C2 PFTeDA	*77*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C3 PFBS	*87*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
18O2 PFHxS	*86*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C4 PFOS	*84*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
13C8 FOSA	*90*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
d3-NMeFOSAA	*79*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
d5-NEtFOSAA	*76*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
M2-6:2 FTS	*86*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*
M2-8:2 FTS	*78*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:32*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-081823-BW-003
Date Collected: 08/18/23 10:53
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-3
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-4:2 FTS	91		25 - 150	08/30/23 12:20	09/01/23 13:32	1
13C3 HFPO-DA	71		25 - 150	08/30/23 12:20	09/01/23 13:32	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-081823-BW-004
Date Collected: 08/18/23 12:11
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-4
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**6.8**	**CI**	5.0	2.4	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluoropentanoic acid (PFPeA)	**1.4**	**J CI**	2.0	0.49	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorohexanoic acid (PFHxA)	**1.7**	**J**	2.0	0.58	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluoroheptanoic acid (PFHpA)	**0.72**	**J**	2.0	0.25	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorooctanoic acid (PFOA)	**3.8**		2.0	0.85	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorononanoic acid (PFNA)	2.0	U	2.0	0.27	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorodecanoic acid (PFDA)	2.0	U	2.0	0.31	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluoroundecanoic acid (PFUnA)	2.0	U	2.0	1.1	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorododecanoic acid (PFDoA)	2.0	U	2.0	0.55	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorotridecanoic acid (PFTrDA)	2.0	U	2.0	1.3	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorotetradecanoic acid (PFTeA)	2.0	U	2.0	0.73	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorobutanesulfonic acid (PFBS)	**0.67**	**J CI**	2.0	0.20	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluoropentanesulfonic acid (PFPeS)	2.0	U	2.0	0.30	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorohexanesulfonic acid (PFHxS)	2.0	U	2.0	0.57	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluoroheptanesulfonic acid (PFHpS)	2.0	U	2.0	0.19	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorooctanesulfonic acid (PFOS)	**4.4**		2.0	0.54	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorononanesulfonic acid (PFNS)	2.0	U	2.0	0.37	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorodecanesulfonic acid (PFDS)	2.0	U	2.0	0.32	ng/L		08/30/23 12:20	09/01/23 13:43	1
Perfluorooctanesulfonamide (FOSA)	2.0	U	2.0	0.98	ng/L		08/30/23 12:20	09/01/23 13:43	1
NMeFOSAA	5.0	U	5.0	1.2	ng/L		08/30/23 12:20	09/01/23 13:43	1
NEtFOSAA	5.0	U	5.0	1.3	ng/L		08/30/23 12:20	09/01/23 13:43	1
4:2 FTS	2.0	U	2.0	0.24	ng/L		08/30/23 12:20	09/01/23 13:43	1
6:2 FTS	5.0	U	5.0	2.5	ng/L		08/30/23 12:20	09/01/23 13:43	1
8:2 FTS	2.0	U	2.0	0.46	ng/L		08/30/23 12:20	09/01/23 13:43	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	2.0	U	2.0	0.40	ng/L		08/30/23 12:20	09/01/23 13:43	1
HFPO-DA (GenX)	4.0	U	4.0	1.5	ng/L		08/30/23 12:20	09/01/23 13:43	1
9Cl-PF3ONS	2.0	U	2.0	0.24	ng/L		08/30/23 12:20	09/01/23 13:43	1
11Cl-PF3OUdS	2.0	U	2.0	0.32	ng/L		08/30/23 12:20	09/01/23 13:43	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*70*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C5 PFPeA	*86*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C2 PFHxA	*92*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C4 PFHpA	*91*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C4 PFOA	*87*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C5 PFNA	*90*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C2 PFDA	*86*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C2 PFUnA	*83*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C2 PFDoA	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C2 PFTeDA	*79*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C3 PFBS	*95*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
18O2 PFHxS	*93*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C4 PFOS	*88*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
13C8 FOSA	*94*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
d3-NMeFOSAA	*82*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*
d5-NEtFOSAA	*87*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:43*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-081823-BW-004
Date Collected: 08/18/23 12:11
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-4
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-6:2 FTS	79		25 - 150	08/30/23 12:20	09/01/23 13:43	1
M2-8:2 FTS	90		25 - 150	08/30/23 12:20	09/01/23 13:43	1
M2-4:2 FTS	78		25 - 150	08/30/23 12:20	09/01/23 13:43	1
13C3 HFPO-DA	81		25 - 150	08/30/23 12:20	09/01/23 13:43	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: GW-11208041-081823-BW-005
Date Collected: 08/18/23 13:30
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-5
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	**11**		4.8	2.3	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluoropentanoic acid (PFPeA)	**1.8**	**J**	1.9	0.47	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorohexanoic acid (PFHxA)	**2.4**		1.9	0.56	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluoroheptanoic acid (PFHpA)	**0.93**	**J**	1.9	0.24	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorooctanoic acid (PFOA)	**5.1**		1.9	0.82	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorononanoic acid (PFNA)	1.9	U	1.9	0.26	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.30	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.1	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.53	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.3	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.71	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorobutanesulfonic acid (PFBS)	**0.80**	**J**	1.9	0.19	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	1.9	0.29	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorohexanesulfonic acid (PFHxS)	**1.2**	**J**	1.9	0.55	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	1.9	0.18	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorooctanesulfonic acid (PFOS)	**12**		1.9	0.52	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.36	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.31	ng/L		08/30/23 12:20	09/01/23 13:54	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.95	ng/L		08/30/23 12:20	09/01/23 13:54	1
NMeFOSAA	4.8	U	4.8	1.2	ng/L		08/30/23 12:20	09/01/23 13:54	1
NEtFOSAA	4.8	U	4.8	1.3	ng/L		08/30/23 12:20	09/01/23 13:54	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 13:54	1
6:2 FTS	4.8	U	4.8	2.4	ng/L		08/30/23 12:20	09/01/23 13:54	1
8:2 FTS	1.9	U	1.9	0.45	ng/L		08/30/23 12:20	09/01/23 13:54	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.39	ng/L		08/30/23 12:20	09/01/23 13:54	1
HFPO-DA (GenX)	3.9	U	3.9	1.5	ng/L		08/30/23 12:20	09/01/23 13:54	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 13:54	1
11Cl-PF3OUdS	1.9	U	1.9	0.31	ng/L		08/30/23 12:20	09/01/23 13:54	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*59*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C5 PFPeA	*77*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C2 PFHxA	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C4 PFHpA	*77*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C4 PFOA	*80*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C5 PFNA	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C2 PFDA	*79*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C2 PFUnA	*72*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C2 PFDoA	*68*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C2 PFTeDA	*54*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C3 PFBS	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
18O2 PFHxS	*79*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C4 PFOS	*73*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
13C8 FOSA	*79*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
d3-NMeFOSAA	*72*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*
d5-NEtFOSAA	*76*		*25 - 150*	*08/30/23 12:20*	*09/01/23 13:54*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: GW-11208041-081823-BW-005
Date Collected: 08/18/23 13:30
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-5
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-6:2 FTS	69		25 - 150	08/30/23 12:20	09/01/23 13:54	1
M2-8:2 FTS	81		25 - 150	08/30/23 12:20	09/01/23 13:54	1
M2-4:2 FTS	72		25 - 150	08/30/23 12:20	09/01/23 13:54	1
13C3 HFPO-DA	70		25 - 150	08/30/23 12:20	09/01/23 13:54	1

Method: EPA 537 (modified) - Fluorinated Alkyl Substances

Client Sample ID: TRIP BLANK
Date Collected: 08/18/23 00:00
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-6
Matrix: Water

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	4.8	U	4.8	2.3	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluoropentanoic acid (PFPeA)	1.9	U	1.9	0.47	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorohexanoic acid (PFHxA)	1.9	U	1.9	0.56	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluoroheptanoic acid (PFHpA)	1.9	U	1.9	0.24	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorooctanoic acid (PFOA)	1.9	U	1.9	0.82	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorononanoic acid (PFNA)	1.9	U	1.9	0.26	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorodecanoic acid (PFDA)	1.9	U	1.9	0.30	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluoroundecanoic acid (PFUnA)	1.9	U	1.9	1.1	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorododecanoic acid (PFDoA)	1.9	U	1.9	0.53	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorotridecanoic acid (PFTrDA)	1.9	U	1.9	1.3	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorotetradecanoic acid (PFTeA)	1.9	U	1.9	0.71	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorobutanesulfonic acid (PFBS)	1.9	U	1.9	0.19	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	1.9	0.29	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorohexanesulfonic acid (PFHxS)	1.9	U	1.9	0.55	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	1.9	0.18	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorooctanesulfonic acid (PFOS)	1.9	U	1.9	0.52	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorononanesulfonic acid (PFNS)	1.9	U	1.9	0.36	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorodecanesulfonic acid (PFDS)	1.9	U	1.9	0.31	ng/L		08/30/23 12:20	09/01/23 14:28	1
Perfluorooctanesulfonamide (FOSA)	1.9	U	1.9	0.95	ng/L		08/30/23 12:20	09/01/23 14:28	1
NMeFOSAA	4.8	U	4.8	1.2	ng/L		08/30/23 12:20	09/01/23 14:28	1
NEtFOSAA	4.8	U	4.8	1.3	ng/L		08/30/23 12:20	09/01/23 14:28	1
4:2 FTS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 14:28	1
6:2 FTS	4.8	U	4.8	2.4	ng/L		08/30/23 12:20	09/01/23 14:28	1
8:2 FTS	1.9	U	1.9	0.45	ng/L		08/30/23 12:20	09/01/23 14:28	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	1.9	0.39	ng/L		08/30/23 12:20	09/01/23 14:28	1
HFPO-DA (GenX)	3.9	U	3.9	1.5	ng/L		08/30/23 12:20	09/01/23 14:28	1
9Cl-PF3ONS	1.9	U	1.9	0.23	ng/L		08/30/23 12:20	09/01/23 14:28	1
11Cl-PF3OUdS	1.9	U	1.9	0.31	ng/L		08/30/23 12:20	09/01/23 14:28	1

Isotope Dilution	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*93*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C5 PFPeA	*86*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C2 PFHxA	*86*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C4 PFHpA	*83*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C4 PFOA	*87*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C5 PFNA	*88*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C2 PFDA	*85*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C2 PFUnA	*78*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C2 PFDoA	*80*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C2 PFTeDA	*78*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C3 PFBS	*85*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
18O2 PFHxS	*84*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C4 PFOS	*87*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
13C8 FOSA	*88*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
d3-NMeFOSAA	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
d5-NEtFOSAA	*86*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
M2-6:2 FTS	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*
M2-8:2 FTS	*87*		*25 - 150*	*08/30/23 12:20*	*09/01/23 14:28*	*1*

Method: EPA 537 (modified) - Fluorinated Alkyl Substances (Continued)

Client Sample ID: TRIP BLANK
Date Collected: 08/18/23 00:00
Date Received: 08/19/23 09:30

Lab Sample ID: 240-190457-6
Matrix: Water

Isotope Dilution	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
M2-4:2 FTS	84		25 - 150	08/30/23 12:20	09/01/23 14:28	1
13C3 HFPO-DA	71		25 - 150	08/30/23 12:20	09/01/23 14:28	1

QC Association Summary

Client: GHD Services Inc.
Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

GC/MS Semi VOA

Prep Batch: 680885

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-190457-1	GW-11208041-081823-BW-001	Total/NA	Water	3510C	
240-190457-3	GW-11208041-081823-BW-003	Total/NA	Water	3510C	
240-190457-4	GW-11208041-081823-BW-004	Total/NA	Water	3510C	
240-190457-5	GW-11208041-081823-BW-005	Total/NA	Water	3510C	
MB 480-680885/1-A	Method Blank	Total/NA	Water	3510C	
LCS 480-680885/2-A	Lab Control Sample	Total/NA	Water	3510C	
240-190457-5 MS	GW-11208041-081823-BW-005	Total/NA	Water	3510C	
240-190457-5 MSD	GW-11208041-081823-BW-005	Total/NA	Water	3510C	

Analysis Batch: 680964

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-190457-1	GW-11208041-081823-BW-001	Total/NA	Water	8270D SIM ID	680885
240-190457-3	GW-11208041-081823-BW-003	Total/NA	Water	8270D SIM ID	680885
240-190457-4	GW-11208041-081823-BW-004	Total/NA	Water	8270D SIM ID	680885
240-190457-5	GW-11208041-081823-BW-005	Total/NA	Water	8270D SIM ID	680885
MB 480-680885/1-A	Method Blank	Total/NA	Water	8270D SIM ID	680885
LCS 480-680885/2-A	Lab Control Sample	Total/NA	Water	8270D SIM ID	680885
240-190457-5 MS	GW-11208041-081823-BW-005	Total/NA	Water	8270D SIM ID	680885
240-190457-5 MSD	GW-11208041-081823-BW-005	Total/NA	Water	8270D SIM ID	680885

LCMS

Prep Batch: 702737

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-190457-1	GW-11208041-081823-BW-001	Total/NA	Water	3535	
240-190457-2	GW-11208041-081823-BW-002	Total/NA	Water	3535	
240-190457-3	GW-11208041-081823-BW-003	Total/NA	Water	3535	
240-190457-4	GW-11208041-081823-BW-004	Total/NA	Water	3535	
240-190457-5	GW-11208041-081823-BW-005	Total/NA	Water	3535	
240-190457-6	TRIP BLANK	Total/NA	Water	3535	
MB 320-702737/1-A	Method Blank	Total/NA	Water	3535	
LCS 320-702737/2-A	Lab Control Sample	Total/NA	Water	3535	
240-190457-5 MS	GW-11208041-081823-BW-005	Total/NA	Water	3535	
240-190457-5 MSD	GW-11208041-081823-BW-005	Total/NA	Water	3535	

Analysis Batch: 703301

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-190457-1	GW-11208041-081823-BW-001	Total/NA	Water	537 (modified)	702737
240-190457-2	GW-11208041-081823-BW-002	Total/NA	Water	537 (modified)	702737
240-190457-3	GW-11208041-081823-BW-003	Total/NA	Water	537 (modified)	702737
240-190457-4	GW-11208041-081823-BW-004	Total/NA	Water	537 (modified)	702737
240-190457-5	GW-11208041-081823-BW-005	Total/NA	Water	537 (modified)	702737
240-190457-6	TRIP BLANK	Total/NA	Water	537 (modified)	702737
MB 320-702737/1-A	Method Blank	Total/NA	Water	537 (modified)	702737
LCS 320-702737/2-A	Lab Control Sample	Total/NA	Water	537 (modified)	702737
240-190457-5 MS	GW-11208041-081823-BW-005	Total/NA	Water	537 (modified)	702737
240-190457-5 MSD	GW-11208041-081823-BW-005	Total/NA	Water	537 (modified)	702737

Method: 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Lab Sample ID: MB 480-680885/1-A
Matrix: Water
Analysis Batch: 680964

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 680885

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dioxane	0.20	U	0.20	0.10	ug/L		08/22/23 11:43	08/23/23 11:22	1

Isotope Dilution	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,4-Dioxane-d8	*42*		*15 - 110*	*08/22/23 11:43*	*08/23/23 11:22*	*1*

Lab Sample ID: LCS 480-680885/2-A
Matrix: Water
Analysis Batch: 680964

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 680885

Analyte			Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec Limits		
1,4-Dioxane			2.00	2.05		ug/L		102	40 - 140		

Isotope Dilution	*LCS %Recovery*	*LCS Qualifier*	*Limits*
1,4-Dioxane-d8	*39*		*15 - 110*

Lab Sample ID: 240-190457-5 MS
Matrix: Water
Analysis Batch: 680964

Client Sample ID: GW-11208041-081823-BW-005
Prep Type: Total/NA
Prep Batch: 680885

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec Limits		
1,4-Dioxane	2.8		2.00	4.96		ug/L		109	40 - 140		

Isotope Dilution	*MS %Recovery*	*MS Qualifier*	*Limits*
1,4-Dioxane-d8	*27*		*15 - 110*

Lab Sample ID: 240-190457-5 MSD
Matrix: Water
Analysis Batch: 680964

Client Sample ID: GW-11208041-081823-BW-005
Prep Type: Total/NA
Prep Batch: 680885

Analyte	Sample Result	Sample Qualifier	Spike Added	MSD Result	MSD Qualifier	Unit	D	%Rec	%Rec Limits	RPD	RPD Limit
1,4-Dioxane	2.8		2.00	4.91		ug/L		106	40 - 140	1	20

Isotope Dilution	*MSD %Recovery*	*MSD Qualifier*	*Limits*
1,4-Dioxane-d8	*26*		*15 - 110*

Method: 537 (modified) - Fluorinated Alkyl Substances

Lab Sample ID: MB 320-702737/1-A
Matrix: Water
Analysis Batch: 703301

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 702737

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorobutanoic acid (PFBA)	5.0	U	5.0	2.4	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluoropentanoic acid (PFPeA)	2.0	U	2.0	0.49	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorohexanoic acid (PFHxA)	2.0	U	2.0	0.58	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluoroheptanoic acid (PFHpA)	2.0	U	2.0	0.25	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorooctanoic acid (PFOA)	2.0	U	2.0	0.85	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorononanoic acid (PFNA)	2.0	U	2.0	0.27	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorodecanoic acid (PFDA)	2.0	U	2.0	0.31	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluoroundecanoic acid (PFUnA)	2.0	U	2.0	1.1	ng/L		08/30/23 12:20	09/01/23 12:46	1

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: MB 320-702737/1-A
Matrix: Water
Analysis Batch: 703301

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 702737

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Perfluorododecanoic acid (PFDoA)	2.0	U	2.0	0.55	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorotridecanoic acid (PFTrDA)	2.0	U	2.0	1.3	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorotetradecanoic acid (PFTeA)	2.0	U	2.0	0.73	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorobutanesulfonic acid (PFBS)	2.0	U	2.0	0.20	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluoropentanesulfonic acid (PFPeS)	2.0	U	2.0	0.30	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorohexanesulfonic acid (PFHxS)	2.0	U	2.0	0.57	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluoroheptanesulfonic acid (PFHpS)	2.0	U	2.0	0.19	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorooctanesulfonic acid (PFOS)	2.0	U	2.0	0.54	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorononanesulfonic acid (PFNS)	2.0	U	2.0	0.37	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorodecanesulfonic acid (PFDS)	2.0	U	2.0	0.32	ng/L		08/30/23 12:20	09/01/23 12:46	1
Perfluorooctanesulfonamide (FOSA)	2.0	U	2.0	0.98	ng/L		08/30/23 12:20	09/01/23 12:46	1
NMeFOSAA	5.0	U	5.0	1.2	ng/L		08/30/23 12:20	09/01/23 12:46	1
NEtFOSAA	5.0	U	5.0	1.3	ng/L		08/30/23 12:20	09/01/23 12:46	1
4:2 FTS	2.0	U	2.0	0.24	ng/L		08/30/23 12:20	09/01/23 12:46	1
6:2 FTS	5.0	U	5.0	2.5	ng/L		08/30/23 12:20	09/01/23 12:46	1
8:2 FTS	2.0	U	2.0	0.46	ng/L		08/30/23 12:20	09/01/23 12:46	1
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	2.0	U	2.0	0.40	ng/L		08/30/23 12:20	09/01/23 12:46	1
HFPO-DA (GenX)	4.0	U	4.0	1.5	ng/L		08/30/23 12:20	09/01/23 12:46	1
9Cl-PF3ONS	2.0	U	2.0	0.24	ng/L		08/30/23 12:20	09/01/23 12:46	1
11Cl-PF3OUdS	2.0	U	2.0	0.32	ng/L		08/30/23 12:20	09/01/23 12:46	1

Isotope Dilution	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
13C4 PFBA	*85*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C5 PFPeA	*85*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C2 PFHxA	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C4 PFHpA	*78*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C4 PFOA	*82*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C5 PFNA	*84*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C2 PFDA	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C2 PFUnA	*74*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C2 PFDoA	*68*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C2 PFTeDA	*66*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C3 PFBS	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
18O2 PFHxS	*77*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C4 PFOS	*77*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C8 FOSA	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
d3-NMeFOSAA	*65*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
d5-NEtFOSAA	*72*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
M2-6:2 FTS	*76*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
M2-8:2 FTS	*79*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
M2-4:2 FTS	*81*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*
13C3 HFPO-DA	*66*		*25 - 150*	*08/30/23 12:20*	*09/01/23 12:46*	*1*

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: LCS 320-702737/2-A
Matrix: Water
Analysis Batch: 703301

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 702737

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec Limits
Perfluorobutanoic acid (PFBA)	40.0	42.2		ng/L		105	76 - 136
Perfluoropentanoic acid (PFPeA)	40.0	44.0		ng/L		110	71 - 131
Perfluorohexanoic acid (PFHxA)	40.0	46.0		ng/L		115	73 - 133
Perfluoroheptanoic acid (PFHpA)	40.0	43.0		ng/L		107	72 - 132
Perfluorooctanoic acid (PFOA)	40.0	41.9		ng/L		105	70 - 130
Perfluorononanoic acid (PFNA)	40.0	44.1		ng/L		110	75 - 135
Perfluorodecanoic acid (PFDA)	40.0	42.5		ng/L		106	76 - 136
Perfluoroundecanoic acid (PFUnA)	40.0	42.8		ng/L		107	68 - 128
Perfluorododecanoic acid (PFDoA)	40.0	44.4		ng/L		111	71 - 131
Perfluorotridecanoic acid (PFTrDA)	40.0	43.4		ng/L		108	71 - 131
Perfluorotetradecanoic acid (PFTeA)	40.0	44.9		ng/L		112	70 - 130
Perfluorobutanesulfonic acid (PFBS)	35.5	39.4		ng/L		111	67 - 127
Perfluoropentanesulfonic acid (PFPeS)	37.6	38.8		ng/L		103	66 - 126
Perfluorohexanesulfonic acid (PFHxS)	36.5	36.5		ng/L		100	59 - 119
Perfluoroheptanesulfonic acid (PFHpS)	38.2	41.5		ng/L		109	76 - 136
Perfluorooctanesulfonic acid (PFOS)	37.2	39.4		ng/L		106	70 - 130
Perfluorononanesulfonic acid (PFNS)	38.5	39.5		ng/L		103	75 - 135
Perfluorodecanesulfonic acid (PFDS)	38.6	36.3		ng/L		94	71 - 131
Perfluorooctanesulfonamide (FOSA)	40.0	40.6		ng/L		101	73 - 133
NMeFOSAA	40.0	42.7		ng/L		107	76 - 136
NEtFOSAA	40.0	40.7		ng/L		102	76 - 136
4:2 FTS	37.5	41.3		ng/L		110	79 - 139
6:2 FTS	38.1	40.4		ng/L		106	59 - 175
8:2 FTS	38.4	45.2		ng/L		118	75 - 135
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	37.8	40.2		ng/L		106	79 - 139
HFPO-DA (GenX)	40.0	44.3		ng/L		111	51 - 173
9Cl-PF3ONS	37.4	41.0		ng/L		110	75 - 135
11Cl-PF3OUdS	37.8	35.1		ng/L		93	54 - 114

Isotope Dilution	*LCS %Recovery*	*LCS Qualifier*	*Limits*
13C4 PFBA	*90*		*25 - 150*
13C5 PFPeA	*89*		*25 - 150*
13C2 PFHxA	*84*		*25 - 150*
13C4 PFHpA	*80*		*25 - 150*
13C4 PFOA	*86*		*25 - 150*
13C5 PFNA	*86*		*25 - 150*
13C2 PFDA	*85*		*25 - 150*
13C2 PFUnA	*77*		*25 - 150*
13C2 PFDoA	*72*		*25 - 150*

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: LCS 320-702737/2-A
Matrix: Water
Analysis Batch: 703301

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 702737

Isotope Dilution	LCS %Recovery	LCS Qualifier	Limits
13C2 PFTeDA	69		25 - 150
13C3 PFBS	84		25 - 150
18O2 PFHxS	83		25 - 150
13C4 PFOS	82		25 - 150
13C8 FOSA	84		25 - 150
d3-NMeFOSAA	80		25 - 150
d5-NEtFOSAA	76		25 - 150
M2-6:2 FTS	83		25 - 150
M2-8:2 FTS	85		25 - 150
M2-4:2 FTS	85		25 - 150
13C3 HFPO-DA	69		25 - 150

Lab Sample ID: 240-190457-5 MS
Matrix: Water
Analysis Batch: 703301

Client Sample ID: GW-11208041-081823-BW-005
Prep Type: Total/NA
Prep Batch: 702737

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec Limits
Perfluorobutanoic acid (PFBA)	11		38.7	52.5		ng/L		107	76 - 136
Perfluoropentanoic acid (PFPeA)	1.8	J	38.7	45.2		ng/L		112	71 - 131
Perfluorohexanoic acid (PFHxA)	2.4		38.7	46.0		ng/L		113	73 - 133
Perfluoroheptanoic acid (PFHpA)	0.93	J	38.7	42.4		ng/L		107	72 - 132
Perfluorooctanoic acid (PFOA)	5.1		38.7	44.2		ng/L		101	70 - 130
Perfluorononanoic acid (PFNA)	1.9	U	38.7	44.2		ng/L		114	75 - 135
Perfluorodecanoic acid (PFDA)	1.9	U	38.7	38.4		ng/L		99	76 - 136
Perfluoroundecanoic acid (PFUnA)	1.9	U	38.7	40.5		ng/L		105	68 - 128
Perfluorododecanoic acid (PFDoA)	1.9	U	38.7	43.9		ng/L		113	71 - 131
Perfluorotridecanoic acid (PFTrDA)	1.9	U	38.7	40.1		ng/L		104	71 - 131
Perfluorotetradecanoic acid (PFTeA)	1.9	U	38.7	41.2		ng/L		107	70 - 130
Perfluorobutanesulfonic acid (PFBS)	0.80	J	34.4	38.1		ng/L		109	67 - 127
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	36.4	40.3		ng/L		111	66 - 126
Perfluorohexanesulfonic acid (PFHxS)	1.2	J	35.3	37.4		ng/L		106	59 - 119
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	36.9	40.2		ng/L		109	76 - 136
Perfluorooctanesulfonic acid (PFOS)	12		36.0	49.5		ng/L		103	70 - 130
Perfluorononanesulfonic acid (PFNS)	1.9	U	37.3	36.0		ng/L		96	75 - 135
Perfluorodecanesulfonic acid (PFDS)	1.9	U	37.3	35.2		ng/L		94	71 - 131
Perfluorooctanesulfonamide (FOSA)	1.9	U	38.7	43.2		ng/L		112	73 - 133
NMeFOSAA	4.8	U	38.7	40.4		ng/L		104	76 - 136
NEtFOSAA	4.8	U	38.7	39.6		ng/L		102	76 - 136
4:2 FTS	1.9	U	36.3	40.0		ng/L		110	79 - 139
6:2 FTS	4.8	U	36.9	40.3		ng/L		109	59 - 175

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: 240-190457-5 MS
Matrix: Water
Analysis Batch: 703301

Client Sample ID: GW-11208041-081823-BW-005
Prep Type: Total/NA
Prep Batch: 702737

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec Limits
8:2 FTS	1.9	U	37.2	44.6		ng/L		120	75 - 135
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	36.6	41.2		ng/L		113	79 - 139
HFPO-DA (GenX)	3.9	U	38.7	43.4		ng/L		112	51 - 173
9Cl-PF3ONS	1.9	U	36.2	39.6		ng/L		110	75 - 135
11Cl-PF3OUdS	1.9	U	36.6	33.6		ng/L		92	54 - 114

Isotope Dilution	*MS %Recovery*	*MS Qualifier*	*Limits*
13C4 PFBA	*66*		*25 - 150*
13C5 PFPeA	*88*		*25 - 150*
13C2 PFHxA	*92*		*25 - 150*
13C4 PFHpA	*89*		*25 - 150*
13C4 PFOA	*93*		*25 - 150*
13C5 PFNA	*92*		*25 - 150*
13C2 PFDA	*91*		*25 - 150*
13C2 PFUnA	*81*		*25 - 150*
13C2 PFDoA	*77*		*25 - 150*
13C2 PFTeDA	*66*		*25 - 150*
13C3 PFBS	*92*		*25 - 150*
18O2 PFHxS	*88*		*25 - 150*
13C4 PFOS	*87*		*25 - 150*
13C8 FOSA	*87*		*25 - 150*
d3-NMeFOSAA	*77*		*25 - 150*
d5-NEtFOSAA	*82*		*25 - 150*
M2-6:2 FTS	*83*		*25 - 150*
M2-8:2 FTS	*87*		*25 - 150*
M2-4:2 FTS	*84*		*25 - 150*
13C3 HFPO-DA	*78*		*25 - 150*

Lab Sample ID: 240-190457-5 MSD
Matrix: Water
Analysis Batch: 703301

Client Sample ID: GW-11208041-081823-BW-005
Prep Type: Total/NA
Prep Batch: 702737

Analyte	Sample Result	Sample Qualifier	Spike Added	MSD Result	MSD Qualifier	Unit	D	%Rec	%Rec Limits	RPD	RPD Limit
Perfluorobutanoic acid (PFBA)	11		39.5	56.8		ng/L		115	76 - 136	8	30
Perfluoropentanoic acid (PFPeA)	1.8	J	39.5	45.5		ng/L		110	71 - 131	1	30
Perfluorohexanoic acid (PFHxA)	2.4		39.5	48.3		ng/L		116	73 - 133	5	30
Perfluoroheptanoic acid (PFHpA)	0.93	J	39.5	43.4		ng/L		108	72 - 132	2	30
Perfluorooctanoic acid (PFOA)	5.1		39.5	47.6		ng/L		107	70 - 130	7	30
Perfluorononanoic acid (PFNA)	1.9	U	39.5	44.6		ng/L		113	75 - 135	1	30
Perfluorodecanoic acid (PFDA)	1.9	U	39.5	42.4		ng/L		107	76 - 136	10	30
Perfluoroundecanoic acid (PFUnA)	1.9	U	39.5	45.1		ng/L		114	68 - 128	11	30
Perfluorododecanoic acid (PFDoA)	1.9	U	39.5	44.5		ng/L		112	71 - 131	1	30
Perfluorotridecanoic acid (PFTrDA)	1.9	U	39.5	42.9		ng/L		108	71 - 131	7	30
Perfluorotetradecanoic acid (PFTeA)	1.9	U	39.5	44.5		ng/L		113	70 - 130	8	30

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Lab Sample ID: 240-190457-5 MSD
Matrix: Water
Analysis Batch: 703301

Client Sample ID: GW-11208041-081823-BW-005
Prep Type: Total/NA
Prep Batch: 702737

Analyte	Sample Result	Sample Qualifier	Spike Added	MSD Result	MSD Qualifier	Unit	D	%Rec	%Rec Limits	RPD	RPD Limit
Perfluorobutanesulfonic acid (PFBS)	0.80	J	35.1	38.3		ng/L		107	67 - 127	1	30
Perfluoropentanesulfonic acid (PFPeS)	1.9	U	37.2	40.9		ng/L		110	66 - 126	2	30
Perfluorohexanesulfonic acid (PFHxS)	1.2	J	36.1	37.7		ng/L		105	59 - 119	1	30
Perfluoroheptanesulfonic acid (PFHpS)	1.9	U	37.7	42.5		ng/L		113	76 - 136	6	30
Perfluorooctanesulfonic acid (PFOS)	12		36.8	52.5		ng/L		109	70 - 130	6	30
Perfluorononanesulfonic acid (PFNS)	1.9	U	38.0	40.2		ng/L		106	75 - 135	11	30
Perfluorodecanesulfonic acid (PFDS)	1.9	U	38.1	37.6		ng/L		99	71 - 131	7	30
Perfluorooctanesulfonamide (FOSA)	1.9	U	39.5	40.8		ng/L		103	73 - 133	6	30
NMeFOSAA	4.8	U	39.5	42.0		ng/L		106	76 - 136	4	30
NEtFOSAA	4.8	U	39.5	41.1		ng/L		104	76 - 136	4	30
4:2 FTS	1.9	U	37.1	39.5		ng/L		106	79 - 139	1	30
6:2 FTS	4.8	U	37.6	43.8		ng/L		116	59 - 175	8	30
8:2 FTS	1.9	U	38.0	46.0		ng/L		121	75 - 135	3	30
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	1.9	U	37.4	43.3		ng/L		116	79 - 139	5	30
HFPO-DA (GenX)	3.9	U	39.5	47.2		ng/L		119	51 - 173	8	30
9Cl-PF3ONS	1.9	U	36.9	42.8		ng/L		116	75 - 135	8	30
11Cl-PF3OUdS	1.9	U	37.3	37.2		ng/L		100	54 - 114	10	30

Isotope Dilution	*MSD %Recovery*	*MSD Qualifier*	*Limits*
13C4 PFBA	*64*		*25 - 150*
13C5 PFPeA	*85*		*25 - 150*
13C2 PFHxA	*92*		*25 - 150*
13C4 PFHpA	*90*		*25 - 150*
13C4 PFOA	*89*		*25 - 150*
13C5 PFNA	*92*		*25 - 150*
13C2 PFDA	*90*		*25 - 150*
13C2 PFUnA	*77*		*25 - 150*
13C2 PFDoA	*80*		*25 - 150*
13C2 PFTeDA	*72*		*25 - 150*
13C3 PFBS	*95*		*25 - 150*
18O2 PFHxS	*90*		*25 - 150*
13C4 PFOS	*88*		*25 - 150*
13C8 FOSA	*94*		*25 - 150*
d3-NMeFOSAA	*83*		*25 - 150*
d5-NEtFOSAA	*82*		*25 - 150*
M2-6:2 FTS	*80*		*25 - 150*
M2-8:2 FTS	*86*		*25 - 150*
M2-4:2 FTS	*80*		*25 - 150*
13C3 HFPO-DA	*79*		*25 - 150*

Lab Chronicle

Client: GHD Services Inc.
Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

Client Sample ID: GW-11208041-081823-BW-001
Lab Sample ID: 240-190457-1
Date Collected: 08/18/23 10:37
Matrix: Water
Date Received: 08/19/23 09:30

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			680885	ER	EET BUF	08/22/23 11:43
Total/NA	Analysis	8270D SIM ID		1	680964	JMM	EET BUF	08/23/23 15:39
Total/NA	Prep	3535			702737	BLR	EET SAC	08/30/23 12:20
Total/NA	Analysis	537 (modified)		1	703301	K1S	EET SAC	09/01/23 13:09

Client Sample ID: GW-11208041-081823-BW-002
Lab Sample ID: 240-190457-2
Date Collected: 08/18/23 10:44
Matrix: Water
Date Received: 08/19/23 09:30

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3535			702737	BLR	EET SAC	08/30/23 12:20
Total/NA	Analysis	537 (modified)		1	703301	K1S	EET SAC	09/01/23 13:20

Client Sample ID: GW-11208041-081823-BW-003
Lab Sample ID: 240-190457-3
Date Collected: 08/18/23 10:53
Matrix: Water
Date Received: 08/19/23 09:30

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			680885	ER	EET BUF	08/22/23 11:43
Total/NA	Analysis	8270D SIM ID		1	680964	JMM	EET BUF	08/23/23 15:56
Total/NA	Prep	3535			702737	BLR	EET SAC	08/30/23 12:20
Total/NA	Analysis	537 (modified)		1	703301	K1S	EET SAC	09/01/23 13:32

Client Sample ID: GW-11208041-081823-BW-004
Lab Sample ID: 240-190457-4
Date Collected: 08/18/23 12:11
Matrix: Water
Date Received: 08/19/23 09:30

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			680885	ER	EET BUF	08/22/23 11:43
Total/NA	Analysis	8270D SIM ID		1	680964	JMM	EET BUF	08/23/23 16:13
Total/NA	Prep	3535			702737	BLR	EET SAC	08/30/23 12:20
Total/NA	Analysis	537 (modified)		1	703301	K1S	EET SAC	09/01/23 13:43

Client Sample ID: GW-11208041-081823-BW-005
Lab Sample ID: 240-190457-5
Date Collected: 08/18/23 13:30
Matrix: Water
Date Received: 08/19/23 09:30

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3510C			680885	ER	EET BUF	08/22/23 11:43
Total/NA	Analysis	8270D SIM ID		1	680964	JMM	EET BUF	08/23/23 12:30
Total/NA	Prep	3535			702737	BLR	EET SAC	08/30/23 12:20
Total/NA	Analysis	537 (modified)		1	703301	K1S	EET SAC	09/01/23 13:54

Lab Chronicle

Client: GHD Services Inc.
Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

Client Sample ID: TRIP BLANK
Lab Sample ID: 240-190457-6
Date Collected: 08/18/23 00:00
Matrix: Water
Date Received: 08/19/23 09:30

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3535			702737	BLR	EET SAC	08/30/23 12:20
Total/NA	Analysis	537 (modified)		1	703301	K1S	EET SAC	09/01/23 14:28

Laboratory References:

EET BUF = Eurofins Buffalo, 10 Hazelwood Drive, Amherst, NY 14228-2298, TEL (716)691-2600

EET SAC = Eurofins Sacramento, 880 Riverside Parkway, West Sacramento, CA 95605, TEL (916)373-5600

Accreditation/Certification Summary

Client: GHD Services Inc.
Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

Laboratory: Eurofins Buffalo

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Arkansas DEQ	State	88-0686	07-06-23 *
Connecticut	State	PH-0568	03-31-24
Florida	NELAP	E87672	06-30-23 *
Georgia	State	10026 (NY)	03-31-24
Georgia	State Program	N/A	03-31-09 *
Illinois	NELAP	200003	09-30-23
Iowa	State	374	03-01-25
Iowa	State Program	374	03-01-09 *
Kansas	NELAP	E-10187	02-01-24
Kentucky (DW)	State	90029	01-01-24
Kentucky (UST)	State	108092	04-01-24
Kentucky (WW)	State	KY90029	12-31-23
Louisiana	NELAP	02031	06-30-23 *
Louisiana (All)	NELAP	02031	06-30-23 *
Maine	State	NY00044	12-04-24
Maryland	State	294	06-30-24
Massachusetts	State	M-NY044	07-01-24
Michigan	State	9937	04-01-24
Michigan	State Program	9937	04-01-09 *
New Hampshire	NELAP	2973	09-11-19 *
New Hampshire	NELAP	2337	11-17-23
New Jersey	NELAP	NY455	06-30-24
New York	NELAP	10026	03-31-24
Pennsylvania	NELAP	68-00281	08-31-24
Rhode Island	State	LAO00328	12-30-23
Texas	NELAP	T104704412-18-10	07-31-23 *
USDA	US Federal Programs	P330-18-00039	03-25-24
Virginia	NELAP	460185	09-14-23
Washington	State	C784	02-10-24
Wisconsin	State	998310390	08-29-23

Laboratory: Eurofins Sacramento

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Alaska (UST)	State	17-020	02-20-24
ANAB	Dept. of Defense ELAP	L2468	01-20-24
ANAB	Dept. of Energy	L2468.01	01-20-24
ANAB	ISO/IEC 17025	L2468	01-20-24
Arizona	State	AZ0708	08-11-24
Arkansas DEQ	State	88-0691	05-18-24
California	State	2897	01-22-24
Colorado	State	CA0004	08-31-24
Florida	NELAP	E87570	06-30-24
Georgia	State	4040	01-29-24
Hawaii	State	<cert No.>	01-29-24
Illinois	NELAP	200060	03-17-24
Kansas	NELAP	E-10375	10-31-23
Louisiana (All)	NELAP	01944	06-30-24
Maine	State	CA00004	04-14-24

* Accreditation/Certification renewal pending - accreditation/certification considered valid.

Accreditation/Certification Summary

Client: GHD Services Inc. Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

Laboratory: Eurofins Sacramento (Continued)

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	Identification Number	Expiration Date
Michigan	State	9947	01-31-24
Nevada	State	CA00044	07-31-24
New Hampshire	NELAP	2997	04-18-24
New Jersey	NELAP	CA005	06-30-24
New York	NELAP	11666	04-01-24
Ohio	State	41252	01-29-24
Oregon	NELAP	4040	01-29-24
Texas	NELAP	T104704399-19-13	05-31-24
US Fish & Wildlife	US Federal Programs	58448	04-30-24
USDA	US Federal Programs	P330-18-00239	02-28-26
Utah	NELAP	CA000442021-12	02-28-24
Virginia	NELAP	460278	03-14-24
Washington	State	C581	05-05-24
West Virginia (DW)	State	9930C	12-31-23
Wisconsin	State	998204680	08-31-23 *
Wyoming	State Program	8TMS-L	01-28-19 *

* Accreditation/Certification renewal pending - accreditation/certification considered valid.

Eurofins Cleveland
180 S. Van Buren Avenue
Barberton, OH 44203
Phone: 330-497-9396 Fax: 330-497-0772

MICHIGAN 190

Chain of Custody Record

13/1.1

eurofins | Environment Testing

Y1

Client Information	Sampler: BMW	Lab PM: Heckler, Denise D	Carrier Tracking No(s):	COC No: 240-111150-39625.1
Client Contact: Ms. Ruth Mickle	Phone: 7342609865	E-Mail: Denise.Heckler@et.eurofinsus.com	State of Origin:	Page: Page 1 of 2
Company: GHD Services Inc.	PWSID:			Job #:
Address: 26850 Haggerty Rd.	Due Date Requested:			
City: Farmington Hills	TAT Requested (days):			
State, Zip: MI, 48331	Compliance Project: Δ Yes Δ No			
Phone: 612-524-6872(Tel)	PO #: Purchase Order Requested			
Email: ruth.mickle@ghd.com	WO #: 58502			
Project Name: 11208041, RACER Nodular Iron	Project #: 24006032			
Site:	SSOW#:			

Analysis Requested

Preservation Codes:

A - HCL, B - NaOH, C - Zn Acetate, D - Nitric Acid, E - NaHSO4, F - MeOH, G - Amchlor, H - Ascorbic Acid, I - Ice, J - DI Water, K - EDTA, L - EDA, M - Hexane, N - None, O - AsNaO2, P - Na2O4S, Q - Na2SO3, R - Na2S2O3, S - H2SO4, T - TSP Dodecahydrate, U - Acetone, V - MCAA, W - pH 4-5, Y - Trizma, Z - other (specify)

Other:

Sample Identification	Sample Date	Sample Time	Sample Type (C=comp, G=grab)	Matrix (W=water, S=solid, O=waste/oil, BT=Tissue, A=Air)	Field Filtered Sample (Yes or No)	Perform MS/MSD (Yes or No)	PFC_IDA - PFAS, Standard List (28 Analytes)	8270D_SIM_MS_ID - 1,4-Dioxane	Total Number of containers	Special Instructions/Note:
			Preservation Code:				N	N		
GW-11208041-081823-BL-001	8.18.23	1037	G	Water			X	X		
002		1044		Water			X			
003		1053		Water			X	X		
004		1211		Water			X	X		
005		1330		Water			X	X		
Trip Blank	-	-	HQ	Water						
				Water						
				Water						
				Water						
				Water						
				Water						

240-190457 Chain of Custody

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☑ Unknown ☐ Radiological

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ Return To Client ☑ Disposal By Lab ☐ Archive For _____ Months

Deliverable Requested: I, II, III, IV, Other (specify)

Special Instructions/QC Requirements:

Empty Kit Relinquished by: Date: Time: Method of Shipment:

Relinquished by	Date/Time	Company	Received by	Date/Time	Company
BMW	8.18.23	GHD		8.19.23 9:30	
Relinquished by	Date/Time	Company	Received by	Date/Time	Company
Relinquished by	Date/Time	Company	Received by	Date/Time	Company

Custody Seals Intact: Δ Yes Δ No | Custody Seal No.: | Cooler Temperature(s) °C and Other Remarks:

Ver: 06/08/2021

9/9/2023

Eurofins – Cleveland Sample Receipt Form/Narrative **Login # :** _____
Barberton Facility

Client GHD Site Name _____ Cooler unpacked by: M. Lou

Cooler Received on 8.19.23 Opened on 8.19.23

FedEx: 1st Grd Exp UPS FAS Waypoint Client Drop Off Eurofins Courier Other _____

Receipt After-hours: Drop-off Date/Time _____ Storage Location _____

Eurofins Cooler # 22 Foam Box Client Cooler Box Other _____

Packing material used: Bubble Wrap Foam Plastic Bag None Other _____

COOLANT: Wet Ice Blue Ice Dry Ice Water None

1. Cooler temperature upon receipt ☐ See Multiple Cooler Form
IR GUN # 21 (CF -0.2 °C) Observed Cooler Temp. 1.3 °C Corrected Cooler Temp. 1.1 °C

2. Were tamper/custody seals on the outside of the cooler(s)? If Yes Quantity _____	Yes	No
-Were the seals on the outside of the cooler(s) signed & dated?	Yes	No NA
-Were tamper/custody seals on the bottle(s) or bottle kits (LLHg/MeHg)?	Yes	No
-Were tamper/custody seals intact and uncompromised?	Yes	No NA
3. Shippers' packing slip attached to the cooler(s)?	Yes	No
4. Did custody papers accompany the sample(s)?	Yes	No
5. Were the custody papers relinquished & signed in the appropriate place?	Yes	No
6. Was/were the person(s) who collected the samples clearly identified on the COC?	Yes	No
7. Did all bottles arrive in good condition (Unbroken)?	Yes	No
8. Could all bottle labels (ID/Date/Time) be reconciled with the COC?	Yes	No
9. For each sample, does the COC specify preservatives (Y/N), # of containers (Y/N), and sample type of grab/comp (Y/N)?		
10. Were correct bottle(s) used for the test(s) indicated?	Yes	No
11. Sufficient quantity received to perform indicated analyses?	Yes	No
12. Are these work share samples and all listed on the COC?	Yes	No
If yes, Questions 13-17 have been checked at the originating laboratory.		
13. Were all preserved sample(s) at the correct pH upon receipt?	Yes	No NA pH Strip Lot# HC312502
14. Were VOAs on the COC?	Yes	No
15. Were air bubbles >6 mm in any VOA vials? ● ← Larger than this.	Yes	No NA
16. Was a VOA trip blank present in the cooler(s)? Trip Blank Lot # _____	Yes	No
17. Was a LL Hg or Me Hg trip blank present? _____	Yes	No

Tests that are not checked for pH by Receiving:
VOAs
Oil and Grease
TOC

Contacted PM _____ Date _____ by _____ via Verbal Voice Mail Other

Concerning _____

18. CHAIN OF CUSTODY & SAMPLE DISCREPANCIES ☐ additional next page | Samples processed by:

_____
_____
_____
_____

19. SAMPLE CONDITION

Sample(s) _____ were received after the recommended holding time had expired.
Sample(s) _____ were received in a broken container.
Sample(s) _____ were received with bubble >6 mm in diameter. (Notify PM)

20. SAMPLE PRESERVATION

Sample(s) _____ were further preserved in the laboratory.
Time preserved: _____ Preservative(s) added/Lot number(s): _____

VOA Sample Preservation - Date/Time VOAs Frozen: _____

Eurofins Cleveland
180 S. Van Buren Avenue
Barberton, OH 44203
Phone: 330-497-9396 Fax 330-497-0772

Chain of Custody Record

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Client Information (Sub Contract Lab)	Sampler:	Lab PM: Heckler Denise D	Carrier Tracking No(s):	COC No: 240-172651 1
Client Contact: Shipping/Receiving	Phone:	E-Mail: Denise.Heckler@et.eurofinsus.com	State of Origin: Michigan	Page: Page 1 of 1
Company: Eurofins Environment Testing Northern Ca		Accreditations Required (See note):		Job #: 240-190457-1
Address: 880 Riverside Parkway	Due Date Requested: 9/18/2023			
City: West Sacramento	TAT Requested (days):			
State, Zip: CA, 95605				
Phone: 916-373-5600(Tel) 916-372-1059(Fax)	PO #:			
Email:	WO #:			
Project Name: 11208041 RACER Nodular Iron	Project #: 24006032			
Site:	SSOW#:			

Analysis Requested

Preservation Codes

A	HCL	M	Hexane
B	NaOH	N	None
C	Zn Acetate	O	AsNaO2
D	Nitric Acid	P	Na2O4S
E	NaHSO4	Q	Na2SO3
F	MeOH	R	Na2S2O3
G	Amchlor	S	H2SO4
H	Ascorbic Acid	T	TSP Dodecahydrate
I	Ice	U	Acetone
J	DI Water	V	MCAA
K	EDTA	W	pH 4-5
L	EDA	Y	Trizma
		Z	other (specify)

Other:

Sample Identification - Client ID (Lab ID)	Sample Date	Sample Time	Sample Type (C=comp, G=grab)	Matrix (W=water, S=solid, O=waste/oil, BT=Tissue, A=Air)	Field Filtered Sample (Yes or No)	Perform MS/MSD (Yes or No)	PFC_IDA/3535_PFC PFAS, Standard List (28 Analytes)	Total Number of containers	Special Instructions/Note
			Preservation Code:						
GW-11208041-081823-BW-001 (240-190457 1)	8/18/23	10:37 Eastern		Water			X	2	
GW 11208041-081823-BW-002 (240-190457-2)	8/18/23	10:44 Eastern		Water			X	2	
GW 11208041-081823-BW-003 (240-190457-3)	8/18/23	10:53 Eastern		Water			X	2	
GW 11208041-081823-BW-004 (240-190457-4)	8/18/23	12:11 Eastern		Water			X	2	
GW 11208041-081823-BW-005 (240-190457-5)	8/18/23	13:30 Eastern		Water			X	4	
GW 11208041-081823-BW-005 (240-190457-5MS)	8/18/23	13:30 Eastern	MS	Water			X	1	
GW 11208041-081823-BW-005 (240-190457-5MSD)	8/18/23	13:30 Eastern	MSD	Water			X	1	
TRIP BLANK (240-190457-6)	8/18/23	Eastern		Water			X	2	

Note: Since laboratory accreditations are subject to change, Eurofins Environment Testing North Central, LLC places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under chain-of-custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analysis/tests/matrix being analyzed, the samples must be shipped back to the Eurofins Environment Testing North Central, LLC laboratory or other instructions will be provided. Any changes to accreditation status should be brought to Eurofins Environment Testing North Central, LLC attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to Eurofins Environment Testing North Central, LLC.

Possible Hazard Identification *Unconfirmed*	***Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)*** ☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For* _____ *Months*
Deliverable Requested I II, III, IV Other (specify) Primary Deliverable Rank. 2	Special Instructions/QC Requirements.

Empty Kit Relinquished by:		Date:	Time		Method of Shipment:	
Relinquished by: Rachell Hacecke	Date/Time: 8/23 1110	Company EETNC	Received by: D.S. Morgan	Date/Time: 08/22/23 0930	Company EETSAC	
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company	
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company	

Custody Seals Intact: Δ Yes Δ No	Custody Seal No.
Cooler Temperature(s) °C and Other Remarks: 2.4°C	

9/9/2023

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Sacramento
Sample Receiving Notes

190457

Tracking # 654910920362

Job _____

SO / PO / FO / SAT / 2-Day / Ground / UPS / CDO / Courier
GSL / OnTrac / Goldstreak / USPS / Other_____

Use this form to record Sample Custody Seal Cooler Custody Seal Temperature & corrected Temperature & other observations.
File in the job folder with the COC.

Therm ID L11 Corr Factor (+ /) NA °C

Ice_____ Wet ✓ Gel _____ Other _____

Cooler Custody Seal _____

Cooler ID _____

Temp Observed 2.4 °C Corrected 2.4 °C
From Temp Blank ☐ Sample ☑

Opening/Processing The Shipment	Yes	No	NA
Cooler compromised/tampered with?	☐	☑	☐
Cooler Temperature is acceptable?	☑	☐	☐
Frozen samples show signs of thaw?	☐	☐	☑

Initials DM Date 08/22/23

Unpacking/Labeling The Samples	Yes	No	NA
Containers are not broken or leaking?	☑	☐	☐
Samples compromised/tampered with?	☐	☑	☐
COC is complete w/o discrepancies	☑	☐	☐
Sample custody seal?	☐	☐	☑
Sample containers have legible labels?	☑	☐	☐
Sample date/times are provided?	☑	☐	☐
Appropriate containers are used?	☑	☐	☐
Sample bottles are completely filled?	☑	☐	☐
Sample preservatives verified?	☐	☐	☑
Is the Field Sampler's name on COC?	☐	☐	☑
Samples w/o discrepancies?	☑	☐	☐
Zero headspace?*	☐	☐	☑
Alkalinity has no headspace?	☐	☐	☑
Perchlorate has headspace? (Methods 314, 331 6850)	☐	☐	☑
Multiphasic samples are not present?	☑	☐	☐

Containers requiring zero headspace have no headspace, or bubble < 6 mm (1/4")

Initials DM Date 08/22/23

Notes _____

Trizma Lot #(s) _____

Ammonium
Acetate Lot #(s) _____

Login Completion	Yes	No	NA
Receipt Temperature on COC?	☑	☐	☐
NCM Filed?	☐	☐	☑
Samples received within hold time?	☐	☐	☑
Log Release checked in TALS?	☐	☐	☑

Initials DM Date 08/22/23

Eurofins Cleveland
180 S. Van Buren Avenue
Barberton, OH 44203
Phone: 330-497-9396 Fax: 330-497-0772

Chain of Custody Record

eurofins | Environment Testing

Client Information (Sub Contract Lab)	Sampler:	Lab PM: Heckler, Denise D	Carrier Tracking No(s):	COC No: 240-172647.1
Client Contact: Shipping/Receiving	Phone:	E-Mail: Denise.Heckler@et.eurofinsus.com	State of Origin: Michigan	Page: Page 1 of 1
Company: Eurofins Environment Testing Northeast,		Accreditations Required (See note):		Job #: 240-190457-1
Address: 10 Hazelwood Drive, ,	Due Date Requested: 9/18/2023			
City: Amherst	TAT Requested (days):			
State, Zip: NY, 14228-2298				
Phone: 716-691-2600(Tel) 716-691-7991(Fax)	PO #:			
Email:	WO #:			
Project Name: 11208041, RACER Nodular Iron	Project #: 24006032			
Site:	SSOW#:			

Analysis Requested

Preservation Codes:
A - HCL, B - NaOH, C - Zn Acetate, D - Nitric Acid, E - NaHSO4, F - MeOH, G - Amchlor, H - Ascorbic Acid, I - Ice, J - DI Water, K - EDTA, L - EDA, M - Hexane, N - None, O - AsNaO2, P - Na2O4S, Q - Na2SO3, R - Na2S2O3, S - H2SO4, T - TSP Dodecahydrate, U - Acetone, V - MCAA, W - pH 4-5, Y - Trizma, Z - other (specify)
Other:

Sample Identification - Client ID (Lab ID)	Sample Date	Sample Time	Sample Type (C=comp, G=grab)	Matrix (W=water, S=solid, O=waste/oil, BT=Tissue, A=Air)	Field Filtered Sample (Yes or No)	Perform MS/MSD (Yes or No)	8270D_SIM_MS_ID/3510C_LVI 1,4-Dioxane	Total Number of containers	Special Instructions/Note:
			Preservation Code:						
GW-11208041-081823-BW-001 (240-190457-1)	8/18/23	10:37 Eastern		Water			X	2	
GW-11208041-081823-BW-003 (240-190457-3)	8/18/23	10:53 Eastern		Water			X	2	
GW-11208041-081823-BW-004 (240-190457-4)	8/18/23	12:11 Eastern		Water			X	2	
GW-11208041-081823-BW-005 (240-190457-5)	8/18/23	13:30 Eastern		Water			X	4	
GW-11208041-081823-BW-005 (240-190457-5MS)	8/18/23	13:30 Eastern	MS	Water			X	1	
GW-11208041-081823-BW-005 (240-190457-5MSD)	8/18/23	13:30 Eastern	MSD	Water			X	1	

Note: Since laboratory accreditations are subject to change, Eurofins Environment Testing North Central, LLC places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under chain-of-custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analysis/tests/matrix being analyzed, the samples must be shipped back to the Eurofins Environment Testing North Central, LLC laboratory or other instructions will be provided. Any changes to accreditation status should be brought to Eurofins Environment Testing North Central, LLC attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to Eurofins Environment Testing North Central, LLC.

Possible Hazard Identification	Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
Unconfirmed	☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months
Deliverable Requested: I, II, III, IV, Other (specify) Primary Deliverable Rank: 2	Special Instructions/QC Requirements:

Empty Kit Relinquished by:	Date:	Time:	Method of Shipment:
Relinquished by: Rachael Herald	Date/Time: 8/23 1030	Company: EET NC	Received by:	Date/Time: 8-22-23 1030	Company: TAB
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company

Custody Seals Intact: Δ Yes Δ No	Custody Seal No.:	Cooler Temperature(s) °C and Other Remarks: 3.2 ICE

Login Sample Receipt Checklist

Client: GHD Services Inc.

Job Number: 240-190457-1

Login Number: 190457
List Number: 2
Creator: Yeager, Brian A

List Source: Eurofins Buffalo
List Creation: 08/22/23 11:31 AM

Question	Answer	Comment
Radioactivity either was not measured or, if measured, is at or below background	True	
The cooler's custody seal, if present, is intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	3.2 ice
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the sample IDs on the containers and the COC.	True	
Samples are received within Holding Time (Excluding tests with immediate HTs)..	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.	True	
If necessary, staff have been informed of any short hold time or quick TAT needs	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Sampling Company provided.	True	
Samples received within 48 hours of sampling.	True	
Samples requiring field filtration have been filtered in the field.	True	
Chlorine Residual checked.	True	

Login Sample Receipt Checklist

Client: GHD Services Inc.

Job Number: 240-190457-1

Login Number: 190457
List Number: 4
Creator: Schick, Robert J

List Source: Eurofins Buffalo
List Creation: 08/23/23 11:20 AM

Question	Answer	Comment
Radioactivity either was not measured or, if measured, is at or below background	True	
The cooler's custody seal, if present, is intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the sample IDs on the containers and the COC.	True	
Samples are received within Holding Time (Excluding tests with immediate HTs)..	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.	True	
If necessary, staff have been informed of any short hold time or quick TAT needs	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Sampling Company provided.	True	
Samples received within 48 hours of sampling.	True	
Samples requiring field filtration have been filtered in the field.	True	
Chlorine Residual checked.	True	

Login Sample Receipt Checklist

Client: GHD Services Inc.

Job Number: 240-190457-1

Login Number: 190457
List Number: 3
Creator: Simmons, Jason C

List Source: Eurofins Sacramento
List Creation: 08/22/23 02:37 PM

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	N/A	Not present
Sample custody seals, if present, are intact.	N/A	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	False	Water present in cooler; indicates evidence of melted ice.
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	2.4
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	N/A	Received project as a subcontract.
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time (excluding tests with immediate HTs)	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	N/A	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

Isotope Dilution Summary

Client: GHD Services Inc.
Project/Site: 11208041, RACER Nodular Iron

Job ID: 240-190457-1

Method: 8270D SIM ID - Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)

Matrix: Water **Prep Type: Total/NA**

		Percent Isotope Dilution Recovery (Acceptance Limits)
Lab Sample ID	Client Sample ID	DXE (15-110)
240-190457-1	GW-11208041-081823-BW-001	38
240-190457-3	GW-11208041-081823-BW-003	36
240-190457-4	GW-11208041-081823-BW-004	38
240-190457-5	GW-11208041-081823-BW-005	26
240-190457-5 MS	GW-11208041-081823-BW-005	27
240-190457-5 MSD	GW-11208041-081823-BW-005	26
LCS 480-680885/2-A	Lab Control Sample	39
MB 480-680885/1-A	Method Blank	42

Surrogate Legend

DXE = 1,4-Dioxane-d8

Method: 537 (modified) - Fluorinated Alkyl Substances

Matrix: Water **Prep Type: Total/NA**

		Percent Isotope Dilution Recovery (Acceptance Limits)							
Lab Sample ID	Client Sample ID	PFBA (25-150)	PFPeA (25-150)	PFHxA (25-150)	C4PFHA (25-150)	PFOA (25-150)	PFNA (25-150)	PFDA (25-150)	PFUnA (25-150)
240-190457-1	GW-11208041-081823-BW-001	42	52	59	59	59	61	58	49
240-190457-2	GW-11208041-081823-BW-002	99	95	92	90	91	96	96	85
240-190457-3	GW-11208041-081823-BW-003	94	91	86	82	87	95	84	85
240-190457-4	GW-11208041-081823-BW-004	70	86	92	91	87	90	86	83
240-190457-5	GW-11208041-081823-BW-005	59	77	81	77	80	81	79	72
240-190457-5 MS	GW-11208041-081823-BW-005	66	88	92	89	93	92	91	81
240-190457-5 MSD	GW-11208041-081823-BW-005	64	85	92	90	89	92	90	77
240-190457-6	TRIP BLANK	93	86	86	83	87	88	85	78
LCS 320-702737/2-A	Lab Control Sample	90	89	84	80	86	86	85	77
MB 320-702737/1-A	Method Blank	85	85	81	78	82	84	81	74

		Percent Isotope Dilution Recovery (Acceptance Limits)							
Lab Sample ID	Client Sample ID	PFDoA (25-150)	PFTDA (25-150)	C3PFBS (25-150)	PFHxS (25-150)	PFOS (25-150)	PFOSA (25-150)	d3NMFOS (25-150)	d5NEFOS (25-150)
240-190457-1	GW-11208041-081823-BW-001	44	41	60	60	56	55	50	49
240-190457-2	GW-11208041-081823-BW-002	83	80	95	90	90	94	82	89
240-190457-3	GW-11208041-081823-BW-003	75	77	87	86	84	90	79	76
240-190457-4	GW-11208041-081823-BW-004	81	79	95	93	88	94	82	87
240-190457-5	GW-11208041-081823-BW-005	68	54	81	79	73	79	72	76
240-190457-5 MS	GW-11208041-081823-BW-005	77	66	92	88	87	87	77	82
240-190457-5 MSD	GW-11208041-081823-BW-005	80	72	95	90	88	94	83	82
240-190457-6	TRIP BLANK	80	78	85	84	87	88	81	86
LCS 320-702737/2-A	Lab Control Sample	72	69	84	83	82	84	80	76
MB 320-702737/1-A	Method Blank	68	66	81	77	77	81	65	72

		Percent Isotope Dilution Recovery (Acceptance Limits)			
Lab Sample ID	Client Sample ID	M262FTS (25-150)	M282FTS (25-150)	M242FTS (25-150)	HFPODA (25-150)
240-190457-1	GW-11208041-081823-BW-001	53	57	60	52
240-190457-2	GW-11208041-081823-BW-002	89	92	92	77
240-190457-3	GW-11208041-081823-BW-003	86	78	91	71
240-190457-4	GW-11208041-081823-BW-004	79	90	78	81
240-190457-5	GW-11208041-081823-BW-005	69	81	72	70
240-190457-5 MS	GW-11208041-081823-BW-005	83	87	84	78

Isotope Dilution Summary

Client: GHD Services Inc. Job ID: 240-190457-1
Project/Site: 11208041, RACER Nodular Iron

Method: 537 (modified) - Fluorinated Alkyl Substances (Continued)

Matrix: Water **Prep Type: Total/NA**

		Percent Isotope Dilution Recovery (Acceptance Limits)			
Lab Sample ID	Client Sample ID	M262FTS (25-150)	M282FTS (25-150)	M242FTS (25-150)	HFPODA (25-150)
240-190457-5 MSD	GW-11208041-081823-BW-005	80	86	80	79
240-190457-6	TRIP BLANK	81	87	84	71
LCS 320-702737/2-A	Lab Control Sample	83	85	85	69
MB 320-702737/1-A	Method Blank	76	79	81	66

Surrogate Legend

PFBA = 13C4 PFBA
PFPeA = 13C5 PFPeA
PFHxA = 13C2 PFHxA
C4PFHA = 13C4 PFHpA
PFOA = 13C4 PFOA
PFNA = 13C5 PFNA
PFDA = 13C2 PFDA
PFUnA = 13C2 PFUnA
PFDoA = 13C2 PFDoA
PFTDA = 13C2 PFTeDA
C3PFBS = 13C3 PFBS
PFHxS = 18O2 PFHxS
PFOS = 13C4 PFOS
PFOSA = 13C8 FOSA
d3NMFOS = d3-NMeFOSAA
d5NEFOS = d5-NEtFOSAA
M262FTS = M2-6:2 FTS
M282FTS = M2-8:2 FTS
M242FTS = M2-4:2 FTS
HFPODA = 13C3 HFPO-DA

Appendix C
Data Validation Memo

Data Verification Report

September 13, 2023

To	John-Eric Pardys, GHD	**Project No.**	11208041
CC	Jessica Gallaway		
From	Ruth Mickle/mg/7	**Contact No.**	612--524-6872
Project Name	RACER Nodular	**Email**	Ruth.Mickle@ghd.com
Subject	Analytical Results and Data Verification PFAS and 1,4-Dioxane Groundwater Sampling RACER Nodular Site Saginaw, Michigan July and August 2023		

The services undertaken by GHD in connection with preparing this report were limited to those specifically detailed in the report and are subject to the scope limitations set out in the report.

1. Introduction

This document details a data verification of analytical results for samples collected for the perfluorinated and polyfluorinated alkyl substances (PFAS) and 1,4-Dioxane Groundwater Sampling at the RACER Nodular during July and August 2023. Samples were submitted to Eurofins Environment Testing (EET) located in Barberton, Ohio. The PFAS samples were transferred via inter-laboratory chain of custody and analyzed at EET's West Sacramento, California laboratory. The 1,4-dioxane were transferred via inter-laboratory chain of custody and analyzed at EET's Buffalo, New York laboratory. A sample collection and analysis summary is presented in Table 1. The validated analytical results are summarized in Table 2. A summary of the analytical methodology is presented in Table 3.

Standard GHD report deliverables were submitted by the laboratory. The final results and supporting quality assurance/quality control (QA/QC) data were assessed. Evaluation of the data was based on information obtained from the chain of custody forms, finished report forms, method blank data, duplicate data, recovery data from isotope dilution analytes/surrogate spikes/laboratory control samples (LCS)/matrix spikes (MS), and field QA/QC samples.

The QA/QC criteria by which these data have been assessed are outlined in the analytical methods referenced in Table 3 and applicable guidance from the document entitled:

1. "National Functional Guidelines for Organic Superfund Methods Data Review", United States Environmental Protection Agency (USEPA)-540-R-20-005, November 2020
2. "Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537", EPA 910-R-18-001, November 2018.

Items 1. and 2. will subsequently be referred to as the "Guidelines" in this Memorandum.

2. Sample Holding Time and Preservation

The sample holding time criteria for the analyses are summarized in Table 3. The sample chain of custody documents and analytical reports were used to determine sample holding times. The samples were prepared and analyzed within the required holding time.

All samples were properly preserved, delivered on ice, and stored by the laboratory at the required temperature (0-6°C).

3. Laboratory Method Blank Analyses

Method blanks are prepared from a purified matrix and analyzed with investigative samples to determine the existence and magnitude of sample contamination introduced during the analytical procedures.

For this study, laboratory method blanks were analyzed at a minimum frequency of 1 per 20 investigative samples and/or 1 per analytical batch.

The method blank results were non-detect, indicating laboratory contamination was not a factor for this investigation.

4. Isotope Dilution Analyte (IDA) Recoveries

IDA data were evaluated for all PFAS and 1,4-dioxane sample analyses. IDAs are isotopically labeled analogs of the analytes of interest added to the investigative and QC samples at the time of extraction. All results are then calculated as a ratio of the IDA responses.

The IDA recovery results for each sample were evaluated against the laboratory established recovery criteria. IDA recoveries must be within laboratory control limits. All isotope dilution analytes yielded acceptable recoveries.

5. Laboratory Control Sample Analyses

LCS or LCS/laboratory control sample duplicates (LCSD) are prepared and analyzed as samples to assess the analytical efficiencies of the methods employed, independent of sample matrix effects. The relative percent difference (RPD) of the LCS/LCSD recoveries is used to evaluate analytical precision.

For this study, LCS or LCS/LCSD were analyzed at a minimum frequency of 1 per 20 investigative samples and/or 1 per analytical batch.

The LCS/LCSD contained all compounds of interest. Most percent recoveries and RPDs were within the laboratory control limits. LCS/LCSD recoveries were assessed per the "Guidelines". All recoveries/RPDs were within the laboratory control limits or yielded recoveries above the control limits that did not result in qualification.

6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

To evaluate the effects of sample matrices on the preparation process, measurement procedures, and accuracy of a particular analysis, samples are spiked with a known concentration of the analyte of concern and analyzed as MS/MSD samples. The RPD between the MS and MSD is used to assess analytical precision. If the original sample concentration is significantly greater than the spike concentration, the recovery is not assessed. If only the MS or MSD recovery was outside of control limits, no qualification of the data was performed based on the acceptable recovery of the companion spike and the acceptable RPD.

MS/MSD analyses were performed on non-project samples.as specified in Table 1.

The MS/MSD samples were spiked with the compounds specified in the method. The percent recoveries and RPD values were within the laboratory control limits.

7. Field QA/QC Samples

The field QA/QC consisted of one trip blank sample, one equipment blank sample, one field blank sample, and one field duplicate sample set.

Trip Blank Sample Analysis

To evaluate contamination from sample collection, transportation, storage, and analytical activities, one trip blank sample was submitted to the laboratory for PFAS analysis. All results were non-detect for the compounds of interest.

Equipment Blank Sample Analysis

To assess field decontamination procedures, ambient conditions at the site, and cleanliness of sample containers, one equipment blank sample was submitted for analysis, as identified in Table 1. All results were non-detect for the analytes of interest.

Field Blank Sample Analysis

To assess ambient conditions at the site, one field blank sample was submitted for analysis, as identified in Table 1. All results were non-detect for the analytes of interest.

Field Duplicate Sample Analysis

To assess the analytical and sampling protocol precision, one field duplicate sample set was collected and submitted "blind" to the laboratory, as specified in Table 1. The RPDs associated with these duplicate samples must be less than 50 percent for water samples. If the reported concentration in either the investigative sample or its duplicate is less than five times the reporting limit (RL), the evaluation criteria is one times the RL value for water samples.

All field duplicate results met the above criteria demonstrating acceptable sampling and analytical precision.

8. Analyte Reporting

The laboratory reported detected results down to the laboratory's sample specific method detection limit (MDL) for each analyte. Positive analyte detections less than the reporting limit (RL) but greater than the MDL were reported as estimated (J) in Table 2 unless qualified otherwise in this memorandum. Non-detect results were presented as non-detect at the RL in Table 2.

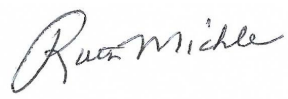
9. Target Compound Identification

To minimize erroneous compound identification during organic analyses, qualitative criteria including compound retention time and mass spectra were evaluated according to the identification criteria established by the method. The lab noted in the case narrative that several results had chromatographic interferences that could adversely impact the identification and quantitation of the PFAS target analyte. As a result, the associated detections were qualified estimated, as noted in Table 4.

10. Conclusion

Based on the assessment detailed in the foregoing, the data summarized in Table 2 are acceptable with the specific qualifications noted herein.

Regards,



Ruth Mickle
Digital Intelligence - Data Management Team Lead- Data Validator

Encl.

Table 1

Sample Collection and Analysis Summary
PFAS and 1,4-Dioxane Groundwater Sampling
RACER Nodular Site
Saginaw, Michigan
July and August 2023

Sample Identification	Location	Matrix	Collection Date (mm/dd/yyyy)	Collection Time (hr:min)	Analysis/Parameters: PFAS	Analysis/Parameters: 1,4-Dioxane	Comments
SDG No.: 240-188340-1							
GW-11208041-071023-BW-001	MW-05038	water	07/10/2023	14:57	X	X	
GW-11208041-071023-BW-002	MW-05038	water	07/10/2023	15:07	X	X	FD(MW-05038)
GW-11208041-071023-BW-003	MW-8R	water	07/10/2023	15:46	X	X	
GW-11208041-071123-BW-004	MW-04438R	water	07/11/2023	09:21	X	X	
GW-11208041-071123-BW-005	MW-04336	water	07/11/2023	10:40	X	X	
GW-11208041-071123-BW-006	MW-05036R	water	07/11/2023	12:05	X	X	
SDG No.: 240-190457-1							
GW-11208041-081823-BW-001	MW-05443	water	08/18/2023	10:37	X	X	
GW-11208041-081823-BW-002	MW-05443	water	08/18/2023	10:44	X		FB
GW-11208041-081823-BW-003	MW-05452	water	08/18/2023	10:53	X	X	EB
GW-11208041-081823-BW-004	MW-05452	water	08/18/2023	12:11	X	X	
GW-11208041-081823-BW-005	MW-05038	water	08/18/2023	13:30	X	X	MS/MSD
TRIP BLANK	Trip Blank	water	08/18/2023	--	X		TB

Notes:
EB - Equipment Blank
FB - Field Blank
FD - Field Duplicate sample of sample in parentheses
MS/MSD - Matrix Spike/Matrix Spike Duplicate
PFAS - Perfluorinated and Polyfluorinated Alkyl Substances
SDG - Sample Delivery Group
TB - Trip Blank

Table 2

Validated Analytical Results Summary
PFAS and 1,4-Dioxane Sampling
RACER Nodular Site
Saginaw, Michigan
July and August 2023

Location ID:		**MW-8R**	**MW-04336**	**MW-04438R**	**MW-05036R**
Sample Name:		**GW-11208041-071023-BW-003**	**GW-11208041-071123-BW-005**	**GW-11208041-071123-BW-004**	**GW-11208041-071123-BW-006**
Sample Date:		**07/10/2023**	**07/11/2023**	**07/11/2023**	**07/11/2023**
Parameters	**Unit**				
Semivolatile Organic Compounds, SIM					
1,4-Dioxane	µg/L	5.2	4.1	0.20 U	0.52
Per/Polyfluoroalkyl Substances					
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propoxy]-propanoic acid (DONA)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Fluorotelomer sulfonic acid (4:2)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Fluorotelomer sulfonic acid (6:2)	ng/L	4.7 U	4.8 U	4.8 U	5.1 U
Fluorotelomer sulfonic acid (8:2)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Hexafluoropropylene oxide dimer acid (HFPO-DA)	ng/L	3.8 U	3.8 U	3.9 U	4.1 U
N-Ethyl perfluorooctane sulfonamido acetic acid (N-EtFOSAA)	ng/L	4.7 U	4.8 U	4.8 U	5.1 U
N-Methyl perfluorooctane sulfonamido acetic acid	ng/L	4.7 U	4.8 U	4.8 U	5.1 U
Perfluorobutane sulfonic acid (PFBS)	ng/L	1.4 J	1.2 J	4.3	3.0
Perfluorobutanoic acid (PFBA)	ng/L	8.7	26	29	22
Perfluorodecanesulfonic acid (PFDS)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Perfluorodecanoic acid (PFDA)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Perfluorododecanoic acid (PFDoDA)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Perfluoroheptane sulfonic acid (PFHpS)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Perfluoroheptanoic acid (PFHpA)	ng/L	1.3 J	2.5	3.4	2.5
Perfluorohexane sulfonic acid (PFHxS)	ng/L	1.4 J	1.9	1.9 U	1.3 J
Perfluorohexanoic acid (PFHxA)	ng/L	1.7 J	4.8	3.2	3.2
Perfluorononane sulfonic acid (PFNS)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Perfluorononanoic acid (PFNA)	ng/L	0.86 J	1.9 U	0.41 J	0.60 J
Perfluorooctane sulfonamide (FOSA)	ng/L	1.9 U	1.9 U	1.9 U	1.1 J
Perfluorooctane sulfonic acid (PFOS)	ng/L	8.2	1.9 U	1.9 U	6.1
Perfluorooctanoic acid (PFOA)	ng/L	4.8	6.0	5.0	4.4
Perfluoropentane sulfonic acid (PFPeS)	ng/L	1.9 U	0.63 J	1.9 U	2.0 U
Perfluoropentanoic acid (PFPeA)	ng/L	1.9 U	4.2 J	2.7 J	2.0 U
Perfluorotetradecanoic acid (PFTeDA)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Perfluorotridecanoic acid (PFTrDA)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U
Perfluoroundecanoic acid (PFUnA)	ng/L	1.9 U	1.9 U	1.9 U	2.0 U

Notes:
U - Not detected at the assoicated reporting limit.
J - Estimated concentration.

Table 2

Validated Analytical Results Summary
PFAS and 1,4-Dioxane Sampling
RACER Nodular Site
Saginaw, Michigan
July and August 2023

Location ID:		**MW-05038**	**MW-05038**	**MW-05038**	**MW-05443**
Sample Name:		**GW-11208041-071023-BW-001**	**GW-11208041-071023-BW-002**	**GW-11208041-081823-BW-005**	**GW-11208041-081823-BW-001**
Sample Date:		**07/10/2023**	**07/10/2023**	**08/18/2023**	**08/18/2023**
			Duplicate		
Parameters	**Unit**				
Semivolatile Organic Compounds, SIM					
1,4-Dioxane	µg/L	3.9	4.1	2.8	2.3
Per/Polyfluoroalkyl Substances					
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propoxy]-propanoic acid (DONA)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Fluorotelomer sulfonic acid (4:2)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Fluorotelomer sulfonic acid (6:2)	ng/L	4.8 U	4.9 U	4.8 U	4.8 U
Fluorotelomer sulfonic acid (8:2)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Hexafluoropropylene oxide dimer acid (HFPO-DA)	ng/L	3.8 U	3.9 U	3.9 U	3.8 U
N-Ethyl perfluorooctane sulfonamido acetic acid (N-EtFOSAA)	ng/L	4.8 U	4.9 U	4.8 U	4.8 U
N-Methyl perfluorooctane sulfonamido acetic acid	ng/L	4.8 U	4.9 U	4.8 U	4.8 U
Perfluorobutane sulfonic acid (PFBS)	ng/L	0.69 J	0.84 J	0.80 J	1.9 U
Perfluorobutanoic acid (PFBA)	ng/L	11	10	11	8.5 J
Perfluorodecanesulfonic acid (PFDS)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluorodecanoic acid (PFDA)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluorododecanoic acid (PFDoDA)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluoroheptane sulfonic acid (PFHpS)	ng/L	0.27 J	0.28 J	1.9 U	1.9 U
Perfluoroheptanoic acid (PFHpA)	ng/L	1.1 J	1.1 J	0.93 J	1.9 U
Perfluorohexane sulfonic acid (PFHxS)	ng/L	1.3 J	1.2 J	1.2 J	1.9 U
Perfluorohexanoic acid (PFHxA)	ng/L	1.6 J	2.0 U	2.4	2.9
Perfluorononane sulfonic acid (PFNS)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluorononanoic acid (PFNA)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluorooctane sulfonamide (FOSA)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluorooctane sulfonic acid (PFOS)	ng/L	13	13	12	1.9 U
Perfluorooctanoic acid (PFOA)	ng/L	5.2	4.9	5.1	1.2 J
Perfluoropentane sulfonic acid (PFPeS)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluoropentanoic acid (PFPeA)	ng/L	1.9 U	1.2 J	1.8 J	2.8 J
Perfluorotetradecanoic acid (PFTeDA)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluorotridecanoic acid (PFTrDA)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U
Perfluoroundecanoic acid (PFUnA)	ng/L	1.9 U	2.0 U	1.9 U	1.9 U

Notes:
U - Not detected at the assoicated reporting limit.
J - Estimated concentration.

Validated Analytical Results Summary
PFAS and 1,4-Dioxane Sampling
RACER Nodular Site
Saginaw, Michigan
July and August 2023

Location ID:		**MW-05452**
Sample Name:		**GW-11208041-081823-BW-004**
Sample Date:		**08/18/2023**
Parameters	**Unit**	
Semivolatile Organic Compounds, SIM		
1,4-Dioxane	µg/L	0.96
Per/Polyfluoroalkyl Substances		
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	ng/L	2.0 U
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propoxy]-propanoic acid (DONA)	ng/L	2.0 U
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	ng/L	2.0 U
Fluorotelomer sulfonic acid (4:2)	ng/L	2.0 U
Fluorotelomer sulfonic acid (6:2)	ng/L	5.0 U
Fluorotelomer sulfonic acid (8:2)	ng/L	2.0 U
Hexafluoropropylene oxide dimer acid (HFPO-DA)	ng/L	4.0 U
N-Ethyl perfluorooctane sulfonamido acetic acid (N-EtFOSAA)	ng/L	5.0 U
N-Methyl perfluorooctane sulfonamido acetic acid	ng/L	5.0 U
Perfluorobutane sulfonic acid (PFBS)	ng/L	0.67 J
Perfluorobutanoic acid (PFBA)	ng/L	6.8 J
Perfluorodecanesulfonic acid (PFDS)	ng/L	2.0 U
Perfluorodecanoic acid (PFDA)	ng/L	2.0 U
Perfluorododecanoic acid (PFDoDA)	ng/L	2.0 U
Perfluoroheptane sulfonic acid (PFHpS)	ng/L	2.0 U
Perfluoroheptanoic acid (PFHpA)	ng/L	0.72 J
Perfluorohexane sulfonic acid (PFHxS)	ng/L	2.0 U
Perfluorohexanoic acid (PFHxA)	ng/L	1.7 J
Perfluorononane sulfonic acid (PFNS)	ng/L	2.0 U
Perfluorononanoic acid (PFNA)	ng/L	2.0 U
Perfluorooctane sulfonamide (FOSA)	ng/L	2.0 U
Perfluorooctane sulfonic acid (PFOS)	ng/L	4.4
Perfluorooctanoic acid (PFOA)	ng/L	3.8
Perfluoropentane sulfonic acid (PFPeS)	ng/L	2.0 U
Perfluoropentanoic acid (PFPeA)	ng/L	1.4 J
Perfluorotetradecanoic acid (PFTeDA)	ng/L	2.0 U
Perfluorotridecanoic acid (PFTrDA)	ng/L	2.0 U
Perfluoroundecanoic acid (PFUnA)	ng/L	2.0 U

Notes:
U - Not detected at the assoicated reporting limit.
J - Estimated concentration.

Table 3

Analytical Methods
PFAS and 1,4-Dioxane Groundwater Sampling
RACER Nodular Site
Saginaw, Michigan
July and August 2023

Parameter	Method	Matrix	Holding Time	
			Collection to Extraction (Days)	Collection or Extraction to Analysis (Days)
Perfluorinated and Polyfluorinated Alkyl Substances (PFAS)	EPA 537 Modified	Water	14	28
1,4-Dioxane	SW 846 8270D SIM	Water	7	40

Method References:
EPA - "Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)", EPA/600/R-18/352, Version 1.0, November 2018
SW - "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Third Edition, 1986, with subsequent revisions

Note:
SIM Selective Ion Monitoring

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Table 4

Qualified Sample Data Due to Chromatographic Interference Issues
PFAS and 1,4-Dioxane Groundwater Sampling
RACER Nodular Site
Saginaw, Michigan
July and August 2023

Parameter	Sample ID	Analyte	Qualified Result	Units
PFAS	GW-11208041-071123-BW-004	Perfluoropentanoic acid (PFPeA)	2.7 J	ng/L
	GW-11208041-071123-BW-005	Perfluoropentanoic acid (PFPeA)	4.2 J	ng/L
	GW-11208041-081823-BW-001	Perfluoropentanoic acid (PFPeA)	2.8 J	ng/L
	GW-11208041-081823-BW-004	Perfluoropentanoic acid (PFPeA)	1.4 J	ng/L
	GW-11208041-081823-BW-001	Perfluorobutanoic acid (PFBA)	8.5 J	ng/L
	GW-11208041-081823-BW-004	Perfluorobutanoic acid (PFBA)	6.8 J	ng/L
	GW-11208041-081823-BW-004	Perfluorobutane sulfonic acid (PFBS)	0.67 J	ng/L

Notes:
PFAS - Per- and Polyfluoroalkyl Substances
J - Estimated concentration