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SITE CHARACTERIZATION WORK PLAN 90-02 QUEENS BOULEVARD NYSDEC SITE NO. 241249 QUEENS, NEW YORK

by Haley & Aldrich of New York New York, New York

for 9002 Commercial Unit LLC New York, New York

File No. 0208933-001 May 2024





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03 May 2024 File No. 0208933

New York State Department of Environmental Conservation Division of Environmental Remediation 625 Broadway Albany, New York 12233

Attention: Mr. Manfred Magloire

Subject: Site Characterization Work Plan 90-02 Queens Boulevard NYSDEC Site 241249 Queens, New York 10034 (Site)

Dear Mr. Magloire,

Haley & Aldrich of New York, on behalf of 9002 Commercial Unit LLC, is submitting for the review and approval of the New York State Department of Environmental Conservation (NYSDEC) this Site Characterization Work Plan (SCWP) for the 90-02 Queens Boulevard Site, NYSDEC Site No. 241249, located at 90-02 Queens Boulevard in Queens, New York (Site). This SCWP has been developed based on the NYSDEC's "Technical Guidance for Site Investigation and Remediation" (DER-10 dated May 2010).

NYSDEC provided comments on the draft SCWP on 07 March, 23 April and 26 April 2024. Comments have been addressed as follows:

NYSDEC Comments:

- 1. **Clarify if the UST is located on site off offsite:** UST was formerly located on the Site and closed in place during the February 2020 Tank Abandonment work by Hydro Tech Environmental Corp.
- 2. What Does LUST and HREC Mean: An Acronym and Abbreviation table has been included.
- 3. **Explain How Soil Boring will be advanced/installed:** The existing concrete slab will be cored and a drill rod with an installed acetate liner will be advanced via the direct push method through the soil stratum. The text has been updated to reflect this.
- 4. **Co-Located soil sampling required at MW-14:** MW-14 has been updated to also include soil analysis.
- 5. **Prior to this please explain how the monitoring wells will be installed and developed:** The order of the text has been updated detailing the installation/development.

6. A third monitoring well is needed to determine groundwater flow during the sampling event: SB-05 has been added as a Monitoring Well.

NYSDOH Comments:

- 1. Verify Existing Site Use: The current Site use includes an interconnected mixed use commercial residential space which occupies approximately 90 percent of entire site footprint. Within the Site limits there are also two adjoining structures with separate entrances along Hoffman Drive. These structures are operating as a closed parking garage and a daycare facility. The adjoining property to the east (Lot 74) is an approximately 26,000 square foot lot. The adjoining property to the west (Lot 22) is improved with a one-story building with affixed parking lot operating as a food establishment.
- 2. Indoor Air Sampling Locations: Interior sub-slab vapor samples will be co-located with an indoor air sample and all sampling will take place over 8 hours.
- **3.** Building questionnaire and chemical inventory: A building questionnaire and chemical inventory will be completed during the sampling event.
- 4. Previous Data Validation: To our knowledge, previous data included in the report by Hydro Tech has not been validated. Data collected by Haley & Aldrich from the August 2023 Limited Phase II will be validated and presented in the Site Characterization Findings Report.
- **5. Community Air Monitoring Plan**: The generic CAMP will be implemented during all outdoor ground intrusive activities. Appendix 1A and 1B from DER-10 have been added as an appendix.
- 6. Include Soil Vapor Reference: Reference section updated to include Guidance for Evaluating Soil Vapor Intrusion in the State of New York.
- **7.** Figure 2: Included five additional SVI samples "to alleviate a potential data gap and characterize nature and extent," including in the daycare.
- 8. Figure 3: Identified sample locations on the figure where LNAPL was observed.
- **9.** Figure 5: Included outdoor ambient air sample from January 2014 sampling event and changed language to indicate AI-1 and AI-2 did not exceed background standards.



Please do not hesitate to contact us if there are any questions regarding this submittal or any other aspects of the project.

Sincerely yours, HALEY & ALDRICH OF NEW YORK

Belle amus James M. Bellew

Principal

Cc: Selim Sabbagh (9002 Commercial Unit LLC) Ralph Chera (9002 Commercial Unit LLC) Jane O'Connell (NYSDEC) Cris-Sandra Maycock (NYSDEC) David Freeman (Gibbons PC) Matthew Sinkman (Gibbons PC)

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Certification

I, James M. Bellew, certify that I am currently a Qualified Environmental Professional as defined in 6 NYCRR Part 375 and that that this Site Characterization Work Plan was prepared in accordance with the applicable statues and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10).

James M. Belle

James M. Bellew

5/03/2024

Date



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List of Acronyms and Abbreviations

A Alpha ASP AWQS	Alpha Analytical Laboratories, Inc. Analytical Services Protocol Ambient Water Quality Standards
B bgs BTEX	Below Ground Surface Benzene, Toluene, Ethylbenzene, Xylenes
C CAMP Coastal COCs CVOCs	Community Air Monitoring Plan Coastal Environmental Solutions, Inc. Contaminants of Concern Chlorinated Volatile Organic Compounds
D DER-10 DOT DUSR	Division of Environmental Remediation-10 (specifically "May 2010 NYSDEC Technical Guidance for Site Investigation and Remediation") Department of Transportation Data Usability Summary Report
E EDD EFR ELAP EPA ESA ESI	Electronic Data Deliverable Enhanced Fluid Recovery Environmental Laboratory Approval Program U.S. Environmental Protection Agency Environmental Site Assessment Environmental Site Investigation
F ft FSP	Feet Field Sampling Plan
G GPR GV	Ground Penetrating Radar Guidance Value
H Haley & Aldrich HASP HAZWOPER HREC Hydro Tech	Haley & Aldrich of New York Health and Safety Plan Hazardous Waste Operations and Emergency Response Historical Recognized Environmental Conditions Hydro Tech Environmental Corp



l IDW	Investigation Derived Waste
L L/min LNAPL LUST	Liters per Minute Light Non Aqueous Phase Liquid Leaking Underground Storage Tank
M MCL MS MSD MTBE MDL mg/kg mg/L MW	Maximum Concentration Limit Matrix Spike Matrix Spike Duplicate Methyl-tert-butyl-ether Method Detection Limit Milligrams per Kilogram Milligrams per Liter Monitoring Well
N NAPL/GCM NPV ng/g ng/L NTU NYCRR NYSDEC NYSDOH	Non-aqueous Phase Liquid/Grossly Contaminated Material Nelson Pope Voorhis Nanograms per Gram Nanograms per liter Nephelometric turbidity unit New York Codes, Rules and Regulations New York State Department of Environmental Conservation New York State Department of Health
O OSHA	Occupational Safety and Health Administration
P PAH PCB PCE PFAS PFOA PFOS PGWSCO PID PPB PPE PPE PPM PVC PQL	Polycyclic aromatic Hydrocarbon Polychlorinated Biphenyl Perchloroethylene/Tetrachloroethene Per- and Polyfluoroalkyl Substances Perfluorooctanoic Acid Perfluorooctanesulfonic Acid Protection of Groundwater Soil Cleanup Objectives Photoionization Detector Parts per Billion Personal Protective Equipment Parts per Million Polyvinyl Chloride Practical Quantitation Limit
Q QA/QC	Quality Assurance/Quality Control



QAPP QHHEA	Quality Assurance Project Plan Qualitative Human Health Exposure Assessment
R RA RAWP RCRA REC RI RIR RIWP RRSCOS RWP	Remedial Action Remedial Action Work Plan Resource Conservation and Recovery Act Recognized Environmental Condition Remedial Investigation Remedial Investigation Report Remedial Investigation Work Plan Restricted-Residential Soil Cleanup Objectives Remediation Work Plan
S SC SCO SCWP SIM Site SMP SVOC	Site Characterization Soil Cleanup Objective Site Characterization Work Plan Selective Ion Monitoring 90-02 Queens Bouelvard Site Management Plan Semi-Volatile Organic Compound
T TAL TCE TCL TCLP TOGS 1.1.1	Total Analyte List Trichloroethene Target Compound List Toxicity Characteristic Leachate Procedure Technical and Operational Guidance Series 1.1.1 <i>(Specifically "June 1998 NYSDEC Division of Water Technical and Operational Guidance Series 1.1.1 Ambient Water Quality Standards and Guidance Values, Class GA for the protection of a source of drinking water modified per the April 2000 addendum")</i>
U μg/kg μg/L μg/m ³ UST UUSCOs	Micrograms per Kilogram Micrograms per Liter Micrograms per Cubic Meter Underground Storage Tank Unrestricted Use Soil Cleanup Objectives
V VOCs	Volatile Organic Compounds



1. Introduction

On behalf of 9002 Commercial Unit LLC, Haley & Aldrich of New York (Haley & Aldrich) has prepared this Site Characterization Work Plan (SCWP) for the 90-02 Queens Boulevard Site located at 90-02 Queens Boulevard (see Figure 1), Queens, New York (Site). This SCWP was prepared in accordance with applicable NYSDEC regulations and guidance.

The Site, identified as Block 2857 Lot 36 on the New York City tax map, is located in the Elmhurst neighborhood of Queens and is comprised of one, approximately 54,500 square foot (sq ft) tax lot. The Site is bound by Queens Boulevard followed by commercial and office buildings to the north; Hoffman Drive followed by a parking facility, a park, and multi-family walk-up residential buildings to the south; a vacant lot to the east; and commercial and office buildings to the west. The Site location is shown in Figure 1. Existing Site features are shown in Figure 2.

The Site is zoned in a residential (R6) zoning district with a C1-2 commercial overlay.

1.1 PURPOSE

Previous investigations conducted at the Site identified the presence of light non-aqueous phase liquid (LNAPL) and elevated concentrations of volatile organic compounds (VOCs) and chlorinated volatile organic compounds (CVOCs) in groundwater and soil vapor at the Site. More specifically, previous investigations related to Spill Case No. 1504881 revealed LNAPL in several permanent groundwater monitoring wells near a closed-in-place underground storage tank (UST). Additionally, petroleum-related VOC and CVOC, including trichloroethene (TCE) and tetrachloroethene (PCE), concentrations in groundwater exceed the New York Technical and Operational Guidance Series (T.O.G.S.) 1.1.1 Class GA Ambient Water Quality Standards (AWQS). Based on a meeting with the NYSDEC on 20 September 2023, due to the elevated VOC concentrations in groundwater at the Site, additional investigation is required in order to understand the extent of contamination on the Site, evaluate potential on-Site sources, and identify remedial options. A summary of the historical soil, groundwater, and soil vapor analytical data collected at the Site is displayed in Figures 3, 4, and 5.



2. Background

2.1 CURRENT LAND USE

The Site is currently developed with a six-story mixed use building. The first two floors of the majority of the building are undergoing renovation for future use as retail and/or office use. The remaining floors are condominiums and are currently occupied.

2.2 SITE HISTORY

According to available historical records, the Site was reportedly developed in the late 1800s with a singlestory building. Between 1902 and 2006, both lots were developed with various one- to two-story sheds, garages, one- to two-story dwellings, one-story buildings, and two-story stores with unlisted uses. In 1950, the eastern portion of Lot 36 was developed with the Horace Harding Hospital. By 1962, Lot 36 was developed with the six-story St. Johns Queens Hospital, which operated until at least 2006. Lot 16 was utilized for parking between 1986 and 2006.

2.3 SURROUNDING LAND USE

The Site is bound by Queens Boulevard followed by commercial and office buildings to the north; Hoffman Drive followed by a parking facility, a park, and multi-family walk-up residential buildings to the south; a vacant lot to the east; and commercial and office buildings to the west. The Site is located within a mixed-use area characterized by commercial, residential, and office buildings. No hospitals are located within a 500 ft radius of the Site.

2.4 GEOLOGY AND HYDROGEOLOGY

According to the Phase I ESA conducted by Hydro Tech Environmental, Corp. (Hydro Tech) dated December 2013, the major landforms of importance to the Site's hydrologic system are the moraines and outwash plains, which originated from glacial activity. The moraines represent the farthest extent of the glacial advances. The moraines consist of till, which is a poorly sorted mixture of sand, silt, clay, gravel and boulders. The till is poor to moderately permeable in most areas. Outwash plains are located to the south of the moraines. The outwash plains were formed by the action of glacial melt water streams, which eroded the headland material of the moraines and laid down deposits of well-sorted sands, silts and gravels. These outwash deposits have a moderate to high permeability.

During the August 2023 Site investigation, fill material generally consisting of light gray to dark brown sand and clay with varying amounts of silt and gravel from surface grade to approximately 6 feet below ground surface (ft bgs). The fill layer was underlain by gray to brown sand extending to at least 9 ft bgs, the maximum depth of exploration.

The Site has slopping topography from a high to the east and lower to the west and a topographic elevation of approximately 25 feet above mean sea level (ft amsl).



Based on a January 2014 Phase II Environmental Site Assessment (ESA) by Hydro Tech, groundwater flow beneath the Site is generally from the west-southwest to east-northeast. During the August 2023 Site investigation, groundwater was encountered at depths ranging from approximately 3 to 6 ft bgs.

2.5 **PREVIOUS INVESTIGATIONS**

To date the following investigations have been performed at the Site:

- 1. 27 December 2013, Phase I Environmental Site Assessment Report, prepared by Hydro Tech.
- 2. 14 January 2014, Phase II Environmental Site Assessment, prepared by Hydro Tech
- 3. 5 August 2015, Focused Subsurface Investigation, prepared by Hydro Tech
- 4. 15 September 2015, Subsurface Investigation Report, prepared by Hydro Tech
- 5. 27 March 2017, Focused Subsurface Investigation, prepared by Hydro Tech
- 6. 17 February 2020, Documentation of Tank Abandonment and Groundwater Quality, prepared by Hydro Tech
- 7. 7 May 2020, Enhanced Fluid Recovery (EFR) Results February and March 2020, prepared by Hydro Tech
- 8. 26 January 2023, Phase I Environmental Site Assessment, prepared by Nelson, Pope & Voorhis, LLC.
- 9. 21 August 2023, Limited Phase II Environmental Site Investigation Report, prepared by Haley & Aldrich of New York.

Investigation findings are included in Appendix A. A summary of environmental findings of these investigations is provided below.

27 December 2013 Phase I Environmental Site Assessment Report Prepared by Hydro Tech Environmental, Corp.

A Phase I ESA was completed by Hydro Tech in December 2013 for the purpose of identifying Recognized Environmental Conditions (RECs) in connection with the Site.

The Phase I ESA identified the following RECs in connection with the Site:

- The presence of in place abandoned underground storage tank (UST) at the Subject Property
- The suspect presence of impact associated with a northwest-adjacent historic gasoline station
- The presence of suspect lead-based paint at the Subject Property
- The presence of mold at the Subject Property

The Phase I ESA did not identify other environmental concerns at the adjoining and surrounding properties that would have potential impacts to the Site condition.

14 January 2014 Phase II Environmental Site Assessment

Prepared by Hydro Tech Environmental, Corp.

In January 2014, a Phase II ESA was completed by Hydro Tech at the Site and consisted of the following:

- Installation of four soil borings and collection of four soil samples analyzed for volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs).
- Installation of one groundwater probe and collection of one groundwater sample analyzed for VOCs.



- Installation of three permanent groundwater monitoring wells and collection of three groundwater samples analyzed for VOCs and SVOCs.
- Installation of three soil vapor probes and collection of three soil vapor samples analyzed for VOCs.
- Collection of two indoor air and one ambient air samples analyzed for VOCs.

The Phase II ESA identified the following:

- Soil analytical results are summarized as follows:
 - No VOCs or SVOCs were detected above the NYSDEC Title 6 New York Code Rules and Regulations (NYCRR) Part 375-6 Unrestricted Use Soil Cleanup Objectives (UUSCOs) in the samples collected.
- Groundwater analytical results are summarized as follows:
 - Chlorinated solvents, including PCE, and its derivative compounds and petroleum-related compounds were detected in two groundwater samples at concentrations exceeding their respective New York Technical and Operational Guidance Series (T.O.G.S.) 1.1.1 Class GA Ambient Water Quality Standards (AWQS).
- Soil vapor and indoor air sample results are summarized as follows:
 - Petroleum hydrocarbons were detected in the sub-slab vapor samples collected beneath the western portion of the Site.
 - No VOCs were identified in indoor air at concentrations exceeding New York State Department of Health (NYSDOH) Indoor Air Guidance levels.

5 August 2015 Focused Subsurface Investigation

Prepared by Hydro Tech Environmental, Corp.

The Focused Subsurface Investigation identified the following:

- The onsite UST in the southeast portion of the Site was not abandoned properly.
- Free product and dissolved VOCs were present near the UST. Spill Case No. 1504881 was issued.
- Dissolved chlorinated solvents were present in groundwater in the southeast portion of the Site.
- Petroleum and chlorinated volatile organic compounds (CVOCs) were present in the sub-slab vapor beneath the building.
- Recommendations included proper UST closure, removal of free product, and reduction of the groundwater contamination. Additionally, a vapor barrier system and a sub-slab depressurization system were recommended.

15 September 2015 Subsurface Investigation Report

Prepared by Hydro Tech Environmental, Corp.

In September 2015, a Subsurface Investigation was completed by Hydro Tech at the Site and consisted of the following:

- Delineation of impacted groundwater associated with the UST located to the south of the property and chlorinated solvents in groundwater beneath the eastern and southeastern portion of the Site.
- Installation and sampling of nine soil probes, five of which were converted to permanent monitoring wells, and the sampling of eight monitoring wells.



The Subsurface Investigation identified the following:

- The soil to the north of the UST exhibited olfactory evidence of petroleum odors and low concentrations of organic vapors ranging between 3 and 61 parts per million (ppm). Additionally, visual evidence of impact (e.g. staining) was observed in the deepest soil samples.
- No CVOCs were identified in soil in the eastern portion of the Site. Free product was observed in MW-3 (north of the UST).
- CVOCs in groundwater in the northern portion of the Site and petroleum-related VOCs in groundwater on the eastern portion of the Site exceed the AWQS.

27 March 2017 Focused Subsurface Investigation

Prepared by Hydro Tech Environmental, Corp.

Following a Remediation Work Plan (RWP) approval from NYSDEC in October 2015, a Focused Subsurface Investigation was completed by Hydro Tech at the Site in March 2017 and consisted of the following:

• Collection of 12 groundwater samples, installation and collection of three sub-slab vapor probes and samples, and collection of one indoor air sample and one outdoor air sample to determine the current levels of CVOCs in groundwater and soil vapors.

The Subsurface Investigation identified the following:

- Free product remains in the subsurface north of the UST.
- CVOCs in groundwater exceeded the AWQS in the vicinity of MW-2 in the eastern portion of the Site.
- The NYSDEC-approved RWP to address the Spill No. 1504881 should be implemented.
- The NYSDEC Hazardous Remediation Section should be contacted to inquire if potential remedies of the CVOCs in groundwater are required and if mitigation measures of soil vapor intrusion need to be implemented.

17 February 2020 Documentation of Tank Abandonment and Groundwater Quality Prepared by Hydro Tech Environmental, Corp.

In May 2017, Hydro Tech closed an abandoned tank abandonment and made observations regarding groundwater quality as follows:

- A 7,500-gallon #2 fuel oil UST located in the southwest portion of the Site was closed and closedin-place.
- A total of 25.52 tons of non-hazardous impacted soil and concrete were removed and disposed.
- A total of 1,030 gallons of an oil/water mixture was removed and disposed.
- Two permanent monitoring wells (MW-13 and MW-14) were installed in the area of the closed tank.
- Free product was observed in MW-3, MW-13, and MW-14. A petroleum sheen was observed in MW-6, MW-8, and MW-9 (north of the closed UST).
- Post-tank closure sidewall end-point samples indicated SVOCs were present to the south of the closed-in place UST at concentrations exceeding the UUSCOs.
- CVOCs remain in groundwater in the southeast portion of the Site in MW-2



7 May 2020 Enhanced Fluid Recovery (EFR) Results – February and March 2020 Prepared by Hydro Tech Environmental, Corp.

In February and March 2020, enhanced fluid recovery (EFR) events were conducted by Hydro Tech, which reported the following:

- A total of 558 gallons of free product and contaminated water were removed from monitoring wells MW-3, MW-8, MW-9, MW-13, and MW-14.
- Free product remaining in MW-3 was thought likely to be an isolated residual impact since none of the surrounding upgradient or downgradient wells exhibited evidence of free product.
- Evidence of petroleum hydrocarbons in the form of sheen or a thin film of product in MW-3, MW-8, MW-13, and MW-14 revealed a relatively minimal impact that Hydro Tech thought would remediate over time via natural biodegradation.

26 January 2023 Phase I Environmental Site Assessment

Prepared by Nelson, Pope & Voorhis, LLC

A Phase I ESA was completed by Nelson Pope Voorhis (NPV) in January 2023 for the purpose of identifying RECs in connection with the Site. The Phase I ESA identified the following RECs in connection with the Site:

- Spill no. 1504881, which was under active investigation by the NYSDEC was considered to be a REC due to the presence of dissolved petroleum and chlorinated solvent contamination in groundwater and soil vapor. The most recent Phase II investigation conducted in 2014 recommended additional investigation, periodic monitoring of groundwater wells for free product, and vapor intrusion mitigation measures such as a sub-slab depressurization system.
- Spill no. 9615014, was considered to be an off-site REC due to the potential for soil vapor impacts to the subject property from former Mobil Station #17, situated at 89-30 Queens Boulevard, adjacent to the west of the subject property, which had identified petroleum impacts in soil and groundwater. The spill incident was closed by NYSDEC in 2012; however, impacts to the subject property from this incident were noted and based on the spill incident report it does not appear that off-site vapor impacts were evaluated. Further, according to the Air Discharge database, a remediation system appeared to be actively operating in response to potential uncontrolled emissions from the former release.

The Phase I ESA identified the following additional environmental concerns at the adjoining and surrounding properties that could have potential impacts on the Site:

- Closed LUST incident, which occurred on the subject property and was assigned Spill no. 8801243, was considered a HREC that had been addressed to the satisfaction of the NYSDEC.
- Closed LUST incident, which occurred on the subject property and was assigned Spill no. 0511055, was considered a HREC that had been addressed to the satisfaction of the NYSDEC.
- Closed Spill incident, which occurred on the subject property and was assigned Spill no. 9908947, was considered a HREC that had been addressed to the satisfaction of the NYSDEC.
- Closed Spill incident, that occurred adjacent to the subject property and was assigned Spill no. 0808306, was considered an off-site HREC that had been addressed to the satisfaction of the NYSDEC.



- Closed Spill incident, which occurred adjacent to the subject property and was assigned Spill no. 0509905, is considered an off-site HREC that had been addressed to the satisfaction of the NYSDEC.
- Closed Spill incident, which occurred adjacent to the subject property and was assigned Spill no. 0302793, was considered an offsite HREC that has been addressed to the satisfaction of the NYSDEC.
- Closed LUST incident, which occurred adjacent to the subject property and was assigned Spill no. 9104895, was considered an offsite HREC that has been addressed to the satisfaction of the NYSDEC.
- Closed LUST incident, which occurred adjacent to the subject property and was assigned Spill no. 908424, was considered an offsite HREC which has been addressed to the satisfaction of the NYSDEC.

21 August 2023 Limited Phase II Environmental Site Investigation Report Prepared by Haley & Aldrich of New York

The Phase II Limited Phase II Environmental Site Investigation Report identified the following environmental findings:

- Fill material generally consisting of light gray to dark brown sand and clay with varying amounts of silt and gravel was observed from surface grade to approximately 6 ft bgs. The fill layer was underlain by gray to brown sand extending to at least 9 ft bgs, the maximum depth of exploration.
- Groundwater was encountered between 3 and 6 ft below the cellar level.
- Approximately one foot of free product was observed in MW-3.
- Soil analytical results were summarized as follows:
 - Acetone was detected above the UUSCO in two soil samples.
 - \circ $\,$ No other VOCs were detected above the UUSCOs in the samples collected.
- Groundwater analytical results were summarized as follows:
 - Chlorinated solvents, including PCE, and its derivative compounds and petroleum-related compounds were detected in two groundwater samples at concentrations exceeding the AWQS.
- Soil vapor sample results were summarized as follows:
 - Petroleum-related VOCs and CVOCs, including PCE, were detected in the sub-slab vapor samples collected.



3. Site Characterization

This section describes the field activities to be conducted during the Site Characterization and provides the sampling scope, objectives, methods, anticipated number of samples, and sample locations. A summary of the sampling and analysis plan is provided in Table 1 and Figure 2. The following activities will be conducted to fill data gaps and determine the nature and extent of contamination at the Site.

3.1 UTILITY MARKOUT

Based on the Site use history and various historical Site investigations, the existing UST closed-in-place, and active utilities, a full Ground Penetrating Radar (GPR) scan will be performed prior to commencement of any intrusive activities.

3.2 SOIL SAMPLING

To further characterize surface soil conditions, additional on-Site soil samples will be collected to meet NYSDEC DER-10 requirements for remedial investigations.

The sampling and analysis plan is summarized in Table 1. A total of five soil borings will be installed to 10 ft bgs by a track-mounted direct-push drill rig (Geoprobe[®]) operated by a licensed operator. The existing concrete slab will be cored and a drill rod with an installed acetate liner will be advanced through the soil stratum. Soil samples will be collected from acetate liners using a stainless-steel trowel or sampling spoon. Samples will be collected using laboratory provided clean bottle ware. VOC grab samples will be collected using terra cores or encores.

Soils will be logged continuously by a geologist or engineer using the Unified Soil Classification System. The presence of staining, odors, and photoionization detector (PID) response will be noted. Samples will be collected using laboratory-provided clean bottle ware. Sampling methods are described in the Field Sampling Plan (FSP) provided as Appendix B. A Quality Assurance Project Plan (QAPP) is provided as Appendix C. Laboratory data will be reported in ASP Category B deliverable format.

Soil samples representative of Site conditions will be collected at five locations, via direct push methods as detailed above, widely distributed across the Site, as shown in Figure 2. Samples will be collected directly underneath the slab and at the interface to the water table (approximately 3 to 6 ft bgs). Additional samples will be collected from any interval exhibiting elevated PID readings or visual and olfactory impacts, specifically for contaminates of concern. Soil samples will be analyzed for:

- Target Compound List (TCL) VOCs using EPA method 8260B;
- TCL SVOCs using EPA method 8270C;
- Total Analyte List (TAL) Metals using EPA method 6010;
- PCBs using EPA method 8082;
- Pesticides using EPA Method 8081;
- Per- and polyfluoroalkyl substances (PFAS) by EPA Method 1633; and
- 1,4-dioxane by EPA Method 8270 SIM;



Samples to be analyzed for PFAS and 1,4-dioxane will be collected and analyzed in accordance with the 'Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs" (April 2023).

3.3 GROUNDWATER SAMPLING

The purpose of groundwater sampling is to obtain current groundwater data and fill in data gaps of the Site conditions observed during previous investigations.

Permanent monitoring wells will be installed utilizing the hollow stem auger method, or approved alternative, to approximately 5 ft below the water table (8 to 14 ft bgs). Monitoring wells will have the appropriate annular space and be installed using either #0 or #00 certified clean sand fill. Wells will be screened starting at least 2 ft above the observed water table to the bottom of the borehole. Groundwater was encountered at approximately 3 to 6 ft bgs during the previous Site investigation completed in August 2023. Monitoring wells will be developed by surging a pump in the well several times to pull fine-grained material from the well. Wells will be developed until the water turbidity is 50 nephelometric turbidity units (NTU) or less or ten well volumes are removed, if possible. Accessible well casings will be surveyed by a New York State licensed surveyor and gauged during a round of synoptic groundwater depth readings to facilitate the preparation of a groundwater contour map and to determine the direction of groundwater flow.

Three Monitoring wells will be installed on the Site. Monitoring wells will be sampled and analyzed for:

- TCL VOCs using EPA method 8260B;
- TCL SVOCs using EPA method 8270C;
- Total and Dissolved Metals using EPA methods 6010/7471;
- PCBs using EPA method 8082;
- Pesticides using EPA Method 8081;
- PFAS using EPA method 1633; and
- 1,4-Dioxane using EPA method 8260B.

The sampling and analysis plan is summarized in Table 1. Proposed and existing monitoring well locations are provided in Figure 2.

Samples to be analyzed for PFAS and 1,4-dioxane will be collected and analyzed in accordance with NYSDEC's April 2023 technical guidance document "Sampling, Analysis and Assessment of PFAS".

Groundwater wells will be sampled using low-flow sampling methods described in the FSP. Following the low-flow purge, samples will be collected from monitoring wells for analysis of the analytes mentioned above. Groundwater sampling will be conducted at least one week after monitoring well development.

The FSP presented in Appendix B details field procedures and protocols that will be followed during field activities. The QAPP presented in Appendix C details the analytical methods and procedures that will be used to analyze samples collected during field activities. Sampling wells for PFAS will be conducted following the purge and according to the sampling method detailed in NYSDEC guidance.



3.4 INVESTIGATION DERIVED WASTE

Following sample collection, boreholes that are not converted to monitoring wells will be backfilled with soil cutting and an upper bentonite plug. Boreholes will be restored to grade with the surrounding area. If soil is identified as grossly contaminated, it will be separated and placed into a sealed and labeled Department of Transportation (DOT) approved 55-gallon drum pending characterization and off-Site disposal. Groundwater purged from the monitoring wells during development and sample collected will be placed into a DOT approved 55-gallon drum pending off-Site disposal.

3.5 SUB-SLAB/SOIL VAPOR SAMPLING

Samples will be collected in accordance with the New York State Department of Health (NYSDOH) Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York (NYSDOH October 2006, Revised 2017 and 2024). Seven sub-slab soil vapor probes will be installed to approximately 1 ft below the slab within the commercial space. Additionally, one sub-slab soil vapor probe will be installed within each occupied space of the property. The vapor implants will be installed with a direct-push drilling rig (e.g., Geoprobe[®]) to advance a stainless-steel probe to the desired sample depth. All interior sub-slab vapor samples will be co-located with indoor air samples to better assess the potential for exposures associated with soil vapor intrusion. Indoor air samples will be collected at a height approximately three feet above the floor to represent a height where occupants are normally seated. Sampling will occur for the duration of eight hours. Additionally, one outdoor ambient air sample will be collected. Access will be requested for adjoining structures within the property boundaries.

Samples will be collected in appropriately sized Summa[®] canisters that have been certified clean by the laboratory, and samples will be analyzed by using United States Environmental Protection Agency (USEPA) Method TO-15. Flow rate for both purging and sampling will not exceed 0.2 L/min. Sampling methods are described in the FSP provided as Appendix B.

A building questionnaire and chemical inventory will be completed during the sampling event to identify preferential pathways, building features, conditions and substances that may affect the interpretation of the sampling results.

3.6 **PROPOSED SAMPLING RATIONALE**

Haley & Aldrich has proposed the sampling plan described herein and as shown in Figure 2, in consideration of the data generated during previous investigations conducted at the Site. Previous investigations observed LNAPL and petroleum-related contamination in groundwater. Additionally, soil vapor samples showed the presence of CVOCs, including PCE, beneath the building. In order to properly characterize the Site and identify potential source areas, all phases of media will be comprehensively investigated as part of this SC, and data gaps will be evaluated.

The Proposed Sample Location Map (included as Figure 2) is designed to generate sufficient data to identify the source of contamination and classify subsurface conditions throughout the Site, as a whole, with a particular focus on sample locations in areas of the Site that have historically revealed evidence of contamination.



4. Quality Assurance and Quality Control

Quality Assurance/Quality Control (QA/QC) procedures will be used to provide performance information with regard to the accuracy, precision, sensitivity, representation, completeness, and comparability associated with the sampling and analysis for this investigation. Field QA/QC procedures will be used (1) to document that samples are representative of actual conditions at the Site and (2) identify possible cross-contamination from field activities or sample transit. Laboratory QA/QC procedures and analyses will be used to demonstrate whether analytical results have been biased either by interfering compounds in the sample matrix or by laboratory techniques that may have introduced systematic or random errors to the analytical process.

QA/QC procedures are defined in the QAPP included in Appendix C.



5. Data Use

5.1 DATA SUBMITTAL

Analytical data will be supplied in ASP Category B Data Packages. If more stringent than those suggested by the United States Environmental Protection Agency, the laboratory's in-house QA/QC limits will be utilized. Validated data will be submitted to the NYSDEC EQUIS database in an EDD package.

5.2 DATA VALIDATION

Data packages will be sent to a qualified data validation specialist to evaluate the accuracy and precision of the analytical results. A Data Usability Summary Report (DUSR) will be created to confirm the compliance of methods with the protocols described in the NYSDEC Analytical service Protocol (ASP). DUSRs will summarize and confirm the usability of the data for project-related decisions. Data validation will be completed in accordance with the DUSR guidelines from the NYSDEC Division of Environmental Remediation. Previous data provided to Haley & Aldrich has not been validated.



6. Health and Safety

6.1 HEALTH AND SAFETY PLAN

A Site-specific Health and Safety Plan (HASP) has been prepared in accordance with NYSDEC and NYSDOH guidelines and is provided as Appendix D of this work plan. The HASP includes a description of health and safety protocols to be followed by Haley & Aldrich field staff during implementation of the remedy, including monitoring within the work area, along with response actions should impacts be observed. The HASP has been developed in accordance with Occupational Health and Safety Administration (OSHA) 40 CFR Part 1910.120 regulatory requirements for use by Haley & Aldrich field staff that will work at the Site during planned activities. Contractors or other personnel who perform work at the Site are required to develop their own health and safety plans and procedures of comparable or stricter content for their respective personnel in accordance with relevant OSHA regulatory requirements for work at hazardous waste sites as well as the general industry as applicable based on the nature of work being performed.

6.2 COMMUNITY AIR MONITORING PLAN

The proposed investigation work will be completed primarily indoors. Where intrusive drilling operations are planned outdoors, generic community air monitoring will be implemented during all outdoor ground intrusive activities to protect downwind receptors. A Haley & Aldrich representative will continually monitor the breathing air in the vicinity of the immediate work area using a PID to measure total volatile organic compounds in the air at concentrations as low as 1 part per million (ppm) and a Dustrak to measure particulates in the air smaller than 10 microns in diameter. The air in the work zone also will be monitored for visible dust generation.

CAMP observations will be reported on a weekly basis and all exceedances will be reported to the NYSDEC and NYSDOH the same day or next business day if after hours along with the reason for the exceedance, what was done to correct it and if it was effective. A copy of the NYSDOH Generic CAMP is included as Appendix E. In addition, investigation work will be in compliance with Fugitive Dust and Particulate Monitoring (included in Appendix E).



7. References

- 1. ASTM Phase I Environmental Site Assessment Report, 90-02 Queens Boulevard, Queens, New York. Prepared by Hydro Techn Environmental, Corp., December 27, 2013.
- 2. Phase II Environmental Site Assessment, 90-02 Queens Boulevard, Queens, New York, Prepared by Hydro Tech Environmental, Corp., January 14, 2014.
- 3. Focused Subsurface Investigation, 90-02 Queens Boulevard, Queens, New York, Prepared by Hydro Tech Environmental, Corp., August 5, 2015.
- 4. Subsurface Investigation Report, 90-02 Queens Boulevard, Queens, New York, Prepared by Hydro Tech Environmental, Corp., September 15, 2015.
- 5. Focused Subsurface Investigation, 90-02 Queens Boulevard, Queens, New York, Prepared by Hydro Tech Environmental, Corp., March 27, 2017.
- 6. Documentation of Tank Abandonment and Groundwater Quality, 90-02 Queens Boulevard, Queens, New York, Prepared by Hydro Tech Environmental, Corp., February 17, 2020.
- 7. Enhanced Fluid Recovery (EFR) Results February and March 2020, 90-02 Queens Boulevard, Queens, New York, Prepared by Hydro Tech Environmental, Corp., May 7, 2020.
- 8. ASTM Phase I Environmental Site Assessment, 90-02 Queens Boulevard, Queens, New York, Prepared by Nelson, Pope & Voorhis, LLC, January 26, 2023.
- 9. Limited Phase II Environmental Site Investigation Report, 90-02 Queens Boulevard, Queens, New York, Prepared by Haley & Aldrich of New York, August 21, 2023.
- 10. Program Policy DER-10, "Technical Guidance for Site Investigation and Remediation," New York State Department of Environmental Conservation, May 2010.
- 11. New York State Department of Environmental Conservation, Part 375 Title 6 of the New York Compilation of Codes, Rules, and Regulations, Effective December 14, 2006.

New York State Department of Environmental Conservation, Division of Environmental Remediation, Sampling, Analysis and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) under NYSDEC Part 375 Remedial Program, Effective April 2023.

Guidance for Evaluating Soil Vapor Intrusion in the State of New York, October 2006 (Revised 2017 and 2024).

https://haleyaldrich.sharepoint.com/sites/MadisonRealtyCapital/Shared Documents/0208933.90-02 Queens Boulevard/Deliverables/2. Site Characterization Work Plan/2. March 2024 Submission/2024-0429-HANY- 90-02 Queens Blvd-SCW-F.docx



TABLES



Table 1. Sample and Analysis Plan90-02 Queens Boulevard SiteQueens, New York

Boring Number	Soil Sample Depth	Target Compound List VOCs (8260B)	Target Compound List SVOCs (8270C)	Total Analyte List Metals (6010)	PCBs (8082)	PFAS (537)	1,4-Dioxane (8270 SIM)	VOCs (TO-15)
				SOIL				
HA SB-04	Beneath the Slab	Х	Х	Х	Х	Х	Х	-
TA 3D-04	At the Water Table	X	Х	Х	Х	Х	Х	-
HA SB-05	Beneath the Slab	Х	Х	Х	Х	Х	Х	-
HA 3B-05	At the Water Table	Х	Х	Х	Х	Х	Х	-
HA SB-06	Beneath the Slab	Х	Х	Х	Х	Х	Х	-
HA 3B-06	At the Water Table	X	Х	Х	Х	Х	Х	-
HA SB-07	Beneath the Slab	Х	Х	Х	Х	Х	Х	-
TA 3D-07	At the Water Table	X	Х	Х	Х	Х	Х	-
				GROUNDWATER				
MW-13	-	Х	Х	Х	Х	Х	Х	-
MW-14	-	X	Х	Х	Х	Х	Х	-
				SOIL VAPOR		·		`
HA SS-04	-	-	-	-	-	-	-	Х
HA SS-05	-	-	-	-	-	-	-	Х
HA SS-06	-	-	-	-	-	-	-	Х
lataci	· ·	0A0C complex include:			•	•	·	•

Notes:

QAQC samples include:

VOCs - Volatile Organic Compounds

SVOCs - Semi-volatile Organic Compounds

PCBs - Polychlorinated biphenyls

PFAS - Per- and Polyfluoroalkyl Substances

X = Run for analysis

LAUC samples include:

MS/MSD - 1 for every 20 samples

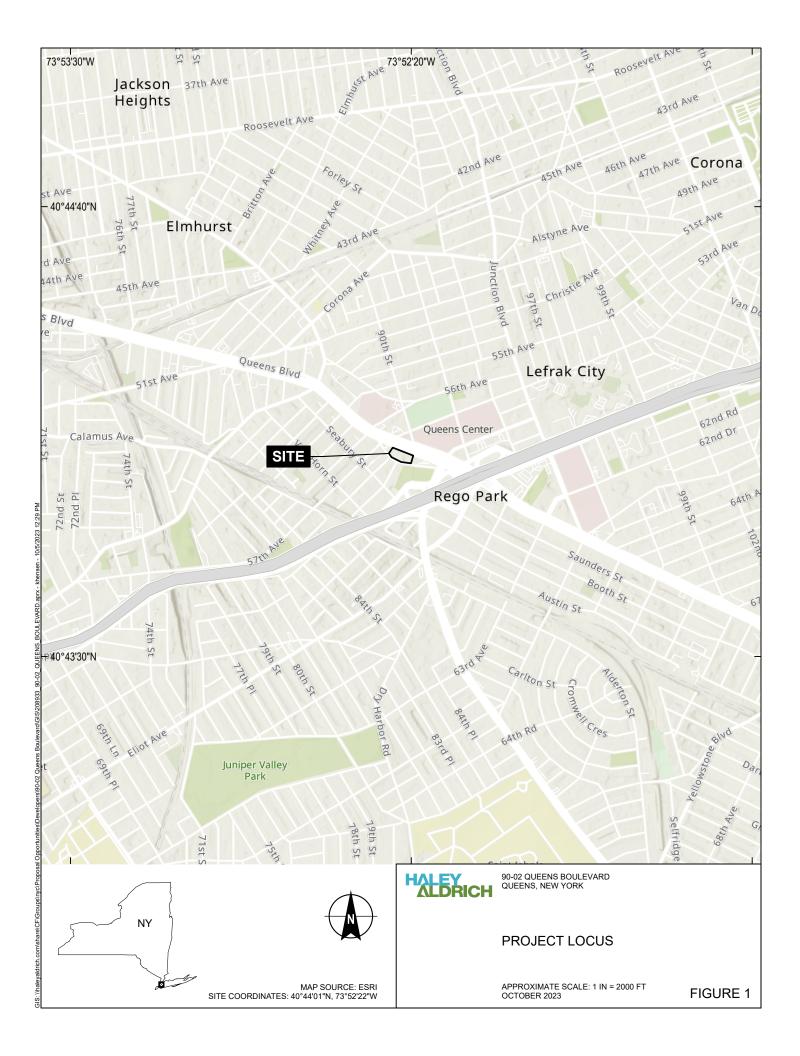
Field Duplicate - 1 for every 20 samples Trip Blanks - 1 per cooler of samples to be analyzed for VOCs

Field Blanks - 1 for every 20 samples

"-" = Not applicable

FIGURES







LEGEND



SITE BOUNDARY

| PARTIAL BASEMENT OUTLINE



PROPOSED SOIL BORING / GROUNDWATER MONITORING WELL LOCATION

PROPOSED SOIL BORING LOCATION



PROPOSED SOIL VAPOR LOCATION

NOTES

1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.

2. PARTIAL BASEMENT OUTLINE SOURCE: 'FIGURE 1: MONITORING WELL LOCATIONS', HYDRO TECH ENVIRONMENTAL CORP., 18 SEPTEMBER 2015

3. ASSESSOR PARCEL DATA SOURCE: NEW YORK CITY PLANNING

4. AERIAL IMAGERY SOURCE: NEARMAP, 28 MAY 2023



0 40 8 SCALE IN FEET

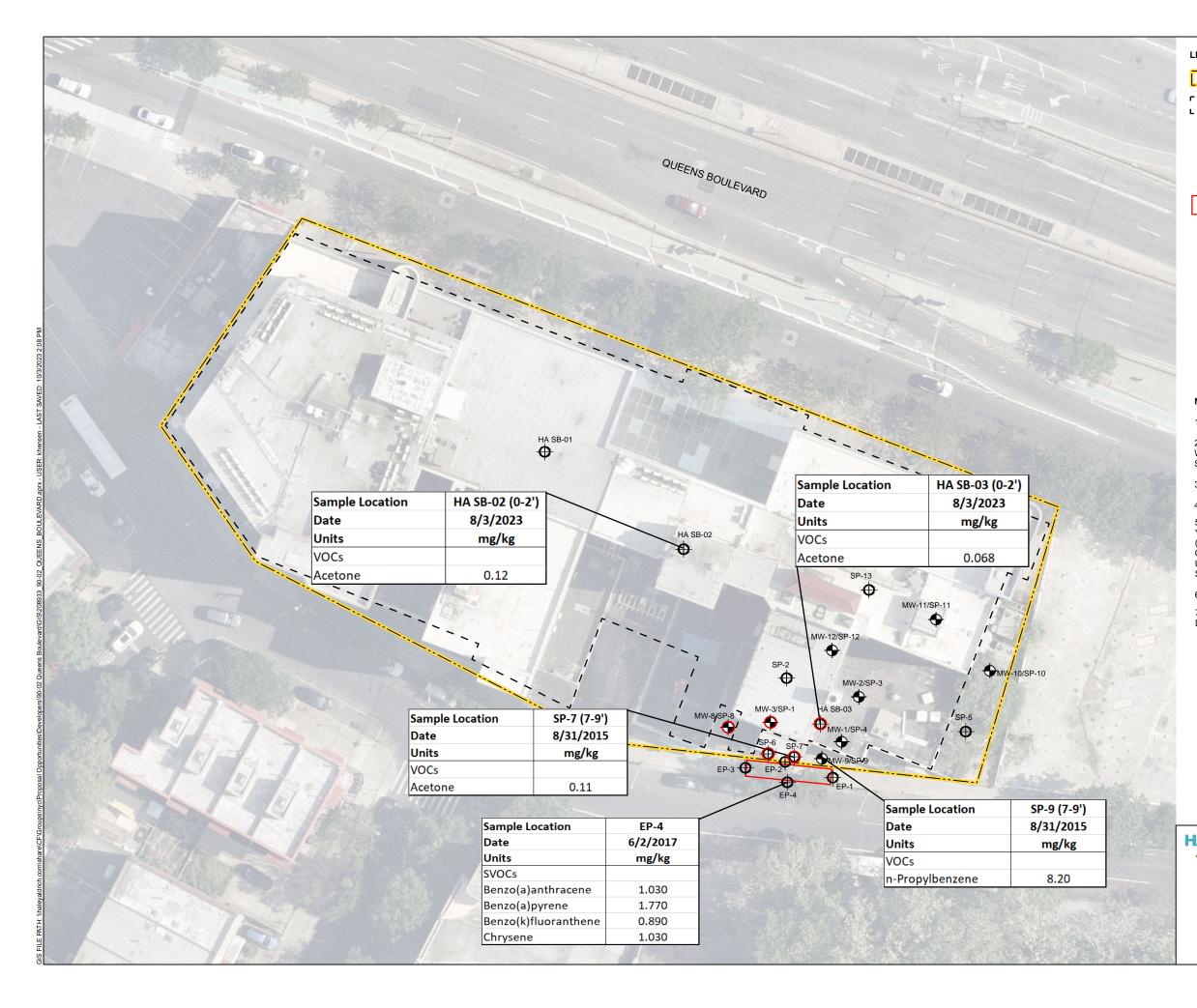
HALEY ALDRICH

90-02 QUEENS BOULEVARD QUEENS, NEW YORK

PROPOSED SAMPLE LOCATION MAP

OCTOBER 2023

FIGURE 2



LEGEND

SITE BOUNDARY





с.

APPROXIMATE LOCATION OF PERMANENT MONITORING WELL/SOIL BORING

 \oplus

APPROXIMATE LOCATION SOIL BORING

Ο

INDICATES THE PRESENCE OF LNAPL DURING INVESTIGATION

CLOSED-IN-PLACE UNDERGROUND STORAGE TANK

	New York Part 375
	UUSCO
VOCs (mg/kg)	
Acetone	0.05
n-Propylbenzene	3.9
SVOCs	
Benzo(a)anthracene	1
Benzo(a)pyrene	1
Benzo(k)fluoranthene	0.8
Chrysene	1

NOTES

1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.

2. PARTIAL BASEMENT OUTLINE SOURCE: 'FIGURE 1: MONITORING WELL LOCATIONS', HYDRO TECH ENVIRONMENTAL CORP., 18 SEPTEMBER 2015

3. ASSESSOR PARCEL DATA SOURCE: NEW YORK CITY PLANNING

4. AERIAL IMAGERY SOURCE: NEARMAP, 28 MAY 2023

5. SOIL SAMPLE ANALYTICAL RESULTS ARE COMPARED TO THE NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION (NYSDEC) TITLE 6 OF THE OFFICIAL COMPILATION OF NEW YORK CODES, RULES, AND REGULATIONS (NYCRR) PART 375 UNRESTRCITED USE SOIL CLEANUP OBJECTIVES (UUSCOS), RESTRICTED-RESIDENTIAL SCOS, AND 40 CFR 261 SUBPART C AND TABLE 1 OF 40 CFR 261.24.

6. RESULTS ARE DISPLAYED IN MILLIGRAMS PER KILOGRAM (mg/kg)

7. ANALYTICAL RESULTS OF SAMPLE LOCATIONS WITHOUT A DATA BOX DID NOT EXCEED THE NYSDEC PART 375 UUSCOS.



SCALE IN FEET

ALDRICH 90-02 QUEENS BOULEVARD QUEENS, NEW YORK

SUMMARY OF HISTORICAL SOIL ANALYTICAL DATA

OCTOBER 2023

FIGURE 3

Sample Location Date	MW-2 1/7/2014	MW-2 8/3/2015	MW-2 9/2/2015	MW-2 3/7/2017	MW-2 1/13/2020	MW-2 8/3/2023
Units	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
VOCs						
1,1,1-Trichloroethane	98	3,400	1,300	150	680	830
1,1,2-Trichloroethane	3.2	4.3	2	1.60	2.1	1.6
1,1-Dichloroethane	800	1,800	790	540	620	490
1,1-Dichloroethylene	270	510	240	300	590	560
1,2-Dichlorobenzene	NE	NE	NE	NE	NE	3.1
1,2-Dichloroethane	0.73	NE	NE	NE	NE	NE
Chlorobenzene	NE	NE	NE	NE	NE	7.7
Choroethane	NE	NE	NE	NE	NE	19
Tetrachloroethene	NE	NE	NE	NE	NE	6.8
Vinyl Chloride	10	21	7.50	NE	19	17

MW-12/SP-12

MW-3/SP-1

 \bullet

MW-9/SI

P-8 >

Φ.

QUEENS BOULEVARD

Sample Location	MW-12	MW-12
Date	9/2/2015	1/13/202
Units	ug/L	ug/L
VOCs		
Naphthalene	25	NE
SVOCs		
Benzo(a)anthracene	NE	0.1
Benzo(a)pyrene	NE	0.1
Benzo(b)fluoranthene	NE	0.1
Benzo(k)fluoranthene	NE	0.1
Chrysene	NE	0.2

MW-

Sample Location Date	MW-6 8/3/2015	MW-6 1/13/2020
Units	ug/L	ug/L
VOCs		
1,2,4-Trimethylbenzene	110	NE
1,3,5-Trimethylbenzene	13	NE
Benzene	6.9	3.4
Ethylbenzene	14	NE
Isopropylbenzene	15	NE
n-Butylbenzene	14	NE
n-Propylbenzene	22	NE
Naphthalene	92	NE
p- & m- Xylenes	7.7	NE
p-Isopropylbenzene	10	NE
sec-Butylbenzene	14	NE

		Contraction of the second second	And the second
	Samp	le Location	MW-11
	Date		8/3/2023
-	Units VOCs		ug/L
the provide the second			
A THE		Trichloroethane	1.3
Sec.	1,1-Dichloroethane		680
and a second	100		
		ichloroethylene	280
D TO SA A		Chloride	17
MW-11/5	SP-11 /		
•	-	51	1
IRS LAND	L P	R. C. C.	
	103	i solo i h	
	1		2.1
MW	10/SP-10	Sample Location	MW-1
MW-2/SP-3		Date	1/7/2014
	si i i	Units	ug/L
	e e <mark>l</mark> e	VOCs	
	i s	1,1,1-Trichloroethane	10,000
IW-1/SP-4 /		1,1,2-Trichloroethane	11
• · ·	i b	1,1-Dichloroethane	2,400
90		1,1-Dichloroethylene	1,800
	+	1,2-Dichlorobenzene	18
I-14 • MW-7		1,2-Dichloroethane	2
and the second		1,4-Dichlorobenzene	19
		2-Butanone	58
		Carbon Tetrachloride	1,700
		Chlorobenzene	67
Real and		Chloroethane	36
		cis-1,2-Dichlorothylene	26
		p- & m- Xylenes	8
		Tetrachloroethylene	66
		Toluene Visud Chlorida	15
		Vinyl Chloride	310

SVOCs

1,2-Dichlorobenzene

1,4-Dichlorobenzene

9

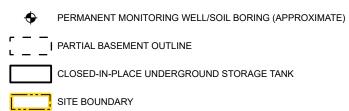
13

	New York TOGS	
	1.1.1 AWQS	
VOCs (ug/L)		
1,1,1-Trichloroethane	5	
1,1,2-Trichloroethane	1	
1,1-Dichloroethane	5	
1,1-Dichloroethylene	5	
1,2,4-Trimethylbenzene	5	
1,2-Dichlorobenzene	3	
1,2-Dichloroethane	0.6	
1,3,5-Trimethylbenzene	5	
1,4-Dichlorobenzene	3	
2-Butanone	50	
Benzene	1	
Carbon Tetrachloride	5	
Chlorobenzene	5	
Chloroethane	5	
cis-1,2-Dichlorothylene	5	
Ethylbenzene	5	
Isopropylbenzene	5	
n-Butylbenzene	5	
n-Propylbenzene	5	
Naphthalene	10	
p- & m- Xylenes	5	
Tetrachloroethylene	5	
Toluene	5	
Vinyl Chloride	2	
SVOCs (ug/L)		
Benzo(a)anthracene	0.002	
Benzo(a)pyrene	0.002	
Benzo(b)fluoranthene	0.002	
Benzo(k)fluoranthene	0.002	

0.002

Chrysene





NOTES

1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.

2. PARTIAL BASEMENT OUTLINE SOURCE: 'FIGURE 1: MONITORING WELL LOCATIONS', HYDRO TECH ENVIRONMENTAL CORP., 18 SEPTEMBER 2015

3. ASSESSOR PARCEL DATA SOURCE: NEW YORK CITY PLANNING

4. GROUNDWATER SAMPLE ANALYTICAL RESULTS ARE COMPARED TO THE NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION (NYSDEC) TECHNICAL AND OPERATIONAL GUIDANCE SERIES (TOGS) 1.1.1 AMBIENT WATER QUALITY STANDARDS (AWQS).

5. RESULTS ARE DISPLAYED IN MICROGRAMS PER LITER (µg/L).

6. NE = NO EXCEEDANCE OF THE NEW YORK TOGS 1.1.1 AWQS

7. ANALYTICAL RESULTS FOR WELLS WITHOUT DATA BOXES DID NOT EXCEED THE NEW YORK TOGS 1.1.1 AWQS.

8. AERIAL IMAGERY SOURCE: NEARMAP, 28 MAY 2023

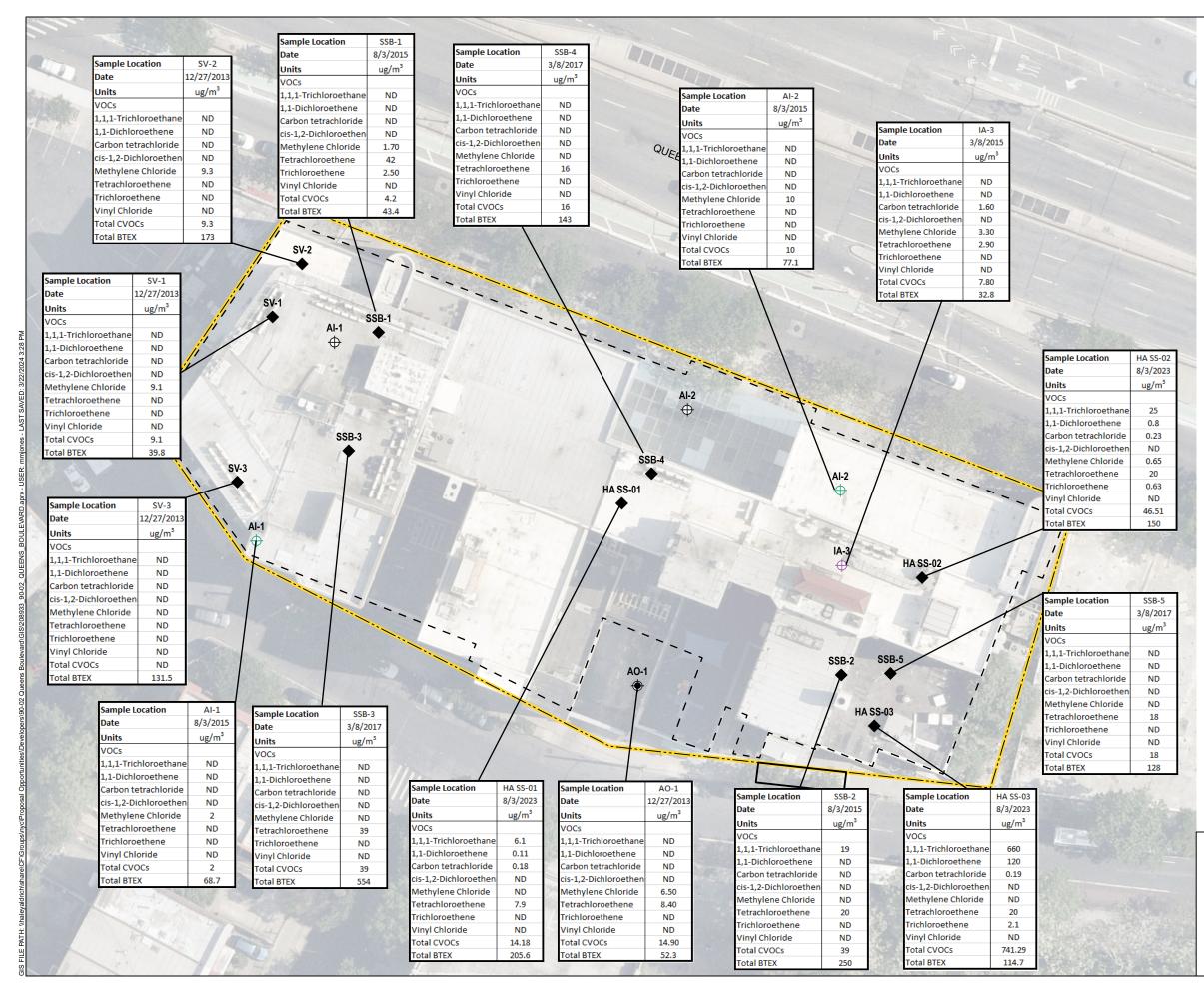


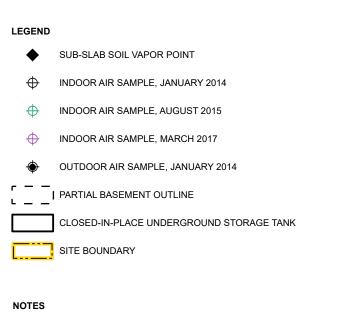
SCALE IN FEET

90-02 QUEENS BOULEVARD QUEENS, NEW YORK

SUMMARY OF HISTORICAL GROUNDWATER ANALYTICAL DATA

MARCH 2024





1. ALL LOCATIONS AND DIMENSIONS ARE APPROXIMATE.

2. PARTIAL BASEMENT OUTLINE SOURCE: 'FIGURE 1: MONITORING WELL LOCATIONS', HYDRO TECH ENVIRONMENTAL CORP., 18 SEPTEMBER 2015

3. ASSESSOR PARCEL DATA SOURCE: NEW YORK CITY PLANNING

4. RESULTS ARE DISPLAYED IN MICROGRAMS PER CUBIC METER $(\mu g/m^3).$

5. TOTAL DETECTED CONCENTRATIONS OF BENZENE, TOLUENE, ETHYLBENZENE AND XYLENES (BTEX).

6. TOTAL CVOCS CONCENTRATIONS OF CARBON TETRACHLORIDE, 1,1-DICHLOROETHENE, CIS-1,2-DICHLOROETHENE, TRICHLOROETHENE, METHYLENE CHLORIDE, TETRACHLOROETHENE, 1,1,1-TRICHLOROETHENE AND VINYL CHLORIDE.

7. NO CVOC OR BTEX CONCENTRATIONS WERE DETECTED AT AI-1 AND AI-2, JANUARY 2014 LOCATIONS.

8. AERIAL IMAGERY SOURCE: NEARMAP, 28 MAY 2023



SCALE IN FEET

HALEY ALDRICH

90-02 QUEENS BOULEVARD QUEENS, NEW YORK

HISTORICAL SOIL VAPOR, INDOOR AIR, AND OUTDOOR AIR ANALYTICAL DATA

MARCH 2024

FIGURE 5

APPENDIX A Previous Reports https://haleyaldrich.sharefile.com/share/view/s3b4a4697462040508adc11368711fe49



APPENDIX B Field Sampling Plan



www.haleyaldrich.com

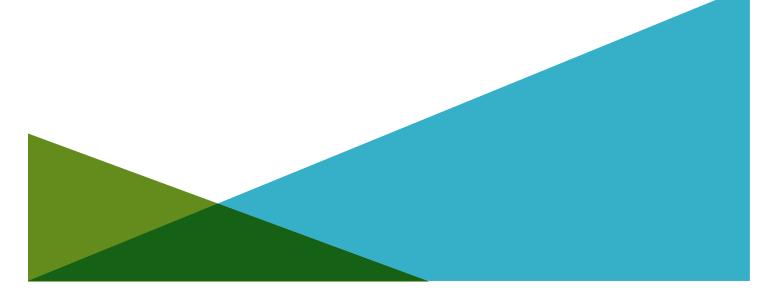


FIELD SAMPLING PLAN 90-02 QUEENS BOULEVARD SITE 90-02 QUEENS BOULEVARD QUEENS, NEW YORK

by Haley & Aldrich of New York New York, New York

for 9002 Commercial Unit LLC New York, New York

File No. 0208933 October 2023



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APPENDIX A – Field Forms

1. Introduction

This Field Sampling Plan (FSP) has been prepared as a component of the Site Characterization Work Plan (SCWP) for the 90-02 Queens Boulevard Site located at 90-02 Queens Boulevard in Queens, New York. This document was prepared to establish field procedures for field data collection to be performed in support of the SCWP for the Site.

The SCWP includes this Field Sampling Plan, a Quality Assurance Project Plan (QAPP) and Health and Safety Plan (HASP), which are included as part of this plan by reference.

The standard operating procedures (SOP) included as components of this plan will provide the procedures necessary to meet the project objectives. The SOPs will be used as reference for the methods to be employed for field sample collection and handling and the management of field data collected in the execution of the approved SCWP. The SOPs include numerous methods to execute the tasks of the SCWP. The Project Manager will select the appropriate method as required by field conditions and/or the objective the respective project task at the time of sample collection. Field procedures will be conducted in general accordance with the New York State Department of Conservation (NYSDEC) Technical Guidance for Site Investigation and Remediation (DER-10) and the Sampling, Analysis and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) under NYSDEC Part 375 Remedial Program when applicable.



2. Field Program

This FSP provides the general purpose of sampling as well as procedural information. The SCWP contains the details on sampling and analysis (locations, depths, frequency, analyte lists, etc.).

The field program has been designed to acquire the necessary data to comply with the SCWP, and includes the following tasks:

- Soil sampling;
- Groundwater sampling;
- Soil vapor and indoor air/ ambient air sampling; and,
- Sampling of investigation of derived wastes (IDW) as needed for disposal.

Multiple Limited Phase II investigations were performed at the Site to investigate the anticipated contaminants of concern identified based on the Site's former uses. The investigations partially determined the nature and extent of light non-aqueous phase liquid (LNAPL) in groundwater and identified the presence of chlorinated volatile organic compounds (CVOCs) in soil vapor. The site characterization did not identify a source of contamination on the Site, therefore additional targeted soil, groundwater, and soil vapor sampling is proposed.

These SOPs presented herein may be changed as required, dependent on-site conditions, or equipment limitations, at the time of sample collection. If the procedures employed differ from the SOP, the deviations will be documented in the associated sampling report.



3. Utility Clearance

Invasive remedial activities such as excavation or remedial construction activities require location of underground utilities prior to initiating work. Such clearance is sound practice in that it minimizes the potential for damage to underground facilities and more importantly, is protective of the health and safety of personnel. Under no circumstances will invasive activities be allowed to proceed without obtaining proper utility clearance by the appropriate public agencies and/or private entities. This clearance requirement applies to all work on both public and private properties, whether located in a dense urban area or a seemingly out-of-the-way rural location.

The drilling contractor performing the work will be responsible for obtaining utility clearance.

Utility clearance is required by law, and obtaining clearance includes contacting a public or private central clearance agency via a "one-call" telephone service and providing the proposed exploration location information. It is important to note that public utility agencies may not, and usually do not have information regarding utility locations on private property.

Before beginning subsurface work at any proposed exploration locations, it is critical that all readily available information on underground utilities and structures be obtained. This includes publicly available information as well as information in the possession of private landowners. Any drawings obtained must be reviewed in detail for information pertaining to underground utilities.

Using the information obtained, the site should be viewed in detail for physical evidence of buried lines or structures, including pavement cuts and patches, variation in or lack of vegetation, variations in grading, etc. Care must also be taken to avoid overhead utilities as well. Presence of surface elements of buried utilities should be documented, such as manholes, gas or water service valves, catch basins, monuments or other evidence.

Overhead utility lines must be considered when choosing exploration and excavation locations. Most states require a minimum of 10 ft of clearance between equipment and energized wires. Such separation requirements may also be voltage-based and may vary depending on state or municipality regulations. In evaluating clearance from overhead lines, the same restrictions may apply to "drops", or wires on a utility pole connecting overhead and underground lines.

Using the information obtained and observations made, proposed exploration or construction locations should be marked in the field. Marking locations can be accomplished using spray paint on the ground, stakes, or other means. All markings of proposed locations should be made in white, in accordance with the generally accepted universal color code for facilities identification (AWMA 4/99):

- White: Proposed Excavation or Drilling location
- Pink: Temporary Survey Markings
- Red: Electrical Power Lines, Cables, Conduit and Lighting Cables
- Yellow: Gas, Oil, Steam, Petroleum or Gaseous Materials
- Orange: Communication, Alarm or Signal Lines, Cables or Conduits
- Blue: Potable Water
- Purple: Reclaimed Water, Irrigation and Slurry Lines
- Green: Sewers and Drain Lines



In order to effectively evaluate the proposed locations with these entities, detailed, accurate measurements between the proposed locations and existing surface features should be obtained. Such features can be buildings, street intersections, utility poles, guardrails, etc.

Obtaining the utility clearance generally involves the designated "One-Call" underground facilities protection organization for the area and the landowner and one or both following entities:

- A third-party utility locator company will be utilized to locate underground utilities outside of the public right-of-way; and/or
- "Soft dig" excavation techniques to confirm or deny the presence of underground utilities in the area.

The proposed locations should be evaluated in light of information available for existing underground facilities. The detailed measurement information described above will be required by the "one call" agency. The owners of the applicable, participating underground utilities are obligated to mark their respective facilities at the site in the colors described above. Utility stake-out activities will typically not commence for approximately 72 hours after the initial request is made.

The public and private utility entities generally only mark the locations of their respective underground facilities within public rights-of-way. Determination of the locations of these facilities on private property will be the responsibility of the property owner or Contractor. If available information does not contain sufficient detail to locate underground facilities with a reasonable amount of confidence, alternate measures may be appropriate, as described below. In some cases, the memory of a long-time employee of a facility on private property may be the best or only source of information. It is incumbent on the Consultant or Contractor to exercise caution and use good judgement when faced with uncertainty.

Note: It is important to note that not all utilities are participants in the "one-call" agency or process. As such, inquiries must be made with the "one-call" agency to determine which entities do not participate, so they can be contacted independently.

Most utility stakeouts have a limited time period for which they remain valid, typically two to three weeks. It is critical that this time period be considered to prevent expiration of clearance prior to completion of the invasive activities, and the need to repeat the stake-out process.

Care must be exercised to document receipt of notice from the involved agencies of the presence or absence of utilities in the vicinity of the proposed locations.

Most agencies will generally provide a telephone or fax communication indicating the lack of facilities in the project area. If contact is not made by all of the agencies identified by the "one-call" process, do not assume that such utilities are not present. Re-contact the "one-call" agency to determine the status.

For complicated sites with multiple proposed locations and multiple utilities, it is advisable to arrange an on-site meeting with utility representatives. This will minimize the potential for miscommunication amongst the involved parties.

Completion of the utility stake out process is not a guarantee that underground facilities will not be encountered in excavations or boreholes; in fact, most "one-call" agencies and individual utilities do not



offer guarantees, nor do they accept liability for damage that might occur. In areas outside the public right-of-way, a utility locating service may be utilized to locate underground utilities. It is advisable that any invasive activities proceed with extreme caution in the upper four to five feet in the event the clearance has failed to identify an existing facility. This may necessitate hand-excavation or probing to confirm potential presence of shallow utilities. If uncertainty exists for any given utility, extra activities can be initiated to solve utility clearance concerns. These options include:

- Screening the proposed work areas with utility locating devices, and/or hiring a utility locating service to perform this task.
- Hand digging, augering or probing to expose or reveal shallow utilities and confirm presence and location. In northern climates, this may require advancing to below frost line, typically at least four feet.
- Using "soft dig" techniques that utilize specialized tools and compressed air to excavate soils and locate utilities. This technique is effective in locating utilities to a depth of four to five feet.

Equipment/Materials:

- White Spray paint
- Wooden stakes, painted white or containing white flagging
- Color-code key
- Available drawings



4. Field Data Recording

This procedure describes protocol for documenting the investigation activities in the field. Field data serves as the cornerstone for an environmental project, not only for site characterization but for additional phases of investigation or remedial design. Producing defensible data includes proper and appropriate recording of field data as it is obtained in a manner to preserve the information for future use. This procedure provides guidelines for accurate, thorough collection and preservation of written and electronic field data.

Field data to be recorded during the project generally includes, but is not limited to, the following:

- general field observations;
- numeric field measurements and instrument readings;
- quantity estimates;
- sample locations and corresponding sample numbers;
- relevant comments and details pertaining to the samples collected;
- documentation of activities, procedures and progress achieved;
- contractor pay item quantities;
- weather conditions;
- a listing of personnel involved in site-related activities;
- a log of conversations, site meetings and other communications; and,
- field decisions and pertinent information associated with the decisions.

4.1 Written Field Data

Written field data will be collected using a standardized, pre-printed field log form. In general, use of a field log form is preferable as it prompts field personnel to make appropriate observations and record data in a standardized format. This promotes completeness and consistency from one person to the next. Otherwise, electronic data collection using a handheld device produces equal completeness and consistency using a preformatted log form.

In the absence of an appropriate pre-printed form, the data should be recorded in an organized and structured manner in a dedicated project field log book. Log books must be hard cover, bound so that pages cannot be added or removed, and should be made from high-grade 50% rag paper with a water-resistant surface.

The following are guidelines for use of field log forms and log books:

- 1. Information must be factual and complete.
- 2. All entries will be made in black indelible ink with a ballpoint pen and will be written legibly. Do not use "rollerball" or felt tip-style pens, since the water-soluble ink can run or smear in the presence of moisture.
- 3. Field log forms should be consecutively numbered.
- 4. Each day's work must start a new form/page.
- 5. At the end of each day, the current log book page or forms must be signed and dated by the field personnel making the entries.



- 6. Make data entries immediately upon obtaining the data. Do not make temporary notes in other locations for later transfer; this only increases the potential for error or loss of data.
- 7. Entry errors are to be crossed out with a single line and initialed by the person making the correction.
- 8. Do not leave blanks on log forms, if no entry is applicable for a given data field, indicate so with "NA" or a dash ("--").
- 9. At the earliest practical time, photocopies or typed versions of log forms and log book pages should be made and placed in the project file as a backup in the event the book or forms are lost or damaged.
- 10. Log books should be dedicated to one project only, i.e., do not record data from multiple projects in one log book.

4.2 Electronic Data

Electronic data recording involves electronic measurement of field information through the use of monitoring instruments, sensors, gauges, and equipment controls. The following is a list of guidelines for proper recording and management of electronic field data:

- 1. Field data management should follow requirements of a project-specific data management plan (DMP), if applicable.
- 2. Use only instruments that have been calibrated in accordance with manufacturer's recommendations.
- 3. Usage of instruments, controls, and computers for the purpose of obtaining field data should only be performed by personnel properly trained and experienced in the use of the equipment and software.
- 4. Use only fully licensed software on personal computers and laptops.
- 5. Loss of electronic files may mean loss of irreplaceable data. Every effort should be made to back up electronic files obtained in the field as soon as practical. A backup file placed on the file server will minimize the potential for loss.
- 6. Electronic files, once transferred from field instruments or laptops to office computers, should be protected if possible, to prevent unwanted or inadvertent manipulation or modification of data. Several levels of protection are usually available for spreadsheets, including making a file "read-only" or assigning a password to access the file.
- 7. Protect CD disks from exposure to moisture, excessive heat or cold, magnetic fields, or other potentially damaging conditions.
- 8. Remote monitoring is often used to obtain stored electronic data from site environmental systems. A thorough discussion of this type of electronic field data recording is beyond the scope of this Section. Such on-site systems are generally capable of storing a limited amount of data as a comma-delimited or spreadsheet file. Users must remotely access the monitoring equipment files via modem or other access and download the data. In order to minimize the potential for loss of data, access and downloading of data should be performed frequently enough to ensure the data storage capacity of the remote equipment is not exceeded.

Equipment/Materials:

- Appropriate field log forms, or iPad[®] or equivalent with preformatted log forms.
- Indelible ball point pen (do not use "rollerball" or felt-tip style pens);
- Straight edge;



- Pocket calculator; and
- Laptop computer (if required).



5. Aquifer Characterization

This procedure describes measurement of water levels in groundwater monitoring.

A synoptic gauging round will be completed to obtain water levels in monitoring wells. Water levels will be acquired in a manner that provides accurate data that can be used to calculate vertical and horizontal hydraulic gradients and other hydrogeologic parameters. Accuracy in obtaining the measurements is critical to ensure the usability of the data.

5.1 Procedure

In order to provide reliable data, water level monitoring events should be collected over as short a period of time as practical. Barometric pressure can affect groundwater levels and, therefore, observation of significant weather changes during the period of water level measurements must be noted. Rainfall events and groundwater pumping can also affect groundwater level measurements. Personnel collecting water level data must note if any of these controls are in effect during the groundwater level collection period. Due to possible changes during the groundwater level collection period, it is imperative that the time of data collection at each station be accurately recorded. Water levels will also be collected prior to any sample collection that day.

The depth to groundwater will be measured with an electronic depth-indicating probe. Prior to obtaining a measurement, a fixed reference point on the well casing will be established for each well to be measured. Unless otherwise established, the reference point is typically established and marked on the north side of the well casing. Do not use protective casings or flush-mounted road boxes as a reference, due to the potential for damage or settlement. The elevation of the reference point shall be obtained by accepted surveying methods, to the nearest 0.01 ft.

The water level probe will be lowered into the well until the meter indicates (via indicator light or tone) the water is reached. The probe will be raised above water level and slowly lowered again until water is indicated. The cable will be held against the side of the inner protective casing at the point designated for water level measurements and a depth reading taken. This procedure will be followed three times or until a consistent value is obtained. The value will be recorded to the nearest 0.01 feet on the Groundwater Level Monitoring Report form.

Upon completion, the probe will be raised to the surface and together with the amount of cable that entered the well casing, will be decontaminated in accordance with methods described in Equipment Decontamination Procedure.

Equipment/Materials:

- Battery-operated, non-stretch electronic water level probe with permanent markings at 0.01 ft. increments, such as the Solinst Model 101 or equivalent.
- The calibrated cable on the depth indicator will be checked against a surveyor's steel tape once per quarter year. A new cable will be installed if the cable has changed by more than 0.01% (0.01 feet for a 100-foot cable). See also the Field Instruments Use and Calibration Procedure.
- Groundwater Level Monitoring Report form.



6. Sample Collection for Laboratory Analysis

6.1 SOIL SAMPLE COLLECTION FOR LABORATORY ANALYSIS

The following procedure is an introduction to soil sampling techniques and an outline of field staff responsibilities. All samples will be collected with dedicated sampling equipment.

6.1.1 Preparatory Requirements

Prior to the beginning of any remedial investigation or remedial measures activities, staff must attend a project briefing for the purpose of reviewing the project work plan, site and utility plans, drawings, applicable regulations, sampling location, depth, and criteria, site contacts, and other related documents. Health and safety concerns will be documented in a site-specific Health & Safety Plan.

A file folder for the field activities should be created and maintained such that all relevant documents and log forms likely to be useful for the completion of field activities by others are readily available in the event of personnel changes.

6.1.2 Soil Classification

The stratigraphic log is a factual description of the soil at the borehole location and is relied upon to interpret the soil characteristics, and their influence and significance in the subsurface environment. The accuracy of the stratigraphic log is to be verified by the person responsible for interpreting subsurface conditions. An accurate description of the soil stratigraphy is essential for a reasonable understanding of the subsurface conditions. Confirmation of the field description by examination of representative soil samples by the project geologist, hydrogeologist, or geotechnical engineer (whenever practicable) is recommended.

The ability to describe and classify soil correctly is a skill that is learned from a person with experience and by systematic training and comparison of laboratory results to field descriptions.

6.1.2.1 Data Recording

Several methods for classifying and describing soils or unconsolidated sediments are in relatively widespread use. The Unified Soil Classification System (USCS) is the most common. With the USCS, a soil is first classified according to whether it is predominantly coarse-grained or fine-grained.

The description of fill soil is similar to that of natural undisturbed soil except that it is identified as fill and not classified by USCS group, relative density, or consistency. Those logging soils must attempt to distinguish between soils that have been placed (i.e., fill) and not naturally present; or soils that have been naturally present but disturbed (i.e., disturbed native).

It is necessary to identify and group soil samples consistently to determine the subsurface pattern or changes and non-conformities in soil stratigraphy in the field at the time of drilling. The stratigraphy in each borehole during drilling is to be compared to the stratigraphy found at the previously completed



boreholes to ensure that pattern or changes in soil stratigraphy are noted and that consistent terminology is used.

Visual examination, physical observations and manual tests (adapted from ASTM D2488, visual-manual procedures) are used to classify and group soil samples in the field and are summarized in this subsection. ASTM D2488 should be reviewed for detailed explanations of the procedures. Visual-manual procedures used for soil identification and classification include:

- visual determination of grain size, soil gradation, and percentage fines;
- dry strength, dilatancy, toughness, and plasticity (thread or ribbon test) tests for identification of inorganic fine-grained soil (e.g., CL, CH, ML, or MH); and
- soil compressive strength and consistency estimates based on thumb indent and pocket penetrometer (preferred) methods.

Soil characteristics like plasticity, strength and dilatancy should be determined using the Haley & Aldrich Soil Identification Field Form.

6.1.2.2 Field Sample Screening

Upon the collection of soil samples, the soil is screened with a photoionization detector (PID) for the presence of organic vapor. This is accomplished by running the PID across the soil sample. The highest reading and sustained readings are recorded.

Note: The PID measurement must be done upwind of the excavating equipment or any running engines so that exhaust fumes will not affect the measurements.

Another method of field screening is head space measurements. This consists of placing a portion of the soil sample in a sealable glass jar, placing aluminum foil over the jar top, and tightening the lid. Alternatively, plastic sealable bags may be utilized for field screen in lieu of glass containers. The jar should only be partially filled. Shake the jar and set aside for at least 30 minutes. After the sample has equilibrated, the lid of the jar can be opened; the foil is punctured with the PID probe and the air (headspace) above the soil sample is monitored. This headspace reading on the field form or in the field book is recorded. All head space measurements must be completed under similar conditions to allow comparability of results. Soil classification and PID readings will be recorded in the daily field report.

Equipment/Materials:

- Pocket knife or small spatula
- Small handheld lens
- Stratigraphic Log (Overburden) (Form 2001)
- Tape Measure
- When sampling for PFAS, acceptable materials for sampling include stainless steel, high density polyethylene (HDPE), PVC, silicone, acetate, and polypropylene.

6.1.3 Soil Sampling

Soil samples will be collected from acetate liners installed by a track-mounted direct push drill rig (Geoprobe®) operated by a licensed operator. Soil samples will be collected using a stainless-steel



trowel or sampling spoon into laboratory provided sample containers. If it is necessary to relocate any proposed sampling location due to terrain, utilities, access, etc., the Project Manager must be notified, and an alternate location will be selected.

Prior to use and between each sampling location at an environmental site, the sampling equipment must be decontaminated. All decontamination must be conducted in accordance with the project specific plans or the methods presented in SOP 7.0.

6.1.4 Sampling Techniques

The following procedure describes typical soil sample collection methods for submission of samples to a laboratory for chemical analysis. The primary goal of soil sampling is to collect representative samples for examination and chemical analysis (if required).

Environmental soil samples obtained for chemical analyses are collected with special attention given to the rationale behind determining the precise zone to sample, the specifics of the method of soil extraction and the requisite decontamination procedures. Preservation, handling and glassware for environmental soil samples varies considerably depending upon several factors including the analytical method to be conducted, and the analytical laboratory being used.

Soil sampling for PFAS will be performed in accordance with NYSDEC, Division of Environmental Remediation, Sampling, Analysis and Assessment of PFAS under NYSDEC Part 375 Remedial Program (April 2023).

6.1.4.1 Grab Versus Composite Samples

A grab sample is collected to identify and quantify conditions at a specific location or interval. The sample is comprised of the minimum amount of soil necessary to make up the volume of sample dictated by the required sample analyses. Composite samples may be obtained from several locations or along a linear trend (in a test pit or excavation). Sampling may occur within or across stratification.

6.2 GROUNDWATER SAMPLE COLLECTION FOR LABORATORY ANALYSIS

The following section describes two techniques for groundwater sampling: "Low Stress/Low Flow Methods" and "Typical Sampling Methods."

"Low Stress/Low Flow" methods will be employed when collecting groundwater samples for the evaluation of volatile constituents (i.e. dissolved oxygen (DO)) or in fine-grained formations where sediment/colloid transport is possible. Analyses typically sensitive to colloidal transport issues include polychlorinated biphenyls (PCBs), polyaromatic hydrocarbons (PAHs) and metals.

The "Typical Sampling Methods" will be employed where the collection of parameters less sensitive to turbidity/sediment issues are being collected (general chemistry, pesticides and other semi-volatile organic compounds (SVOCs)).

NOTE: If non-aqueous phase liquids (NAPL) (light or dense) are detected in a monitoring well, groundwater sample collection will not be conducted, and the Project Manager must be contacted to determine a course of action.



6.2.1 Preparatory Requirements

- Verify well identification and location using borehole log details and location layout figures. Note the condition of the well and record any necessary repair work required.
- Prior to opening the well cap, measure the breathing space above the well casing with a handheld organic vapor analyzer to establish baseline breathing space VOC levels. Repeat this measurement once the well cap is opened. If either of these measurements exceeds the air quality criteria in the HASP, field personnel should adjust their PPE accordingly.
- Prior to commencing the groundwater purging/sampling, a water level must be obtained to
 determine the well volume for hydraulic purposes. In some settings, it may be necessary to
 allow the water level time to equilibrate. This condition exists if a watertight seal exists at the
 well cap and the water level has fluctuated above the top of screen; creating a vacuum or
 pressurized area in this air space. Three water level checks will verify static water level
 conditions have been achieved.
- Calculate the volume of water in the well. Typically overburden well volumes consider only the quantity of water standing in the well screen and riser; bedrock well volumes are calculated on the quantity of water within the open core hole and within the overburden casing.

6.2.2 Well Development

Well development is completed to remove fine grained materials from the well but in such a manner as to not introduce fines from the formation into the sand pack. Well development continues until the well responds to water level changes in the formation (i.e., a good hydraulic connection is established between the well and formation) and the well produces clear, sediment-free water to the extent practical.

- Attach appropriate pump and lower tubing into well.
- Gauge well and calculate one well volume. Turn on pump. If well runs dry, shut off pump and allow to recover.
- Surging will be performed by raising and lowering the pump several times to pull fine-grained material from the well. Periodically measure turbidity level using a La Motte turbidity reader.
- The second and third steps will be repeated until turbidity is less than 50 nephelometric turbidity units (NTU) or when 10 well volumes have been removed.
- All water generated during cleaning and development procedures will be collected and contained on site in 55-gallon drums for future analysis and appropriate disposal.

Equipment:

- Appropriate health and safety equipment
- Knife



- Power source (generator)
- Field book
- Well Development Form (Form 3006)
- Well keys
- Graduated pails
- Pump and tubing
- Cleaning supplies (including non-phosphate soap, buckets, brushes, laboratory-supplied distilled/deionized water, tap water, cleaning solvent, aluminum foil, plastic sheeting, etc.) Water level meter

6.2.3 Well Purging and Stabilization Monitoring (Low Stress/Low Flow Method)

The preferred method for groundwater sampling will be the low stress/low flow method described below.

- Slowly lower the pump, safety cable, tubing and electrical lines into the well to the depth specified by the project requirements. The pump intake must be at the midpoint of the well screen to prevent disturbance and resuspension of any sediment in the screen base.
- Before starting the pump, measure the water level again with the pump in the well leaving the water level measuring device in the well when completed.
- Purge the well at 100 to a maximum of 500 milliliters per minute (mL/min). During purging, the
 water level should be monitored approximately every 5 minutes, or as appropriate. A steady
 flow rate should be maintained that results in drawdown of 0.3 feet or less. The rate of
 pumping should not exceed the natural flow rate conditions of the well. Care should be taken to
 maintain pump suction and to avoid entrainment of air in the tubing. Record adjustments made
 to the pumping rates and water levels immediately after each adjustment.
- During the purging of the well, monitor and record the field indicator parameters (pH, temperature, conductivity, oxidation-reduction (redox) reaction potential (ORP), dissolved oxygen (DO), and turbidity) approximately every five minutes. Stabilization is considered to be achieved when the final groundwater flow rate is achieved, and three consecutive readings for each parameter 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; and
 - Turbidity: 10 percent of the average value of the three readings, or a final value of less than 50 nephelometric turbidity units (NTU).
- The pump must not be removed from the well between purging and sampling.



6.2.4 Sampling Techniques

- If an alternate pump is utilized, the first pump discharge volumes should be discarded to allow the equipment a period of acclimation to the groundwater.
- Samples are collected directly from the pump with the groundwater being discharged directly into the appropriate sample container. Avoid handling the interior of the bottle or bottle cap and don new gloves for each well sampled to avoid contamination of the sample.
- Order of sample collection:
 - Polyfluoroalkyl substances (PFAS)
 - Volatile organic compounds (VOC)
 - 1,4-Dioxane
 - Semi-volatile organic compounds (SVOC)
 - Total Analyte List (TAL) metals
 - PCBs, pesticides, and herbicides
- No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including plumbers tape and sample bottle cap liners with a PTFE layer.
- For low stress/low flow sampling, samples should be collected at a flow rate between 100 and 500 mL/min and such that drawdown of the water level within the well does not exceed the maximum allowable drawdown of 0.3 feet.
- The pumping rate used to collect a sample for VOC should not exceed 100 mL/min. Samples should be transferred directly to the final container 40 mL glass vials completely full and topped with a Teflon cap. Once capped the vial must be inverted and tapped to check for headspace/air presence (bubbles). If air is present, the sample will be discarded, and recollected until free of air.
- Groundwater sampling for PFAS will be performed in accordance with NYSDEC, Division of Environmental Remediation, Sampling, Analysis and Assessment of PFAS under NYSDEC Part 375 Remedial Program (June 2021)
- All samples must be labeled with:
 - A unique sample number
 - Date and time
 - Parameters to be analyzed
 - Project Reference ID
 - Samplers initials
- Labels should be written in indelible ink and secured to the bottle with clear tape.

Equipment/Materials:



- pH meter, conductivity meter, DO meter, ORP meter, nephelometer, temperature gauge
- Field filtration units (if required)
- Purging/sampling equipment
 - Peristaltic Pump
- Water level probe
- Sampling materials (containers, log book/forms, coolers, chain of custody)
- Work Plan
- Health and Safety Plan
- When sampling for PFAS, acceptable materials for sampling include stainless steel, HDPE, PVC, silicone, acetate, and polypropylene.

Note: Peristaltic pump use for VOC collection is not acceptable on NYSDEC/EPA/RCRA sites; this technique has gained acceptance in select areas where it is permissible to collect VOCs using a peristaltic pump at a low flow rate (e.g. Michigan).

Note: 1,4-Dioxane and PFAS purge and sample techniques will be conducted following the NYSDEC guidance documents (see Appendix D of the RIWP). Acceptable groundwater pumps include stainless steel inertia pump with HDPE tubing, peristaltic pump equipped with HDPE tubing and silicone tubing, stainless steel bailer with stainless steel ball or bladder pump (identified as PFAS-free) with HDPE tubing.

Field Notes:

- Field notes must document all the events, equipment used, and measurements collected during the sampling activities. Section 2.0 describes the data/recording procedure for field activities.
- The log book should document the following for each well sampled:
 - Identification of well
 - Well depth
 - Static water level depth and measurement technique
 - Sounded well depth
 - Presence of immiscible layers and detection/collection method
 - Well yield high or low
 - Purge volume and pumping rate
 - Time well purged
 - Measured field parameters
 - Purge/sampling device used
 - Well sampling sequence
 - Sampling appearance
 - Sample odors
 - Sample volume
 - Types of sample containers and sample identification
 - Preservative(s) used
 - Parameters requested for analysis
 - Field analysis data and method(s)
 - Sample distribution and transporter
 - Laboratory shipped to
 - Chain of custody number for shipment to laboratory
 - Field observations on sampling event



- Name collector(s)
- Climatic conditions including air temperature
- Problems encountered and any deviations made from the established sampling protocol.

A standard log form for documentation and reporting groundwater purging and sampling events are presented on the Groundwater Sampling Record, Low Flow Groundwater Sampling Form, and Low Flow Monitored Natural Attenuation (MNA) Field Sampling Form. Refer to Appendix A for example field forms.

Groundwater/Decon Fluid Disposal:

- Groundwater disposal methods will vary on a case-by-case basis but may range from:
 - Off-site treatment at private treatment/disposal facilities or public owned treatment facilities
 - On-site treatment at Facility operated facilities
 - Direct discharge to the surrounding ground surface, allowing groundwater infiltration to the underlying subsurface regime
- Decontamination fluids should be segregated and collected separately from wash waters/groundwater containers.

6.3 SOIL VAPOR SAMPLING

The following procedure is an introduction to soil vapor sampling techniques and an outline of field staff responsibilities.

6.3.1 Preparatory Requirements

Prior to collecting the field sample, ensure the stainless-steel or polyethylene soil vapor probe has been installed to the desired depth and sealed completely to the surface using a material such as bentonite. As part of the vapor intrusion evaluation, a tracer gas should be used in accordance with NYSDOH protocols to serve as a quality assurance/quality control (QA/QC) to verify the integrity of the soil vapor probe seal. A container (box, plastic pail, etc.) will serve to keep the tracer gas in contact with the probe during testing. A portable monitoring device will be used to analyze a sample of soil vapor for the tracer gas prior to sampling. If the tracer sample results show a significant presence of the tracer, the probe seals will be adjusted to prevent infiltration. At the conclusion of the sampling round, tracer monitoring should be performed a second time to confirm the integrity of the probe seals.

6.3.2 Sampling Techniques

Samples will be collected in appropriately sized Summa canisters that have been certified clean by the laboratory and samples will be analyzed by using USEPA Method TO-15. Flow rate for both purging and sampling will not exceed 0.2 L/min. One to three implant volumes shall be purged prior to the collection of any soil-gas samples. A sample log sheet will be maintained summarizing sample identification, date and time of sample collection, sampling depth, identity of samplers, sampling methods and devices, soil vapor purge volumes, volume of the soil vapor extracted, vacuum of canisters before and after the samples are collected, apparent moisture content of the sampling zone, and chain of custody protocols.



6.4 SAMPLE HANDLING AND SHIPPING

Sample management is the continuous care given to each sample from the point of collection to receipt at the analytical laboratory. Good sample management ensures that samples are properly recorded, properly labeled, and not lost, broken, or exposed to conditions which may affect the sample's integrity.

All sample submissions must be accompanied with a chain of custody (COC) document to record sample collection and submission. Personnel performing sampling tasks must check the sample preparation and preservation requirements to ensure compliance with the Quality Assurance Project Plan.

The following sections provide the minimum standards for sample management.

6.4.1 Sample Handling

Prior to entering the field area where sampling is to be conducted, especially at sites with defined exclusion zones, the sampler should ensure that all materials necessary to complete the sampling are on hand. If samples must be maintained at a specified temperature after collection, dedicated coolers and ice must be available for use. Conversely, when sampling in cold weather, proper protection of water samples, trip blanks, and field blanks must be considered. Sample preservation will involve pH adjustment, cooling to 4°C, and sample filtration and preservation.

6.4.2 Sample Labeling

Samples must be properly labeled immediately upon collection.

Note that the data shown on the sample label is the minimum data required. The sample label data requirements are listed below for clarity.

- Project name
- Sample name/number/unique identifier
- Sampler's initials
- Date of sample collection
- Time of sample collection
- Analysis required
- Preservatives

To ensure that samples are not confused, a clear notation should be made on the container with a permanent marker. If the containers are too soiled for marking, the container can be put into a "zip lock" bag which can then be labeled.

All sample names will be as follows:

- Sample unique identifier: Enter the sample name or number. There should be NO slashes, spaces or periods in the date.
- Date: Enter the six-digit date when the sample was collected. Note that for one-digit days, months, and/or years, add zeros so that the format is MMDDYY (050210). There should be NO slashes, dashes, or periods in the date.



The QA/QC samples will be numbered consecutively as collected with a sample name, date and number of sample collected throughout the day (i.e. when multiple QA/QC samples are collected in one day).



Examples of this naming convention are as follows:

Sample Name:	Comments
TB-050202-0001	TRIP BLANK
TB-050202-0002	TRIP BLANK
FD-050202-0001	FIELD DUPLICATE
FD-050202-0002	FIELD DUPLICATE

NOTE: The QA/QC Sample # resets to 0001 EACH DAY, this will avoid having to look back to the previous day for the correct sequential number.

6.4.3 Field Code

The field code will be written in the 'Comments' field on the chain of custody for EVERY sample but will not be a part of the actual sample name. Enter the one/two-character code for type of sample (must be in CAPITALS):

- N Normal Field Sample
- FD Field Duplicate (note sample number (i.e. 0001) substituted for time)
- TB Trip Blank (note sample number (i.e. 0001) substituted for time)
- EB Equipment Blank (note sample number (i.e. 0001) substituted for time)
- FB Field Blank (note sample number (i.e. 0001) substituted for time)
- KD Known Duplicate
- FS Field Spike Sample
- MS Matrix Spike Sample (note on 'Comments' field of COC laboratory to spike matrix.
- MD Matrix Spike Duplicate Sample (note on 'Comments' field of COC laboratory to spike matrix.
- RM Reference Material

The sample labeling – both chain and sample bottles must be EXACTLY as detailed above. In addition, the Field Sample Key for each sample collected must be filled out.

6.4.4 Packaging

Sample container preparation and packing for shipment should be completed in a well-organized and clean area, free of any potential cross contamination. The following is a list of standard guidelines which must be followed when packing samples for shipment.

- Double bag ice in "Zip Lock" bags.
- Double check to ensure trip and temperature blanks have been included for all shipments containing VOCs, or where otherwise specified in the QAPP.
- Enclose the Chain of Custody form in a "Zip Lock" bag.
- Ensure custody seals (two, minimum) are placed on each cooler. Coolers with hinged lids should have both seals placed on the opening edge of the lid. Coolers with "free" lids should have seals placed on opposite diagonal corners of the lid. Place clear tape over custody seals.
- Containers should be wiped clean of all debris/water using paper towels (paper towels must be disposed of with other contaminated materials).
- Clear, wide packing tape should be placed over the sample label for protection.



- Do not bulk pack. Each sample must be individually padded.
- Large glass containers (1 liter and up) require much more space between containers.
- Ice is not a packing material due to the reduction in volume when it melts.

Note: Never store sterile sample containers in enclosures containing equipment which use any form of fuel or volatile petroleum-based product. When conducting sampling in freezing conditions at sites without a heated storage area (free of potential cross contaminants), unused trip blanks should be isolated from coolers immediately after receipt. Trip blanks should be double bagged and kept from freezing.

6.4.5 Chain-of-Custody Records

Chain of custody (COC) forms will be completed for all samples collected. The form documents the transfer of sample containers. The COC record, completed at the time of sampling, will contain, but not be limited to, the sample number, date and time of sampling, and the name of the sampler. The COC document will be signed and dated by the sampler when transferring the samples.

Each sample cooler being shipped to the laboratory will contain a COC form. The cooler will be sealed properly for shipment. The laboratory will maintain a copy for their records. One copy will be returned with the data deliverables package.

The following list provides guidance for the completion and handling of all COCs:

- COCs used should be a Haley & Aldrich standard form or supplied by the analytical laboratory.
- COCs must be completed in black ball point ink only.
- COCs must be completed neatly using printed text.
- If a simple mistake is made, cross out the error with a single line and initial and date the correction.
- Each separate sample entry must be sequentially numbered.
- If numerous repetitive entries must be made in the same column, place a continuous vertical arrow between the first entry and the next different entry.
- When more than one COC form is used for a single shipment, each form must be consecutively numbered using the "Page ____ of ____" format.
- If necessary, place additional instructions directly onto the COC in the Comment Section. Do not enclose separate instructions.
- Include a contact name and phone number on the COC in case there is a problem with the shipment.
- Before using an acronym on a COC, define clearly the full interpretation of your designation [i.e., polychlorinated biphenyls (PCBs)].

6.4.6 Shipment

Prior to the start of the field sampling, the carrier should be contacted to determine if pickup will be at the field site location. If pick-up is not available at the Site, the nearest pick-up or drop off location should be determined. Sample shipments must not be left at unsecured drop locations.

Copies of all shipment manifests must be maintained in the field file.



7. Field Instruments – Use and Calibration

A significant number of field activities involve usage of electronic instruments to monitor for environmental conditions and health and safety purposes. It is imperative the instruments are used and maintained properly to optimize their performance and minimize the potential for inaccuracies in the data obtained. This section provides guidance on the usage, maintenance and calibration of electronic field equipment.

- All monitoring equipment will be in proper working order and operated in accordance with manufacturer's recommendations.
- Field personnel will be responsible for ensuring that the equipment is maintained and calibrated in the field in accordance with manufacturer's recommendations.
- Instruments will be operated only by personnel trained in the proper usage and calibration.
- Personnel must be aware of the range of conditions such as temperature and humidity for instrument operation. Usage of instruments in conditions outside these ranges will only proceed with approval of the Project Manager and/or Health and Safety Officer as appropriate.
- Instruments that contain radioactive source material, such as x-ray fluorescence (XRF) analyzers
 or moisture-density gauges require specific transportation, handling and usage procedures that
 are generally associated with a license from the Nuclear Regulatory Commission (NRC) or an
 NRC-Agreement State. Under no circumstance will operation of such instruments be allowed on
 site unless by properly authorized and trained personnel, using the proper personal dosimetry
 badges or monitoring instruments.

7.1 GENERAL PROCEDURE DISCUSSION

Care must be taken to minimize the potential for transfer of contaminated materials to the ground or onto other materials. Regardless of the size or nature of the equipment being decontaminated, the process will utilize a series of steps that involve removal of gross material (dirt, grease, oil etc.), washing with a detergent, and multiple rinsing steps. In lieu of a series of washes and rinse steps, steam cleaning with low-volume, high-pressure equipment (i.e., steam cleaner) is acceptable.

Exploration equipment, and all monitoring equipment in contact with the sampling media must be decontaminated prior to initiating site activities, in between exploration locations to minimize cross-contamination, and prior to mobilizing off site after completion of site work.

The following specific decontamination procedure is recommended for sampling equipment and tools:

- Brush loose soil off equipment;
- Wash equipment with laboratory grade detergent (i.e., Alconox or equivalent);
- Rinse with tap water;
- Rinse equipment with distilled water;
- Allow water to evaporate before reusing equipment; and



• Wrap equipment in aluminum foil when not being used.

7.2 DECONTAMINATION OF MONITORING EQUIPMENT

Because monitoring equipment is difficult to decontaminate, care should be exercised to prevent contamination. Sensitive monitoring instruments should be protected when they are at risk of exposure to contaminants. This may include enclosing them in plastic bags allowing an opening for the sample intake. Ventilation ports should not be covered.

If contamination does occur, decontamination of the equipment will be required; however, immersion in decontamination fluids is not possible. As such, care must be taken to wipe the instruments down with detergent-wetted wipes or sponges, and then with de-ionized water-wetted wipes or sponges.

7.3 DISPOSAL OF WASH SOLUTIONS AND CONTAMINATED EQUIPMENT

All contaminated wash water, rinses, solids, and materials used in the decontamination process that cannot be effectively decontaminated (such as polyethylene sheeting) will be containerized and disposed of in accordance with applicable regulations. All containers will be labeled with an indelible marker as to contents and date of placement in the container, and any appropriate stickers required (such as PCBs). Storage of decontamination wastes on site will not exceed 90 days under any circumstances.

Equipment/Materials:

Decontamination equipment and solutions are generally selected based on ease of decontamination and disposability.

- Polyethylene sheeting;
- Metal racks to hold equipment;
- Soft-bristle scrub brushes or long-handle brushes for removing gross contamination and scrubbing with wash solutions;
- Large galvanized wash tubs, stock tanks, or wading pools for wash and rinse solutions;
- Plastic buckets or garden sprayers for rinse solutions;
- Large plastic garbage cans or other similar containers lined with plastic bags can be used to store contaminated clothing;
- Contaminated liquids and solids should be segregated and containerized in DOT-approved plastic or metal drums, appropriate for offsite shipping/disposal if necessary.



8. Investigation Derived Waste Disposal

8.1 RATIONALE/ASSUMPTIONS

This procedure applies to the disposition of investigation derived waste (IDW) including soils and/or groundwater. IDW is dealt with the following "Best Management Practices" and is not considered a listed waste due to the lack of generator knowledge concerning chemical source, chemical origin, and timing of chemical introduction to the subsurface.

Consequently, waste sampling and characterization is performed to determine if the wastes exhibit a characteristic of hazardous waste. The disposal of soil cuttings, test pit soils and/or purged groundwater will be reviewed on a case-by-case basis prior to initiation of field activities. Two scenarios typically exist:

- When no information is available in the area of activity or investigation, and impacted media/soils are identified. Activities such as new construction and /or maintenance below grade may encounter environmental conditions that were unknown.
- Disposal Required/Containerization Required When sufficient Site information regarding the investigative Site conditions warrant that all materials handled will be contained and disposed.

If a known listed hazardous and/or characteristically hazardous waste/contaminated environmental media is being handled, then handling must be performed in accordance with RCRA Subtitle C (reference 2, Part V, Section 1(a),(b),(c)).

The following outlines the waste characterization procedures to be employed when IDW disposal is required.

The following procedure describes the techniques for characterization of IDW for disposal purposes. IDW may consist of soil cuttings (augering, boring, well installation soils, test pit soils), rock core or rock flour (from coring, reaming operations), groundwater (from well development, purging and sampling activities), decontamination fluids, personal protective equipment (PPE), and disposal equipment (DE).

8.2 PROCEDURE

The procedures for handling and characterization of field activity generated wastes are:

- A.) Soil Cuttings Soils removed from boring activities will be contained within an approved container, suitable for transportation and disposal.
 - Once placed into the approved container, any free liquids (i.e., groundwater) will be removed for disposal as waste fluids or solidified within the approved container using a solidification agent such as Speedy Dri (or equivalent).
 - Contained soils will be screened for the presence of Volatile Organic Compounds (VOCs), using a Photo ionization detector (PID); this data will be logged for future reference.
 - Once screened, full and closed; the container will be labeled and placed into the container storage area. At a minimum, the following information will be shown on each container



label: date of filling/generation, Site name, source of soils (i.e., borehole or well), and contact.

- Prior to container closure, representative samples from the containers will be collected for waste characterization purposes and submitted to the project laboratory.
- Typically, at a location where an undetermined site-specific parameter group exists, sampling and analysis may consist of the full RCRA Waste Characterization (ignitability, corrosivity, reactivity, toxicity), or a subset of the above based upon data collected, historical information, and generator knowledge.
- B.) Groundwater purging, and sampling groundwater, which requires disposal, will be contained.
 - Containment may be performed in 55-gallon drums, tanks suitable for temporary storage (i.e., Nalgene tanks 500 to 1,000 gallons) or if large volumes of groundwater are anticipated, tanker trailer (5,000 to 10,000 gallons ±), or drilling "Frac" tanks may be utilized (20,000 gallons ±). In all cases the container/tank used for groundwater storage must be clean before use such that cross contamination does not occur.
- C.) Decon Waters/Decon Fluids Decon waters and/or fluids will be segregated, contained, and disposed accordingly.
 - Decon waters may be disposed of with the containerized groundwater once analytical results have been acquired.
- D.) PPE/DE A number of disposal options exists for spent PPE/DE generated from investigation tasks. The options typically employed are:
 - Immediately disposed of within on-Site dumpster/municipal trash; or
 - If known to be contaminated with RCRA hazardous waste, dispose off-Site at a RCRA Subtitle C facility.
 - Spent Solvent/Acid Rinses The need for sampling must be determined in consultation with the waste management organization handling the materials. If known that only the solvent and/or acids are present, then direct disposal/treatment using media specific options may be possible without sampling (i.e., incineration).
 - PPE/DE Typically not sampled and included with the disposal of the solid wastes.

Equipment/Materials:

- Sample spoons, trier, auger,
- Sample mixing bowl,
- Sampling bailer, or pump,
- Sample glassware.



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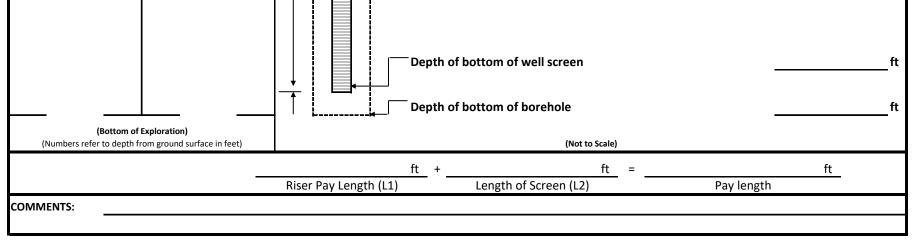


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APPENDIX A Field Forms

SOIL BORING LOG									Pag	BORING NO. Je 1 of									
PROJECT												F	PROJ	ECT #				1. 442	•
LOCATIO	N													ECT MG	R.				
CLIENT CONTRAC	TOR													REP.	FD				
DRILLER												-		FINISH					
Elevation		ft.	Datum		Boring	Location													
ltem		Casing		Sampler	Rig Ma	ke & Mode							Han	nmer Typ	oe 🛛	Dr	illing Mu		Casing Advance
Type Inside Dian	neter (in)				Tru		Tripoo Geop				-Head			Safety Doughr	out		Bentor Polym		Type Method Depth
Hammer W					🗌 Tra	ck	Air Tr				er Bit			Automa			None		
Hammer Fa	all (in.)				Ski	d	Other			Cut	ting Head	C	Drilling	g Notes:					
Depth (ft.)	Recovery (in/tot)	PID (ppm)	Sample Depth (ft)	Sample ID		(Color, prim	ary compone				ntification &				noisture	∍ [SYM	BOL])		Remarks
- 0 -																			
1																			
2																			
2																			
3																			
4																			
- 5 -																			
6																			
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29																			
20																			
_ 30 _		Weter	Level Data			1			Sample				-				C	marri	
		vvater		epth in feet to:		<u> </u>		:	Sample I	U							Sum	ndry	
Date	Time	Elapsed Time (hr.)		Water		O Open End Rod T Thin Wall Tube U Undisturbed Sample S Split Spoon Sample G Geoprobe					Overburden (Linear ft.) Rock Cored (Linear ft.) Number of Samples								
											BORING NO.								
<u> </u>			*Ni	OTE: Maximum Pa	ticle Siz	e is deter	mined by d	irect o	bservati	ion with	in the limitat	tions	of sar	npler siz	e.				
For	n #3000	N		criptions based on												drich,	nc.		

							Well No.
ALDRICH	PER	MAN	IENT	WELL INSTAL	LATION REPO	DRT	Boring No.
PROJECT					H&A FILE NO.		
LOCATION					PROJECT MGI	R.	
CLIENT					FIELD REP.		
CONTRACTOR					DATE INSTALI		
DRILLER					WATER LEVEL		
Ground El. El. Datum	ft	Locatio	n		Drilling Equipment		Guard Pipe
SOIL/ROCK	BOREHOLE						
CONDITIONS	BACKFILL			Type of protective cov	ver/lock (circle one): Per	nt.bolt 9/16" hex. dlock key no	
				Height /Depth	of top of guard pipe/roadv		ft
				above/below g			"
	-			Height/Depth	of top of riser pipe		ft
				above/below g			
				Type of protect	tive casing:		
				Length			ft
				Inside Diam	eter		in
				Depth of botto	m of guard pipe/roadway	box	ft
					Type of Seals	<u>Top of Seal (ft)</u>	<u>Thickness (ft)</u>
							·
		L:					·
				Type of riser pi	201		
					eter of riser pipe		in
					kfill around riser		
				.,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,			
	-			Diameter of bo	rehole		in
				Depth to top o	f well screen		ft
				Type of screen		Machine	Slotted PVC
					ge or size of openings		in
		Li I		Diameter o			in
				Type of backfil	around screen		



HALEY	LOW-FLOW GROUNDWATER SAMPLING RECORD												
PROJECT				H&A FILE NO.									
LOCATION				PROJECT MGR.									
CLIENT				FIELD REP									
CONTRACTOR				DATE									
			GROUNDWATE	R SAMPLING INFO	RMATION								
Well ID:		-	Well Volume:		-	Start Time:							
Well Depth:		_	Equipment:		-	Sample Time:							
Depth to Water:		-											
Time	Volume purged, gallons	Temp, C (+/-3%)	Conductivity, us/cm (+/- 3%)	Dissolved Oxygen, mg/L (+/- 10%)	рН (+/-0.1)	ORP/eH, mv (+/-10mv)	Turbidity, NTU (<5 NTU)	Depth to Water (ft)					
<u> </u>													
	1												

HALEY ALDR	ICH	SOIL VAPOR SAMPLING LOG								
		Project Name/	Location:			-	Project Number:		-	
Site: Date Collected: Personnel: Weather: Humidity:			- - - -							
Sample ID	Canister ID	Caniser Size	Flow Controller ID	Sample Start Time	Canister Start Pressure ("Hg)	Sample End Time	Canister End Pressure ("Hg)	Sample Start Date	Sample Type	Analyses Method

Notes:

Summas and flow regulators provided by <NAME OF LAB> Analyses for VOCs by Method TO-15/TO-15SIM (circle one) **APPENDIX C** Quality Assurance Project Plan



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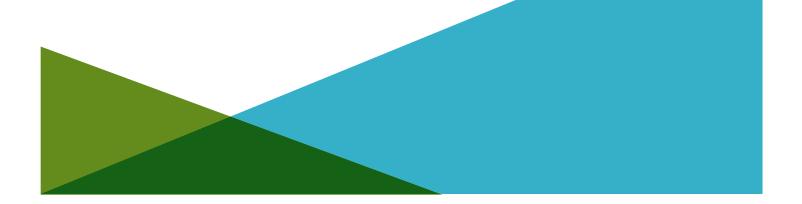
ALDRICH

QUALITY ASSURANCE PROJECT PLAN 90-02 QUEENS BOULEVARD SITE 90-02 QUEENS BOULEVARD QUEENS, NEW YORK

by Haley & Aldrich of New York New York, New York

for 9002 Commercial Unit LLC New York, New York

File No. 0208933 May 2024



Executive Summary

This Quality Assurance Project Plan (QAPP) outlines the scope of the quality assurance and quality control (QA/QC) activities associated with the site monitoring activities associated with the Site Characterization Work Plan (SCWP) for the 90-02 Queens Boulevard Site (Site) located at 90-02 Queens Boulevard in Queens, New York.

Protocols for sample collection, sample handling and storage, chain-of-custody procedures, and laboratory and field analyses are described herein or specifically referenced to related project documents.



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В	Project Team Resumes
С	Laboratory PFAS SOPs



1. **Project Description**

This Quality Assurance Project Plan (QAPP) has been prepared as a component of the SCWP for the 90-02 Queens Boulevard Site located at 90-02 Queens Boulevard in Queens, New York.

1.1 PROJECT OBJECTIVES

The primary objective for data collection activities is to collect sufficient data necessary to confirm the results of the previous site characterization activities, potentially identify an on-site source, and to determine a course for remedial action. In addition, a qualitative exposure assessment will be conducted and will consider the nature of populations currently exposed or that have the potential to be exposed to Site-related contaminants both on- and off-site, along with describing the reasonably anticipated future land use of the site and affected off-site areas.

1.2 SITE DESCRIPTION AND HISTORY

The general Site description and Site history is provided in the Site Description and History Summary that accompanies the SCWP for the Site and incorporated herein by reference.

1.3 LABORATORY PARAMETERS

The laboratory parameters for soil include:

- Target Compound List volatile organic compounds (VOCs) using USEPA method 8260C/5035
- Target Compound List semi-volatile organic compounds (SVOCs) using USEPA method 8270D
- Total Analyte List (TAL) Metals (including hexavalent chromium, and cyanide) using USEPA method 6010C/7471B/9010C/7196A
- Polychlorinated biphenyls (PCBs) using USEPA method 8082A
- TCL Pesticides using USEPA method 8081B for historic fill samples
- Per- and polyfluoroalkyl substances (PFAS) using USEPA method 1633
- 1,4-Dioxane using USEPA method 8270 SIM

The laboratory parameters for groundwater include:

- Target Compound List VOCs using USEPA method 8260B
- Target Compound List SVOCs using USEPA method 8270C
- Total Analyte List (TAL) Metals using USEPA method 6010/7471
- PCBs using USEPA method 8082
- Pesticides by USEPA method 8081B
- PFAS using USEPA method 1633
- 1,4-Dioxane using USEPA method 8270D SIM isotope dilution



PFAS Analyte List:

PFAS Analyte Description	Laboratory RL for solids (µg/kg)	Laboratory MDL for solids (µg/kg)	Laboratory RL for aqueous (ng/L)	Laboratory MDLs for aqueous (ng/L)
Perfluorobutanoic acid (PFBA)	0.800	0.200	8.00	2.13
Perfluoropentanoic acid (PFPeA)	0.400	0.100	4.00	1.00
Perfluorohexanoic acid (PFHxA)	0.200	0.0500	2.00	0.500
Perfluoroheptanoic acid (PFHpA)	0.200	0.0500	2.00	0.500
Perfluorooctanoic acid (PFOA)	0.200	0.0500	2.00	0.500
Perfluorononanoic acid (PFNA)	0.200	0.0500	2.00	0.500
Perfluorodecanoic acid (PFDA)	0.200	0.0500	2.00	0.500
Perfluoroundecanoic acid (PFUnA)	0.200	0.0500	2.00	0.500
Perfluorododecanoic acid (PFDoA)	0.200	0.0500	2.00	0.500
Perfluorotridecanoic acid (PFTrDA)	0.200	0.0500	2.00	0.500
Perfluorotetradecanoic acid (PFTeDA)	0.200	0.0500	2.00	0.500
Perfluorobutanesulfonic acid (PFBS)	0.200	0.0500	2.00	0.500
Perfluoropentanesulfonic acid (PFPeS)	0.200	0.0500	2.00	0.500
Perfluorohexanesulfonic acid (PFHxS)	0.200	0.0500	2.00	0.517
Perfluoroheptanesulfonic acid (PFHpS)	0.200	0.0500	2.00	0.500
Perfluorooctanesulfonic acid (PFOS)	0.200	0.0500	2.00	0.500
Perfluorononanesulfonic acid (PFNS)	0.200	0.0500	2.00	0.500
Perfluorododecanesulfonic acid (PFDoS)	0.200	0.0500	2.00	0.500
1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2 FTS)	0.800	0.200	8.00	2.00
1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2 FTS)	0.800	0.200	8.00	2.00
1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2 FTS)	0.800	0.200	8.00	2.00
Perfluorooctanesulfonamide (PFOSA)	0.200	0.0500	2.00	0.500
N-methylperfluorooctane sulfonamide (NMeFOSA)	0.200	0.0500	2.00	0.500
N-ethylperfluorooctane sulfonamide (NEtFOSA)	0.200	0.0500	2.00	0.500
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	0.200	0.0500	2.00	0.620
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	0.200	0.0500	2.00	0.521
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	2.00	0.500	20.0	5.00
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	2.00	0.500	20.0	5.00
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	0.800	0.200	8.00	2.00
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	0.800	0.200	8.00	2.00
Perfluoro-4-methoxybutanoic acid (PFMBA)	0.400	0.100	4.00	1.00
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	0.400	0.100	4.00	1.22
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	0.800	0.200	8.00	2.00
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	0.800	0.200	8.00	2.00
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	0.400	0.100	4.00	1.00
3-Perfluoropropylpropanoic acid (3:3 FTCA)	1.00	0.250	10.0	2.50
3-Perfluoropentylpropanoic acid (5:3 FTCA)	5.00	1.25	50.0	12.5
3-Perfluoroheptylpropanoic acid (7:3 FTCA)	5.00	1.25	50.0	14.9
Perfluorodecanesulfonic acid (PFDS)	0.200	0.0500	2.00	0.500
Perfluoro-3-methoxypropanoic acid (PFMPA)	0.400	0.100	4.00	1.00

Notes: RL – Reporting Limit, MDL – Method Detection Limit; All RLs and MDLs provided by Eurofins Scientific



1,4-Dioxane	Laboratory RL for solids (mg/kg)	Laboratory MDL for solids (mg/kg)	Laboratory RL for aqueous (μg/L)	Laboratory MDLs for aqueous (µg/L)
1,4-Dioxane	0.0330	0.0289	0.200	0.0720

Note: 1,4-Dioxane and PFAS sampling techniques will be conducted following the NYSDEC Collection of Groundwater Samples for Per- and Polyfluoroalkyl Substances (PFAS) from Monitoring Wells Sample Protocol (April 2023).

During the collection of groundwater samples, pH, specific conductivity, temperature, dissolved oxygen (DO), and oxidation/reduction potential (ORP) will be measured until stabilized.

The laboratory parameter for soil vapor, indoor air and ambient air includes:

• VOCs using EPA method TO-15

Laboratory parameters for disposal samples will be determined by the disposal facility after an approved facility has been determined.

1.4 SAMPLING LOCATIONS

The SCWP provides the locations of soil borings, soil vapor points, indoor air/ambient air locations and groundwater monitoring wells that will be sampled.



2. Project Organization and Responsibilities

This section defines the roles and responsibilities of the individuals who will perform the SCWP monitoring activities. A NYSDOH certified analytical laboratory will perform the analyses of environmental samples collected at the Site. See Appendix A for data validation team resumes and Appendix B for project team resumes.

2.1 MANAGEMENT RESPONSIBILITIES

James Bellew will be the Principal in Charge and Qualified Environmental Professional (QEP) for this work. In this role, Mr. Bellew will be responsible for the overall completion of each task as per requirements outlined in this work plan and in accordance with the DER-10 guidance.

Mari Conlon P.G. will be the Project Manager for this work. In this role, Ms. Conlon will manage the dayto-day tasks, including coordination and supervision of field engineers and scientists, adherence to the work plan and oversight of project schedule. As the Project Manager, Ms. Conlon will also be responsible for communications with the NYSDEC Case Manager regarding project status, schedule, issues, and updates for project work.

The Project Manager will be responsible for managing the implementation of the SCWP and monitoring and coordinating the collection of data. The Project Manager will be responsible for technical quality control and project oversight. The Project Manager responsibilities include the following:

- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule restraints;
- Review work performed to ensure quality, responsiveness, and timeliness;
- Communicate with the client point of contact concerning the progress of the monitoring activities;
- Assure corrective actions are taken for deficiencies cited during audits of SCWP monitoring activities; and
- Overall Site health and safety plan compliance.

Hailey Russell will be the field engineer responsible for implementing the field effort for this work. His responsibilities will include implementing the work plan activities and directing the subcontractors to ensure successful completion of all field activities.

2.2 QUALITY ASSURANCE RESPONSIBILITIES

The Quality Assurance team will consist of a Quality Assurance Officer and the Data Validation staff. Quality Assurance responsibilities are described as follows:

2.2.1 Quality Assurance (QA) Officer

The QA Officer will be Sebastian Sotomayor and he will report directly to the Project Manager and will be responsible for overseeing the review of field and laboratory data. Additional responsibilities include the following:



- Assure the application and effectiveness of the QAPP by the analytical laboratory and the project staff;
- Provide input to the Project Manager as to corrective actions that may be required as a result of the above-mentioned evaluations;
- Prepare and/or review data validation and audit reports.

The QA Officer will be assisted by the data validation staff in the evaluation and validation of field and laboratory generated data.

2.2.2 Data Validation Staff

The data validation staff will be Oscar Cervantes and Kirkland Broadwell will be independent of the laboratory and familiar with the analytical procedures performed. The validation will include a review of each validation criterion as prescribed by the guidelines presented in Section 9.2 of this document and be presented in a Data Usability Summary Report (DUSR) for submittal to the QA Officer.

2.3 LABORATORY RESPONSIBILITIES

Laboratory services provided by Eurofins Scientific (an ELAP approved lab) in support of the SCWP include the following personnel:

2.3.1 Laboratory Project Manager

The Laboratory Project Manager will report directly to the QA Officer and Project Manager and will be responsible for ensuring all resources of the laboratory are available on an as-required basis. The Laboratory Project Manager will also be responsible for the approval of the final analytical reports.

2.3.2 Laboratory Operations Manager

The Laboratory Operations Manager will report to the Laboratory Project Manager and will be responsible for coordinating laboratory analysis, supervising in-house chain-of-custody reports, scheduling sample analyses, overseeing data review and overseeing preparation of analytical reports.

2.3.3 Laboratory QA Officer

The Laboratory QA Officer will have sole responsibility for review and validation of the analytical laboratory data. The Laboratory QA Officer will provide Case Narrative descriptions of any data quality issues encountered during the analyses conducted by the laboratory. The QA Officer will also define appropriate QA procedures, overseeing QA/QC documentation.

2.3.4 Laboratory Sample Custodian

The Laboratory Sample Custodian will report to the Laboratory Operations Manager and will be responsible for the following:

- Receive and inspect the incoming sample containers;
- Record the condition of the incoming sample containers;
- Sign appropriate documents;



- Verify chain-of-custody and its correctness;
- Notify the Project Manager and Operations Manager of sample receipt and inspection;
- Assign a unique identification number and enter each into the sample receiving log;
- Initiate transfer of samples to laboratory analytical sections; and
- Control and monitor access/storage of samples and extracts.

2.3.5 Laboratory Technical Personnel

The laboratory technical staff will have the primary responsibility in the performance of sample analysis and the execution of the QA procedures developed to determine the data quality. These activities will include the proper preparation and analysis of the project samples in accordance with the laboratory's Quality Assurance Manual (QAM) and associated Standard Operating Procedures (SOPs). Laboratory SOPs are included in Attachment C.



2.4 FIELD RESPONSIBILITIES

2.4.1 Field Coordinator

The Field Coordinator is responsible for the overall operation of the field team and reports directly to the Project Manager. The Field Coordinator works with the project Health & Safety Officer (HSO) to conduct operations in compliance with the project Health & Safety Plan (HASP). The Field Coordinator will facilitate communication and coordinate efforts between the Project Manager and the field team members.

Other responsibilities include the following:

- Develop and implement field-related work plans, ensuring schedule compliance, and adhering to management-developed project requirements;
- Coordinate and manage field staff;
- Perform field system audits;
- Oversee quality control for technical data provided by the field staff;
- Prepare and approve text and graphics required for field team efforts;
- Coordinate and oversee technical efforts of subcontractors assisting the field team;
- Identify problems in the field; resolve difficulties in consultation with the Project QAO, and Project Manager; implement and document corrective action procedures; and,
- Participate in preparation of the final reports.

2.4.2 Field Team Personnel

Field Team Personnel will be responsible for the following:

- Perform field activities as detailed in the SCWP and in compliance with the Field Sampling Plan (FSP) and QAPP.
- Immediately report any accidents and/or unsafe conditions to the Site Health & Safety Officer and take reasonable precautions to prevent injury.



3. Sampling Procedures

The FSP provides the SOPs for sampling required by the SCWP. Sampling will be conducted in general accordance with the New York State Department of Conservation (NYSDEC) Technical Guidance for Site Investigation and Remediation (DER-10) and the Sampling, Analysis and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) under NYSDEC Part 375 Remedial Program when applicable.

3.1 SAMPLE CONTAINERS

Sample containers for each sampling task will be provided by the laboratory performing the analysis. The containers will be cleaned by the manufacturer to meet or exceed the analyte specifications established in the U.S. EPA, "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers", April 1992, OSWER Directive #9240.0-0.5A. Certificates of analysis for each lot of sample containers used will be maintained by the laboratory.

The appropriate sample containers, preservation method, maximum holding times, and handling requirements for each sampling task are provided in Table I.

3.2 SAMPLE LABELING

Each sample will be labeled with a unique sample identifier that will facilitate tracking and crossreferencing of sample information. Equipment rinse blank and field duplicate samples also will be numbered with a unique sample identifier to prevent analytical bias of field QC samples.

Refer to the FSP for the sample labeling procedures.

3.3 PFAS SAMPLING PROTOCOL

Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633.

3.3.1 PFAS Sample Containers and Storage

Sample containers will be high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory. No sampling equipment components or sample containers should come in to contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon[™]) materials including sample bottle cap liners with a PTFE layer. Only regular ice may be used for sample shipment.

3.3.2 PFAS Sampling Equipment

Materials for sampling may include stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation. No sampling equipment components or sample containers will come in to contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon[™]) materials including sample bottle cap liners with a PTFE layer.



Sample equipment may include the below, but other equipment may be considered appropriate based on sampling conditions. All groundwater sampling pumps will utilize PFAS-Free tubing and parts.

- stainless steel spoon
- stainless steel bowl
- steel hand auger or shovel without any coatings
- bladder pump
- monsoon pump
- peristaltic pump

3.3.3 **PFAS Equipment Decontamination**

Standard two step decontamination using detergent (i.e. Alconox) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

3.4 FIELD QC SAMPLE COLLECTION

3.4.1 Field Duplicate Sample Collection

3.4.1.1 Water Samples

Field duplicate samples will be collected by filling the first sample container to the proper level and sealing and then repeated for the second set of sample container.

- 1. The samples are properly labeled as specified in Section 3.2.
- 2. Steps 1 through 4 are repeated for the bottles for each analysis. The samples are collected in order of decreasing analyte volatility as detailed in Section 3.3.1.
- 3. Chain-of-custody documents are executed.
- 4. The samples will be handled as specified in Table I.

3.4.1.2 Soil Samples

Soil field duplicates will be collected as specified in the following procedure:

- 1. Soils will be sampling directly from acetate liners.
- 2. Soil for VOC analysis will be removed from the sampling device as specified in the FSP.
- 3. Soil for non-VOC analysis will be removed from the sampling device and collected into clean laboratory provided containers.



4. Custody Procedures

Sample custody is addressed in three parts: field sample collection, laboratory analysis and final project files. Custody of a sample begins when it is collected by or transferred to an individual and ends when that individual relinquishes or disposes of the sample.

A sample is under custody if:

- 1. The item is in actual possession of a person;
- 2. The item is in the view of the person after being in actual possession of the person;
- 3. The item was in actual possession and subsequently stored to prevent tampering; or
- 4. The item is in a designated and identified secure area.

4.1 FIELD CUSTODY PROCEDURES

Field personnel will keep written records of field activities on applicable preprinted field forms or in a bound field notebook to record data collecting activities. These records will be written legibly in ink and will contain pertinent field data and observations. Entry errors or changes will be crossed out with a single line, dated, and initialed by the person making the correction. Field forms and notebooks will be periodically reviewed by the Field Coordinator.

The beginning of each entry in the logbook or preprinted field form will contain the following information:

- Date
- Start time
- Weather
- Names of field personnel (including subcontractors)
- Level of personal protection used at the Site
- Names of all visitors and the purpose of their visit.

For each measurement and sample collected, the following information will be recorded:

- Detailed description of sample location,
- Equipment used to collect sample or make measurement and the date equipment was calibrated,
- Time sample was collected,
- Description of the sample conditions,
- Depth sample was collected (if applicable),
- Volume and number of containers filled with the sample; and,
- Sampler's identification.



4.1.1 Field Procedures

The following procedure describes the process to maintain the integrity of the samples:

- Upon collection samples are placed in the proper containers. In general, samples collected for organic analysis will be placed in pre-cleaned glass containers and samples collected for inorganic analysis will be placed in pre-cleaned plastic (polyethylene) bottles. Refer to the FSP for sample packaging procedures.
- Samples will be assigned a unique sample number and will be affixed to a sample label. Refer to the FSP for sample labeling procedures.
- Samples will be properly and appropriately preserved by field personnel in order to minimize loss of the constituent(s) of interest due to physical, chemical or biological mechanisms.
- Appropriate volumes will be collected to ensure that the appropriate reporting limits can be successfully achieved and that the required QC sample analyses can be performed.

4.1.2 Transfer of Custody and Shipment Procedures

- A chain-of-custody (COC) record will be completed at the time of sample collection and will accompany each shipment of project samples to the laboratory. The field personnel collecting the samples will be responsible for the custody of the samples until the samples are relinquished to the laboratory. Sample transfer will require the individuals relinquishing and receiving the samples to sign, date and note the time of sample transfer on the COC record.
- Samples will be shipped or delivered in a timely fashion to the laboratory so that holding times and/or analysis times as prescribed by the methodology can be met.
- Samples will be transported in containers (coolers) which will maintain the refrigeration temperature for those parameters for which refrigeration is required in the prescribed preservation protocols.
- Samples will be placed in an upright position and limited to one layer of samples per cooler. Additional bubble wrap or packaging material will be added to fill the cooler. Shipping containers will be secured with strapping tape and custody tape for shipment to the laboratory.
- When samples are split with the NYSDEC representatives, a separate chain-of-custody will be prepared and marked to indicate with whom the samples are shared. The person relinquishing the samples will require the representative's signature acknowledging sample receipt.
- If samples are sent by a commercial carrier, a bill of lading will be used. A copy of the bill of lading will be retained as part of the permanent record. Commercial carriers will not sign the custody record as long as the custody record is sealed inside the sample cooler and the custody tape remains intact.
- Samples will be picked up by a laboratory courier or transported to the laboratory the same day they are collected unless collected on a weekend or holiday. In these cases, the samples will be



stored in a secure location until delivery to the laboratory. Additional ice will be added to the cooler as needed to maintain proper preservation temperatures.

4.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

A sample custodian will be designated by the laboratory and will have the responsibility to receive all incoming samples. Once received, the custodian will document if the sample is received in good condition (i.e., unbroken, cooled, etc.) and that the associated paperwork, such as chain-of-custody forms have been completed. The custodian will sign the chain-of-custody forms.

The custodian will also document if sufficient sample volume has been received to complete the analytical program. The sample custodian will then place the samples into secure, limited access storage (refrigerated storage, if required). The sample custodian will assign a unique number to each incoming sample for use in the laboratory. The unique number will then be entered into the sample-receiving log with the verified time and date of receipt also noted.

Consistent with the analyses requested on the chain-of-custody form, analyses by the laboratory's analysts will begin in accordance with the appropriate methodologies. Samples will be removed from secure storage with internal chain-of-custody sign-out procedures followed.

4.3 STORAGE OF SAMPLES

Empty sample bottles will be returned to secure and limited access storage after the available volume has been consumed by the analysis. Upon completion of the entire analytical work effort, samples will be disposed of by the sample custodian. The length of time that samples are held will be at least thirty (30) days after reports have been submitted. Disposal of remaining samples will be completed in compliance with all Federal, State, and local requirements.

4.4 FINAL PROJECT FILES CUSTODY PROCEDURES

The final project files will be the central repository for all documents with information relevant to sampling and analysis activities as described in this QAPP. The Haley & Aldrich Project Manager will be the custodian of the project file. The project files including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews will be maintained in a secured, limited access area and under custody of the Project Director or his designee.

The final project file will include the following:

- Project plans and drawings
- Field data records
- Sample identification documents and soil boring/monitoring well logs
- All chain-of-custody documentation
- Correspondence
- References, literature
- Laboratory data deliverables
- Data validation and assessment reports
- Progress reports, QA reports
- Final report



The laboratory will be responsible for maintaining analytical logbooks, laboratory data and sample chain of custody documents. Raw laboratory data files and copies of hard copy reports will be inventoried and maintained by the laboratory for a period of six (6) years at which time the laboratory will contact the Haley & Aldrich Project Manager regarding the disposition of the project related files.



5. Calibration Procedures and Frequency

5.1 FIELD INSTRUMENT CALIBRATION PROCEDURES

Several field instruments will be used for both on-site screening of samples and for health and safety monitoring, as described in the Health and Safety Plan (HASP). On-site air monitoring for health and safety purposes may be accomplished using a vapor detection device, such as a photoionization detector (PID).

Field instruments will be calibrated at the beginning of each day and checked during field activities to verify performance. Instrument specific calibration procedures will be performed in accordance with the instrument manufacturer's requirements.

5.2 LABORATORY INSTRUMENT CALIBRATION PROCEDURES

Reference materials of known purity and quality will be utilized for the analysis of environmental samples. The laboratory will carefully monitor the preparation and use of reference materials including solutions, standards, and reagents through well-documented procedures.

All solid chemicals and acids/bases used by the laboratory will be rated as "reagent grade" or better. All gases will be "high" purity or better. All Standard Reference Materials (SRMs) or Performance Evaluation (PE) materials will be obtained from approved vendors of the National Institute of Standards and Technology (formerly National Bureau of Standards), the U.S. EPA Environmental Monitoring Support Laboratories (EMSL), or reliable Cooperative Research and Development Agreement (CRADA) certified commercial sources.



6. Analytical Procedures

Analytical procedures to be utilized for analysis of environmental samples will be based on referenced USEPA analytical protocols and/or project specific SOP.

6.1 FIELD ANALYTICAL PROCEDURES

Field analytical procedures include the measurement of pH, temperature, ORP, DO and specific conductivity during sampling of groundwater, and the qualitative measurement of volatile organic compounds (VOC) during the collection of soil samples.

6.2 LABORATORY ANALYTICAL PROCEDURES

Laboratory analyses will be based on the U.S. EPA methodology requirements promulgated in:

• "Test Methods for Evaluating Solid Waste," SW-846 EPA, Office of Solid Waste, and promulgated updates, 1986.

6.2.1 List of Project Target Compounds and Laboratory Detection Limits

The laboratory reporting limits (RLs) and associated method detection limits (MDLs) for the target analytes and compounds for the environmental media to be analyzed are presented in Table I. MDLs have been experimentally determined by the project laboratory using the method provided in 40 CFR, Part 136 Appendix B.

Laboratory parameters for soil samples are listed in the SCWP. Laboratory parameters for disposal samples will be determined by the disposal facility after an approved facility has been determined.

6.2.2 List of Method Specific Quality Control (QC) Criteria

The laboratory SOPs include a section that presents the minimum QC requirements for the project analyses. Section 7.0 references the frequency of the associated QC samples for each sampling effort and matrix.



7. Internal Quality Control Checks

This section presents the internal quality control checks that will be employed for field and laboratory measurements.

7.1 FIELD QUALITY CONTROL

7.1.1 Field Blanks

Internal quality control checks will include analysis of field blanks to validate equipment cleanliness. Whenever possible, dedicated equipment will be employed to reduce the possibility of cross-contamination of samples.

7.1.2 Trip Blanks

Trip blanks samples will be prepared by the project laboratory using ASTM Type II or equivalent water placed within pre-cleaned 40 milliliter (ml) VOC vials equipped with Teflon septa. Trip blanks will accompany each sample delivery group (SDG) of environmental samples collected for analysis of VOCs.

Trip blank samples will be placed in each cooler that stores and transports project samples that are to be analyzed for VOCs.

7.1.3 Equipment Blanks

Internal quality control checks for PFAS sampling will include analysis of equipment blanks once per day per site for each matrix to validate equipment cleanliness.

7.2 LABORATORY PROCEDURES

Procedures which contribute to maintenance of overall laboratory quality assurance and control include appropriately cleaned sample containers, proper sample identification and logging, applicable sample preservation, storage, and analysis within prescribed holding times, and use of controlled materials.

7.2.1 Field Duplicate Samples

The precision or reproducibility of the data generated will be monitored through the use of field duplicate samples. Field duplicate analysis will be performed at a frequency of 1 in 20 project samples.

Precision will be measured in terms of the absolute value of the relative percent difference (RPD) as expressed by the following equation:

$RPD = [|R1-R2|/[(R1+R2)/2]] \times 100\%$

Acceptance criteria for duplicate analyses performed on solid matrices will be 100% and aqueous matrices will be 35%. RPD values outside these limits will require an evaluation of the sampling and/or analysis



procedures by the project QA Officer and/or laboratory QA Director. Corrective actions may include reanalysis of additional sample aliquots and/or qualification of the data for use.



7.2.2 Matrix Spike Samples

Ten percent of each project sample matrix for each analytical method performed will be spiked with known concentrations of the specific target compounds/analytes.

The amount of the compound recovered from the sample compared to the amount added will be expressed as a percent recovery. The percent recovery of an analyte is an indication of the accuracy of an analysis within the site-specific sample matrix. Percent recovery will be calculated for MS/MSD using the following equation.

% Recovery =
$$\frac{Spiked \ Sample - Background}{KnownValue \ of \ Spike} \times 100\%$$

If the quality control value falls outside the control limits (UCL or LCL) due to sample matrix effects, the results will be reported with appropriate data qualifiers. To determine the effect a non-compliant MS recovery has on the reported results, the recovery data will be evaluated as part of the validation process.

7.2.3 Laboratory Control Sample (LCS) Analyses

The laboratory will perform LCS analyses prepared from Standard Reference Materials (SRMs). The SRMs will be supplied from an independent manufacturer and traceable to NIST materials with known concentrations of each target analyte to be determined by the analytical methods performed. In cases where an independently supplied SRM is not available, the LCS may be prepared by the laboratory from a reagent lot other than that used for instrument calibration.

The laboratory will evaluate LCS analyses in terms of percent recovery using the most recent laboratory generated control limits.

LCS recoveries that do not meet acceptance criteria will be deemed invalid. Analysis of project samples will cease until an acceptable LCS analysis has been performed. If sample analysis is performed in association with an out-of-control LCS sample analysis, the data will be deemed invalid.

Corrective actions will be initiated by the Haley & Aldrich QA Officer and/or Laboratory QA Officer to investigate the problem. After the problem has been identified and corrected, the solution will be noted in the instrument run logbook and re-analysis of project samples will be performed, if possible.

The analytical anomaly will be noted in the sample delivery group (SDG) Case Narrative and reviewed by the data validator. The data validator will confirm that appropriate corrective actions were implemented and recommend the applicable use of the affected data.

7.2.4 Surrogate Compound/Internal Standard Recoveries

For VOCs, surrogates will be added to each sample prior to analysis to establish purge and trap efficiency. Quantitation will be accomplished via internal standardization techniques.

The recovery of surrogate compounds and internal standards will be monitored by laboratory personnel to assess possible site-specific matrix effects on instrument performance.



For semi-volatile organics analyses, surrogates will be added to the raw sample to assess extraction efficiency. Internal standards will be added to all sample extracts and instrument calibration standard immediately before analysis for quantitation via internal standardization techniques.

Method specific quality control (QC) limits are provided in the attached laboratory method SOPs. Surrogate compound/internal standard recoveries that do not fall within accepted QC limits for the analytical methodology performed will have the analytical results flagged with data qualifiers as appropriate by the laboratory and will not be noted in the laboratory report Case Narrative.

To ascertain the effect non-compliant surrogate compound/internal standard recoveries may have on the reported results, the recovery data will be evaluated as part of the validation process. The data validator will provide recommendations for corrective actions including but not limited to additional data qualification.

7.2.5 Calibration Verification Standards

Calibration verification (CV) standards will be utilized to confirm instrument calibrations and performance throughout the analytical process. CV standards will be prepared as prescribed by the respective analytical protocols. Continuing calibration will be verified by compliance with method-specific criteria prior to additional analysis of project samples.

Non-compliant analysis of CV standards will require immediate corrective action by the project laboratory QA officer and/or designated personnel. Corrective action may include re-analysis of each affected project sample, a detailed description of the problem, the corrective action undertaken, the person who performed the action, and the resolution of the problem.

7.2.6 Laboratory Method Blank Analyses

Method blank sample analysis will be performed as part of each analytical batch for each methodology performed. If target compounds are detected in the method blank samples, the reported results will be flagged by the laboratory in accordance with standard operating procedures. The data validator will provide recommendations for corrective actions including but not limited to additional data qualification.



8. Data Quality Objectives

Sampling that will be performed as described in the SCWP is designed to produce data of the quality necessary to achieve the minimum standard requirements of the field and laboratory analytical objectives described below. These data are being obtained with the primary objective to assess levels of contaminants of concern associated with the Site.

The overall project data quality objective (DQO) is to implement procedures for field data collection, sample collection, handling, and laboratory analysis and reporting that achieve the project objectives. The following section is a general discussion of the criteria that will be used to measure achievement of the project DQO.

8.1 PRECISION

8.1.1 Definition

Precision is defined as a quantitative measure of the degree to which two or more measurements are in agreement. Precision will be determined by collecting and analyzing field duplicate samples and by creating and analyzing laboratory duplicates from one or more of the field samples. The overall precision of measurement data is a mixture of sampling and analytical factors. The analytical results from the field duplicate samples will provide data on sampling precision. The results from duplicate samples created by the laboratory will provide data on analytical precision. The measurement of precision will be stated in terms of relative percent difference (RPD).

8.1.2 Field Precision Sample Objectives

Field precision will be assessed through collection and measurement of field duplicate samples at a rate of 1 duplicate per 20 investigative samples. The RPD criteria for the project field duplicate samples will be +/- 100% for soil, +/- 35 % for groundwater for parameters of analysis detected at concentrations greater than 5 times (5X) the laboratory reporting limit (RL).

8.1.3 Laboratory Precision Sample Objectives

Laboratory precision will be assessed through the analysis of laboratory control and laboratory control duplicate samples (LCS/LCSD) and matrix spike and matrix spike duplicate (MS/MSD) samples for groundwater and soil samples and the analysis of laboratory duplicate samples for air and soil vapor samples. Air and soil vapor laboratory duplicate sample analyses will be performed by analyzing the same SUMMA canister twice. The RPD criteria for the air/soil vapor laboratory duplicate samples will be +/- 35 % for parameters of analysis detected at concentrations greater than 5 times (5X) the laboratory reporting limit (RL).



8.2 ACCURACY

8.2.1 Definition

Accuracy relates to the bias in a measurement system. Bias is the difference between the observed and the "true" value. Sources of error are the sampling process, field contamination, preservation techniques, sample handling, sample matrix, sample preparation and analytical procedure limitations.

8.2.2 Field Accuracy Objectives

Sampling bias will be assessed by evaluating the results of field equipment rinse and trip blanks. Equipment rinse and trip blanks will be collected as appropriate based on sampling and analytical methods for each sampling effort.

If non-dedicated sampling equipment is used, equipment rinse blanks will be collected by passing ASTM Type II water over and/or through the respective sampling equipment utilized during each sampling effort. One equipment rinse blank will be collected for each type of non-dedicated sampling equipment used for the sampling effort. Equipment rinse blanks will be analyzed for each target parameter for the respective sampling effort for which environmental media have been collected. (Note: If dedicated or disposable sampling equipment is used, equipment rinse samples will not be collected as part of that field effort.)

Trip blank samples will be prepared by the laboratory and provided with each shipping container that includes containers for the collection of groundwater samples for the analysis of VOC. Trip blank samples will be analyzed for each VOC for which groundwater samples have been collected for analysis.

8.3 LABORATORY ACCURACY OBJECTIVES

Analytical bias will be assessed through the use of laboratory control samples (LCS) and Site-specific matrix spike (MS) sample analyses. LCS analyses will be performed with each analytical batch of project samples to determine the accuracy of the analytical system.

One (1) set of MS/MSD analyses will be performed with each batch of 20 project samples collected for analysis to assess the accuracy of the identification and quantification of analytes within the Site-specific sample matrices. Additional sample volume will be collected at sample locations selected for the preparation of MS/MSD samples so that the standard laboratory reporting limits (RLs) are achieved.

The accuracy of analyses that include a sample extraction procedure will be evaluated through the use of system monitoring or surrogate compounds. Surrogate compounds will be added to each sample, standard, blank, and QC sample prior to sample preparation and analysis. Surrogate compound percent recoveries will provide information on the effect of the sample matrix on the accuracy of the analyses.



8.4 **REPRESENTATIVENESS**

8.4.1 Definition

Representativeness expresses the degree to which sample data represent a characteristic of a population, a parameter variation at a sampling point or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the design of the sampling program. The representativeness criterion is satisfied through the proper selection of sampling locations, the quantity of samples and the use of appropriate procedures to collect and analyze the samples.

8.4.2 Measures to Ensure Representativeness of Field Data

Representativeness will be addressed by prescribing sampling techniques and the rationale used to select sampling locations. Sampling locations may be biased (based on existing data, instrument surveys, observations, etc.) or unbiased (completely random or stratified-random approaches).

8.5 COMPLETENESS

8.5.1 Definition

Completeness is a measure of the amount of valid (usable) data obtained from a measuring system compared to the total amount of the anticipated to be obtained. The completeness goal for all data uses is that a sufficient amount of valid data be generated so that determinations can be made related to the intended data use with a sufficient degree of confidence.

8.5.2 Field Completeness Objectives

Completeness is a measure of the amount of valid measurements obtained from measurements taken in this project versus the number planned. Field completeness objective for this project will be greater than (>) 90%.

8.5.3 Laboratory Completeness Objectives

Laboratory data completeness objective is a measure of the amount of valid data obtained from laboratory measurements. The evaluation of the data completeness will be performed at the conclusion of each sampling and analysis effort.

The completeness of the data generated will be determined by comparing the amount of valid data, based on independent validation, with the total laboratory data set. The completeness goal will be >90%.

8.6 COMPARABILITY

8.6.1 Definition

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another.



8.6.2 Measures to Ensure Comparability of Laboratory Data

Comparability of laboratory data will be measured from the analysis of Standard Reference Materials (SRM) obtained from either EPA Cooperative Research and Development Agreement (CRADA) suppliers or the National Institute of Standards and Technology (NIST). The reported analytical data will also be presented in standard units of mass of contaminant within a known volume of environmental media. The standard units for various sample matrices are as follows:

- Solid Matrices mg/kg of media (Dry Weight).
- Aqueous Matrices ng/L for PFAS analyses, ug/L of media for organic analyses, and mg/L for inorganic analyses.

8.7 LEVEL OF QUALITY CONTROL EFFORT

If non-dedicated sampling equipment is used, equipment rinse blanks will be prepared by field personnel and submitted for analysis of target parameters. Equipment rinse blank samples will be analyzed to check for potential cross-contamination between sampling locations that may be introduced during the investigation. One (1) equipment rinse blank will be collected per sampling event to the extent that non-dedicated sampling equipment is used.

If necessary, A separate equipment rinse blank sample will be collected for PFAS using the sample collection procedure described in Section 8.1.1 of the NYSDEC-approved Field Sampling Plan. (Note: If dedicated or disposable sampling equipment is used, equipment rinse samples will not be collected as part of that field effort.)

Trip blanks will be used to assess the potential for contamination during sample storage and shipment. Trip blanks will be provided with the sample containers to be used for the collection of groundwater samples for the analysis of VOC. Trip blanks will be preserved and handled in the same manner as the project samples. One (1) trip blank will be included along with each shipping container containing project samples to be analyzed for VOC.

Method blank samples will be prepared by the laboratory and analyzed concurrently with all project samples to assess potential contamination introduced during the analytical process.

Field duplicate samples will be collected and analyzed to determine sampling and analytical reproducibility. One (1) field duplicate will be collected for every 20 or fewer investigative samples collected for off-Site laboratory analysis.

Matrix spikes will provide information to assess the precision and accuracy of the analysis of the target parameters within the environmental media collected. One (1) matrix spike/matrix spike duplicate (MS/MSD) will be collected for every 20 or fewer investigative samples per sample matrix.

(Note: Soil MS/MSD samples require triple sample volume for VOC only. Aqueous MS/MSD samples require triple the normal sample volume for VOC analysis and double the volume for the remaining parameters.)



9. Data Reduction, Validation and Reporting

Data generated by the laboratory operation will be reduced and validated prior to reporting in accordance with the following procedures:

9.1 DATA REDUCTION

9.1.1 Field Data Reduction Procedures

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. The pH, conductivity, temperature, turbidity, DO, ORP and breathing zone VOC readings collected in the field will be generated from direct read instruments. The data will be written into field logbooks immediately after measurements are taken. If errors are made, data will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original entry.

9.1.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures are provided by the appropriate chapter of USEPA, "Test Methods for Evaluating Solid Waste", SW-846, Third Edition. Errors will be noted; corrections made with the original notations crossed out legibly. Analytical results for soil samples will be calculated and reported on a dry weight basis.

9.1.3 Quality Control Data

Quality control data (e.g., laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates) will be compared to the method acceptance criteria. Data determined to be acceptable will be entered into the laboratory information management system.

Unacceptable data will be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits and any other anomalous conditions encountered during sample analysis.

9.2 DATA VALIDATION

Data validation procedures of the analytical data will be performed by the Haley & Aldrich QA Officer or designee using the following documents as guidance for the review process:

- "U.S. EPA National Functional Guidelines for Organic Data Review", and the "U.S. EPA National Functional Guidelines for Inorganic Data Review".
- The specific data qualifiers used will be applied to the reported results as presented and defined in the EPA National Functional Guidelines. Validation will be performed by qualified personnel at the direction of the Haley & Aldrich QAO.



The completeness of each data package will be evaluated by the Data Validator. Completeness
checks will be administered on all data to determine that the deliverables are consistent with the
NYSDEC Analytical Services Protocol (ASP) Category A and Category B data package requirements.
The validator will determine whether the required items are present and request copies of missing
deliverables (if necessary) from the laboratory.

9.3 DATA REPORTING

Data reporting procedures will be carried out for field and laboratory operations as indicated below:

- Field Data Reporting: Field data reporting will be conducted principally through the transmission of report sheets containing tabulated results of measurements made in the field and documentation of field calibration activities.
- Laboratory Data Reporting: The laboratory data reporting package will enable data validation based on the protocols described above. The final laboratory data report format will include the QA/QC sample analysis deliverables to enable the development of a data usability summary report (DUSR) based on Department DER-10 Appendix 2B.



10. Performance and System Audits

A performance audit is an independent quantitative comparison with data routinely obtained in the field or the laboratory. Performance audits include two separate, independent parts: internal and external audits.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

10.1.1 Internal Field Audit Responsibilities

Internal audits of field activities will be initiated at the discretion of the Project Manager and will include the review of sampling and field measurements. The audits will verify that all procedures are being followed. Internal field audits will be conducted periodically during the project. The audits will include examination of the following:

- Field sampling records, screening results, instrument operating records
- Sample collection
- Handling and packaging in compliance with procedures
- Maintenance of QA procedures
- Chain-of-custody reports

10.1.2 External Field Audit Responsibilities

External audits may be conducted by the Project Coordinator at any time during the field operations. These audits may or may not be announced and are at the discretion of the NYSDEC. The external field audits can include (but are not limited to) the following:

- Sampling equipment decontamination procedures
- Sample bottle preparation procedures
- Sampling procedures
- Examination of health and safety plans
- Procedures for verification of field duplicates
- Field screening practices

10.2 LABORATORY PERFORMANCE AND SYSTEM AUDITS

10.2.1 Internal Laboratory Audit Responsibilities

The laboratory system audits are typically conducted by the laboratory QA Officer or designee on an annual basis. The system audit will include an examination of laboratory documentation including sample receiving logs, sample storage, chain-of-custody procedures, sample preparation and analysis and instrument operating records.

At the conclusion of internal system audits, reports will be provided to the laboratory's operating divisions for appropriate comment and remedial/corrective action where necessary. Records of audits and corrective actions will be maintained by the Laboratory QA Officer.



10.2.2 External Laboratory Audit Responsibilities

External audits will be conducted as required, by the NYSDOH or designee. External audits may include any of the following:

- Review of laboratory analytical procedures
- Laboratory on-site visits
- Submission of performance evaluation samples for analysis

Failure of any of the above audit procedures can lead to laboratory de-certification. An audit may consist of but not limited to:

- Sample receipt procedures
- Custody, sample security and log-in procedures
- Review of instrument calibration logs
- Review of QA procedures
- Review of log books
- Review of analytical SOPs
- Personnel interviews

A review of a data package from samples recently analyzed by the laboratory can include (but not be limited to) the following:

- Comparison of resulting data to the SOP or method
- Verification of initial and continuing calibrations within control limits
- Verification of surrogate recoveries and instrument timing results
- Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable
- Assurance that samples are run within holding times



11. Preventive Maintenance

11.1 FIELD INSTRUMENT PREVENTIVE MAINTENANCE

The field equipment preventive maintenance program is designed to ensure the effective completion of the sampling effort and to minimize equipment down time. Program implementation is concentrated in three areas:

- Maintenance responsibilities
- Maintenance schedules
- Inventory of critical spare parts and equipment

The maintenance responsibilities for field equipment will be assigned to the task leaders in charge of specific field operations. Field personnel will be responsible for daily field checks and calibrations and for reporting any problems with the equipment. The maintenance schedule will follow the manufacturer's recommendations. In addition, the field personnel will be responsible for determining that an inventory of spare parts will be maintained with the field equipment. The inventory will primarily contain parts that are subject to frequent failure, have limited useful lifetimes and/or cannot be obtained in a timely manner.

11.2 LABORATORY INSTRUMENT PREVENTIVE MAINTENANCE

Analytical instruments at the laboratory will undergo routine and/or preventive maintenance. The extent of the preventive maintenance will be a function of the complexity of the equipment.

Generally, annual preventive maintenance service will involve cleaning, adjusting, inspecting and testing procedures designed to deduce instrument failure and/or extend useful instrument life. Between visits, routine operator maintenance and cleaning will be performed according to manufacturer's specifications by laboratory personnel.



12. Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness

12.1 FIELD MEASUREMENTS

Field generated information will be reviewed by the Field Coordinator and typically include evaluation of bound logbooks/forms, data entry and calculation checks. Field data will be assessed by the Project Coordinator who will review the field results for compliance with the established QC criteria that are specified in Section 7.0 of this QAPP. The accuracy of pH and specific conductance will be assessed using daily instrument calibration, calibration check, and blank data. Accuracy will be measured by determining the percent recovery (% R) of calibration check standards. Precision of the pH and specific conductance measurements will be assessed on the basis of the reproducibility of duplicate readings of a field sample and will be measured by determining the relative percent difference (RPD). Accuracy and precision of the soil VOC screening will be determined using duplicate readings of calibration checks. Field data completeness will be calculated using the following equation:

 $Completeness = \frac{Valid (usable) Data Obtained}{Total Data Planned} X 100$

12.2 LABORATORY DATA

Surrogate, internal standard and matrix spike recoveries will be used to evaluate data quality. The laboratory quality assurance/quality control program will include the following elements:

- Precision, in terms of relative percent difference (RPD), will be determined by relative sample analysis at a frequency of one duplicate analysis for each batch of ten project samples or a frequency of 10 percent (10%). RPD is defined as the absolute difference of duplicate measurements divided by the mean of these analyses normalized to percentage.
- Accuracy, in terms of percent recovery (recovery of known constituent additions or surrogate recoveries), will be determined by the analysis of spiked and unspiked samples. MS/MSD will be used to determine analytical accuracy. The frequency of MS/MSD analyses will be one project sample MS/MSD per set of 20 project samples.
- One method blank will be prepared and analyzed with each batch of project samples. The total number of method blank sample analyses will be determined by the laboratory analytical batch size.
- Standard Reference Materials (SRMs) will be used for each analysis. Sources of SRM's include the U.S. EPA, commercially available material from CRADA certified vendors and/or laboratory produced solutions. SRMs, when available and appropriate, will be processed and analyzed on a frequency of one per set of samples.
- Completeness is the evaluation of the amount of valid data generated versus the total set of data produced from a particular sampling and analysis event. Valid data is determined by independent



confirmation of compliance with method-specific and project-specific data quality objectives. The calculation of data set completeness will be performed by the following equation.

 $\frac{Number of Valid Sample Results}{Total Number of Samples Planned} X 100 = \% Complete$



13. Quality Assurance (QA) Reports

Critically important to the successful implementation of the QA Plan is a reporting system that provides the means by which the program can be reviewed, problems identified, and programmatic changes made to improve the plan.

QA reports to management can include:

- Audit reports, internal and external audits with responses
- Performance evaluation sample results; internal and external sources
- Daily QA/QC exception reports/corrective actions

QA/QC corrective action reports will be prepared by the Haley & Aldrich QA Officer when appropriate and presented to the project and/or laboratory management personnel so that performance criteria can be monitored for all analyses from each analytical department. The updated trend/QA charts prepared by the laboratory QA personnel will be distributed and reviewed by various levels of the laboratory management.



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- 3. United States Environmental Protection Agency, (1993). Data Quality Objectives Process for Superfund Interim Final Guidance. U.S. EPA/540/R-93-071, Office of Solid Waste and Emergency Response (OSWER), September 1993.
- 4. United States Environmental Protection Agency, (1992). Specifications and Guidance for Contaminant-Free Sample Containers. OSWER Directive 9240.0-05A, April 1992.
- 5. United States Environmental Protection Agency. U.S. EPA National Functional Guidelines for Organic Data Review. U.S. EPA 540/R-2017-002.
- 6. United States Environmental Protection Agency. U.S. EPA National Functional Guidelines for Organic Data Review. U.S. EPA 540/R-2017-001.
- 7. United States Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Office of Solid Waste, U.S. EPA, SW-846, November 1986, with updates.
- 8. New York State Department of Environmental Conservation, NYSDEC Analytical Services Protocol (ASP), Bureau of Environmental Investigation, 1991 with updates.
- 9. New York State Department of Environmental Conservation, NYSDEC, Division of Environmental Remediation, Technical Guidance for Site Investigation and Remediation, DER-10, May 2010.
- 10. New York State Department of Environmental Conservation, NYSDEC, Division of Environmental Remediation, Sampling, Analysis and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) under NYSDEC's Part 375 Remedial Programs, April 2023.



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TABLES



TABLE I SUMMARY OF ANALYSIS METHOD, PRESERVATION METHOD, HOLDING TIME, SAMPLE SIZE REQUIREMENTS AND SAMPLE CONTAINERS 90-02 Queens Boulevard Site Queens NY

Analysis/Method	Sample Type	Number of Expected Samples	Preservation	Holding Time	Volume/Weight	Container
Volatile Organic Compounds/8260C	Soil	Ten	1 - 1 Vial MeOH/2 Vial Water, Cool, 4 ± 2 °C	14 days ¹	120 mL	3 - 40ml glass vials
Semivolatile Organic Compounds/8270D	Soil	Ten	Cool, 4 ± 2 °C	14 days	250 mL	1 - 8 oz glass
Pesticides (8081B)	Soil	Ten	Cool, 4 ± 2 °C	14 days	250 mL	1 - 8 oz glass
Polychlorinated Biphenyls/8082A	Soil	Ten	Cool, 4 ± 2 °C	14 days	250 mL	1 - 8 oz glass
Metals/6010D	Soil	Ten	Cool, 4 ± 2 °C	180 days	60 mL	1 - 2 oz glass
PFAS 1633	Soil	Ten	Cool, 4 ± 2 °C	14 days	250 mL	1 - 8 oz HDPE container
1,4-Dioxane 8270	Soil	Ten	Cool, 4 ± 2 °C	14 days	250 mL	1 - 8 oz glass
Volatile Organic Compounds/8260C	Groundwater	Three	HCl, Cool, 4 ± 2 °C	14 days	120 mL	3 - 40ml glass vials
Semivolatile Organic Compounds/8270D	Groundwater	Three	Cool, 4 ± 2 °C	7 days	500 mL	2 - 250 mL amber glass
TAL Metals 6020	Groundwater	Three	HNO_3Cool , 4 ± 2 $^{\circ}C$	180 days	500 mL	1 - 500 mL plastic bottle
PFAS 1633	Groundwater	Three	H2O Cool, 4 ± 2 $^{\circ}$ C	14 days	500 mL	2 - 250 ml HDPE containers
1,4-Dioxane 8270	Groundwater	Three	Cool, 4 ± 2 °C	7 days	500 mL	1 - 500 mL plastic bottle
Volatile Organic Compounds/TO-15	Soil Vapor	Eight	N/A	30 days	2.7 - 6 L	1 2.7 or 6 L Summa Canister

Notes:

1. Terracores and encores must be frozen within 48 hours of collection

2. Refer to text for additional information.

3. HDPE - High Density Polyethylene

4. Number of expected samples excludes QAQC samples

QAQC samples include:

MS/MSD - 1 for every 20 samples Field Duplicate - 1 for every 20 samples Trip Blanks - 1 per cooler of samples to be analyzed for VOCs Field Blanks - 1 for every 20 samples Equipment Blanks - 1 per day per matrix for PFAS

Appendix A: Data Validation Team Resumes





KIRKLAND BROADWELL, GIT

Project Geologist

EDUCATION

Ph.D., Philosophy, Geosciences, Virginia Polytechnic Institute and State University (Virginia Tech) B.S., Geology, The College of William & Mary-Williamsburg

PROFESSIONAL REGISTRATIONS

2022/ VA: Certified Geologist in Training (GIT) (Reg. No.: 2801002362)

SPECIAL STUDIES AND COURSES

40-Hour OSHA Hazardous Waste Operations and Emergency Response Training (29 CFR 1910.120)
8-Hour OSHA HAZWOPER Refresher Training
10-Hour OSHA Construction
24-Hour MSHA/New Miner Training (Part 48)
24-Hour MSHA/New Miner Training Surface Mine Operations
American Red Cross First Aid Training and CPR Course

Kirkland is a project geologist with field and laboratory experience and is currently working in the Energy & Mining sector at Haley & Aldrich. He has experience in geologic data collection, analysis, and interpretation, as well as technical communication. He holds a doctorate in philosophy in geosciences from Virginia Tech, where he studied metamorphic processes and specialized in igneous and metamorphic geochemistry and petrology. He has extensive experience with petrography, electron microscopy, and thermodynamic modeling of geologic systems. He also has experience with compliance and corrective action groundwater monitoring programs, including field activities (slug testing and groundwater, surface water, and stormwater sampling), and data analysis (data verification, validation, and regulatory reporting).

RELEVANT PROJECT EXPERIENCE

Waste Management, Inc., Environmental Services, Virginia. Kirkland served as a field geologist for environmental services which included compliance and corrective action groundwater, surface water, and stormwater monitoring activities, landfill gas monitoring and data management, and data verification, analysis, and reporting.

Iluka Resources, Inc., Environmental Services, Stony Creek, Virginia. Kirkland served as a geologist for environmental services which included aquifer testing and compliance groundwater monitoring activities.

ExTerra Field Institute and Research Endeavor, Western Alps, Europe. Kirkland worked as a graduate researcher and used geochemistry and petrology to trace fluid and element cycling within subduction zone systems and quantify the timing and conditions of metamorphic processes that occur in subduction zones.

PUBLICATIONS

"From the Appalachians to the Alps: Constraints on the timing, duration, and conditions of metamorphism at convergent margins," dissertation for Doctor of Philosophy, Virginia Polytechnic Institute and State University (Virginia Tech), Blackburg, Virginia, 2020.

"Deciphering poly-metamorphism and fluid alteration in the central Appalachians with monazite and allanite petrochronology," with M.J. Caddick, J.S. Beard, W. Henika, and A. Kylander-Clark, A., in review with *American Journal of Science*.

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"Meta-rodingite dikes as recorders of subduction zone metamorphism and serpentinite dehydration: Voltri Ophiolite, Italy," with A.A. Haws, P.G. Starr, B. Dragovic, M. Scambelluri, D. Delmonte, M.J. Caddick, J.J Ague, and E.F. Baxter, *Chemical Geology*, 2021. <u>https://doi.org/10.1016/j.chemgeo.2021.120077</u>

"The subduction and exhumation history of the Voltri Ophiolite, Italy: Evaluating exhumation mechanisms for highpressure metamorphic massifs," P.G. Starr, B. Dragovic, M. Scambelluri, A.A. Haws, M.J. Caddick, A.J. Smye, and E.F. Baxter, In press at *Lithos*, 2020. <u>https://doi.org/10.1016/j.lithos.2020.105767</u>

"Polymetamorphism in high-T metamorphic rocks: An example from the central Appalachians," with M.J. Caddick, and J.S. Beard, *Journal of Metamorphic Geology*, 2019. <u>https://doi.org/10.1111/jmg.12509</u>

"Transient and periodic brittle deformation of eclogites during intermediate-depth subduction," with M. Locatelli, A. Verlaguet, P. Agard, and M.J. Caddick, Earth and Planetary Science Letters, Pages 91-102, September 2019. https://doi.org/10.1016/j.epsl.2019.06.008

"A Comparative Climate Analysis of Virginia During the 20th and 21st centuries," thesis for Bachelor of Science, The College of William & Mary, Williamsburg, Virginia, 2013.



OSCAR CERVANTES

Staff Professional

EDUCATION B.S., Environmental Engineering, Arizona State University

PROFESSIONAL REGISTRATIONS

2021/ AZ: Environmental Engineering F.E. Exam (NCEES ID 22-408-39)

SPECIAL STUDIES AND COURSES (DELETE IF NOT APPLICABLE)

40-Hour OSHA Hazardous Waste Operations and Emergency Response Training (29 CFR 1910.120)] 24-Hour MSHA/New Miner Training (Part 48) EPA Region 9: TRI Basic Concepts Training EPA Region 9: TRI Advanced Concepts Training

Oscar is a recently graduated staff professional with experience in environmental remediation programs and data quality assurance/quality control and will focus his time at Haley & Aldrich serving the Energy and Mining markets. He is skilled in groundwater, air, and soil sampling methods. He participates in the operating and maintaining of high-volume air samplers. He is also skilled in data quality assurance and quality control, regularly validating field and laboratory data. Oscar will also become familiar in overseeing operations and maintenance for water treatment systems.

RELEVANT PROJECT EXPERIENCE

Airports

Goodyear, Phoenix Goodyear Airport Groundwater Monitoring, Goodyear, Arizona. Oscar served as an engineer for environmental services which included measuring the water levels at all wells, collecting suitable samples at the treatment compounds and wells, completing field tests such as pH and turbidity, and performing maintenance on wells.

Mining Sites

Asarco, Air Monitoring Activities, Hayden, Arizona Oscar has worked as a field technician at the Hayden site. His main responsibility in this role is operating and maintaining high-volume air sampling machines. This includes collecting suitable air samples and performing monthly audits and verifications. Oscar also validates field and lab data for the project.

Colleges and Universities

Arizona State University, Design Project, Tempe, Arizona Oscar participated in a design project during his capstone course at Arizona State University. The group designed an entire drinking water treatment plant/system including coagulation/flocculation, sedimentation, granular media filtration, granular activated carbon filtration, disinfection, advanced treatment, and storage/distribution. Oscar was specifically responsible for the storage/distribution of the treated water. This involved considerations/calculations such as location, piping size/material, water demands, length of pipes, residence time, total storage required, and of course, costs (capital and operational). Sustainability was an emphasis on the design of the treatment plant: each student had to provide ways in which their design choices implemented sustainability concepts.

Appendix B: Project Team Resumes







JAMES BELLEW

Principal

EDUCATION

M.S., Environmental Geology, Queens College B.S., Geology, Pre-Law, Environmental Science, Binghamton University

PROFESSIONAL SOCIETIES

American Council of Engineering Companies, Member, 2017 Urban Land Institute, Member, 2016 Business Council of New York, Member, 2018

SPECIAL STUDIES AND COURSES

40-Hour OSHA Hazardous Waste Operations and Emergency Response Training (29 CFR 1910.120)
30-Hour OSHA Construction Safety and Heath
8-hour OSHA Site Supervisor Certification
OSHA Confined Space Entry Training Certification
Erosion and Sediment Control, New York, No. 006925
USDOT/IATA Training on the Shipping and/or Transportation of Hazardous Materials

James has a hands-on approach to every project. He believes that being present and putting himself into his clients' shoes is the best way to understand their needs. As a Principal, James's expertise includes due diligence, environmental risk development, building surveys, remedial investigations, remedial design, and technical oversight. Mr. Bellew has completed over 50 NYCOER E-Designation Sites and NYSDEC Brownfield Cleanup Program Sites which include preparation of all reports through to the certificate of completion and a certificate of occupancy.

Clients appreciate James' strategies from the inception of a project through closure under various regulatory programs nationwide. That comprehensive approach is what James loves the most about his job. He enjoys taking on complex projects and finding rational, cost-effective, remedial solutions. His biggest reward? When he can bring a client cost relief through value engineering.

RELEVANT PROJECT EXPERIENCE

Development, NYCDDC Shirley Chisholm Recreational Center, Brooklyn, New York. Principal for the project released by the New York City Department of Design and Construction, on behalf of the NYC Parks Department, for the design and construction of a new recreational center located at 3002 Foster Avenue in Brooklyn New York. Scope of services included execution of a Phase II Environmental Site Assessment, soil characterization, remedial oversight, geotechnical percolation testing and closure with the NYC Department of Environmental Protection.

Development's, New York State Superfund Site, Former NuHart Plastics Site, New York State Superfund Site (NuHart West) and Brownfield Cleanup Program Site (NuHart East), Brooklyn, New York. Principal for the preparation of the feasibility study, offsite investigation reports, RCRA (Resource Conservation and Recovery Act) Closure Work Plan, execution of the RCRA Closure, preparation of the Brownfield Cleanup Application (NuHart East), 100% Remedial Design, preparation of all BCP related work plans (NuHart East), coordination to vest the Site for 421-a and all community outreach programs for a former plasticizer facility with on- and off-site pollutant concerns. Responsible for all remedial cost and alternative analysis with the client to bring the Site to a certificate of completion. NuHart is a high-profile Site that requires coordination with the New York State Department of Environmental Conservation (NYSDEC), the New York City Office of Environmental Remediation (NYCOER), local regulatory agencies, community stakeholders and local elected officials. The NuHart East Site has completed the remediation and received the Certificate of Completion with the NYSDEC and the NuHart West Site is close to completion with an anticipated 2024 transition from a Class 2 to a Class 4 Inactive Hazardous waste Site.

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Development's, 101 Fleet Place, Brooklyn, New York. Principal responsible for the due diligence during acquisition, preparation of the Brownfield Cleanup Program Application, Change of Use Documents, BCA Amendments, remedial investigation, and remedial action design (BCP and OER) for a former bus depot Site under the New York State Brownfield Cleanup program and NYCOER E-Designation Programs (Air/Noise). The Site has a footprint of 20,000 SF with a planned development of a 21-story mixed use building with approximately 292 units which include affordable housing.

Development's, Speedway Portfolio, Multiple Boroughs, New York. Principal responsible for the expedited due diligence during acquisition of 5 former Speedway Sites of Phase I ESA's and Limited Phase II ESI's, preparation of the Brownfield Cleanup Program Applications, Remedial Investigation Work Plans, Interim Remedial Measure Work Plans and Air/Noise Remedial Action Work Plans (NYCOER). Five of the Sites were accepted into the NYSDEC Brownfield Cleanup program. Remedial Investigations for compliance with the Brownfield Cleanup Program have been completed and the remedial design on the Sites include a variety of remedial approaches which include in situ chemical treatment for groundwater, soil vapor extraction, excavation and dewatering removal and treatment.

Development, 138 Bruckner Boulevard, Bronx, New York. Principal responsible for the due diligence during acquisition, preparation of the Brownfield Cleanup Program Application, Change of Use Documents, coordination to vest the Site for 421-a, BCA Amendments, remedial investigation, and remedial action design (BCP and OER) for the former Zaro's Bakery Site under the New York State Brownfield Cleanup program and NYCOER E-Designation Programs (Air/Noise). The Site has a footprint of 50,000 SF with a planned development of a 12-story mixed use building with approximately 447 units which include affordable housing.

Development, 310 Grand Concourse, Bronx, New York. Principal responsible for environmental and construction management services required to successfully navigate this two-building redevelopment project through the NYSDEC Brownfield Cleanup Program (BCP) and NYCOER E-Designation Program (Air/Noise). Project included site investigation, design, and remediation for development of two buildings within a 30,000 square-foot lot in the Bronx, New York. Remediation included excavation of approximately 20,000 cubic yards of soil, groundwater extraction and treatment, underground storage tank (UST) removal, design, and installation an ex-situ chemical in situ soil stabilization process for elevated levels of metals.

Development, 40 Bruckner Boulevard, Bronx, New York. Principal responsible for the due diligence during acquisition, preparation of the Brownfield Cleanup Program Application, Change of Use Documents, BCA Amendments, remedial investigation, and remedial action design (BCP and OER) for the former Mill Sanitary Wiping Cloth Site under the New York State Brownfield Cleanup program and NYCOER E-Designation Programs (Air/Noise). The Site has a footprint of 45,000 SF with a planned development of a 12-story mixed use building with approximately 480 units which include affordable housing.

Development, 297 Wallabout Street, Brooklyn New York. Principal responsible for the due diligence during acquisition, preparation of the Brownfield Cleanup Program Application, Change of Use Documents, BCA Amendments, remedial investigation, and remedial action design (BCP and OER) for the 297 Wallabout Street Site under the New York State Brownfield Cleanup program and NYCOER E-Designation Programs (Air). Successfully delineated the onsite tetrachloroethene (PCE) plume in soil and groundwater. The Site is currently in the remedial implementation phase.

Developments, 89-91 Gerry & 93 Gerry Street, Brooklyn New York. Principal responsible for the due diligence during acquisition, preparation of the Brownfield Cleanup Program Application, Change of Use Documents, BCA Amendments, remedial investigation, and remedial action design (BCP and OER) for two Sites (adjacent to each other) located at 89-91 Gerry Street and 93 Gerry Street under the New York State Brownfield Cleanup program and NYCOER E-Designation Programs (Air). The Sites are currently preparing to execute the remedial action.

Development, Former Techtronics Site (8 Walworth Street), Brooklyn, New York. Principal for the remedial investigation, remedial action design and remedial action implementation for the former Techtronics Site under the New York State Brownfield Cleanup program as a Participant where trichloroethene (TCE) and tetrachloroethene (PCE)

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JAMES BELLEW PAGE 3

were encountered in soil and groundwater. James successfully delineated the vertical and lateral extents of the plumes which were identified as an upgradient, on-site. For this Site we have designed source removal to 20'bgs, Zero Valent Iron (ZVI) Reactive Barrier Wall, in situ ZVI injections sitewide and a vertical vapor mitigation system. The Site is currently in the remedial implementation phase.

Development, 346 Grand Concourse, Bronx, New York. Principal for the proposed 9-story, 60 key commercial building with one-level deep cellar. Design phase environmental services consist of guiding the Site through the New York City Office of Environmental of Remediation Voluntary Cleanup and E-Designation Programs, including Hazmat, Air Quality and Noise requirements. This program included submission of a Remedial Investigation Work Plan, implementation of a Remedial Investigation, submittal of a Remedial Investigation Report, Remedial Action Work Plans (Hazmat Air and Noise) and the Final Installation Report for the Certificate of Occupancy.

Development, 3294 Atlantic Avenue, Brooklyn, New York. Principal for the proposed 12-story, 80 key commercial building with one-level deep cellar. Design phase environmental services consist of guiding the Site through the New York City Office of Environmental of Remediation Voluntary Cleanup and E-Designation Programs, including Hazmat, Air Quality and Noise requirements. This program included submission of a Remedial Investigation Work Plan, implementation of a Remedial Investigation, submittal of a Remedial Investigation Report, Remedial Action Work Plans (Hazmat Air and Noise) and the Final Installation Report for the Certificate of Occupancy.

590-594 Myrtle Avenue, Brooklyn, New York. Principal for the proposed 6-story, 12-unit residential building with onelevel deep cellar. Design phase environmental services consist of guiding the Site through the New York City Office of Environmental of Remediation Voluntary Cleanup and E-Designation Programs, including Hazmat, Air Quality and Noise requirements. This program included submission of a Remedial Investigation Work Plan, implementation of a Remedial Investigation, submittal of a Remedial Investigation Report, Remedial Action Work Plans (Hazmat Air and Noise) and the Final Installation Report for the Certificate of Occupancy.

Development, 3530 Webster Avenue, Bronx, New York. Principal for the proposed 8-story, 75 key commercial building with one-level deep cellar. Design phase environmental services consist of guiding the Site through the New York City Office of Environmental of Remediation Voluntary Cleanup and E-Designation Programs, including Hazmat, Air Quality and Noise requirements. This program included submission of a Remedial Investigation Work Plan, implementation of a Remedial Investigation, submittal of a Remedial Investigation Report, Remedial Action Work Plans (Hazmat Air and Noise). The project is currently in the construction phase of the NYCOER program.

Development, Former BP Station, Elmhurst Queens, New York. Principal for the preparation of a full environmental impact statement with respect to a mixed-use development proposed in Elmhurst Queens for submission to the NYC Department of City Planning to rezone the project. The work included a full impact assessment of the proposed construction with respect to the neighborhood, evaluation of green/open spaces for the community and environmental site investigation and remediation services.

New York State Brownfield Site, Former Delta Metals Site, Brooklyn, New York. Senior Project manager for the remedial investigation and remedial action design for the former Delta Metal Products Company. Project is under the New York State Brownfield Cleanup program as a Participant where TCE and tetrachloroethene (PCE) were encountered in soil and groundwater. James successfully delineated the vertical and lateral extents of the plumes which were identified as an upgradient, on-site and downgradient plume. Investigation results triggered the NYSDEC to utilize its call-out contract to perform a plume track down for the immediate area and identify additional Potentially Responsible Parties. The design for an Air Sparge Soil Vapor Extraction system has been accepted and the project is currently under construction.

Manufacturing-Industrial, Hess Amerada, Bogota and Edgewater, New Jersey. Senior Project Manager and technical Lead for the construction management services for the demolition of two waterfront terminals on the Hackensack and Hudson rivers. Services included demolition design, submittal review, site execution and coordination of activities

related to asbestos abatement, demolition of buildings, thirty holding tanks, piping structures, containment structures and storm water structures.

Manufacturing-Industrial, PQ Corporation, Northeastern United States. Senior Project Manager responsible for the design and implementation of a three phased program for handling polychlorinated biphenyl (PCB) containing materials on approximately 100 tank structures at large, active industrial sites, which included coating removal, encapsulation, demolition, and Toxic Substances Control Act (TSCA) remediation. He was responsible for development of the overall program, specifications, drawings, bid packages, construction oversight and project administration until closure. The program also included design and oversight of a new façade and roof upgrades completed concurrently to client operations.

Development, New York State Brownfield Site, Former Cascade Laundry, Brooklyn, New York. Senior Project Manager responsible for environmental and construction management services required to successfully navigate a seven-building redevelopment project through the NYSDEC Brownfield Cleanup Program (BCP) and NYCOER E-Designation Program (Air/Noise). Project included site investigation, design, and remediation for development of seven buildings within a 2-acre site in Brooklyn, New York. Remediation included excavation of approximately 40,000 cubic yards of soil, groundwater extraction and treatment, underground storage tank (UST) removal, design, and installation of a sub slab depressurization system (SSDS) and ex situ chemical oxidation of groundwater impacted by petroleum.

Development, New York City Brownfield Site - 520-534 West 29th Street, New York, New York. James was responsible for environmental site investigation and remediation activities required to successfully navigate the project through the NYCOER's E-Designation and Voluntary Cleanup Programs. This program included submission of a Remedial Investigation Work Plan, implementation of a Remedial Investigation, submittal of a Remedial Investigation Report, Remedial Action Work Plans (Hazmat Air and Noise). The project is currently in the construction phase of the NYCOER program.

Development, New York State Brownfield Site, BJ's Wholesale, Brooklyn, New York. Senior Project Manager for the remedial execution within the NYSDEC BCP and NYCOER E-Designation programs at an 8-acre peninsula in Gravesend Bay being redeveloped by BJ's Wholesale Club (BJ's) into a "big-box" warehouse and parking garage, and a publicly accessible, waterfront open space. He implemented a comprehensive community air monitoring plan (CAMP), managed the design and installation of a passive sub slab depressurization system, and oversaw handling and off-site disposal of impacted material generated by BJ's (the Lessee for the subject site) during their foundation construction activities.

Development, New York State Brownfield Site, Coney Island, Brooklyn, New York. Senior Project Manager responsible for the environmental design during the rehabilitation and expansion of a 1970s-era mixed-use complex, which covers an area equivalent to three city blocks. He facilitated the BCP applications for two adjacent parcels within the complex impacted by historic dry-cleaning uses. Site investigations performed had documented the presence of PCE in soil gas and was delineated over three separate structural slabs in commercial and residential space utilizing a mobile laboratory. He designed and installed two sub-slab depressurization systems and prepared Remedial Investigation Work Plan which outlined work required to delineate the vertical and horizontal extent of the impacted soils, soil vapor and groundwater at both BCP sites. The system was designed with below slab suction pits, remote sensing vacuum monitoring points, and a variable frequency drive blower tied into the monitoring points for optimization and power savings.

Development, New York City Brownfield Site, Hospitals, Memorial Sloan Kettering Cancer Center (MSKCC), New York, New York. Project Manager for environmental remediation for this MSKCC development project. James was solely responsible for subsurface investigation and remediation activities, large, manufactured gas plant (MGP) gas holder removal (from former Con Edison Operations), UST removal, daily status updates to the NYCOER, implementation of the CAMP and the management, handling, characterization, and off-site disposal of MGP impacted soil and dewatering fluids.

New York State Spill Remediation, Metropolitan Transportation Agency Bridges and Tunnels, New York, New York. Project Manager responsible for execution of a remedial action scope which included UST removal, excavation of 600 cubic yards of petroleum impacted soil, design and installation of a groundwater extraction and treatment system and post remediation samples. He implemented the In Situ Chemical Oxidation program for the injection of 54,000 gallons of 8 percent solution Fenton's Reagent and the O&M (Operation & Maintenance) of the petroleum spill with respect to Fenton's performance and the plume migration.

Various Public Schools, New York City School Construction Authority, New York, New York. Project Manager responsible for environmental remediation proposed several school developments sites, including PS 312, P.S. 281, and PS 27K. Assisted in the design and implementation of the remediation programs for the sites for petroleum spills, PCB TSCA contamination and hazardous lead hot spots.

Development, i.Park Edgewater, Edgewater, New Jersey. Project Manager responsible for the design and environmental remediation on-site. Implemented the construction plan for remediation of arsenic, pitch- and PCB-impacted soil for excavation and off-site disposal of 20,000 tons. He managed the air monitoring system on-site which consisted of four permanent stations set upwind and downwind on-site for volatile organic compounds (VOCs) and particulate migration off-site. Also, James performed redesigns throughout the project to keep within the current schedule and budget.

Development, New York State Brownfield, Queens West, Long Island City, New York. Project Manager responsible for oversight of the Environmental Remediation on-site. James implemented the construction plan for remediation of 20,000 cubic yards of LNAPL on the Site; he assisted in design and oversight of the In Situ Chemical Oxidation mixing on-site. The project was eventually developed into three large towers and a new school.

Manufactured Gas Plant, National Grid, Rockaway, New York. James aided in the design and implementation of the soil characterization plan for MGP impacted sands. After delineation of the contamination plume, drafted work plans and site layout of the negative pressure tent. He performed and trained the on-site staff on the use of personal air monitoring equipment and aided with design considerations on the installation of a waterloo barrier to be advanced to minus 80 feet below grade surface. James also helped with the design and permitting for the groundwater treatment system installed on-site.

Manufactured Gas Plant, Con Edison, New York, New York. Environmental engineer for responsible party for all environmental issues associated with this job, including transportation and disposal of 8,000 tons of MGP contaminated soil from former Con Edison operations. James scheduled weekly work for all civil and environmental tasks on the job. He was responsible for the design and installation of the dewatering treatment system with a daily discharge of 25,000 gallons per day of MGP -impacted water.

New York State Superfund Project, NYSDEC, Hicksville, New York. James performed O&M and reporting on the Site's Potassium Permanganate Injection system, which was on a timed system; maintained the system, troubleshooting problems and ensuring that the proper ratios were being injected. He performed the fieldwork for analysis and drafted interim reports for the project manager.

Retail Petroleum, New York State Spills Program, Hess Amerada, Various Locations, New York. Environmental Engineer responsible for the design and installation of groundwater and soil vapor remedial systems at over 30 retail petroleum stations for Hess. Responsible for ensuring that the remedial systems were operating properly and performing repairs as necessary during operation. He performed groundwater and soil vapor monitoring and drafted O&M reports for the NYSDEC. Plume size ranged from within the retail station property with monitoring off-site impacts in local neighborhoods greater than a 3-mile radius.

Retail Petroleum, New York State Spills Program, British Petroleum (BP), Various Locations, New York. Environmental Engineer responsible for the design and installation of groundwater and soil vapor remedial systems at over 10 retail petroleum stations for BP. He was responsible for ensuring that the remedial systems were operating properly and performing repairs necessary during operation. He performed groundwater and soil vapor monitoring JAMES BELLEW PAGE 6

and drafted O&M reports for the NYSDEC. Plume size ranged from within the retail station property with monitoring off-site impacts in local neighborhoods greater than a 2-mile radius.

Development, 524 West 19th Street, New York, NY (Metal Shutter Homes). Project Engineer responsible party for all environmental and civil issues associated with this job, including transportation and disposal of 5,000 tons of MGP contaminated soil from former Con Edison operations. James scheduled weekly work for all civil and environmental tasks on the job. He successfully redesigned the grout cutoff wall connections to the installed steel sheeting with a secant wall installed off-site. He provided technical guidance for drilling 4-foot diameter exploratory casings for subsurface anomalies. Additionally, James was responsible for the design and installation of the dewatering treatment system with a daily discharge of 25,000 gallons per day of MGP impacted water.

EPA Superfund Site, Newtown Creek Superfund, Brooklyn, New York. Environmental Engineer who aided in the design of the pump and treat system installed at Peerless Importers. He also aided in the design and installation of the harbor boom set up. Operated and Maintained groundwater/LNAPL extraction systems on-site and performed monthly site gauging as part of the O&M plan.



MARI C. CONLON

Senior Client Account Manager

EDUCATION

MS, Geology, Boston College BS, Geology with a minor in Economics and Business, Lafayette College

PROFESSIONAL REGISTRATIONS NY: Professional Geologist (License No. 000769)

PROFESSIONAL SOCIETIES Big Apple Brownfield Awards, Co-Chair, 2018-2019 Big Apple Brownfield Awards Nomination Committee, 2016-2017

SPECIAL STUDIES AND COURSES

40-Hour OSHA Hazardous Waste Operations and Emergency Response Training (29 CFR 1910.120)
10-Hour OSHA Construction Safety
8-Hour OSHA Supervisor of Hazardous Waste (29 CFR 1910.120 & 29 CFR 1926.65)

Mari is a project manager with experience in soil, groundwater and soil vapor investigation and a focus on remedial design and implementation. She is also experienced in completion of numerous Phase I Environmental Site Assessments and Phase II Environmental Site Investigations, site characterization, hazardous materials analysis, regulatory closure reports as well as remedial design and implementation.

Mari has experience in composing site closure documentation including Remedial Closure Reports and Noise Installation Reports reviewed by the Office of Environmental Remediation as well as Final Engineering Reports reviewed by the New York State Department of Environmental Conservation. Her background includes developing and complying with approved site management plans overseeing the operation and maintenance of on-site engineering controls and ensuring the protection of human health and the environment.

Mari has also worked on city rezoning proposals by performing work associated with and composing the Hazardous Materials Analysis chapter included in Final Environmental Impact Statements published by New York City Department of Planning. Analysis methods were performed in accordance with the City Environmental Quality Review (CEQR) guidelines for neighborhoods including East New York, Brooklyn, Jerome Avenue, Brooklyn, Inwood, and Manhattan.

RELEVANT PROJECT EXPERIENCE

State and City Agencies

School Construction Authority, Waste Characterization and Excavation Materials Disposal Plan, Brooklyn, New York. Project manager for consulting services for New York Public School 127. Services included composition of an Excavated Materials Disposal Plan, collection of waste characterization samples and preparation of and preparation of a findings and recommendations report.

Department of City Planning, Rezoning Environmental Impact Statement, Bronx, New York. Project lead for analysis and composing the Hazardous Materials Chapter as per City Environmental Quality Review (CEQR) Technical Manual guidelines included in the Final Environmental Impact Statement (FEIS) for an approximately 92-block area primarily along Jerome Avenue and its east-west commercial corridors in the Bronx. The review assessed the potential for the presence of hazardous materials in soil and/or groundwater at both the projected and potential development sites identified in the reasonable worst-case development scenario under the proposed East New York Rezoning Proposal. Procedures involved site inspections and review of historic Sanborn fire insurance maps, city directories and city/state regulatory databases. The assessment identified that each of the 146 projected and potential development sites has some associated concern regarding environmental conditions. As a result, the proposed zoning map actions include (E) designations (E-366) for all privately-held projected and potential development sites.

Department of City Planning, Rezoning Environmental Impact Statement, Brooklyn, New York. Project lead for performance analysis and composing the Hazardous Materials Chapter as per CEQR Technical Manual guidelines included in the FEIS for an approximately 190-block area of East New York, Cypress Hills, and Ocean Hill neighborhoods of Brooklyn, New York. The review assessed the potential for the presence of hazardous materials in soil and/or groundwater at both the projected and potential development sites identified in the reasonable worst-case development scenario under the proposed East New York Rezoning Proposal. Procedures involved site inspections and review of historic Sanborn fire insurance maps, city directories and city/state regulatory databases. The assessment identified that each of the 186 projected and potential development sites has some associated concern regarding environmental conditions. As a result, the proposed zoning map actions include (E) designations (E-366) for all privately-held projected and potential development sites.

Redevelopment and Remediation

Titan Equity Group, Hotel Redevelopment, Bronx, New York. Project manager for a hotel redevelopment in the south Bronx. The site has been assigned New York City Office of Environmental Remediation (NYC OER) E-Designation status for hazardous materials, noise, and air quality. Services included completion of a remedial investigation, composition of a Remedial Investigation Report and development of Hazardous Material Remedial Action Work Plan and Air Quality/Noise Remedial Action Plan as per NYC OER requirements.

The Related Companies, Chelsea Mixed-Use Redevelopment, New York, New York. Field geologist for oversight of the remediation of a mixed-use residential and commercial building, the second of a two-building development on 30th Street. Contaminants of concern included volatile and semi-volatile organic compounds associated with historic operations and underground storage tanks (USTs) located on the Site. The Site was given an E-designation (E-142) for hazardous materials and noise as part of the Highline/West Chelsea rezoning proposal. To satisfy the requirements of the E-designation program, soil was excavated to at least 12 feet below grade and bottom endpoint collected showing no contaminants of concern exceeding the New York State Department of Environmental Conservation (NYSDEC) Unrestricted Use Soil Cleanup Objectives (SCO). By achieving Unrestricted Use SCOs, no engineering controls were necessary, although the building slab was included as part of development, and removal of the hazardous materials E-designation was requested.

Tishman Speyer, Long Island City Residential Development, Long Island City, New York. Field geologist for remedial oversight and implementation of a Community Air Monitoring Program during concurrent remediation and development of three Brownfield Cleanup Program (BCP) sites located in Long Island City, New York. The Sites were grossly contaminated with creosote, a carcinogenic chemical formed from the distillation of various tars. Remediation strategies included soil excavation and in-situ soil stabilization. To prevent migration of groundwater off-site, a temporary and later a permanent capture well system was installed on the western boundary of the property. The BCP site located on the western portion of the property left residual contamination in place requiring installation of a sub-slab depressurization system.

Queens West Development Corporation, Queens Waterfront Development, Long Island City, New York. Field geologist for performance of site management post remedial action. Services included annual groundwater monitoring, evaluation of engineering and institutional controls completion and Period Review Reports. In addition to conducting annual site management activities, responsibilities included composing a work plan to evaluate the transition from active sub-slab depressurization systems to passive. Upon NYSDEC approval, active systems were shut down for 30 days prior to a sub-slab vapor sampling event evaluation soil vapor, indoor and outdoor air conditions for potential vapor intrusion risk. As results indicated no evidence of vapor intrusion, continued pressure monitoring was conducted for from the existing monitoring ports for one year assessing whether negative pressure was held by the existing slab by stack-effect or other passive processes.

Jim Beam Brands Co., Brownfield Cleanup Program Remediation Site, Long Island City, New York. Field geologist for oversight of the installation of an Electrical Resistive Heating (ERH) system implemented in order to remediate trichloroethylene groundwater plumes in shallow/intermediate and deep groundwater on- and off-site. The Site, a

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former stapler manufacturing facility, underwent various remedies, including a Soil Vapor Extraction system, air sparging, ozone injection and chemical oxidation using potassium permanganate injections, which resulted in little reduction to contamination levels and rebounding chlorinated solvents. Components of the ERH system installed included electrodes for delivery of steam, vapor recovery wells, and groundwater monitoring wells. The site is currently under remediation in the state BCP program.

Due Diligence and Site Characterization

Manufacturing Plants, Multiple Investors, Environmental and Compliance Assessment Portfolio United States. Project lead for completion of Phase I Environmental Site Assessments (ESAs) and Limited Compliance Reviews for multiple auto parts manufacturing facilities throughout the United States. Services included completion of Phase I ESAs in accordance with the American Society for Testing and Materials E1527-13 requirements and a limited review of each facility's compliance liabilities including issues pertaining to the Resource Conservation and Recovery Act, Greenhouse Gas Emission Standards and Tier II Emergency and Hazardous Chemical Inventory reporting requirements.

ARM Parking, Environmental Site Assessment and Subsurface Investigation, Brooklyn, New York. Project manager for site assessment and subsurface investigation of parking facility in Sunset Park neighborhood, Brooklyn, New York. Services included ground penetrating radar survey for former and current petroleum USTs, completion of a subsurface investigation of soils and composition of Limited Subsurface Investigation Report.

Spill Consulting

The Trump Organization, Spill Consulting Services, New York, New York. Project manager for consulting services provided after incidental release of calcium carbonate ice rink paint to the Central Park Pond from Wollman Rink. Services included liaising with NYSDEC regarding violations, consent order and required corrective action. Corrective action included designing alterations to the existing on-site drainage plans and routing all meltwater containing paint into the combined sewer system. Coordination was required with property owner, operations personnel, New York City Department of Parks and NYSDEC.

Richmond Gardens Apartments, Spill Management and Closure Services, Staten Island, New York. Project lead responsible for spill closure activities and reporting for Spill 1105661 located at the Richmond Gardens Apartment Complex in the Richmond neighborhood of Staten Island, New York. The spill was opened in 2011 when several underground storage tanks were identified adjacent to the apartments at Jersey Street and Hendricks Avenue. The tanks were cleaned and removed and impacted soils surrounding the tank area excavated to the extent possible. Excavation of all impacted material was not feasible due to the proximity of the tanks to the apartment buildings. Residual contamination in soil and groundwater remained and was monitored through 2016. Upon reviewing the groundwater monitoring data from over 12 consecutive quarters, it was apparent monitored natural attenuation was not a feasible option and an in situ chemical oxidation (ISCO) remedy was approved by NYSDEC. Due to success of the pilot test, the ISCO injection event was implemented utilizing pressure pulse technology to deliver the alkaline activated persulfate solution to the subsurface.



HAILEY RUSSELL

Staff Professional

EDUCATION

B.S., Chemical Engineering with a concentration: Sustainable Engineering, Energy, and the Environment, North Carolina State University

PROFESSIONAL SOCIETIES

Tau Beta Pi Engineering Honors Society (NC Alpha), Treasurer, 2017-2018

SPECIAL STUDIES AND COURSES

40-Hour OSHA Hazardous Waste Operations and Emergency Response Training (29 CFR 1910.120) 10-Hour OSHA Construction Safety Training

8-Hour DOT Hazmat Employee & RCRA Hazardous Waste Generator Training

Hailey is a motivated, experienced chemical and environmental engineer who is knowledgeable in testing, process scale-up and data analysis. She also has experience in soil, groundwater, and soil vapor investigation, and preparation of technical documents. She has focused her time at Haley & Aldrich serving the Building & Infrastructure markets. In this effort, she has performed site reconnaissance to observe existing conditions and assessing site access for subsurface explorations. She has also monitored subsurface exploration activities, conducted soil and groundwater sampling, and conducted routine data collection, synthesis and analysis.

RELEVANT PROJECT EXPERIENCE

Environmental Experience

Madison Realty Capital, Former NuHart Plastics Site, New York State Superfund Site (NuHart West), Brooklyn, New York. Hailey served as a staff engineer during the demolition phase of Nuhart West. Her responsibilities included demolition oversight, well gauging, and air monitoring. NuHart is a high-profile site that requires coordination with the New York State Department of Environmental Conservation (NYSDEC), the New York City Office of Environmental Remediation (NYCOER), local regulatory agencies, community stakeholders and local elected officials.

The Jay Group, Speedway Portfolio, Brooklyn, New York. As a staff engineer, Hailey was responsible for a waste characterization, a lead delineation sampling event, and a supplemental remedial investigation, where she successfully determined the extent of contamination in one of the former Speedway sites. Additionally, she helped draft an in-situ chemical oxidation (ISCO) scope for the sites to treat the petroleum-based contamination in the groundwater. She also oversaw the installation of monitoring and injection wells for two of the sites in preparation for the ISCO treatment.

Mazel Mit Brucha 104 LLC, 817-819 Bedford Avenue, Brooklyn, New York. Hailey conducted a Limited Phase II Environmental Site Assessment (ESA) sampling event at 817 Bedford Avenue, which included soil characterization and sampling, groundwater and soil vapor sampling and well installation. In addition, she conducted a waste characterization sampling event for 817 Bedford Avenue and prepared a Brownfield Cleanup Program (BCP) application for 817-819 Bedford Avenue, which was submitted to the NYSDEC.

Toldos Yehuda, Former Techtronics Site (8 Walworth Street), Brooklyn, New York. Hailey served as staff engineer for environmental waste characterization services for the former Techtronics Site under the NYSBCP as a participant where trichloroethene (TCE) and tetrachloroethene (PCE) were encountered in soil and groundwater.

Lendlease (US) Construction LMB Inc, Shirley Chisholm Recreation Center, Brooklyn, New York. Hailey served as a staff engineer for a remedial investigation at the site. The investigation included the oversight of ten soil borings,

HAILEY RUSSELL PAGE 2

installation of nine soil vapor points, and installation of six temporary wells. Her responsibilities included classifying soil, developing wells, collecting environmental soil samples, collecting soil vapor samples, and conducting groundwater sampling for various analyses

Madison Realty Capital, River North, Staten Island, New York. As a staff engineer, Hailey performed environmental waste characterization services for the approximately two-acre site, as required by the disposal facility. In addition to characterizing soil and collecting samples, she collected bedrock samples for various analyses and drafted the waste characterization letter for the client.

Riverside Developers USA Inc, 43 Franklin Avenue, Brooklyn, New York. Hailey performed environmental waste characterization services for the site, as required by the disposal facility. In addition to characterizing soil and collecting samples for the waste characterization, she conducted lead delineation sampling, which determined the extent of lead contamination at one location of the site.

Manhattan Management Realty, 91 Bruckner Boulevard, Bronx, New York. Hailey served as a staff engineer for a remedial investigation at the site. The investigation included the oversight of 14 soil borings, installation of 11 soil vapor points, and installation of seven permanent wells. Her responsibilities included classifying soil, developing wells, collecting environmental soil samples, and collecting soil vapor samples.

Upton Metropolitan LLC, 808 Metropolitan Avenue, Bronx, New York. Hailey served as a staff engineer for a remedial investigation at the site. Her responsibilities included low-flow groundwater sampling for emerging contaminants such as PFAS.

BCP Applications for NYSDEC. Hailey has completed writing several BCP Applications for various clients in New York State. In writing the applications, Hailey reviews previous subsurface investigations of the site, and historical information to help get underutilized and abandoned contaminated properties into the BCP to be remediated and redeveloped under NYSDEC.

Multiple Clients, Phase I ESAs and Due Diligence, Multiple Locations in New York. Hailey conducted Phase I ESAs, for buyers on a variety of properties including commercial and residential sites in New York. She has experience conducting site reconnaissance and reviewing historical site documentation to identify recognized environmental conditions at the sites.



SEBASTIAN SOTOMAYOR

Staff Environmental Engineer

EDUCATION

B.S., Geological Sciences, Rutgers University

SPECIAL STUDIES AND COURSES

40-Hour OSHA Hazardous Waste Operations and Emergency Response Training (29 CFR 1910.120)
8-Hour OSHA Hazardous Waste Worker Refresher Training (29 CFR 1910.120)
OSHA HAZWOPER Site Supervisor
8-Hour DOT Hazmat Employee
10-Hour OSHA Construction Safety Training
NY SST Training
RCRA Hazardous Waste Generator Training

Sebastian is an engineer with four years of experience in environmental consulting including soil, groundwater, and soil vapor investigations, subsurface investigations, in-situ chemical oxidation remediation, preparation of technical reports, and data collection and analysis. He has extensive experience conducting Phase I Environmental Site Assessments (ESAs) and Phase II ESAs. He has performed soil sampling events, groundwater sampling events, soil gas/vapor surveys, and has drafted remedial investigation reports, along with remedial investigation work plans.

He focuses his time at Haley & Aldrich leading remedial investigations, monitoring subsurface exploration activities, completing technical reports, and assisting in the development of remedial work plans.

RELEVANT PROJECT EXPERIENCE

Environmental

Waterfront Management of NY, 89-91/93 Gerry Street, Brooklyn, New York. Sebastian conducted waste characterization events for both sites, and successfully delineated the vertical and horizonal extent of lead contamination on site. He was responsible for data collection, analysis, and drafted both waste characterization reports. Sebastian also served as a field engineer during the remedial oversight phase of the project, and was responsible for excavation oversight, collection of endpoint samples, implementation of community air monitoring, and proper disposal of on-site soil required for the installation of foundation elements.

Madison Realty Capital, River North, Staten Island, New York. As the lead field engineer, Sebastian was responsible for the execution of the remedial investigation, at this approximately 2-acre site. He coordinated with drillers for the installation of approximately fifty soil borings, twenty soil vapor points, including soil borings to bedrock. Sebastian was also responsible for data collection and analysis, and competition of the remedial investigation report (RIR). Sebastian also led field staff through the waste characterization event, which included the installation of 115 soil borings to bedrock.

Madison Realty Capital, 644 East 14th Street, New York, New York. Sebastian was the lead field engineer during both the remedial investigation and waste characterization event. He successfully delineated the vertical and lateral extents of lead contamination on site and was responsible for data collection and analysis.

Panoramic Hudson LLC, 541 West 37th Street, Hudson Yards, New York. Sebastian served as a staff engineer for the preparation of offsite investigative reports which included a limited ESA, and a remedial investigation work plan (RIWP). He also led waste characterization activities which included monitoring drilling activities, recording soil recovered by the drilling rig, and collecting suitable samples of soil to be analyzed.

SEBASTIAN SOTOMAYOR

PAGE 2

The Jay Group, Speedway Portfolio, Multiple Boroughs, New York. As a staff engineer, Sebastian was responsible for the preparation of remedial investigations and leading field staff through said investigations. He was also responsible for data collection and analysis, competition of the remedial investigation reports (RIR). Sebastian also led remedial oversight efforts, observed and documented excavation activities, implemented community air monitoring, and proper disposal of on-site soil required for the installation of foundation elements.

Appendix C: Laboratory SOPs



Always check on-line for validity. Analysis of Per and Polyfluoroalkyl Substances (PFAS) Aqueous Samples by LC-MS/MS Using Draft Method 16 PFAS-SOP63699	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in	Standard
	Operating Procedure	
Old Reference:		
Version: 3		Organisation level: 5-Sub-BU
Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS AII	Responsible: CLE PFAS All

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Revision Log Reference	
Cross Reference	
Scope	
Basic Principles	
Interferences	
Precaution to Minimize Method Interference	
Safety Precautions and Waste Handling	
Personnel Training and Qualifications	
Sample Collection, Preservation, and Handlin	ng
Apparatus and Equipment	
Reagents and Standards	
Calibration	
Procedure	
Calculations	
Statistical Information/Method Performance	
Quality Assurance/Quality Control	

Revision Log

Revision: 02	Effective date:	This version
Section	Justification	Changes
	en l'internet i	Update procedure.

Reference

1. US EPA Method 1633, Analysis of Per and Polyfluoroalkyl Substances(PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, Version 3rd DRAFT, December 2022.

Cross Reference

Document	Document Title	_
reconstruction of the second		

Curofins Comment number: PFAS-SOP63699 Old Reference: Version: 3 Approved by: HWD4	Ana Aque	ways check on-line for validity. Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633			
Effective Date: 20- NOV-2023	GLEI	PFAS All	CLE PFAS All		
Document		Document Title			
NC-WC-092		Determination of Solids in Waters and Wastes (Total Suspend Solids)	ded		
NDSC-QA-SOP42	2091	Detection and Quantitation Limits			
NDSC-QA-SOP4	3862	Manual Integrations			
NC-QA-015		Support Equipment Calibration and Verification			
NC-QA-018		Statistical Evaluation of Data and Development of Control C	harts		
QA-003		Quality Control Program			
NC-QA-017		Standards and Reagents			
NDSC-QA-QP44	940	Calibration Curves and the Selection of Calibration Points			
NDSC-US-EHS QP46060		NDSC EH&S Manual			
NC-QAM-001		QA Manual			

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8

US Eurofins Cleveland - Analysis of Per and Polyfluoroalky! Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633

Image: Second system Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 Old Reference: Version: 3 3			Standard Operating Procedure Organisation level 5-Sub-BU		
Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS AII				
	Analyte	Acronym	CAS#		
Perf	uoro-n-butanoic acid	PFBA	375-22-4		
Perfl	uoro-n-pentanoic acid	PFPeA	2706-90-3		
8:2 - Fl	uorotelomersulfonic acid	8:2FTS	39108-34-4	n ge - M	
N-methylperflu	ioro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9	86.	
N-ethylperfluoro	-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6		
4:2-Flu	orotelomersulfonic acid	4:2-FTS	757124-72-4	1941 - 1	
Perfluc	propentanesulfonic acid	PFPeS	2706-91-4	1.1.1.1	
6:2-Flu	orotelomersulfonic acid	6:2-FTS	27619-97-2		
Perfluc	proheptanesulfonic acid	PFHpS	375-92-8		
Perfluorononanesulfonic acid		PFNS	68259-12-1		
Perfluorodecanesulfonic acid		PFDS	335-77-3		
Perfluorododecanesulfonic acid		PFDoDS	79780-39-5		
Perflu	orooctanesulfonamide	PFOSA	754-91-6		
2-(N-methylperfluoro-1-octanesulfonamido)- ethanol		NMePFOSAE	24448-09-7	1. Jan	
N-methylperfluoro-1-octanesulfonamide		NMePFOSA	31506-32-8		
2-(N-ethylper	fluoro-1-octanesulfonamido)- ethanol	NEtPFOSAE	1691-99-2	10000	
N-ethylper	luoro-1-octanesulfonamide	NEtPFOSA	4151-50-2		
heptafluor	trafluoro-2-(1,1,2,2,3,3,3- opropoxy)-propanoic acid; propylene oxide dimer acid)	HFPODA	13252-13-6		
Ammonium 4,8-	dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *		
Potassium 9-chlo	prohexadecafluoro-3-oxanonane- 1-sulfonic acid	9CI-PF3ONS, F53B major	756426-58-1 *		
Potassium 11-ch	loroeicosafluoro-3-oxaundecane- 1-sulfonic acid	11CI-PF3OUdS, F53B minor	763051-92-9 *		
3-Perflu	oropropylpropanoic acid	3:3 FTCA	356-02-5		
3-Perflu	oropentylpropanoic acid	5:3 FTCA	914637-49-3		
3-Perflu	oroheptylpropanoic acid	7:3 FTCA	812-70-4		
Perfluoro-	3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1		

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Curofins Document number: PFAS-SOP63699 Old Reference:	Aqueous Samples by LC-MS/MS Using Draft Method 1633			Level: Standard Operating Procedure
Version: 3	_			Organisation level: 5-Sub-BU
Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS All			Responsible: CLE PFAS All
	Analyte	Acronym	CAS#	
Perfluor	o-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5	
Nonafluo	ro-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6	
Perfluoro(2	2-ethoxyethane)sulfonic acid	PFEESA, PES	113507-82-7	1

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

An aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. Carbon cleanup is performed on each sample extract. The extract is filtered and fortified with Isotopically-labeled injection internal standards. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

- 1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK[™] solvent frits and tubing where possible.
- 2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.
- 3. All part of the SPE manifold must be cleaned every 2 weeks per manufacturer recommendations.

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Document number: PFAS-SOP63699		Operating Procedure
Old Reference:		
Version: 3		Organisation level: 5-Sub-BU
Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS AII	Responsible: CLE PFAS All

Safety Precautions and Waste Handling

See NDSC EH&S Manual, the Facility Addendum to the NDSC EH&S Manual, and this document for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level. IDOC must meet the requirements for the IPR as listed in Table 5 of method 1633 draft, V3. See *Attachment 19*.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 125-mL HDPE containers. All sample containers must have linerless HDPE or polypropylene caps. Keep the sample sealed from time of collection until extraction.

Cocument number: PFAS-SOP63699 Old Reference:	Always check on-line for validity. Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633	Level: Standard Operating Procedure
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Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS AII	Responsible: CLE PFAS All

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

- B. Sample Storage and Shipment
- 1. Samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.
- Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, and protected from light until extraction. Alternatively, to meet project requirements, samples may be stored at ≤ -20°C and protected from light until extraction.
- 3. Water samples must be extracted within 28 days when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Water samples must be extracted within 90 days when stored at a temperature < -20°C and protected from light. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light.

Apparatus and Equipment

- A. Apparatus
- 1. 125 mL HDPE bottles: Environmental Sampling Supply; #0125-1902-QC, or equivalent.
- Centrifuge tubes 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 14955237 or equivalent
- 3. 10-mL polypropylene volumetric flask, Class A Fisher Scientific, Cat. No. S02288 or equivalent.
- HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC, or equivalent.
- 5. Analytical Balance Capable of weighing to 0.0001 g
- 5. Top-Loading Balance Capable of weighing to 0.01 g

7.Waters Oasis Wax-150 mg/6cc (Part # 186002493) or equivalent with 10mg carbon added.

- 3. Large-volume SPE Reservoir (25-mL) Millipore-Sigma; Product # 54258-U.
- 9. SPE Tube Adapter Millipore-Sigma; Product # 57020-U.
- 10. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.
- 11. Polypropylene SPE delivery needles Agilent; Cat. No. 12234511.
- 12. Vacuum manifold for Solid Phase Extraction (SPE).
- 13. Centrifuge "Thermo Sorvall ST4" with TX-750 rotor or equivalent, capable of a maximum rotational force of 3800 rcf.

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Old Reference:		140
Version: 3		Organisation level: 5-Sub-BU
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14. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. FisherScientific cat # 14-287-150, or equivalent.

15. Polypropylene pipette tips: 0-200µl. Fisher; Cat. No. 22491539

- 16. Polypropylene pipette tips: 101-1000µl. Fisher, Cat. No. 5414006
- 17. Pipettes Disposable transfer. Fisher Scientific, Cat. No. 13-711-7M
- 18. Vortex mixer, variable speed, Fisher Scientific or equivalent.
- 19. N-Evap sample extract concentrator with N₂ supply and water bath for temperature control. Organomation, Inc. Cat. #11250-NT, or equivalent.
- 20. Thermo Target PP Polyspring inserts, catalog number C4010-630P, or equivalent
- 21. Thermo Target 9mm PP vials, catalog number C4000-14, or equivalent
- 22. Agilent 9mm vial caps, polypropylene/silicone septa, catalog 5191-8151, or equivalent
- 23. Centrifuge tubes 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent
- 24. Polypropylene bottles for standard storage 4 mL; Fisher Scientific, Cat. No. 2006-9125

25. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product # 11-865-130.

- 26. pH paper, range 0-14, Supelco # 1.09533.0001 or equivalent, 0.5 unit readability
- 27. Syringe filter Acrodisc, Syringe Filter, 25 mm, 0.2 µm, 50/pkg, Fisher part # 50786068.
- 28. Silanized glass wool, Supelco # 20411 or equivalent
- 29.Wheaton Bottle, 15ml, narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Cat. No. 209044SP, or equivalent
- B. Equipment
 - 1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

ExionLC Controller ExionLC AC Pump ExionLC AC Autosampler Exion AC Column Oven Data system -Analyst 1.6.3

- PromoChrom automated extractor, model, SPE-03, or equivalent

 Promochrom Filters, Catalog Number F-HC-30
- 3. HPLC columns
 - Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-Y0 or equivalent
 - b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

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Document number: PFAS-SOP63699	Adactus camples by Le Moy Ms Using Drait Method 1055	Operating Procedure
Old Reference:		
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Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions. All reagents are verified before use by the associated Certificate of Analysis or by analysis of the Method Blank.

A. Reagents:

- 1. Methanol (MeOH) J.T. Baker Ultra Resi-Analyzed, Cat. No. UN1230 or equivalent
- 2. Acetonitrile (ACN) J.T. Baker Ultra Resi-Analyzed, Cat. No. UN1648 or equivalent
- 3. Water Fisher Scientific, Optima, Cat. No. W6-4 or equivalent
- 3. Ammonium acetate Fisher Scientific, Cat. No. A637-500 or equivalent
- 4. Methanolic ammonium hydroxide (1%) add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
- Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
- 6. Acetic Acid ACS grade or equivalent, store at room temperature
- 7. Acetic Acid (0.1%) dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
- 8. Formic acid-(greater than 96% purity or equivalent), store at room temperature.
 - a. Formic acid (aqueous, 0.1 M) dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years
 - b. Formic acid (aqueous, 0.3 M) dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years
 - c. Formic acid (aqueous, 5% v/v) mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years
 - d. Formic acid (aqueous, 50% v/v) mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years
 - e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years
- 9. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.

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- 10. Eluent A: nt20 mM ammonium acetate solution in 95:5(v/v) reagent water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL reagent water and mix well to dissolve the ammonium acetate. Add 100 mL acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.
- 11. Eluent B: 20 mM ammonium acetate solution in 90:10 acetonitrile/reagent water Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 200 mL of reagent water and mix well to dissolve the Ammonium Acetate. Add 1800 mL of acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

B. Standards:

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10 ml Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent. After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 μ l should be avoided in routine production operations.

All stock, intermediate and spiking solutions are prepared using Methanol.

All initial calibration, initial calibration verification, and linear branched working standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

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The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 04*.

If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent, i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.

- Attachment 05 Native PFAS Intermediate A Attachment 06 - Native PFAS Intermediate B Attachment 07 - Native PFAS Intermediate M Attachment 08 - Extraction Standard Mix Attachment 09 - Internal Standard Mix Attachment 10 - Initial Calibration Standards Preparation Attachment 11 - Initial Calibration Standards Concentrations Attachment 12 - Bile Salts Stock Solutions Attachment 13 - Bile Salts Working Solution A Attachment 14 - Bile Salts Working Solution B Attachment 15 - PFAS Linear/Branched Mix A Attachment 16 - PFAS Linear/Branched Mix B
- Attachment 17 Linear/Branched Bile Salts Solution

Attachment 18 - ICV Working Standard

Calibration

A. Initial Calibration

- 1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order(quadratic) curve fit. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.
- 2. Analyze a Cal4 level standard that contains Bile Salt retention time markers and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMEFOSA, NEtFOSA, NMEFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the

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Document number: PFAS-SOP63699		Operating Procedure
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calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

Example Initial Calibration Sequence:

- 1. Instrument Blank
- 2. Instrument Blank
- 3. Instrument Blank
- 4. CAL 1
- 5. CAL 2
- 6. CAL 3
- 7. CAL 4
- 8. CAL 5
- 9. CAL 6
- 10. CAL 7
- 11. ICB (Instrument Blank)
- 12. ICV
- 13. WDM (Linear Branched/Bile Salts standard)
- Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See <u>Attachment 02</u> for additional information about compound relationships.
- 4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic acid (TCDCA), and Tauroursodeoxycholic acid (TUDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

NOTE: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA.

Compound	Extraction Standard	
PFBA	13C4-PFBA	
PFPeA	The second second	
3:3FTCA	1205 050-4	
PFMPA	13C5-PFPeA	
PFMBA	1. N. P.	
PFHxA	A DEPOSIT	
NFDHA		
5:3FTCA	13C5-PFHxA	
7:3FTCA		
PFEESA		
PFHpA	13C4-PFHpA	
PFOA	13C8-PFOA	

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	PFNA	13C9-PFNA		
	PFDA	13C6-PFDA		
	PFUnA	13C7-PFUnA		
	PFDoA	13C2-PFDoA		
8	PFTrDA	Avg 13C2- PFTeDA and 13C2-PFDoA		
	PFTeDA	13C2-PFTeDA		
	PFBS	13C3-PFBS		
PFPeS		1202.05040		
PFHxS		13C3-PFHxS		
PFHpS				
	PFOS		13C8-PFOS	
PFNS PFDS		13C8-PFOS		
	PFDoS			
2	1:2-FTS	13C2-4:2-FTS		
6	5:2-FTS	13C2-6:2-FTS		
٤	3:2-FTS	13C2-8:2-FTS		
	PFOSA	13C8-PFOSA		
N	MeFOSA	D3-NMeFOSA		
N	IEtFOSA	D5-NEtFOSA		
N	1eFOSAA	D3-NMeFOSAA		
NEtFOSAA		D5-N-EtFOSAA		
NMeFOSE		D7-NMeFOSE		
N	IEtFOSE	D9-NEtFOSE		
HFPO-DA DONA 9CI-PF3ONS				
		1302 4500 54		
		13C3-HFPO-DA		
11C	I-PF3OUdS			

5. Fit the curve

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- a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.
- b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.
- c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within $\pm 30\%$ of its true value.
- d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See <u>Attachment 04</u>.

6. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must be within $\pm 30\%$ of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

- B. Continuing calibration
 - 1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.
 - 2. Acceptance criteria
 - a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.
 - b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

All water samples should be evaluated for Total Suspended Solids (TSS) as per the TSS SOP. Although the full container volume of sample should be extracted, this method is applicable to aqueous samples containing up to 100 mg/L of suspended solids. For samples containing >100mg/L of suspended solids, or when unavoidable due to high levels of PFAS, smaller samples volumes may be analyzed.

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However, ultimately, the TSS results, visual inspection, and analyst experience are all used to determine the volume to be used to extract the sample.

A. Sample Preparation

- 1. Weigh sample container with contents on a calibrated top loading balance, and record the first reading in the automated prep entry system.
 - a. For all samples, the full bottle must be extracted. The sample must remain in the original sample container until after spiking solutions have been added.
 - b. If limited sample is submitted, spike sample in original container, then add reagent water to bring to final volume of 125 mL prior to SPE extraction (see B.6 for spiking details).
 - c. If the sample has dissolved and/or settleable solids content (i.e; is cloudy or has a layer of sediment/solids at the bottom of the bottle), the sample must be centrifuged in order to minimize the difficulty of passing throug the SPE sorbent bed. In order to preserve the integrity of the sample and ensure the full voume of the container is used, see Procedure C.
- Use a 125 mL HDPE bottle for the method blank, the laboratory control sample (LCS) and the low level laboratory control sample (LLCS). Fill each bottle with 125 mL of reagent water. Record 125 mL as the volume for the batch QC samples on the batchlog.
- 3. Check that the pH is 4.0±0.5. If necessary, adjust the pH with 50% formic acid (or with 5% formic acid). NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.
- B. Solid Phase Extraction (SPE) Manual Sample Extraction
 - 1. Pack clean silanized glass wool to a quarter of the height of the SPE cartridge barrel.
 - 2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batch log.
 - 3. Condition each SPE cartridge with the following reagents in the following order without allowing the cartridges to go dry:
 - a. 15 mL 1% methanolic ammonium hydroxide
 - b. 5 mL 0.3M formic acid
 - c. Discard conditioning eluent(s)
 - 4. Label each sample bottle, cap and reservoir with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).
 - 5. Vortex all spike solutions prior to use.
 - Spike QC and all samples with 25 µl of Working labeled extraction standard spike solution (PFAS EIS Mix). Spike LCS/MS/MSD with 200 µl of native 1633 mid-level spike solution (PFAS Native M). Spike LLCS with 200 µl of native 1633 low-level spike solution (PFAS Native B). Vortex/Shake containers to mix thoroughly.
 - 7. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges.
 - 8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5 mL 1:1 0.1M formic acid/methanol and pass the rinses through the cartridge using vacuum. Apply full

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vacuum (not exceeding 20" Hg) to the cartridges and dry until they are visually similar to an unused cartridge. This may take up to 15 minutes for some cartridges. Discard the rinse solution.

- NOTE: If a given sample is too problematic to extract using one cartridge with full vacuum, a second cartridge is created following Procedure B.1-3 above.
- 9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge ensuring the delivery needles to do not touch the sides of the tubes.
- 10. Rinse the inside of each empty sample/QC bottle with 5mL of 1% methanolic ammonium hydroxide.
- 11. Using a glass pipette, transfer the rinse from the bottles to the SPE reservoirs, washing the walls of the reservoirs. Set empty bottles aside to air dry.
- 12. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.
- 13. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.
- Note: If a second cartridge was required, the elutant from the second cartridge is added to the first cartridge elutant with a methanol rinse and then blown down to 5 mL on an N-evap prior to step 14 below.
- 14. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix.
- 15. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.
- 16. Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to a clean 15-mL polypropylene centrifuge collection tube.
- 17. Place a syringe filter (25-mm filter, 0.2-μm nylon membrane) on a 5 mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the plunger and filter the entire extract into the new collection tube containing the internal standard.
- 18. Bring each sample extract to final volume 5ml using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.
- 19. Cap and vortex to mix.
- 20. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.
- 21. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for dilution or reinjection if needed.

C. Samples containing Dissolved and/or Settleable Solids

1. Check that the pH is 4.0 ± 0.5 . If necessary, adjust the pH with 50% formic acid (or with 5% formic acid). **NOTE:** Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.

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- 2. Spike sample with appropriate spikes as in Procedure B.6.
- 3. Centrifuge the full bottle.
- 4. DO NOT SHAKE BOTTLE FOLLOWING THE CENTRIFUGE STEP.
- 5. Follow steps in Procedure B, 1 through 4.

6. Attach a 25-mL SPE reservoir to each cartridge. Decant the centrifuged sample onto its respective SPE cartridge. Allow the full volume to pass through each cartridge by gravity if possible. Apply a light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.

7. Rinse the sample bottle with 5mL of reagent water, add the rinseate to the cartridge, and repeat.

8. Continue extraction process with Procedure B. 8.

D. Solid Phase Extraction (SPE) - Automated Sample Extraction using Promochrom

This procedure can be used in place of the manual extraction described above.

Note: all samples must be prescreened prior to use of the promochrom to avoid carryover and contamination.

- 1. Pack clean silanized glass wool to a quarter of the height of the SPE cartridge barrel.
- 2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.
- 3. Check to make sure all reagent bottles are filled and that reagents are not expired.
- 4. Label each sample bottle and cap with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).
- 5. Vortex all spike solutions prior to use.
- Spike QC and all samples with 25 µl of Working labeled extraction standard spike solution (PFAS EIS Mix). Spike LCS/MS/MSD with 200 µl of native 1633 mid-level spike solution (PFAS Native M). Spike LLCS with 200 µl of native 1633 low-level spike solution (PFAS Native B). Vortex/Shake containers to mix thoroughly.
- 7. Select "Clean sys-004" from the drop down menu to start the cleaning cycle prior to SPE. Make sure the "Clean" cartridges and bottles are attached for this cycle. After the "Clean sys-004" has finished, attach Promochrom filters to the bottles, select "Clean Filter" from the drop down menu to clean filters prior to SPE.
- 8. Remove "Clean" cartridges and attach the labeled cartridges containing a graphitized carbon that will be used when running the samples when the system cleaning is complete. Disconnect the "Clean" bottles. Attach the labeled sample bottles by twisting the bottles and not the cap. Place sample bottles upside down in corresponding numbered location on shaker. Poke two holes into the lip of each bottle. Load labeled centrifuge tubes in the moving tray beneath the cartridges.

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- 9. Select "EPA 1633-004" from the drop down menu and press the green check mark to start the procedure. Note: N2 dry down should be 15 minutes long.
- 10. Once the cycle is complete, remove centrifuge tubes and discard the used cartridges and bottles. Reattach "Clean" bottles and cartridges, select "clean sys-004" from menu, select "start". This must be done between each batch and at the beginning and the end of the day.
- 11. Add 25ul of Acetic acid to sample extracts and vortex to mix thoroughly. Extracts must be stored in a cooler.
- Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to a clean 15-mL polypropylene centrifuge collection tube. Using a syringe, push ~ 3mL of MeOH through each syringe filter and allow to dry..
- Place a syringe filter (25-mm filter, 0.2-μm nylon membrane) on a 5 mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the plunger and filter the entire extract into the new collection tube containing the internal standard.
- 14. Bring each sample extract to final volume 5ml using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.
- 15. Cap and vortex to mix.
- 16. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.
- 17. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for reinjection if needed.
- E. LC/MS/MS Analysis
 - 1. Mass Calibration and Tuning
 - a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.
 - b. When masses fall outside of the +/- 0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.
 - 2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.
 - 3. Example acquisition method: See Attachment 03. Mass Transitions: See Attachment 01.
 - 4. Instrument Sensitivity Check (ISC) and Instrument Blanks
 - a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the

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LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within $\pm 30\%$ of their true values. The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.

- b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
- 5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.
- 6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salt RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

Example Sample Sequence:

- 1. Instrument blank
- 2. Instrument blank
- 3. Instrument blank
- 4. Instrument Sensitivity Check (CCV 0_CAL1)
- 5. CCV 2_CAL4
- 6. Linear Branched/Bile Salts marker (WDM)
- 7. Instrument Blank (ICB)
- 8. Method Blank (MB)
- 9. Low Level LCS (LLCS)
- 10. LCS
- 11. Sample (10 or less)
- 12. CCV 3_CAL4
- 13. Instrument Blank
- 7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.
- 8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.
- 9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

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- a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.
- b. If LLCS and LCSD recoveries are acceptable, proceed to sample quantitation.
- c. If the LCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS must be reextracted.
- d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10.

- Calculate the relative percent difference(RPD) between the sample and the matrix duplicate(DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data.
- 11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. (See Statistical Infromation/Method Performance section of this SOP.) If the extraction standard recovery(ies) is(are) outside the QC limit(s), revial a fresh aliquot and reanalyze. If the extraction standard recovery(ies) are still outside the QC limit(s), reanalyze the sample at a dilution. If the extraction standard recovery(ies) are still outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) are still outside the QC limit(s), are still outside the QC limit(s), narrate with an NCM and report the data.
- 12. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.
- 13. Compare the retention times of all of the analytes, surrogates, and internals standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than
 - a. 0.4 minutes for isotopically-labeled compounds
 - b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound
 - c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

14. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of 13C4-PFBA, 13C5-PFPeA, 13C4-PFHpA, 13C8-PFOA, 13C9-PFNA, 13C6-PFDA, 13C7-PFUnA, 13C2-PFDA,13C2-PFDoDA 13C2-PFTeDA, 13C8-PFOSA, D3-NMePFOSA, D5-NEtFOSAA, D3-NMeFOSAA, D5-NEtPFOSA, D7-NMePFOSAE, D9-NEtPFOSAE, 13C3-PFBA, 13C4-

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PFOA, 13C5-PFNA, 13C2-PFOA, 18O2-PFHxS, PFBA, PFECA F(PFMPA), PFECA A(PFMBA), NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier "I" is placed on the raw data and on the sample report. No corrective action is required in prepped samples. Ion ratios must be in control in calibration solutions. If they are outside of limits, stop the analysis and correct the issues.

- 15. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.
- 16. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of internal standard spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring dilutions greater than 10X should be reextracted using a reduced aliquot.

Dilution Example 1/10: Mix 895 μ l of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 μ l of sample extract and 5 μ l of internal standard spike solution. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio

Peak Area Ratio = <u>Analyte Response</u> Labeled Analyte Response

2. On-Column Analyte Concentration using average RRF

On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

On-column Concentration = (peak area ratio - intercept) ÷ slope

4. Sample Concentration

Sample concentration (ng/l) = (On-column concentration x Final Sample Volume x DF) \div Initial Sample Volume

5. Ion Ratio

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ion ratio = (peak area or height of quantifier)/(peak area or height of qualifier)

6. See cross-referenced SOPs for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS(OPR) and LLCS(LLOPR) should contain all compounds of interest. LCS, LLCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are method defined. The LCS(OPR) and LLCS(LLOPR) and their respective EIS limits are from Table 5 of Draft 1633, V3. See *Attachment 19*. The MS/MSD native recoveries are compared to the OPR limits listed in Table 5 Draft 1633, V3. See *Attachment 19*. The EIS limits for the MS/MSD, DUP, Method Blank, and samples are listed in Table 8 from Draft 1633,V3. See *Attachment 20*.

QC parameter	Lower acceptance limit	High acceptance limit
Non-extracted Internal Standard (NIS)	>30% of the average NIS from the initial calibration	200%
Sample/DUP	=30%</td <td>NA</td>	NA

Records of the standard deviation of the percent recovery (SR) of EIS and NIS compounds from samples must be maintained and should be assessed periodically. It is recommended that this assessment is done quarterly. The assessment should be expressed as the average percent recovery +/- 2SR for each isotopically labeled compound, and evaluated at least annually.

Historical data for MS/Ds, LCSs, EIS, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to the SOP for Determining Method Detection Limits and Limits of Quantitation for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank, an LCS/LLCS (Milli Q water spiked with all compounds to be determined carried through the entire procedure), and a sample/DUP, must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Attachment:
Attachment 01 - Mass Transitions (.doc)
Attachment 02 - Standard Relationships (.docx)
Attachment 03 - Acquisition Parameters (.pdf)
Attachment 04 - Example Certificate of Analysis (.pdf)
Attachment 05 - Native PFAS Intermediate A (.pdf)
Attachment 06 - Native PFAS Intermediate B (.pdf)
Attachment 07 - Native PFAS Intermediate M (.pdf)

	Always check on-line for validity.	Level:	
Cocument number: PFAS-SOP63699 Old Reference:	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633	Standard Operating Procedure	
Version: 3		Organisation level: 5-Sub-BU	
Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS AII	Responsible: CLE PFAS All	
Attachment 10 - I Attachment 11 - I Attachment 12 - E Attachment 13 - E Attachment 14 - E Attachment 15 - F Attachment 16 - F Attachment 17 - L Attachment 18 - I Attachment 19 - T Attachment 20 - T	Internal Standard Mix (.pdf) Initial Calibration Standards Preparation (.pdf) Initial Calibration Standard Concentrations (.pdf) Bile Salt Stock Solutions (.pdf) Bile Salts Working Solution A (.pdf) Bile Salts Working Solution B (.pdf) PFAS Linear Branched Mix A (.pdf) PFAS Linear Branched Mix B (.pdf) Linear Branched Bile Salts Solution (.pdf) ICV Working Standard (.pdf) Fable 5 (1633 Draft V3) (.docx) Fable 8 (1633 Draft V3) (.docx)		
Attachment: Attac Attachment: Attac	chment 01 - Mass Transitions (doc) chment 02 - Standard Relationships (docx) chment 03 - Acquisition Parameters (pdf) chment 04 - Example Certificate of Analysis (pdf) chment 05 - Native PFAS Intermediate A (pdf) chment 06 - Native PFAS Intermediate B (pdf) chment 07 - Native PFAS Intermediate M (pdf) chment 08 - Extraction Standard Mix (pdf) chment 09 - Internal Standard Mix (pdf) chment 10 - Initial Calibration Standards Preparation (pdf) chment 11 - Initial Calibration Standard Concentrations (pdf) chment 12 - Bile Salt Stock Solutions (pdf) chment 13 - Bile Salts Working Solution A (pdf) chment 14 - Bile Salts Working Solution B (pdf) chment 15 - PFAS Linear Branched Mix A (pdf) chment 16 - PFAS Linear Branched Mix B (pdf) chment 17 - Linear Branched Bile Salts Solution (pdf) chment 18 - ICV Working Standard (pdf) chment 19 - Table 5 (1633 Draft V3) (docx) chment 20 - Table 8 (1633 Draft V3) (docx)		

End of document

Version history

Version	Approval	Revision information
0	24.AUG.2023	
2	03.NOV.2023	
3	20.NOV.2023	

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
1802-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

Attachment 1

Compound	Parent Ion	Daughter Ion
13C8-PFOA	421.1	376.0
PFOA	413.0	369.0
PFOA (2)	413.0	169.0
13C4-PFOS	502.8	79.9
13C4-PFOS (2)	502.8	98.9
13C8-PFOS	507.1	79.9
13C8-PFOS (2)	507.1	98.9
PFOS	498.9	79.9
PFOS (2)	498.9	98.8
13C5-PFNA	468.0	423.0
13C9-PFNA	472.1	427.0
PFNA	463.0	419.0
PFNA (2)	463.0	219.0
13C8-PFOSA	506.1	77.8
PFOSA	498.1	77.9
PFOSA (2)	498.1	478.0
PFNS	548.8	79.9
PFNS (2)	548.8	98.8
13C2-PFDA	515.1	470.1
13C6-PFDA	519.1	474.1
PFDA	512.9	469.0
PFDA (2)	512.9	219.0
13C2-8:2-FTS	529.1	80.9
13C2-8:2-FTS (2)	529.1	509.0
8:2-FTS	527.1	507.0
8:2-FTS (2)	527.1	80.8
d7-NMePFOSAE	623.2	58.9
NMePFOSAE	616.1	58.9
d3-NMePFOSA	515.0	219.0
NMEPFOSA	511.9	219.0
NMEPFOSA (2)	511.9	169.0
d3-NMeFOSAA	573.2	419.0
NMeFOSAA	570.1	419.0
NMeFOSAA (2)	570.1	483.0
d9-NEtPFOSAE	639.2	58.9
NEtPFOSAE	630.0	58.9
d5-NETPFOSA	531.1	219.0
NEtPFOSA	526.0	219.0
NEtPFOSA (2)	526.0	169.0
PFDS	599.0	79.9

Attachment 1

Compound	Parent lon	Daughter Ion
PFDS (2)	599.0	98.8
13C7-PFUnDA	570.0	525.1
PFUnDA	563.1	519.0
PFUnDA (2)	563.1	269.1
d5-NEtFOSAA	589.2	419.0
NEtFOSAA	584.2	419.1
NEtFOSAA (2)	584.2	526.0
13C2-PFDoDA	615.1	570.0
PFDoDA	613.1	569.0
PFDoDA (2)	613.1	319.0
PFDoS	699.1	79.9
PFDoS (2)	699.1	98.8
PFTrDA	663.0	619.0
PFTrDA (2)	663.0	168.9
13C2-PFTeDA	715.2	670.0
PFTeDA	713.1	669.0
PFTeDA (2)	713.1	168.9
13C3-HFPODA	286.9	168.9
13C3-HFPODA (2)	286.9	184.9
HFPODA	284.9	168.9
HFPODA (2)	284.9	184.9
DONA	376.9	250.9
DONA (2)	376.9	84.8
9CI-PF3ONS	530.8	351.0
9CI-PF3ONS (2)	532.8	353.0
11CI-PF3OUdS	630.9	450.9
11CI-PF3OUdS (2)	632.9	452.9
PFECA B (NFDHA)	295.0	201.0
PFECA B(NFDHA) (2)	295.0	84.9
PFECA F (PFMPA)	229.0	84.9
3:3 FTCA	241.0	177.0
3:3 FTCA (2)	241.0	117.0
PFECA A (PFMBA)	279.0	85.1
PFEESA (PES)	314.8	134.9
PFEESA (PES) (2)	314.8	82.9
5:3 FTCA	341.0	237.1
5:3 FTCA (2)	341.0	217.0
7:3 FTCA	441.0	316.9
7:3 FTCA (2)	441.0	336.9

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

Inj Std	Internal Standard/Injection Standard
I13C3-PFBA	13C3-PFBA
I13C2-PFHxA	13C2-PFHxA
I13C4-PFOA	13C4-PFOA
I13C5-PFNA	13C5-PFNA
I13C2-PFDA	13C2-PFDA
I18O2-PFHxS	18O2-PFHxS
I13C4-PFOS	13C4-PFOS

Extraction Standards

Extraction Standard	Internal Standard
E13C4-PFBA	13C3-PFBA
E13C5-PFPeA	
E13C5-PFHxA	1202 DELL. A
E13C4-PFHpA	13C2-PFHxA
E13C3-HFPO-DA	
E13C8-PFOA	13C4-PFOA
E13C9-PFNA	13C5-PFNA
E13C6-PFDA	
E13C7-PFUnA	13C2-PFDA
E13C2-PFDoA	ISC2-PFDA
E13C2-PFTeDA	
E13C3-PFBS	
E13C3-PFHxS	
E13C2-4:2-FTS	18O2-PFHxS
E13C2-6:2-FTS	
E13C2-8:2-FTS	

Page 1 of 3

Extraction Standard	Internal Standard
E13C8-PFOS	
E13C8-PFOSA	
Ed3-NMeFOSA	
Ed5-NEtFOSA	13C4-PFOS
Ed3-NMeFOSAA	1504-1105
Ed5-NEtFOSAA	
Ed7-NMeFOSE	
Ed9-NEtFOSE	

Native PFAS Compounds

Native	Extraction Standard
PFBA	13C4-PFBA
PFPeA	
3:3FTCA	13C5-PFPeA
PFMPA	ISCS-FFFEA
PFMBA	
PFHxA	
NFDHA	
5:3FTCA	13C5-PFHxA
7:3FTCA	
PFEESA	
РҒНрА	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	1202 DELL-9
PFHxS	13C3-PFHxS
PFHpS	
PFOS	1000 0000
PFNS	13C8-PFOS
PFDS	

Page 2 of 3

Native	Extraction Standard
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	
DONA	
9Cl-PF3ONS	- 13C3-HFPO-DA
11Cl-PF3OUdS	

Attachment "Attachment 06 - Native PFAS Intermediate B" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Page 28 of 87 Samples by LC-MS/MS Using Draft Method 1633 "

Attachment "Attachment 07 - Native PFAS Intermediate M" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 * Printed by Dorothy Love. d. Mon 20 Nov 2023 15:06 FST Page 29 of 87

			633 ass Spec 12.500 min Period 12.500 mi -NRM C System Equilibrate Injection	Acquisition Method
01 Mass (Da) 03 Mass 241.000 177.000	01 Mass (Da) 03 Mass 229.000 84.900	01 Mass (Da) 03 Mass 212.800 168.900	Period 1: Scans in Period: Min. Dwell Time: Relative Start Time: Scheduled Ionization: Experiments in Period: Use target Cycle Time: Target Cycle Time: Target Cycle Time: Scheduled MRM: Period 1 Experiment Scan Type: Scan Mode: Ion Source: sMRM QLQ3 Resolution: MRM detection window: Target Scan Time: Resolution Q1: Resolution Q1: Resolution Q3: Intensity Thres.: Settling Time: MR Pause: MR Pause: MR Pause: MR Pause:	Mass Spectrometer Method Properties
(Da) RT (min) 2.98	(Da) RT (min) 2.94	(Da) RT (min) 2.71	1250 3 ms 250 ms 0.00 msec Off 1 No N/A N/A 1: 1: 1: No N/A Nurbo Spray No 60 sec 0.6000 sec Unit 0.000 msec 5.0070 msec No 0.000 Da	od Properties
Paran DF	Paran DF	Paran DF		
Start -50.00 CE	Start -40,00 Œ	Start -40.00 CE		
Stop ID -50.00 3:3 FICA -12.00-12.00	Stop ID -40.00 PFECA F -25.00-25.00	Stop: ID -40.00 PFBA -14.00-14.00		

01 Mass	Q1 Mass	01 Mass	01 Mass	01 Mass	Q1 Mass	01 Mass
313.000	327.100	295.000	314.800	279.000	298.700	263.000
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)) (Da)
Q3 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)
269.000	307.000	201.000	134.900	85.100	79.900	219.000
Rf (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)
3.55	3.41	3.52	3,84	3.32	3.67	3.21
Paran	Paran	Paran	Paran	Paran	Paran	Paran
Df	D E	DF	DE	Df	DF	DF
Start Stop ID -30.00 -30.00 PFHxA CE -15.00-15.00 Start Stor ID	Start Stop ID -110.00-110.00 4:2-FIS CE -28.00-28.00	Start Stop ID -30.00 -30.00 PFECA B CE -25.00-25.00	Start Stor ID -70.00 -70.00 PFEESA CE -30.00-30.00	Start Stor ID -40.00 -40.00 PRECA A CE -20.00-20.00	Start Stoy ID -90.00 -90.00 PTBS CE -65.00-65.00	Start Stop ID -40.00 -40.00 PEPEA CE -14.00-14.00

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01 Mass 449.000	 Q1 Mass 427.100	01 Mass 341.000	Q1 Mass 376.900		Q1 Mass 398.700	Q1 Mass 363.100	Q1 Mass (Da) 284.900	349.100 79.900 4.00
(Da)	(Da)	(Da)	(Da)		(Da)	(Da)	(Da)	79.90
Q3 Mass (Da) 79.900	03 Mass 407.000	Q3 Mass 237.100	<u>0</u> 3 Mass 250.900		<u>Q</u> 3 Mass 79,900	Q3 Mass 319.000	03 Mass (Da) 168.900	0 4.00
(Da)	(Da)	(Da)	(Da)		(Da)	(Da)	(Da)	DF
RT (min) 4.68	RT (min) 3.98	RT (min) 3.75	RT (min) 3.96		RT (min) 4.30	RT (min) 3.85	RT (min) 3.68	-90.00 CE
Paran DF	Paran DF	Paran DF	Paran DF		Paran DF	Paran Df	Paran DF	-90.00 -70.00
Start -110.00 CE	-90,00 CE	Start -70.00 CE	Start -40.00 CE		Start -100.00 CE	Start -40.00 CE	Start -20.00 CE	0 PEPes 0 -70.00
Start Stor ID -110.00 -110.00 PF CE -90.00 -90.00	Stop ID -90.00 6:2-FIS -35.00-35.00	Stop ID -70.00 5: -20.00-20.00	Stop: ID -40.00 DO -20.00-20.00		Start Stop ID -100.00 -100.00 PF CE -80.00 -80.00	Stop ID -40.00 PE -15.00-15.00	Stop: ID -20.00 旺 -10.00-10.00	
PEHpS 0.00	6:2-FI 5.00	5:3 FICA 0.00	0.00		PFHAS 0.00	PEHpA 5.00	HEPODA	
	N	CA						
1								

01 Mass (Da) 512.900 01 Mass (Da) 548.800 Q1 Mass (Da) 530,800 Q1 Mass (Da) 463.000 Q1 Mass (Da) 498.900 01 Mass (Da) 413.000 Q1 Mass 441.000 (Da) Q3 Mass (Da) 469.000 <u>Q</u>3 Mass (Da) 79.900 Q3 Mass (Da) 419.000 Q3 Mass (Da) 79.900 Q3 Mass (Da) 351.000 Q3 Mass (Da) 316,900 Q3 Mass (Da) 369.000 RT (min) 4.85 RT (min) 5.61 RT (min) 5.72 RT (min) 4.12 RC (min) 4.40 RT (min) 4.43 RC (min) 5.22 Paran DF Paran DE Paran DE Paran DF Paran DF Paran DF Paran DE Start Stop ID -100.00-100.00 CE -110.00 Start Stop ID -90.00 -90.00 CE -110.00 Start Stop ID -60.00 -60.00 PFDA CE -18.00-18.00 Start Stop ID -100.00 -100.00 9C1-PE3CNS CE -38.00 -38.00 Start Stop ID -80.00 -80.00 7:3 FICA CE -20.00-20.00 Start Stop ID -50.00 -50.00 PENA CE -18.00-18.00 Start Stop ID -50.00 -50.00 PFOA CE -16.00-16.00 PENS -110.00 PFOS -110.00

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Attachment "Attachment 11 - Initial Calibration Standard Concentrations" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 33 of 87 1633 "

01 Mass (Da) 613,100	8	Q1 Mass (Da) 630,900	 Q1 Mass (Da) 584.200	Q1 Mass (Da) 563.100	Q1 Mass (Da) 599.000	1	01 Mass (Da) 570,100	01 Mass (Da) 498.100	Q1 Mass (Da) 527.100
Q3 Mass (Da) 569.000		03 Mass (Da) 450.900	Q3 Mass (Da) 419,100	Q3 Mass (Da) 519.000	03 Mass (Da) 79.900		03 Mass (Da) 419.000	Q3 Mass (Da) 77.900	03 Mass (Da) 507.000
FT (min) 5.87		RT (min) 6.49	RT (min) 5.04	RT (min) 5.39	RT (min) 6.16		RT (min) 4.79	RT (min) 6.14	RT (min) 4.58
Paran DF		Param DF	Paran DE	Paran DE	Paran Df		Paran DF	Paran DE	Paran DF
Start Stor ID -60.00 -60.00 PFDDDA CE -20.00-20.00		Start Stop ID -90.00 -90.00 11CL-FF30UdS CE -43.00-43.00	Start Stop ID -90.00 -90.00 NECEOSAA CE -30.00-30.00	Start Stop ID -70.00 -70.00 PFUNDA CE -19.00 -19.00	Start Stop ID -90.00 -90.00 PFDS CE -130.00 -130.00		Start Stop ID -80.00 -80.00 NMAEFOSAA CE -27.00-27.00	Start Stop ID -110.00 -110.00 PFOSA CE -80.00 -80.00	Start Stop ID -100.00 -100.00 8:2-FTS CE -42.00-42.00

Q1 Mass	01 Mas	01 Mas	01 Mass	Q1 Mass	Q1 Mass	Q1 Mass
713.100	663.00	526.00	630.000	699,100	511.900	616.100
Q1 Mass (Da) 713.100	01 Mass (Da) 663.000	01 Mass (Da) 526.000	ss (Da)	ss (Da)	ss (Da)	ss (Da)
Q3 Mass (Da)	Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	03 Mass (Da)	03 Mass (Da)
669.000	619.000	219.000	58.900	79.900	219.000	58.900
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)
6.71	6.3C	7.36	7.24	6.94	7.08	6.94
Paran	Paran	Paran	Paran	Paran	Paran	Paran
D£	DF	De	DF	DF	DF	DF
Start -60.00	-60.00	rr Start Stop ID -90.00 -90.00 NEtPFOSA CF -38.00 -88.00	n Start Stop ID -45.00 -45.00 NEtPFCSAE CE -70.00-70.00	m Start Stop ID -100.00 -100.00 PFDoS CE -150.00 -150.00	rr Start Stop ID -90.00 -90.00 IMAEPEOSA CE -37.00-37.00	#T Start Stop ID -50.00 -50.00 NMEPFOSAE CE -70.00-70.00

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Attachment "Attachment 13 - Bile Salts Working Solution A" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalky/ Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 "
Printed by Dorothy Love. d. Mon 20 Nov 2023 15:06 EST Page 35 of 87

Q1 Mass (398.700	Q1 Mass (363.100	Q1 Mass (284.900	01 Mass (349.100	01 Mass (313.000	01 Mass (327.100	01 Mass (295.000	Q1 Mass (298.700
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
Q3 Mass (98.900	Q3 Mass (169.000	Q3 Mass (184.900	03 Mass (98,900	Q3 Mass (118.900	03 Mass (80.900	03 Mass (84,900	03 Mass (98.800
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
RT (min) 4.30	RT (min) 3.85	RT (min) 3.68	RT (min) 4.0C	PCr (min) 3.55	RT (min) 3.41	RT (min) 3.52	RT (min) 3.66
Paran Df	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF	Paran Df
Start Stop ID -90.00 -90.00 FFHAS 2	Start Stop ID -50.00 -50.00 PFHpA_2 CE -25.00-25.00	Start Stop ID -75.00 -75.00 HEPODA 2 CE -10.00-10.00	Start Stop ID -110.00 -110.00 PFPeS 2 CE -40.00 -40.00	Start Stop ID -40.00 -40.00 PFHxA 2 CE -31.00-31.00	Start Stop ID -100.00 -100.00 4:2 FTS_2 CE -50.00-50.00	Start Stop ID -10.00 -10.00 PFECA B_2 CE -35.00-35.00	Start Stor ID -110.00 -110.00 PFBS_2 CE -35.00-35.00

Q1 Mass (Da) 548,800 Q1 Mass (Da) 463.000 Q1 Mass (Da) 498.900 Q1 Mass (Da) 413.000 Q1 Mass (Da) 449.000 Q1 Mass (Da) 427.100 Q1 Mass (Da) 341.000 Q3 Mass (Da) 98,800 Q3 Mass (Da) 219.000 Q3 Mass (Da) 98.800 Q3 Mass (Da) 169.000 Q3 Mass (Da) 98.800 Q3 Mass (Da) 217.000 <u>Q</u>3 Mass (Da) 80.900 RT (min) 4.43 RT (min) 4.12 RT (min) 4.68 RT (min) 3.75 RT (min) 5.72 RT (min) 5.21 RT (min) 3.98 А Paran Start Stop ID DF -100.00-100.00 PFNS_2 CE -90.00-90.00 Paran DE Paran D€ Paran DF Paran DF Paran DE Paran DE -70.00 -70.00 Start Stop ID -50.00 -50.00 PENA_2 CE -30.00-30.00 Start Stop ID -110.00 -110.00 PFCS_2 CE -80.00 -80.00 Start Stop ID -100.00-100.00 PFHps_2 CE -70.00-70.00 Start Stop ID -50.00 -50.00 PROM_2 CE -26.00-26.00 Start Stop ID -80.00 -80.00 5:3 FTCA_2 CE -20.00-20.00 Start Stop ID -120.00-120.00 6:2 FTS_2 CE -70.00-70.00

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Attachment *Attachment 15 - PFAS Linear Branched Mix A* to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 * Printed by Dorothy Love. d. Mon 20 Nov 2023 15:06 FST Page 37 of 87

Q1 Mass (Da)	Q1 Mass	Q1 Mass	Q1 Mass	01 Mass	Q1 Mass	01 Mass	01 Mass
	613.100	584.200	563.100	599.000	570.100	527.100	512.900
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
Q3 Mass (Da)	03 Mass ()	03 Mass ()	03 Mass (1	Q3 Mass ()	03 Mass (1	Q3 Mass (1	03 Mass (1
	319.000	526.000	269.100	98.800	483,000	80_800	219.000
(B)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)
	5.87	5.04	5.39	6.16	4.79	4.58	4.85
Paran	Paran	Paran	Paran	Paran	Paran	Paran	Paran
	DF	DF	DF	De	Df	Df	D£
Start	Start -60.00	Start -100.00	Start -70.00 CE	Start -100.00 CE	Start -90.00	Start -100.00 CE	Start -60.00 CE
Stop ID	Stor ID	Start Stop ID	Stop ID	Start Stop ID	Stop ID	Start Stop ID	Stop ID
	-60.00 PEDODA 2	-100.00 -100.00 NECEOSAA 2	-70.00 PFUNDA 2	-100.00 -100.00 PFDS_2	-90.00 NMEROSAA 2	-100.00 -100.00 8:2 FTS_2	-60.00 PEDA_2
	-38.00-38.00	CE -30.00-30.00	-35.00-35.00	CE -100.00 -100.00	-24.00 -24.00	CE -90.00-90.00	-31.00 -31.00

Start Stop ID -100.00 -100.00 9C1-PF3QNS 2 CE -38.00-38.00	Paran Df	RT (min) 5.6C	s (Da) C	Q3 Mass (Da) 353.000	(Da)	Q1 Mass 532,800
Start Stop ID -40.00 -40.00 DOWN_2 CE -40.00-40.00	Paran DF	RT (min) 3.95	s (Da)	Q3 Mass 84.800	(Da)	<u>0</u> 1 Mass 376.900
Start Stop ID -90.00 -90.00 NECPFOSA_2 CE -40.00 -40.00	Paran DF	RT (min) 7.35	ic (Da)	03 Mass 169.000	(Da)	01 Mass 526.000
Start Stop ID -110.00 -110.00 NMEPFCSA_2 CE -37.00-37.00	Paran DF	RT (min) 7.08	35 (Da)	Q3 Mass 169.000	(Da)	Q1 Mass 511.900
Start Stop ID -130.00 -130.00 PFOSA_2 CE -35.00 -35.00	Paran DF	RT (min) 6.13	03 Mass (Da) 478.000	03 Mass 478.000	(Da)	Q1 Mass 498.100
Start Stop ID -40.00 -40.00 PFPeA_2 CE -55.00-55.00	Paran DF	RT (min) 3.21) J	Q3 Mass (Da) 68.900	(Da)	Q1 Mass 263.000
Start Stor ID -60.00 -60.00 PFTEDA_2 CE -40.00-40.00	Paran DE	RT (min) 6.71)0 (Da)	03 Mass (Da) 168,900	(Da)	01 Mass 713.100
0 PETRDA 2 0 -40.00	0 -60.00 -40.00	-60.00	Я.	663.000 168.900 6.30	168.9	663.000

Attachment "Attachment 17 - Linear Branched Bile Salts Solution" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 * Page 39 of 87 Printed by Dorothy Love. d. Mon 20 Nov 2023 15:06 EST

 01 Mass (1 216.800	Q1 Mass (1 216.000	Q1 Mass (I 699,100	×	01 Mass (I 314.800	•	Q1 Mass (1 441.000	Q1 Mass (I 241.000	01 Mass (I 632.900	2
(Da)	(Da)	(Da)		(Da)		(Da)	(Da)	(Da)	
Q3 Mass (Da) 171.900	03 Mass (Da) 172.000	Q3 Mass (Da) 98.800		Q3 Mass (Da) 82.900		<u>Q</u> 3 Mass (Da) 336.900	<u>Q</u> 3 Mass (Da) 117.000	Q3 Mass (Da) 452.900	
RT (min) 2.71	RT (min) 2.71	RT (min) 6.94		RT (min) 3.83		RT (min) 4.4C	RT (min) 2.98	RT (min) 6.48	
Paran DF	Paran DF	Paran DF		Paran Df		Paran DF	Paran DF	Paran DF	
Start -40.00 CE	Start -40.00	Start -90.00 CE		Start -70.00 CE		Start -80.00 CF	Start -50.00 OE	Start -90,00 CE	
Stor ID -40.00 13C4-PFBA -14.00-14.00	Stop ID -40.00 13C3-PFBA -14.00 -14.00	Stop: ID -90.00 PFDos:2 -140.00 -140.00		Stop ID -70.00 PFEESA_2 -30.00-30.00		Stop ID -80.00 7:3 FTCA 2 -20.00 -20.00	Stop ID -50.00 3:3 FICA_2 -45.00-45.00	Stop ID -90.00 11C1-PF300ds_2 -40.00 -40.00	

Q1 Mass (Da) 402.100 Q1 Mass (Da) 367.100 Q1 Mass (Da) 286.900 Q1 Mass (Da) 318.000 Q1 Mass (Da) 315.100 Q1 Mass (Da) 329.100 01 Mass (Da) 302.100 01 Mass (Da) 268.300 Q3 Mass (Da) 168.900 Q3 Mass (Da) 79.900 03 Mass (Da) 322.000 Q3 Mass (Da) 273.000 <u>Q</u>3 Mass (Da) 270.000 03 Mass (Da) 80.900 Q3 Mass (Da) 79,900 Q3 Mass 223.000 (Da) RT (min) Param Start Stop ID 4.3C DF -100.00-100.00 13C3-PFHbcS RT (min) 3.85 RT (min) 3.68 RT (min) 3.54 RT (min) 3.54 RT (min) 3.41 RT (min) 3.66 RT (min) 3.21 Paran DF Paran DF Paran DE Paran DE Paran DF Paran DF Ракап DF Start Stop ID -30.00 -30.00 13C5 CE -15.00-15.00 Start Stop ID -30.00 -30.00 13C2 CE -15.00-15.00 Start Stop ID -40.00 -40.00 13C4-PFHpA CE -15.00-15.00 Start Stop ID -20.00 -20.00 13C3-HEPODA CE -10.00-10.00 Start Stop ID -100.00 -100.00 13C2-4:2-FTS CE -28.00 -28.00 Start Stop ID -90.00 -90.00 130 CE -65.00 -65.00 A Start Stop ID -40.00 -40.00 130 CE -14.00-14.00 -80.00-80.00 1302-PETHAA 13C3-PFBS 13C5-PFPeA 13C5-PFHkA

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Q1 Mass (Da)	01 Mass (Da)	Q1 Mass (Da)	Q1 Mass (Da)	01 Mass (Da)	01 Mass (Da)	01 Mass (Da)
403.000	429.100	417.100	421.100	502.800	507.100	472.100
03 Mass (Da)	03 Mass (Da)	03 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)
83.900	80,900	172.000	376.000	79,900	79.900	427.000
RT (min)	PT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)
4.3C	3.97	4.12	4.12	5.21	5.21	4.42
Paran	Paran	Paran	Paran	Paran	Paran	Paran
DF	DF	DF	DF	D€	DF	DF
Start Stop ID	Start Stor ID	Start Stop ID	Start Stop ID	Start Stop ID	Start Stor ID	Start Stop ID
-100.00 -100.00 1802-PFHxS	-90.00 -90.00 13C2-6:2-FTS	-50.00 -50.00 13C4-PFOA	-50.00 -50.00 13C8-PFOA	-90.00 -90.00 13C4-PECS	-90.00 -90.00 1308-PFCS	-50,00 -50.00 13C9-PENA
CE -80.00 -80.00	CE -35.00-35.00	CE -16.00-16.00	CE -16.00-16.00	CE -110.00 -110.00	CE -110.00 -110.00	CE -18.00-18.00

Q1 Mass 570.000 Q1 Mass	Q1 Mass 573.200	Q1 Mass 506.100	01 Mass 529.100	01 Mass 515.100	Q1 Mass 519,100	Q1 Mass 468.000
(Da)	s (Da)	o (Da)	s (Da)	s (Da)	0 (Da)	o (Da)
Q3 Mass (Da) 525.100 Q3 Mass (Da)	Q3 Mass (Da) 419.000	Q3 Mass (Da) 77.800	Q3 Mass (Da) 80.900	Q3 Mass (Da) 470.100	Q3 Mass (Da) 474.100	<u>0</u> 3 Mass (Da) 423.000
RT (min)	PCT (min) 4.78	RT (min) 6.13	RT (min) 4.58	RT (min) 4.85	RT (min) 4.85	RT (min) 4.42
Paran DF Paran	Paran DE	Paran DF	Param DF	Paran DF	Paran DF	Paran Df
r Start Stop ID -70.00 -70.00 13C7-PEUNDA CE -19.00-19.00 Start Stop ID	r Start Stop ID -90.00 -90.00 d3-NMEFOSAA CE -27.00-27.00	r Start Stop ID -110.00 -110.00 13C8-PFCSA CE -80.00-80.00	r Start Stop ID -100.00 -100.00 13C2-8:2-FTS CE -42.00-42.00	r Start Stop ID -60.00 -60.00 13C2-PFDA CE -18.00-18.00	r Start Stop ID -60.00 -60.00 13C6-PFDA CE -18.00-18.00	r Start Stop ID -50.00 -50.00 13C5-PENNA CE -18.00-18.00

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Q1 Mass (Da) 286.900 Q1 Mass (Da)	01 Mass (Da) 529.100	01 Mass (Da) 429.100	Q1 Mass (Da) 329,100	Q1 Mass (Da) 507.100	Q1 Mass (Da) 402.100	01 Mass (Da) 302.100
Q3 Mass (Da) 184.900 Q3 Mass (Da)	03 Mass (Da) 509.000	03 Mass (Da) 409.000	03 Mass (Da) 309.000	Q3 Mass (Da) 96,900	03 Mass (Da) 98.800	03 Mass (Da) 98,900
RT (min) 3.68 RT (min)	RT (min) 4.58	RT (min) 3,97	RT (min) 3.41	RT (min) 5.20	RT (min) 4.29	RT (min) 3.66
Paran DF Paran	Paran Df	Paran DF	Paran DF	Paran D£	Paran DF	Paran DF
Start Stop ID -75.00 -75.00 13C3-HEFODA_2 CE -10.00-10.00 Start Stop ID	Start Stop ID -100.00 1302-8:2-FTS_2 CE -42.00-42.00	Start Stop ID -100.00 1302-6:2-FTS_2 CE -35.00-35.00	Start Stop ID -100.00 102-4:2-FTS_2 CE -28.00-28.00	Start Stor ID -110.00 -110.00 13C8-PFOS_2 CE -80.00 -80.00	Start Stop ID -90.00 -90.00 13C3-PFHAS_2 CE -70.00-70.00	Start Stop ID -110.00 -110.00 13C3-PFES_2 CE -35.00-35.00

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	Paramet CUR: IS: IS: IS: IS: IS: IS: IS: IS: IS: IS	Q1 Mass 498.200	Q1 Mass (Da) 498.100	01 Mass (Da) 498.000	Q1 Mass (Da) 502,800	313.100 119.400 3.54
	er Tab	(Da)	(Da)			1.
	Parameter Table (Period 1 CUR: 35.00 IS: -3000.00 IS: 450.00 TEM: 40.00 GS1: 40.00 GS2: 50.00 CAD: 10.00 EF -10.00 CXF -14.00	Q3 Mass (80.000	03 Mass (80.000	03 Mass (80.000	03 Mass (Da) 98,900	ین د ا
		(Da)	(Da)	(Da)		ĥ
	Experiment	RT (min) 3.39	RT (min) 3.70	RT (min) 3.78	RT (min) 5.21	Ĥ
	1):	Paran DF	Paran DF	Paran DF	Paran DF	-31.00
		Start Stop ID -100.00 -100.00 CE -100.00	Start Stop ID -100.00 -100.00 CE -100.00	Start Stop ID -100.00 -100.00 CE -100.00	Start Stop ID -110.00 -110.00 13 CE -80.00 -80.00	0 -31.00
		-100.00	-100.00	-100.00	13C4-PFOS_2	

	C System Equilibrate Injection	ass Spec 12,500 min Period 12,500 min -VRM ntegrated Valve	633	Acquisition Method

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Attachment " to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 " Printed by Dorothy Love d. Mon 20 Nov 2023 15:06 EST

1 Mass (Da) 03 Mass (Da) 12.800 168.900	esolution Ql: esolution Q3: ntensity Thres.: ettling Time: R Pause: CA: tep Size:	e: d MRM: : : : : : : : : : : : : : : : : : :	eriod 1: cans in Period: in. Dwell Time: ax. Dwell Time: elative Start Time: checkuled Ionization: xperiments in Period: se target Cycle Time: arget Cycle Time: eriod 1 Exceriment 1:	reated: ast Modified; amment: ynchronization Mode: uto-Equilibration: cquisition Duration: unber Of Scans: eriods In File: cquisition Module: oftware version	Acquisition Information: cquisition Method:
RT (min) 2.71		X			
Paran DF					
Start Stop ID -40.00 -40.00 PFBA CE -14.00 -14.00	Unit Unit 0.00 cps 0.0000 msec 5.0070 msec No 0.00 Da	MRM (MRM) Yes Negative N/A Turbo Spray No 60 sec 0.6000 sec	1250 3 ms 250 ms 0.00 msec Off 1 No N/A	Wechessday May 24 2023 14:48:39 FM Wechessday June 14 2023 11:54:50 AM LC Sync Off 12min30sec 1250 1 Acquisition Method Analyst 1.7.3	1633.dam

S									
	1 Mass (Da) 27.100	1 Mass (Da) 95.000	1 Mass (Da) 14.800	1 Mass (Da) 79.000	1 Mass (Da) 98.700	1 Mass (Da) 63.000	1 Mass (Da) 41.000	1 Mass (Da) 29,000	
	03 Mass (Da) 307.000	03 Mass (Da) 201.000	Q3 Mass (Da) 1.34.900	03 Mass (Da) 85.100	Q3 Mass (Da) 79,900	Q3 Mass (Da) 219.000	Q3 Mass (Da) 177.000	03 Mass (Da) 84,900	
4 5 1) RT (min) 3.41	87 (mi.n) 3.52) RT (min) 3.84) RT (min) 3.32) RT (min) 3.67) RT (min) 3.21) RT (min) 2.98) RT (min) 2.94	
2	Paran DF	Paran DF		Paran DF		Paran DF	Paran DF	Paran DF	

	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)
	41.000	76.900	98.700	63.100	84.900	49.100	13.000
Attachment "" to "	Q3 Mass (Da)	03 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	<u>0</u> 3 Mass (Da)	03 Mass (Da)
Printed by Doroth	237.100	250.900	79.900	319.000	168.900	79.900	269.000
'US Eurofins I	RT (min)	F47 (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)
IV Love. d. Ma	3.75	3.96	4.30	3.85	3.68	4.00	3.55
Cleveland	Paran	Paran	Paran	Paran	Paran	Paran	Paran
20 Nov	Df	DE	D F	DF	Df	DF	Df
Attachment ^{***} to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalky Printed by Dorothy Love. d. Mon 20 Nov 2023 15:06 EST	Start Stop ID -70.00 -70.00 5: CE -20.00-20.00	Start Stop ID -40.00 -40.00 DC CE -20.00 -20.00	Start Stop ID -100.00 -100.00 PE CE -80.00 -80.00	Start Stop ID -40.00 -40.00 PE CE -15.00 -15.00	Start Stop ID -20.00 -20.00 HE CE -10.00-10.00	Start Stop ID -90.00 -90.00 - PE CE -70.00 -70.00	Start Stop ID -30.00 -30.00 PF CE -15.00-15.00
nd Polyfluoroalky	ID 5:3 FTCA -20.00	ID DONA 20.00	D PETHAS -80.00	ID REHDA -15.00	D HEPODA 10+00	ID PFTeS -70.00	D PFłbcA 15.00

1 Mass (Da) 48.800	1 Mass (Da) 30.800	1 Mass (Da) 41.000	1 Mass (Da) 63.000	1 Mass (Da) 98.900	1 Mass (Da) 13.000	1 Mass (Da) 49.000	1 Mass (Da) 27.100	
Q3 Mass (Da) 79,900	Q3 Mass (Da) 351.000	03 Mass (Da) 316.900	03 Mass (Da) 419.000	Q3 Mass (Da) 79.900	Q3 Mass (Da) 369,000	Q3 Mass (Da) 79,900	03 Mass (Da) 407,000	
RT (min) 5.72	RT (min) 5.61	RT (min) 4.40	RT (min) 4.43	RC" (min) 5.22	RT (min) 4.12	RT (min) 4.68	RT (min) 3.98	
Parar Start Stor ID DF -100.00-100.00 PENS								

Attachment "" to "IS Funding Claveland - Analysis of Der and Dologunnally Cubetrance (DEAC) is Associate but to 10 Monate Livin Bure to the 1 and a	- Analysis of Bor and Dolya inmalled Substances (DEAS)	Cleveland	1 X Fimfine	Attachment *** to .	3
	Start Stor ID -90.00 -90.00 NEtEOSAA CE -30.00 -30.00	Paran DF	RT (min) 5.04	Q3 Mass (Da) 419,100	1 Mass (Da) 84.200
	Start Stop ID -70.00 -70.00 PEUNDA CE -19.00-19.00	Paran DF	RT (min) 5.39	Q3 Mass (Da) 519.000	1 Mass (Da) 63.100
	Start Stop ID -90.00 -90.00 PEDS CE -130.00 -130.00	Paran DF	RT (min) 6.16	<u>0</u> 3 Mass (Da) 79,900	1 Mass (Da) 99.000
	Start Stor ID -80.00 -80.00 NMEEOSPA CE -27.00 -27.00	Paran DF	RT (min) 4.79	Q3 Mass (Da) 419,000	1 Mass (Da) 70.100
	Start Stor ID -110.00 -110.00 PFCSA CE -80.00 -80.00	Paran DF	RT (min) 6.14	Q3 Mass (Da) 77.900	1 Mass (Da) 98.100
	Start Stop ID -100.00 -100.00 8:2-FTS CE -42.00 -42.00	Paran	RT (min) 4.58	03 Mass (Da) 507.000	1 Mass (Da) 27.100
	Start Stop ID -60.00 -60.00 PEDA CE -18.00-18.00	Paran Df	RT (min) 4.85	03 Mass (Da) 469.000	1 Mass (Da) 12.900
	-110.00 -110.00	-110.00	A		

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1 Mass (Da) 26_000	1 Mass 30.000	1 Mass 99.100	1 Mass 11.900	1 Mass 16.100	1 Mass 13.100	1 Mass 30,900
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
Q3 Mass (Da) 219.000 Q3 Mass (Da)	03 Mass 58.900	Q3 Mass (Da) 79,900	Q3 Mass (Da) 219.000	Q3 Mass 58,900	Q3 Mass (Da) 569,000	03 Mass (Da) 450,900
	(Da)			(Da)		
RT (min) 7.36 RT (min)	RT (min) 7.24	PT (min) 6.94	RT (min) 7.08	PCT (min) 6.94	RT (min) 5.87	RT (min) 6.49
Paran DF Paran	Paran DF	Paran Df	Paran DF	Paran DF	Paran DF	Paran Df
Start Stop ID -90.00 -90.00 NEtPROSA CE -38.00 -38.00 Start Stop ID	Start Stop ID -45.00 -45.00 NELEFOSAE CE -70.00-70.00	- Start Stop ID -100.00 PFDoS CE -150.00 -150.00	Start Stop ID -90.00 -90.00 NMAPFOSA CE -37.00-37.00	- Start Stop ID -50.00 -50.00 NMEPEOSAE CE -70.00 -70.00	r Start Stop ID -60.00 PEDODA CE -20.00-20.00	- Start Stop ID -90.00 -90.00 11C1-FF30UdS CE -43.00-43.00

Start Stop ID -75.00 -75.00 HEFODA_2 CE -10.00-10.00	i) DF	RT (min) 3.68	Q3 Mass (Da) 184.900	1 Mass (Da) (84.900 1
Start Stop ID -110.00-110.00 PFRes 2 CE -40.00-40.00	i) Paran DF	RT (min) 4.00	03 Mass (Da) 98,900	1 Mass (Da) (49.100
Start Stop ID -40.00 -40.00 PEHAA_2 CE -31.00 -31.00	i) Paran DF	RT (min) 3.55	Q3 Mass (Da) 118,900	1 Mass (Da) (13.000]
Start Stop ID -100.00 -100.00 4:2 FTS 2 CE -50.00 -50.00	i) Paran DF	RT (mirs) 3,41	Q3 Mass (Da) 80,900	1 Mass (Da) (27.100 8
Start Stop ID -10.00 -10.00 Preca B_2 CE -35.00 -35.00) Paran DF	RT (min) 3.52	03 Mass (Da) 84,900	1 Mass (Da) 95.000
Start Stop ID -110.00 -110.00 PFBS_2 CE -35.00 -35.00) Paran D£	RT (min) 3.66	Q3 Mass (Da) 96.800	1 Mass (Da) 98.700 9
Start Stop ID -60.00 -60.00 PFTeDA CE -22.00 -22.00) Paran DF	RT (min) 6.71	Q3 Mass (Da) 669.000	1 Mass (Da) 6 13.100 6
00 PFILDA 00 -21.00	00 -60.00 -21.00	-60.00 Œ	6.30 DF	63.000 619.000 6.30

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1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	
98,900	13,000	49.000	27,100	41.000	98.700	63.100	
Q3 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	03 Mass (Da)	
98.800	169,000	98.800	80.900	217,000	98,900	169.000	
(Da) RT (min) 5.21	RT (min) 4,12	RT (min) 4.68	RT (min) 3.98	RT (mins) 3.75	4.30 PT (min)) RT (min) 3.85	
Paran	Paran	Paran	Paran	Paran	Paran	Paran	
DF	Df	Df	DF	DF	DF	DF	
ram Start Stop ID -110.00-110.00 PROS_2 CE -80.00-80.00 CE -80.00-80.00	ran Start Stor ID -50.00 -50.00 PFCA_2 CE -26.00-26.00	uran Start Stop ID -100.00 -100.00 PFHpS_2 CE -70.00 -70.00	uran Start Stop ID -120.00 -120.00 6:2 FTS_2 CE -70.00 -70.00	uran Start Stop ID -80.00 5:3 FTCA_2 CE -20.00-20.00	uran Start Stop ID -90.00 -90.00 PFHbcs_2 CE -70.00-70.00	uran Start Stop ID -50.00 -50.00 PEHpA_2 CE -25.00-25.00	

Attachment " to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 * Printed by Dorothy Love d. Mon 20 Nov 2023 15:06 FST	d - Analysis of Per and Polyfluoroal v 2023 15:06 FST	Cleveland	"US Eurofins 1V Love. d. N	Attachment " to Printed by Dorott	
A 2	Start Stor ID -100.00-100.00 NEtFOSAA_2 CE -30.00-30.00	Paran DF	RT (min) 5.04	03 Mass (Da) 526,000	1 Mass (Da) 84.200
2	Start Stop ID -70.00 -70.00 PEUnDA_2 CE -35.00-35.00	Paran D f	RT (min) 5.39	Q3 Mass (Da) 269,100	1 Mass (Da) 63.100
	Start Stop ID -100.00 -100.00 PEDS_2 CE -100.00 -100.00	Paran DF	RT (min) 6.16	Q3 Mass (Da) 98.800	1 Mass (Da) 99.000
A_2	Start Stop ID -90.00 -90.00 NMeFOSAA_2 CE -24.00-24.00	Paran DF	RT (min) 4.79	Q3 Mass (Da) 483.000	1 Mass (Da) 70.100
	Start Stop ID -100.00 -100.00 8:2 FTS_2 CE -90.00-90.00	Paran DF	RT (min) 4.58	Q3 Mass (Da) 80.800	1 Mass (Da) 27.100
	Start Stop ID -60.00 -60.00 PEDA 2 CE -31.00-31.00	Paran DF	RT (min) 4.85	03 Mass (Da) 219.000	1 Mass (Da) 12.900

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1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 48.800 98.800 5.72 DF -100.00-100.00 PFNS_2 CE -90.00-90.00 1 Mass (Da) Q3 Mass (Da) 63.000 219.000

RT (min) 4.43

Paran DF

Start Stop ID -50.00 -50.00 PENA_2 OE -30.00 -30.00

1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)
26,000	11.900	98.100	63.000	13.100	63.000	13.100
Q3 Mass	Q3 Mass	<u>0</u> 3 Mass	03 Mass	<u>0</u> 3 Mass	Q3 Mass	Q3 Mass
169,000	169,000	478.000	68,900	168.900	168.900	319,000
(Da) RT (min)	(Da) RT (min)	(Da) RT (min)	(Da) RT (min)	(Da) RT (min)	(Da) RT (min)	(Da) RT (min)
7.35	7.08	6.13	3.21	6.71	6.30	5.87
Paran DF	Paran DF	Paran DE	DF	Param	Paran DE) Paran Df
Start Stop ID	Start Stop ID	Start Stop ID	Start Stor ID	Start Stor ID	Start Stop ID	Start Stop ID
-90.00 -90.00 NEtPFOSA_2	-110.00 -110.00 NMEPFOSA 2	-130.00 -130.00 PROSA_2	-40.00 -40.00 PETEA 2	-60.00 -60.00 PFTeDA_2	-60.00 -60.00 PETrDA_2	-60.00 -60.00 PEDODA_2
CE -40.00-40.00	CF -37.00 -37.00	CE -35.00 -35.00	CE -55.00 -55.00	CE -40.00-40.00	CE -40.00 -40.00	CE -38.00-38.00

Attachment ^{***} to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 * Printed by Dorothy Love rd Mon 20 Nov 2023 15:06 EST	1 Mass (Da) Q3 Mass (Da) RT (min) Paran Start Stop ID 16.000 172.000 2.71 DF -40.00 -40.00 13C3-PFBA	1 Mass (Da) Q3 Mass (Da) RT (min) Paran Start Stop ID 99.100 98.800 6.94 DF -90.00 -90.00 PFDoS_2 CE -140.00 -140.00	1 Mass (Da) Q3 Mass (Da) RT (min) Param Start Stop ID 14.800 82.900 3.83 DF -70.00 -70.00 PRESA_2 CE -30.00-30.00	1 Mass (Da) Q3 Mass (Da) RT (min) Farar Start Stop ID 41.000 336.900 4.40 DF -80.00 -7:3 FTCA_2 CE -20.00-20.00	1 Mass (Da) Q3 Mass (Da) RT (min) Param Start Stop ID 41.000 117.000 2.98 DF -50.0C -50.00 3:3 FTCA_2 CE -45.00-45.00	1 Mass (Da) Q3 Mass (Da) RT (min) Pararr Start Stop ID 32.900 452.900 6.48 DF -90.00 -90.00 11Cl-PF300dS_2 CE -40.00-40.00	1 Mass (Da) Q3 Mass (Da) RT (min) Faran Start Stop ID 32.800 353.000 5.60 DF -100.00 -100.00 9C1-PF3CNS_2 CE -38.00-38.00	1 Mass (Da) Q3 Mass (Da) FCT (min) Parar Start Stop ID 76.900 84.800 3.95 DF -40.00 -40.00 DOWA_2 CE -40.00 -40.00	
Ising Draft Method 1633 * Page 57 of 87									

1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	
86.900	18.000	15.100	29,100	02,100	68.300	16,800	
Q3 Mass (Da)	03 Mass (Da)	03 Mass (Da)	03 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	
168.900	273,000	270.000	80.900	79,900	223.000	171.900	
RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	A
3.68	3.54	3,54	3.41	3.66	3.21	2.71	
Paran	Paran	Paran	Paran	Paran	Paran	Paran	~14.00
Df	DF	DF	Df	Df	DF	DF	
Start Stop ID	Start Stop ID	Start Stop ID	Start Stor ID	Start Stop ID	Start Stop ID	Start Stop ID	0 -14.00
-20.00 -20.00 13C3-HFFODA	-30.00 -30.00 13C5-PFHbA	-30.00 -30.00 13C2-PFHxA	-100.00 -100.00 13C2-4:2-FIS	-90.00 -90.00 13C3-PEBS	-40.00 -40.00 13C5-PFPeA	-40.00 -40.00 13C4-PEBA	
CE -10.00-10.00	CE -15.00-15.00	CE -15.00-15.00	CE -28.00-28.00	CE -65.00 -65.00	CE -14.00 -14.00	CE -14.00-14.00	

4

Attachment "" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalky! Substance	s of Per and	d - Analysi	Cievelan	US Eurofins	ment to	Attach		
	Stop: ID	Start	Paran	RT (min)	lass (Da)	Q3 Mass	(Da)	1 Mass
13C4-PEOS 110,00	Stop ID 90.00 110.00	Start -90.00 CE	Paran DF	RT (min) 5.21	bass (Da) 100	Q3 Mass 79.900	(Da)	1 Mass 02.800
1308-PF0A 1.00	Stor ID -50.00 13 -16.00 -16.00	Start -50,00 CE	Param DF	RT (min) 4.12	bass (Da) 000	03 Mass 376.000	(Da)	1 Mass 21.100
13C4-PFOA 00	Stop ID -50.00 13 -16.00 -16.00	Start -50.00 CE	Paran DE	RT (min) 4,12	脑 SS (Da) 000	03 Mass 172.000	(Da)	1 Mass 17.100
1302-6:2-FTS	Stop: ID -90.00 13 -35.00 -35.00	Start -90.00 CE	Paran D£	RT (min) 3.97	bass (Da) 00	03 Mass 80.900	(Da)	1 Mass 29.100
1802-PFHXS	Start Stop ID -100.00 -100.00 18 CE -80.00 -80.00	Start 100.00	Paran D£	RT (min) 4.30	ADC ADC	03 Mass 83,900	(Da)	1 Mass 03.000
13C3-PFHxS	Start Stop ID -100.00 -100.00 13 CE -80.00 -80.00	Start -100.00 CE	Paran DF	RT (min) 4.30	00 00	03 Mass 79.900	(Da)	1 Mass 02.100
13C4-PFHpA 00	Stop: ID -40.00 13 -15.00-15.00	Start -40.00	Paran DE	RT (min) 3.85	ass (Da) 000	Q3 Mass 322.000	(Da)	1 Mass 67,100

	1 Mass (Da) 73.200	1 Mass (Da) 06,100	1 Mass (Da) 29,100	1 Mass (Da) 15.100	1 Mass (Da) 19.100	1 Mass (Da) 68.000	1 Mass (Da) 72,100	07,100 79,900
	03 Mass 419.000	03 Mass 77.800	03 Mass 80.,900	Q3 Mass 470.100	Q3 Mass 474.100	a) Q3 Mass (Da) 423.000	Q3 Mass 427.000	900 5.21
	(Da)	(Da)	(Da)	(Da)	(Da)		(Da)	뭐
1	RT (min) 4.78	RT (min) 6.13	RT (min) 4.58	RT (min) 4.85	RT (min) 4.85	RT (min) 4.42	RT (min) 4,42	-90,00
	Paran DF	Paran DF	Paran DE	Paran DF	Paran DF	Paran DF	Paran Df	
	Start Stop ID -90.00 -90.00 d3-NMAFOSAA CE -27.00-27.00	Start Stop ID -110.00 -110.00 13C8-PFOSA CE -80.00 -80.00	Start Stop ID -100.00-100.00 13C2-8:2-FTS CE -42.00-42.00	C Start Stor ID -60.00 -60.00 13C2-PFDA CE -18.00-18.00	r Start Stop ID -60.00 -60.00 13C6-PFDA CE -18.00-18.00	r Start Stop ID -50.00 -50.00 13C5-PENA CE -18.00-18.00	r Start Stop ID -50.00 -50.00 13C9-PENA CE -18.00-18.00	-90.00 13C3-PFCS -110.00 -110.00
2								
						1.410		

1 Mass 31.100	1 Mass 39,200	1 Mass 15.000	1 Mass 23.200	1 Mass 15.100	1 Mass 89.200		1 Mass 70.000	
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)		(Da)	
<u>Q</u> 3 Mass 219.000	03 Mass 58,900	03 Mass 219.000	03 Mass 58.900	<u>Q</u> 3 Mass 570.000	03 Mass 419.000		Q3 Mass 525.100	
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)		(Da)	
RT (min) 7.34	RT (min) 7.21	RT (min) 7.07	RT (min) 6.92	RT (min) 5.86	RT (min) 5.02		RT (min) 5.38	
Paran DF	Paran DF	Param DF	Paran DF	Paran Df	Paran DF		Paraw DE	
Start -90.00 CE	Start -45,00 Œ	Start -90.00 CE	Start -50,00 CE	Start -60.00 CE	Start -90.00 CE	ŕ	Start -70.00	
Stop ID -90.00 d5 -38.00 -38.00	Stop: ID -45.00 d9 -70.00-70.00	Stop: ID -90,00 d3 -37,00 -37,00	Stop: ID -50.00 d7 -70.00 -70.00	Stop ID -60.00 13 -20.00-20.00	Stop ID -90.00 d5 -30.00-30.00	1000	Stop ID -70.00 13	
d5-NELPFOSA 3.00	d 9 NELPFOSAE).00	d3-NMEPEOSA 7.00	d7-NMEPFOSAE),00	13C2-PFDoDA 1.00	d5-NELFOSAA).00		13C7-PFUnDA	

Printed by Dorothy Love. d. Mon 20 Nov 2023 15:06 EST FAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 *

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Q3 Mass (Da) RT (min) Param Start Stor DE Q3 Mass (Da) RT (min) DE -40.00 -40.00 -22.00-22 Q3 Mass (Da) RT (min) DE -40.00 -40.00 -22.00-22 Q3 Mass (Da) RT (min) DE -40.00 -40.00 -31.00-31 Q3 Mass (Da) RT (min) Param Start Stor 1D Q3 Mass (Da) RT (min) Param Start Stor -35.00-35 Q3 Mass (Da) RT (min) Param Start Stor 1D 98.900 (Da) StT (min) Param Start Stor 1D Q3 Mass (Da) RT (min) Param Start Stor 1D Q3 Mass (Da) RT (min) Param Start Stor 1D Q3 Mass (Da) 3.97 Dr -28.00-28 -28.00-30 Q3 Mass (Da) <th>Q3 Mass (Da) Per (min) Perame DE Start Stop ID Q3 Mass (Da) RT (min) DE -60.00 -60.00 -22.00 -23.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -35.00 -35.00 -35.00 -35.00 -35.00 -35.00 -35.00 -35.00 -30.00 -100.00 -100.00 -100.00 -100.00 -100.00 -100.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00</th> <th>1 Mass (Da) 29,100</th> <th>1 Mass (Da) 29,100</th> <th>1 Mass (Da) 29.100</th> <th>1 Mass (Da) 07,100</th> <th>1 Mass (Da) 02.100</th> <th>1 Mass (Da) 02.100</th> <th>1 Mass 18.000</th> <th>1 Mass (Da) 15.200</th>	Q3 Mass (Da) Per (min) Perame DE Start Stop ID Q3 Mass (Da) RT (min) DE -60.00 -60.00 -22.00 -23.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -33.00 -35.00 -35.00 -35.00 -35.00 -35.00 -35.00 -35.00 -35.00 -30.00 -100.00 -100.00 -100.00 -100.00 -100.00 -100.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00 -20.00	1 Mass (Da) 29,100	1 Mass (Da) 29,100	1 Mass (Da) 29.100	1 Mass (Da) 07,100	1 Mass (Da) 02.100	1 Mass (Da) 02.100	1 Mass 18.000	1 Mass (Da) 15.200
RT (min) Parar Start Stop ID 8.70 DE -60.00 -60.00 -60.00 -22.00-22 RT (min) Parar Start Stop ID 3.54 DE -40.00 -40.00 -40.00 RT (min) Parar Start Stop ID RT (min) Parar -90.00 -90.00 -35.00-35 RT (min) Parar Start Stop ID A.29 DE -90.00 -90.00 -90.00 RT (min) Parar Start Stop ID Star Stop ID -110.00 -100.00 Star Start Stop ID RT (min) Parar Start Stop ID Star Stop ID -28.00-28 ID RT (min) DE Start Stop ID 3.97 DE -100.00 -28.00-35 -28.00-35 RT (min)	RT (min) Parar Start Stop: ID RT (min) Parar Start Stop: ID 3.54 DE -40.00 -40.00 -22.00-22 RT (min) Parar Start Stop: ID 3.54 DE -40.00 -20.00 -22.00-23 RT (min) Parar Start Stop: ID 3.66 DE -110.00 -110.00 -100.00 RT (min) Parar Start Stop: ID RT (min) Parar Start Stop: ID 3.41 DE -110.00 -100.00 -28.00-28 RT (min) Parar Start Stop: ID 3.97 DE -100.00 -28.00-28 -28.00-28 RT (min) Parar Start Stop: ID 3.97 DE -100.00 -28.00-35 -28.00-35 RT (min) Parar Start Stop: 1D							(Da)	
RT (min) Parar Start Stop ID RT (min) Parar Start Stop ID S.54 DF -40.00 -40.00 -40.00 RT (min) Parar Start Stop ID S.66 DF -110.00 -110.00 -110.00 RT (min) Parar Start Stop ID S.20 DF -90.00 -90.00 -90.00 RT (min) Parar Start Stop ID S.20 DF -110.00 -110.00 -100.00 RT (min) Parar Start Stop ID S.41 DF -100.00 -28.00 -28 RT (min) Parar Start Stop ID S.97 DF -100.00 -100.00 -28 RT (min) Parar Start Stop ID S.97 DF -100.00 -35.00 -35.00 S.97 DF	RT (min) Parar Start Stop: ID RT (min) Parar Start Stop: ID 3.54 DE -40.00 -40.00 -22.00-22 RT (min) Parar Start Stop: ID 3.66 DE -40.00 -40.00 -31.00-31 RT (min) Parar Start Stop: ID S.20 DE -90.00 -90.00 -90.00 RT (min) Parar Start Stop: ID S.20 DE -110.00 -110.00 -100.00 RT (min) Parar Start Stop: ID S.20 DE -100.00 -28.00 -28 RT (min) Parar Start Stop: ID S.397 DE -100.00 -100.00 -35.00 RT (min) Parar Start Stop: ID S.97 DE -28.00 -35.00 -35.00 S.97	3 Mass 09.000	3 Mass 09.000	3 Mass 09.000	3 Mass 8.900	13 Mass 8,800	8,900	13 Mass 20,300)3 Mass 570.000
Param Start Stop: ID Param Start Stop: ID Param Start Stop: ID Param Start Stop: ID DE -40.00 -40.00 -40.00 DE -31.00 -31.00 -31.00 DE -110.00 -110.00 -110.00 DE -90.00 -90.00 -90.00 DE -110.00 -110.00 -100.00 DE -110.00 -100.00 -100.00 DE Start Stop: ID Param Start Stop: ID DE -100.00 -28.00 -28 DE -28.00 -28 -28.00 DE -28.00 -28 -28 DE -100.00 -100.00 -35.00 DE -100.00 -35.00 -35.00 DE -100.00 -35.00 -35.00 DE -35.00	Param Start Stop: ID Param Start Stop: ID Param Start Stop: ID Param Start Stop: ID DE -40.00 -40.00 -22.00 -22 Param Start Stop: ID DE -31.00 -31 Param Start Stop: ID DE -31.00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -31 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 00 -35 </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>								
Start Stop ID -60.00 -60.00 CE -22.00-22 Start Stop ID -40.00 CE -31.00-31 Start Stop ID -110.00 -110.00 CE -70.00 -35.00-35 Start Stop ID -100.00 -100.00 CE -80.00-80 CE -80.00-80 CE -80.00-80 CE -80.00 -80 CE -80.00 -100.00 CE -28.00 -28 Start Stop ID -100.00 -100.00 CE -35.00 -35 Start Stop ID -100.00 -100.00 CE -35.00 -35	Start Stop ID -60.00 -60.00 CE -22.00-22 Start Stop ID -40.00 -40.00 CE -31.00 -31 Start Stop ID -110.00 -110.00 CE -35.00 -35 CE -80.00 -80 CE -80.00 -80 CE -80.00 -80 CE -80.00 -80 CE -80.00 -80 CE -80.00 -100.00 CE -28.00 -28 Start Stop ID -100.00 -100.00 CE -35.00 -35 Start Stop ID -100.00 -100.00 CE -35.00 -35	تر (min) 1,58	27 (min) 3.97	27 (min) 3.41	5.20	27 (min) 1.29	21" (min) 3.66	3.54 3.54	70 (min) 6.70
Start Stop ID -60.00 -60.00 CE -22.00-22 Start Stop ID -40.00 -40.00 CE -31.00 -31 -110.00 -110.00 CE -35.00 -35 CE -70.00 -70 CE -70.00 -70 CE -80.00 -80 CE -80.00 -80 CE -28.00 -28 Start Stop ID -100.00 -100.00 CE -35.00 -35 Start Stop ID -100.00 -100.00 CE -35.00 -35 CE -42.00 -42	Start Stop ID -60.00 -60.00 CE -22.00-22 Start Stop ID -40.00 -40.00 CE -31.00-31 -110.00 -110.00 CE -35.00-35 CE -70.00 -70 CE -70.00 -70 CE -80.00-80 CE -80.00-80 CE -28.00 -28 Start Stop ID -100.00 -100.00 CE -35.00 -35 Start Stop ID -100.00 -100.00 CE -35.00 -35 CE -42.00 -42	Paran DF	Paran DF	Paran D£	Paran DF	Paran DF	Paran DF	Paran DF	Paran DE
		Start Stop ID -100.00 -100.00 CE -42.00 -42.	Start Stop ID -100.00 -100.00 CE -35.00 -35	Start Stor ID -100.00 -100.00 CE -28.00 -28	Start Stop ID -110.00 -110.00 CE -80.00 -80	Start Stop ID -90.00 -90.00 CE -70.00 -70.	Start Stop ID -110,00 -110,00 CE -35,00 -35	Start Stop ID -40.00 -40.00 CE -31.00 -31.	Start Stop ID -60.00 -60.00 CE -22.00 -22.

arameter 1 UR: S: EM: S1: S2: S2: F	1 Mass (Da) 98.200	1 Mass (Da) 98.100	1 Mass (Da) 98.000	1 Mass (Da) 02-800	1 Mass (Da) 15.100	1 Mass (Da) 86.900
arameter Table (Period 1 UR: 35.00 S: -3000.00 EM: 450.00 S1: 40.00 S2: 50.00 AD: 10.00 F -10.00	03 Mass 80.000	03 Mass (Da) 80.000	03 Mass 80.000	03 Mass 98,900	03 Mass 119.400	Q3 Mass 184.900
	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
Experiment	RT (min) 3.39	RT (min) 3.70	RT (min) 3.78	RT (min) 5.21	RT (min) 3.54	RT (min) 3.68
1):	Paran De	Paran DF	Paran DF	Paran DF	Paran DF	Paran Df
	Start Stop ID -100.00 -100.00 CE -100.00	Start Stop ID -100,00 -100,00 CE -100.00	Start Stop ID -100.00 -100.00 CE -100.00	Start Stop ID -110.00-110.00 13 CE -80.00-80.00	Start Stop ID -40.00 -40.00 13 CE -31.00-31.00	Start Stop ID -75.00 -75.00 13 CE -10.00-10.00
	TUDCA 100,00	TCDCA 100.00	TDCA -100.00	1304-PEOS_2 .00	13C2-РЕНИА_2 .00	13C3-HEPODA 2

Artacoment To TUS Eurones Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 " Printed by Dorothy Love, d. Mon 20 Nov 2023 15:06 FST

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LPGE cycle: <punip8></punip8>	<advanced></advanced>	A		Port Mob	Set co	Ambile p	Select	Anaphile p	<pmpb></pmpb>	12 SU U 2000	0.00 0.000	3-80 0.5000	3.00 0.5000	0.20 0.5000	0.00 0.5000		3	Time Flow	<pre> Gradient> </pre>	Pressure	Pressine	B Outro	B.Conc:			FI mu/Processine •		Mode:	Gradient start adjustment>	top time:	unip mode:	odel:	L Info	C system Equiperation time = 0.00 min C system Injection Volume = 10.00 ul	C mentar ruberties	C Mathod Down	1.0	Total T	ntegrated Valve Method Properties alve Diverter	X∓ -14.00
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Standard	2																			6500 psi	_				0 2000 mT					12.50 min	Binary gradient	D XP B: LC-40D XR								
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Wait for temperature equilibrat	Measuring line purge volume:	Runsing volume:	Runsing speed:	Runse time:	Rinse method:	Rinse dip time:		Rinse type:	Rinse settings>	Out off loop settings>	Pretreatment start timing:	Acquisition cycle time optimization>	Use low dispersion injection:	Use air oap volume:	Use cooler temperature:	Sampling speed:	Injection settings>	Specify needle stroke:	Specify plate:	Sample plate settings>	Use Autosampler:	General>		ucosanjoter	unit and and	Turn pump on after	Turn oven on after autopurge:	<execute after="" autopurge=""></execute>	B.GEL flow:	Wait time:	Community	Unit conc-replacement>	Execute sampler purge:	<pre><autosampler purge="" settings=""></autosampler></pre>		Autopurge settings>	LAGE cycle:	ancec	A Mobile phase A	Port Mobile phase name	Set compressibility:	Abbile phase settings>	Selected mobile phase port:	
Wait for temperature equilibration before run (sample cooler):	100 JL	500 ptL	35 µL/s	2.0 s	Rinse port only	s 0	Before and after aspiration	External only	ž	F	Off		No	LU C		1.0 µL/s		No	No		Yes		STI-ADC VD			≥: No	2: No		0.0000 mL/min	0 min	- Mo	5°)	No		sis: No		Standard				No		h: ₽	,

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No	Event 2:
	over: External output settings>
	ystem Controller
Auto	Cooler mode: Time program>
Auto	Fan speed:
	Wait for temperature equilibration
	Temperature linit (Maximum) :
AD PC	Use Column Oven:
	Column Oven A> <general></general>
A: CTO-40C	odel:
	ven
Standard	ridde: Time program>
	Pretreatment>

ELLINGTON BORATORIES

CERTIFICATE OF ANALYSIS DOCUMENTATION

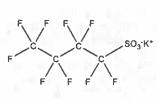
PRODUCT CODE: COMPOUND:

L-PFBS Potassium perfluoro-1-butanesulfonate LOT NUMBER: LPFBS1022

CAS #:

29420-49-3

STRUCTURE:



MOLECULAR FORMULA: CONCENTRATION:

CHEMICAL PURITY: LAST TESTED: (mm/dd/yyyy) EXPIRY_DATE: (mm/dd/yyyy) **RECOMMENDED STORAGE:**

C,F,SO,K 50.0 ± 2.5 µg/mL (K salt) 44.4 ± 2.2 µg/mL (PFBS acid) 44.2 ± 2.2 µg/mL (PFBS anion) >98% 10/04/2022 10/04/2027 Store ampoule in a cool, dark place **MOLECULAR WEIGHT:** 338.19 SOLVENT(S):

Methanol

DOCUMENTATION/ DATA ATTACHED:

Figure 1: LC/MS Data (Full Scan and Mass Spectrum) Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

See page 2 for further details.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Certified By:

B.G. Chittim, General Manager

Date: 10/14/202

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA 519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

Form#:27, Issued 2004-11-10

LPFBS1022 (1 of 4)

Attachment *** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 67 of 87 1633 '

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compound it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products are compared to older lots in the same manner, which further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters

 x_1, x_2, \dots, x_n on which it depends is:

$$u_{c}(y(x_{1}, x_{2}, ..., x_{n})) = \sqrt{\sum_{i=1}^{n} u(y, x_{i})^{2}}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of $\pm 5\%$ (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A1226), and ISO 17034 by ANSI National Accreditation Board (ANAB; AR-1523).





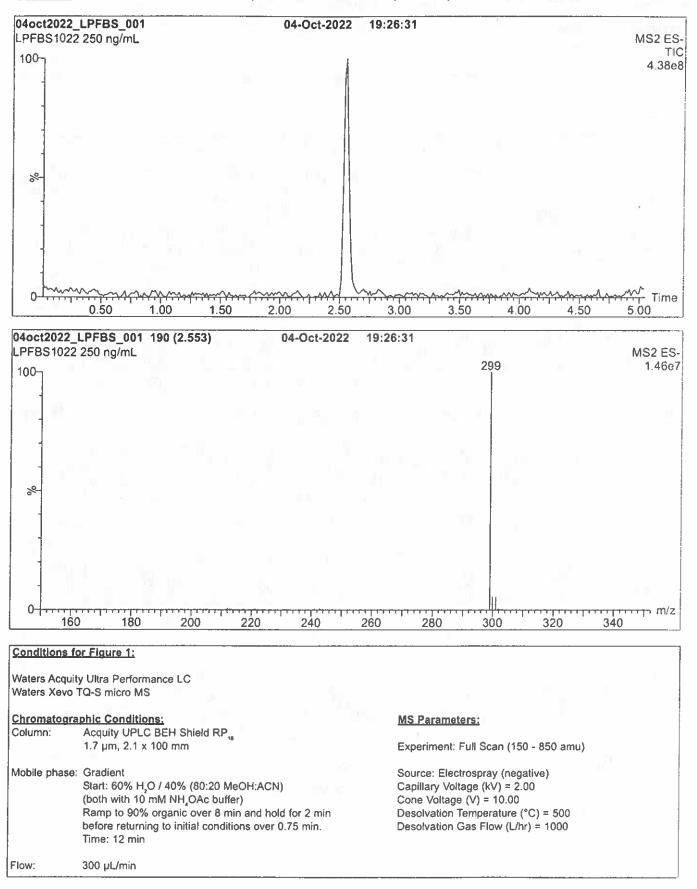
For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at <u>www.well-labs.com</u> or contact us directly at <u>info@well-labs.com</u>

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Attachment ** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 68 of 87 1633 *





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Attachment *** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 69 of 87 1633 *

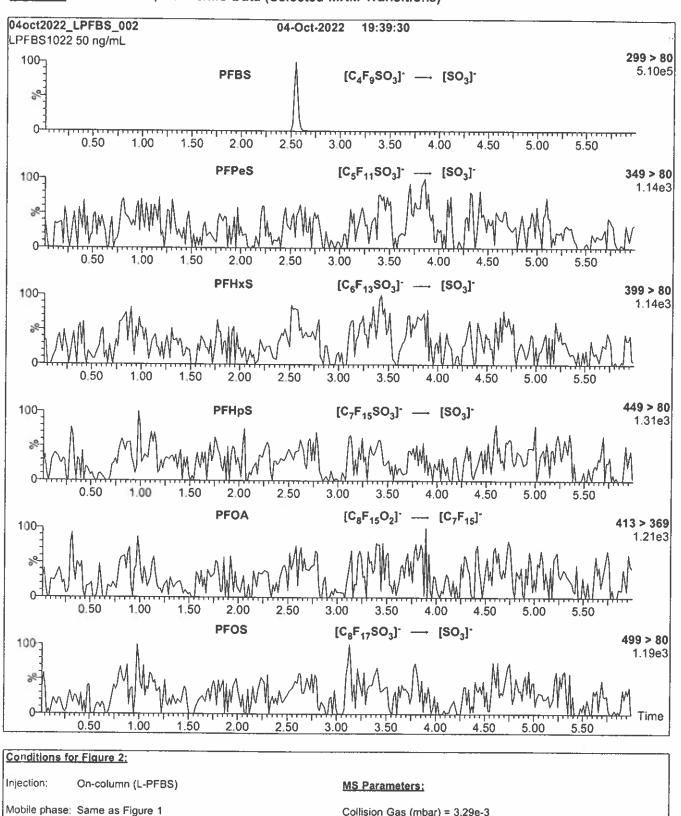


Figure 2: L-PFBS; LC/MS/MS Data (Selected MRM Transitions)

Collision Gas (mbar) = 3.29e-3 Collision Energy (eV) = 30

Form# 27, Issued 2004-11-10

Flow:

300 µL/min

Attachment ** to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 70 of 87 1633 *

					1		Final	Final Conc.
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Volume (ml) Methanol	Native PFAS Intermediate A (ng/mi)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.1	-	500
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.05	1.97	250
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.025	1.11	125
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.025		125
Wellington	PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.025		125
Wellington	PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.025	1.145	125
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.025	1.1	125
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000	0.025		125
Wellington	PFDoA	Perfluorododecanoic acid	307-55-1	PFDoA	50000	0.025		125
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.025	1.1	125
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.025		125
Weilington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.025		111
Wellington		Perfluoropentansulfonic acid	2706-91-4	PFPeS	46900	0.025	W	117
_	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.025	1.5	114
Vellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.025		119
Vellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.025		117
Vellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.025	120	120
Vellington		Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.025	Guine	121
Vellington	2	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400	0.025	-	121
Vellington		1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	46700	0.1	U. 0	467
Vellington		1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	47400	0.1	40	474
Vellington		1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	47900	0.1	10	479
Vellington	-	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.025		125
	N-MeFOSA-M	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	50000	0.025		125
	N-EtFOSA-M	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	50000	0.025		125
	br-NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.025		125
Vellington	br-NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.025		125
Vellington	N-MeFOSE-M	N-methyl perfluorocctanesulfonamidoethanol	24448-09-7	NMePFOSAE	50000	0.25		1250
Vellington	N-EtFOSE-M	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEIPFOSAE	50000	0.25		1250
Vellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	50000	0.1		500
Vellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	47100	0.1	10.00	471
Vellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.05		250
Vellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50000	0.05		250
Vellington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	50000	0.05		250
Vellington	9CI-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1- sulfonic acid	756426-58-1	9CI-PF3ONS	46600	0.1		466
	11CI-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1- sulfonic acid		11CI-PF3OUdS	47100	0.1	=	471
Vellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.05	u l	223
Vellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625
Vellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000	0.625		3125
Vellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000	0.625		3125

	Nati	ve PFAS Inter	mediate B				
Solution Name	Anatyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (mi) Methanol	Final Conc. Native PFAS Intermediate B (ng/ml)
	Perfluorobutanoic acid	375-22-4	PFBA	500			5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250	1		2.5
	Perfluorohexanoic acid	307-24-4	PFHxA	125			1.25
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			1.25
	Perfluorooctanoic acid	335-67-1	PFOA	125			1.25
	Perfluorononanoic acid	375-95-1	PFNA	125			1.25
	Perfluorodecanoic acid	335-76-2	PFDA	125			1.25
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125	1		1.25
	Perfluorododecanoic acid	307-55-1	PFDoA	125			1.25
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			1.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			1.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5	1		1.11
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117.25			1.17
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75	1		1.14
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119	1		- 1.19
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5	1		1.17
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			1.20
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5			1.21
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121	1		1,21
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467	1		4.67
Native PFAS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474	0.1	10	4.74
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479	1		4.79
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			1.25
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			1.25
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			1.25
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			1.25
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			12.5
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			12.5
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			5
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			4.71
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			2.5
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			2.5
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			2.5
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9CI-PF3ONS	466			4.66
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid		11CI-PF3OUdS	471			4.71
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			2.23
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			6.25
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			31.3
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			31.3

		Native PFAS	ntermediate M	V			
Solution Name	Analyte	CAS#	Acronym	Concentration (ng/mi)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Native PFAS Intermediate M (ng/mi)
1	Perfluorobutanoic acid	375-22-4	PFBA	500			62.5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250] i	1.1	31.3
	Perfluorohexanoic acid	307-24-4	PFHxA	125			15.6
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			15.6
	Perfluorooctanoic acid	335-67-1	PFOA	125			15.6
	Perfluorononanoic acid	375-95-1	PFNA	125	-		15.6
	Perfluorodecanoic acid	335-76-2	PFDA	125			15.6
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			15.6
	Perfluorododecanoic acid	307-55-1	PFDoA	125			15.6
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			15.6
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125		51 B	15.6
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			13.8
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117.25			14.7
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75			14.2
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119		- C P-	14.9
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5			14.6
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120	-		15.0
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5	1.1	- 1	15.1
	Perfluorododecanesulfonic acid	79780-39-5	PFDo\$	121	1.1		15.1
	1H,1H, 2H, 2H-Perfluorohexane suffonic acid	757124-72-4	4:2-FTS	467	- R.C.	4 K - 1	58.4
Native PFAS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474	1.25	10	59.3
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479	~ Q		59.9
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			15.6
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			15.6
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEIPFOSA	125		17	15.6
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125		- 1	15.6
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125	_		15.6
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250		l j	156
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEIPFOSAE	1250	H _ 10		156
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			62.5
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471	1.000		58.9
	Perfluoro-3-methoxypropanoic acid	377-73-1	PEMPA	250		-	31.3
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			31.3
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250		ŀ	31.3
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9CI-PF3ONS	466			58.3
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11CI-PF3OUdS	471			58.9
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5		L	27.8
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			78.1
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			391
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125		ſ	391

		Extractio	n Standard	Mix				
Vendor	Catalog Number	Analyte	CAS#	Acronym	Concentration (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Extraction Standard Mix (ng/mi)
Wellington	MPFBA	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	50000	0.1		500
Wellington	M5PFPeA	Perfluoro-n-(13C5)pentanoic acid	STL01893	13C5-PFPeA	50000	0.05		250
Wellington	M5PFHxA	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	50000	0.025		125
Wellington	M4PFHpA	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	50000	0.025		125
Wellington	M8PFOA	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	50000	0.025		125
Wellington	M9PFNA	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	50000	0.0125		62.5
Wellington	M6PFDA	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	50000	0.0125		62.5
Wellington	M7PFUdA	Perfluoro-n-{1,2,3,4,5,6,7- 13C7]undecanoic acid	STL02580	13C7-PFUnA	50000	0.0125		62.5
Wellington	MPFDoA	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	50000	0.0125		62.5
Wellington	M2PFTeDA	Perfluoro-n-[1,2-13C2]letradecanoic acid	STL02116	13C2-PFTeDA	50000	0.0125		62.5
Wellington	M3PFBS	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	46500	0.025		116
Wellington	M3PFHxS	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	47300	0.025	:	118
Wellington	M8PFOS	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	47800	0.025	10	120
Wellington	M8FOSA-I	Perfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	50000	0.025	10	125
Wellington	d3-N-MeFOSAA	N-methyl-d3-perfluoro-1- octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	50000	0.05		250
Wellington	d5-N-EtFOSAA	N-ethyl-d5-perfluoro-1- octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	50000	0.05		250
Wellington	M2-4;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2- 13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	46700	0.05		234
Wellington	M2-6;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2- 13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	47500	0.05		238
Wellington	M2-8;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2- 13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	47900	0.05		240
Wellington	M3HFPO-DA	Tetrafluoro-2-heptafluoropropoxy-13C3- propanoic acid	STL02255	13C3-HFPO-DA	50000	0.1		500
Wellington	d7-N-MeFOSE-M	N-methyl-d7- perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	50000	0.25		1250
Wellington	d9-N-EtFOSE-M	N-ethyl-d9- perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	50000	0.25		1250
Wellington	d-N-MeFOSA-M	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	50000	0.025		125
Wellington	d-N-EIFOSA-M	N-methyl-d3-perfluoro-1- octanesulfonamide	STL02705	D3-NMeFOSA	50000	0.025		125

		Interna	I Standard I	Aix				
Vendor	Catalog Number	Analyte	CAS#	Acronym	Concentration (ng/ml)	Aliquot (mi)	Final Volume (ml) Methanol	Final Conc. Internal Standard Mix (ng/ml)
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	50000	50		250
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	50000	25		125
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	50000	12.5		62.5
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	47800	25	10	120
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	50000	12.5		62.5
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]haxanolc acid	STL00993	13C2-PFHxA	50000	25		125
Wellington	MPFHxS	Perfluoro-1-hexane[1802]sulfonic acid	STL00994	1802-PFHxS	47300	25		118

	1633 in	itial Calibrati	on Standards	Preparation			
Solution Name	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Native PFAS Intermediate A (ml)	N/A	N/A	N/A	0.01	0.02	0.05	0.25
Native PFAS Intermediate B (ml)	0.08	0.2	0.5	N/A	N/A	N/A	N/A
Extraction Standard Mix (ml)	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Internal Standard Mix (ml)	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Final Volume (ml) (methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid)	2	2	2	2	2	2	2

Attachment "" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 76 of 87 1633 "

Attacl	nment	11	
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			Initial Calibration S				
	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Compound Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.200	0.500	1,25	2.50	5.00	12.5	62.5
PFpeA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFHxA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFHpA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFOA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFNA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDA	0.050	0.125	0.313	0.625	1.25	3,13	15.6
PFUnA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDoA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTrDA	0.050	0.125	0.313	0.625	1.25	3,13	15.6
PFTeDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFBS	0.044	0.111	0.276	0.553	1.11	2.76	13.8
PFPeS	0.047	0.117	0.293	0.586	1.17	2,93	14.7
PFHxS	0.046	0.114	0.284	0.569	1.14	2.84	14.2
PFHpS	0.048	0.119	0.298	0.595	1.19	2.98	14.9
PFOS	0.047	0.117	0.291	0.583	1.17	2.91	14.6
PFNS	0.048	0.120	0.300	0.600	1.20	3.00	15.0
PFDS	0.048	0.121	0.301	0.603	1.21	3.01	15.1
PFDoS	0.048	0.121	0.303	0.605	1.21	3.03	15.1
4:2 FTS	0.187	0.467	1.17	2.34	4.67	11.7	58.4
6:2 FTS	0.190	0.474	1,19	2.37	4.74	11.9	59.3
3:2 FTS	0.192	0.479	1.20	2.40	4.79	12.0	59_9
PFOSA	0.050	0.125	0.313	0.625	1.3	3,13	15.6
MeFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSA	0.050	0.125	0.313	0.625	1.3	3,13	15.6
MeFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
MeFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
NETFOSE	0.500	1.25	3.13	6.25	12,5	31,3	156
IFPO-DA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
DONA	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PEMPA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFMBA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
VEDHA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
CI-PF3ONS	0.186	0.466	1.17	2.33	4.66	11.7	58.3
1CI-PF3OUdS	0.188	0.400	1.18	2.36	4.71	11.8	58.9
PFEESA	0.089	0.223	0.56	1.11	2.23	5.56	27.8
3:3 FTCA	0.250	0.625	1.56	3.13	6.25	15.6	78.1
5:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
7:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
I3C4-PFBA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
I3C5-PFPeA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
3C5-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3C4-PFHpA				0.625	0.625	0.625	0.625
	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3C8-PFOA	0.625		0.625				
3C9-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C6-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C7-PFUnA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C2-PFDoA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C2-PFTeDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C3-PFBS	0.581	0.581	0.581	0.581	0.581	0.581	0.581
3C3-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591
3C8-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
3C8-PFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3-NMeFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
5-NEIFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
3C2-4:2FTS	1.17	1.17	1.17	1.17	1.17	1.17	1,17
3C2-6:2FTS	1.19	1.19	1.19	1.19	1.19	1.19	1.19
3C2-8:2FTS	1.20	1.20	1.20	1.20	1.20	1.20	1.20
3C3-HFPO-DA	2.50	2.50	2.50	2.50	2.50	2.50	2,50
7-NMeFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
9-NEIFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
5-NEtFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3-NMeFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3C3-PFBA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
3C4-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3C2-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C4-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
3C5-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C2-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
802-PFHxS	0.591	0.591	0.525	0.525	0.591	0.591	0.591

Attachment "" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 77 of 87 1633 "

	Bile Salts Stock Solutions								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Amount (mg)	Aliquot (g)	Final Volume (ml) Methanol	Final Conc. Bile Saits Stock Solutions (ng/mi)	
Sigma Aldrich	T0557-500MG	Taurodeoxycholic acid	516-50-7	TDCA	500	0.05	50	1000000	
Sigma Aldrich	T6260-1G	Taurochenodeoxycholic acid	516-35-8	TCDCA	1000	0.05	50	1000000	
Sigma Aldrich	580549-1GM	Tauroursodeoxycholic acid	14605-22-2	TUDCA	1000	0.05	50	1000000	

Attachment ** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 78 of 87 1633 *

Bile Salts Working Solution A							
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Bile Salts Working Solution A (ng/ml)
TDCA Stock Solution	Taurodeoxycholic acid	516-50-7	TDCA	1000000	- 0.1	1	250000
TCDCA Stock Solution	Taurochenodeoxycholic acid	516-35-8	TCDCA	1000000	1	4	250000
TUDCA Stock Solution	Tauroursodeoxycholic acid	14605-22-2	TUDCA	1000000			250000

	Bile Salts Working Solution B								
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Bile Salts Working Solution B (ng/ml)		
	Taurodeoxycholic acid	516-50-7	TDCA	250000			5000		
Bile Salts Working Solution A	Taurochenodeoxycholic acid	516-35-8	TCDCA	250000	0.1	5	5000		
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	250000			5000		

Attachment "" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method Page 80 of 87 1633 "

		PFAS Linear/	Branched M	lix A				
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. PFAS Linear/Branched Mix A (ng/mi)
Wellington	T-PFOA	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	50000	0.02		500
		Perfluorooctanoic acid	335-67-1 PFOA		50000	0.02		500
Cambridge Isotope Laboratories	ULM-11036-S	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEIPFOSAE	50000	0 02	12	500
Cambridge Isotope Laboratories	ULM-11034-S	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	50000	0.01		500
Cambridge Isotope Laboratories	ULM-10780-S	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	100000	0.01	2	500
Cambridge Isotope Laboratories	ULM-10779-S	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	100000	0.02		500
Cambridge Isotope Laboratories	ULM-10977-S	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.02	11 M 1	500
Wellington	ipPFNA	Perflucro-7-methylpoctanoic acid	15899-31-7	PF7MOA	50000	0.02	· · · ·	500
Wellington	PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.02		500

	PFAS Linear/Branched Mix B							
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. PFAS Linear/Branched Mix B (ng/ml)	
	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	500			125	
	Perfluorooctanoic acid		PFOA	500			125	
	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500			125	
	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500			125	
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500	0.5	2	125	
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			125	
	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			125	
	Perfluoro-7-methylpoctanoic acid	15899-31-7	PF7MOA	500		1	125	
	Perfluorononanoic acid	375-95-1	PFNA	500			125	

Attachment 17	
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				Conc.	Aliquot	Final	Final Conc. 1633
Solution Name	Analyte	CAS#	Acronym	(ng/ml)	(m))	Volumə* (ml)	Linear/Branched Bild Salts Solution (ng/mi
Bile Salts	Taurodeoxycholic acid	516-50-7	TDCA	5000	T	- 5	100
Vorking	Taurochenodeoxycholic acld	516-35-8	TCDCA	5000	0.1		100
Solution B	Tauroursodeoxycholic acid	14605-22-2	TUDCA	5000			100
	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	125		- 11	1.25
	Perfluorooctanoic acid	335-67-1	PFOA	125			1.25
	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	125		1.0	1.25
FAS	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	125	1.1		1.25
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	125	0.02		1.25
lix B	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	125		STITE:	1.25
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	Perfluoro-7-methylpoctanoic acid	15899-31-7	PF7MOA	125			1.25
	Perfluorononanoic acid	375-95-1	PFNA	125			1.25
	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500			2.5
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250			1.25
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid STL02577 13C5-PFHxA 125						0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125	2.1		0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125	111		0.625
	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	62.5	orm -		0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5	- Kapitan (0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5		1.1	0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5		2	0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PF8S	116	1.1		0.581
xtraction	Perfluoro-1-(1,2,3-13C3)hexanesulfonic acid	STL02581	13C3-PFHxS	118	0.01		0.591
tandard Mix	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	120	0.01		0.598
	Perfluoro-1-{13C8}octanesulfonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250	≤ 0	1.00	1.25
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	250			1.25
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	234	5 C		1.17
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	238			1.19
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	240			1.20
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	500			2.5
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	1250			6.25
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250		O	6.25
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	125	200		0.625
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250	=		1.25
i	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
	Perfluoro-n-{1,2-13C2}decanoic acid	STL00996	13C2-PFDA	62.5	.2		0.313
ternal andard Mix	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	120	0.01		0.598
SHOULD MIA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[1802]sulfonic acid	STL00994	18O2-PFHxS	118			0.591

*Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

Attachment 1	18
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-		Vorking Stand				Ele-1	Eleal Ocean 4000
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume* (ml)	Final Conc. 1633 ICV Working Standard (ng/ml)
	Perfluorobutanoic acid	375-22-4	PFBA	500		(()))	2.50
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			1.25
	Perfluorohexanoic acid	307-24-4	PFHxA	125			0.625
	Perfluoroheptanoic acid Perfluorooctanoic acid	375-85-9	PFHpA	125			0.625
	Perfluorononanoic acid	335-67-1 375-95-1	PFOA PFNA	125 125			0.625
	Perfluorodecanoic acid	335-76-2	PFDA	125			0.625
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			0.625
	Perfluorododecanoic acid	307-55-1	PFDoA	125			0.625
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			0.625
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			0.625
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			0.553
	Perfluoropentansulfonic acid Perfluorohexanesulfonic acid	2708-91-4	PFPeS	117			0.586
	Perfluoroheptanesulfonic acid	355-46-4 375-92-8	PFHxS	114 119			0.569
	Perfluorooctanesulfonic acid	1763-32-0	PFHpS PFOS	117			0.595
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			0.600
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120			0.603
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			0.605
Native PFAS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467	0.04		2.34
A anneolate	1H,1H, 2H, 2H-Perituorooctane sulfonic acid	27619-97-2	6:2-FTS	474	0.01		2.37
•	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			2.40
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			0.625
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			0.625
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			0.625
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			0.625
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			0.625
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			6.25
	N-ethyl perfluorooctanesulfonamidoethanol Hexafluoropropylene oxide dimer acid	1691-99-2	NEIPFOSAE	1250			6.25
	4,8-Dioxa-3H-perfluorononanoic acid	13252-13-6 919005-14-4	HFPODA DONA	500 471	1.1		2.50
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			2.36
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			1.25
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6		250			1.25
	9-Chiorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1		466			2.33
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11CI-PF3OUdS	471		2	2.36
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			1.11
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			3.13
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			15.6
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			15.6
	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500			2.5
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250			1.25
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01892 STL01052	13C4-PFHpA 13C8-PFOA	125 125			0.625
	Perfluoro-n-[13C9]nonanoic acid	STL01052	13C9-PFNA	62.5			0.625
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PF8S	116			0.581
Extraction	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	118	0.01		0.591
Standard Mix	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	120	0.01		0.598
	Perfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	125		.	0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250			1.25
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEIFOSAA	250			1.25
	1H,1H,2H,2H-Perfluoro-1-(1,2-13C2)hexane sulfonic acid	STL02395	13C2-4:2FTS	234			1.17
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid 1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02279	13C2-6:2FTS	238			1.19
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02280	13C2-8:2FTS	240			1.20
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02255 STL02277	13C3-HFPO-DA D7-NMeFOSE	500 1250			2.5
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02277	D9-NEIFOSE	1250			<u> 6.25</u> 6.25
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02278	D5-NE(FOSA	1250			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250		1	1.25
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
atomai	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
nternal Standard Mix	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	120	0.01		0.598
AND A DOM AND	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	1802-PFHxS	118			0.591

*Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

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ATTACHMENT 21

	Aqueous Matrix ^{1, 2}							
	IPR	37.3		10				
Compounds	Recovery (%)	RSD (%)	OPR Recovery (%)	LLOPR Recovery (%)				
PFBA	60 - 147	20	58-148	44 - 157				
PFPeA	56-150	20	54-152	57 - 148				
PFHxA	59 - 148	25	55-152	62 - 149				
PFHpA	60-149	25	54-154	56 - 150				
PFOA	55-158	25	52 - 161	57 - 161				
PFNA	64 - 144	25	59 - 149	53 - 157				
PFDA	57-142	25	52-147	43 - 158				
PFUnA	54-153	30	48-159	50 - 155				
PFDoA	73 - 133	25	64 - 142	60 - 141				
PFTrDA	52 - 145	25	49 - 148	52 - 140				
PFTeDA	49 - 158	25	47 - 161	52 - 156				
PFBS	66-141	20	62 - 144	63 - 145				
PFPeS	66 - 144	25	59-151	58 - 144				
PFHxS	62 - 141	25	57 146	44 - 158				
PFHpS	59-148	25	55-152	51 - 150				
PFOS	61-145	20	58-149	43 - 162				
PFNS	57-143	25	52-148	46 - 151				
PFDS	56-142	25	51-147	50 - 144				
PFDoS	41 - 140	30	36-145	30 - 138				
4:2FTS	77 - 135	25	67 - 146	52 - 158				
6:2FTS	75-137	30	61 - 151	48 - 158				
8:2FTS	79 - 136	30	63 - 152	46 - 165				
PFOSA	65-144	20	61 - 148	47 - 163				
NMeFOSA	76-132	25	63 - 145	54 - 155				
NEtFOSA	75 - 129	25	65-139	49 - 156				
NMeFOSAA	69 - 134	25	58-144	32 - 160				
NEtFOSAA	65-140	25	59 - 146	51 - 154				
NMeFOSE	79 - 129	20	71 - 136	56 - 151				
NEtFOSE	79 - 126	25	69-137	60 - 147				
HFPO-DA	72 - 135	25	63 - 144	58 - 154				
ADONA	75 - 138	20	68-146	61 - 148				
PFMPA	55 - 141	25	51-145	48 - 150				
PFMBA	59-141	20	55-148	49 - 154				
NFDHA	63 - 146	35	48-161	47 - 160				
9CI-PF3ONS	72 - 140	30	56-156	44 - 167				
11C1-PF3OUds	61 - 140	35	46-156	36 - 158				
PFEESA	57 - 149	20	56-151	56 - 144				
3:3FTCA	66 - 126	20	62 - 129	32 - 161				
5:3FTCA	68 - 130	20	63-134	39 - 156				
7:3FTCA	55 - 133	25	50-138	36 - 149				
¹³ C ₄ -PFBA	10-130	30	10-130	10-130				
¹³ C ₅ -PFPeA	35 - 150	30	40 -150	40 -150				
¹³ C ₃ -PFHxA	55 - 150	30	40 -150	40 -150				
¹³ C ₄ -PFHpA	55 - 150	30	40 - 150	40 -150				
¹³ C ₈ -PFOA	60 - 140	30	30-140	30-140				
¹³ C ₉ -PFNA	55 - 140	30	30-140	30-140				
¹³ C ₆ -PFDA	50 - 140	30	20-140	20-140				

Table 5. IPR/OPR/LLOPR Acceptance Limits for Wastewater Samples

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ATTACHMENT 21

			Aqueous Matrix ^{1, 2}				
	IPR						
Compounds	Recovery (%)	RSD (%)	OPR Recovery (%)	LLOPR Recovery (%)			
¹³ C ₇ -PFUnA	30 - 140	30	20-140	20-140			
¹³ C ₂ -PFDoA	10-150	30	10-150	10-150			
¹³ C ₂ -PFTeDA	10-130	30	10-130 =	10-130			
¹³ C ₃ -PFBS	55 - 150	30	25-150	25-150			
¹³ C ₃ -PFHxS	55-150	30	25-150	25-150			
¹³ C ₄ -PFOS	45-140	30	20-140	20-140			
13C2-4:2FTS	60 - 200	30	25-200	25-200			
¹³ C ₂ -6:2FTS	60 - 200	30	25-200	25-200			
¹³ C ₂ -8:2FTS	50 - 200	30	25-200	25-200			
¹³ C ₆ -PFOSA	30 - 130	30	10-130	10-130			
D3-NMeFOSA	15-130	30	10-130	10-130			
D3-NEtFOSA	10-130	30	10-130	10-130			
D3-NMeFOSAA	45 - 200	30	10-200	10-200			
D3-NEtFOSAA	10-200	30	10-200	10-200			
D7-NMeFOSE	10-150	30	10-150	10-150			
D9-NEtFOSE	10-150	30	10-150	10-150			
¹³ C ₃ -HFPO-DA	25-160	30	25-160	25-160			

Table 5.	IPR/OPR/LLOPR	Acceptance Limits fo	r Wastewater Samples
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¹ The recovery limits apply to the target analyte results for IPR, OPR, and LLOPR samples for wastewater matrices. Data for this matrix type are derived from the multi-laboratory validation study and are therefore the limits required for this method.

² The recovery limits for the EIS compounds were derived by EPA from the wastewater sample data from multilaboratory validation study. To simplify laboratory operations, EPA has applied the same EIS recovery limits used for field sample analyses to the EIS recoveries in the IPR, OPR, and LLOPR samples.

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ATTACHMENT 22

Recoveries in Wastewater Samples			
EIS Compound	Recovery Range (%)		
¹³ C ₄ -PFBA	10-130*		
¹³ C ₅ -PFPeA	35 - 150		
¹³ C5-PFHxA	55 - 150		
¹³ C ₄ -PFHpA	55 - 150		
¹³ C ₈ -PFOA	60 - 140		
¹³ C ₉ -PFNA	55 - 140		
¹³ C ₆ -PFDA	50 - 140		
¹³ C ₇ -PFUnA	30 - 140		
¹³ C ₂ -PFDoA	10 - 150		
¹³ C ₂ -PFTeDA	10-130 *		
¹³ C ₃ -PFBS	55 - 150		
¹³ C ₃ -PFHxS	55 - 150		
¹³ C ₈ -PFOS	45 - 140		
¹³ C ₂ -4:2FTS	60 - 200 *		
¹³ C ₂ -6:2FTS	60 - 200 *		
¹³ C ₂ -8:2FTS	50 - 200 *		
¹³ C ₈ -PFOSA	30-130		
D3-NMeFOSA	15-130		
D5-NEtFOSA	10-130		
D3-NMeFOSAA	45 - 200 *		
D ₅ -NEtFOSAA	10-200		
D7-NMeFOSE	10-150 *		
D9-NEtFOSE	10-150 *		
¹³ C ₃ -HFPO-DA	25 - 160		

Table 8.	QC Acceptance Limits for EIS
	Recoveries in Wastewater Samples

* In the multi-laboratory validation study data for wastewater matrices, some laboratories had difficulties achieving EIS recoveries in this range.

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Revision Log
Reference
Cross Reference
Scope
Basic Principles
Interferences
Precaution to Minimize Method Interference
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision: 02	Effective date:	This version
Section	Justification	Changes
	1.5	Update Procedure

Reference

1. US EPA Method 1633, Analysis of Per and Polyfluoroalkyl Substances(PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, Version 3nd DRAFT, December 2022.

Cross Reference

Document	Document Title
NDSC-QA-SOP42091	Detection and Quantitation Limits
NDSC-QA-SOP43862	Manual Integrations

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Curofins	Aways check on-line for validity. Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633		Level: Standard Operating Procedure
Old Reference: Version: 2			
Approved by: HWD4 Effective Date: 20- NOV-2023	ve Date: 20- CLE PFAS All		Responsible: CLE PFAS All
Document		Document Title	
NC-QA-015		Support Equipment Calibration and Verification	
NC-QA-018		Statistical Evaluation of Data and Development of Control Charts	
QA-003		Quality Control Program	
NC-QA-017	7 Standards and Reagents		
NDSC-QA-QP44	€40	40 Calibration Curves and the Selection of Calibration Points	
NDSC-US- EHSQP46060		NDSC EH&S Manual	
NC-QAM-001		QA Manual	

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in solid. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA -	2706-90-3
8:2 - Fluorotelomersulfonic acid	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9

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	Analyte	Acronym	CAS#	
N-ethylperfluoro	-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6	
4:2-Flu	orotelomersulfonic acid	4:2-FTS	757124-72-4	1.000
Perfluc	propentanesulfonic acid	PFPeS	2706-91-4	
6:2-Flu	orotelomersulfonic acid	6:2-FTS	27619-97-2	
Perfluc	proheptanesulfonic acid	PFHpS	375-92-8	
Perflue	prononanesulfonic acid	PFNS	68259-12-1	
Perflue	prodecanesulfonic acid	PFDS	335-77-3	
Perfluo	rododecanesulfonic acid	PFDoDS	79780-39-5	
Perfluorooctanesulfonamide		PFOSA	754-91-6	i Libre
2-(N-methylpe	rfluoro-1-octanesulfonamido)- ethanol	NMePFOSAE	24448-09-7	
N-methylperfluoro-1-octanesulfonamide		NMePFOSA	31506-32-8	
2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol		NEtPFOSAE	1691-99-2	
N-ethylperf	luoro-1-octanesulfonamide	NEtPFOSA	4151-50-2	
heptafluor	trafluoro-2-(1,1,2,2,3,3,3- opropoxy)-propanoic acid; propylene oxide dimer acid)	HFPODA	13252-13-6	
Ammonium 4,8-	dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *	
Potassium 9-chlo	prohexadecafluoro-3-oxanonane- 1-sulfonic acid	9CI-PF3ONS, F53B major	756426-58-1 *	
Potassium 11-ch	oroeicosafluoro-3-oxaundecane- 1-sulfonic acid	11CI-PF3OUdS, F53B minor	763051-92-9 *	
3-Perflu	oropropylpropanoic 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	121 1
Perfluoro-3-methoxypropanoic acid		PFMPA, PFECA F	377-73-1	
Perfluoro	-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5	
Nonafluor	o-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6	
Perfluoro(2-ethoxyethane)sulfonic acid		PFEESA, PES	113507-82-7	de en Sa

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*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

A solid sample is fortified with isotopically-labeled extraction standards. The sample extract is shaken, centrifuged, and the supernatant decanted. Carbon cleanup is performed on each sample extract. Sample extract is diluted to volume and then concentrated. The sample is then passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. The extract is fortified with Isotopically-labeled injection internal standards and filtered. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

- 1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK[™] solvent frits and tubing where possible.
- 2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.
- 3. All parts of the SPE manifold must be cleaned every 2 weeks per manufacturer recommendations.

Safety Precautions and Waste Handling

See NDSC EH&S Manual, the Facility Addendum to the NDSC EH&S Manual, and this document for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

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Version: 2		Organisation level: 5-Sub-BU
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Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 4oz PP specimen containers, with polyethylene screw caps. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Solid and Biosolid samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.

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- Solid and Biosolid samples stored in the lab must be protected from light and held at a temperature of 0° to 6°C, or ≤ -20°C, until extraction.
- Solid and Biosolid samples must be extracted within 90 days. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light.

NOTE: Biosolid samples stored under refrigeration may produce gases that may cause sample to be expelled from the container when opened. This may produce noxious odors. It is recommended to store frozen if extraction will not occur for a few days.

Apparatus and Equipment

A. Apparatus

- Specimen container; PP; natural; graduated; non-sterile; separate 1/4-turn red screwcap; 4 oz; 600 EA, Greenwood Products, Inc. Part No. HP30011
- Centrifuge tubes 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 14955237 or equivalent
- 3. 10-mL polypropylene volumetric flask, Class A Fisher Scientific, Cat. No. S02288 or equivalent.
- HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 0125-1902-PC.
- 5. Analytical Balance Capable of weighing to 0.0001 g
- 6. Top-Loading Balance Capable of weighing to 0.01 g
- 7. Waters Oasis WAX 150 mg/6cc (part # 186002493) with 20 mg loose carbon added.
- Phenomenex Strata PFAS WAX/GCB, 500mg/50mg/6cc (part # CS0-9208) or equivalent. This cartridge incorporates graphitized carbon.
- 9. Large-volume SPE Reservoir (25-mL) Millipore-Sigma; Product # 54258-U.
- 10. SPE Tube Adapter Millipore-Sigma; Product # 57020-U.
- 11. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.
- 12. Polypropylene SPE delivery needles Agilent; Cat. No. 12234511.
- 13. Centrifuge "Thermo Sorvall ST4" with TX-750 rotor or equivalent, capable of a minimum rotational force of 3800 rcf.
- 14. Disposable polyethylene pipette Fisher Scientific, Cat. No. S30467-1 or equivalent.
- 15. Auto Pipettes Eppendorf; capable of accurately dispensing 10- to 1000-μL. FisherScientific cat # 14-287-150, or equivalent.
- 16. Polypropylene pipette tips: 0-200µl. Fisher; Cat. No. 22491539

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17. Polypropylene pipette tips: 101-1000µl. Fisher, Cat. No. 5414006

- 18. Pipettes Disposable transfer. Fisher Scientific, Cat. No. 13-711-7M
- 19. Vortex mixer, variable speed, Fisher Scientific or equivalent.
- 20. N-Evap sample extract concentrator with N₂ supply and water bath for temperature control. Organomation, Inc. Cat. #11250-NT, or equivalent.
- 21. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTPF08831.

22. Thermo Target PP Polyspring inserts, catalog number C4010-630P, or equivalent

23. Thermo Target 9mm PP vials, catalog number C4000-14, or equivalent

24. Agilent 9mm vial caps, polypropylene/silicone septa, catalog 5191-8151, or equivalent

- 25. Centrifuge tubes 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent
- 26. Polypropylene bottles for standard storage 4 mL; Fisher Scientific, Cat. No. 2006-9125
- 27. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product # 11-865-130.

28. pH paper, range 0-14, Supelco # 1.09533.0001 or equivalent, 0.5 unit readability

- 29. Syringe filter Acrodisc, Syringe Filter, GHP,13 mm, 0.2 μm, Aqueous, 100/pkg, Part # WAT097962.
- 30. Silanized glass wool (Sigma-Alrich, Cat #20411 or equivalent)
- 31. Syringe filter Acrodisc, Syringe Filter, 25 mm, 0.2 µm, 50/pkg, Fisher part # 50786068.
- **32**. Variable speed mixing table (FisherbrandTM Nutating mixer or equivalent)
- 33. Evaporation/concentrator tubes: 60 mL clear glass vial, 30x125 mm, without caps (Wheaton Cat # W226060 or equivalent).
- 34. Wooden Tongue Depressors Fisher; Cat. # 11-700-555, or equivalent.
- 35.Wheaton Bottle, 15ml, Narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Cat. No. 209044SP, or equivalent
- B. Equipment
 - 1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

ExionLC Controller ExionLC AC Pump ExionLC AC Autosampler Exion AC Column Oven Data system –Analyst 1.6.3

2. Promochrom automated extraction unit, model SPE-03 or equivalent

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- 3. HPLC columns
 - a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-YO or equivalent
 - b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

- A. Reagents:
 - 1. Methanol (MeOH) J.T. Baker Ultra Resi-Analyzed, Cat. No. UN1230 or equivalent
 - 2. Acetonitrile (ACN) J.T. Baker Ultra Resi-Analyzed, Cat. No. UN1648 or equivalent
 - 3. Water Fisher Scientific, Optima, Cat. No. W6-4 or equivalent
 - 4. Ammonium acetate Fisher Scientific, Cat. No. A637-500 or equivalent
 - 5. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
 - 6. Methanolic ammonium hydroxide (0.3%) add ammonium hydroxide (10 mL, 30%) to methanol (990 mL), store at room temperature, replace after 1 month
 - 7. Methanolic ammonium hydroxide (1%) add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
 - Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
 - 9. Acetic Acid ACS grade or equivalent, store at room temperature
 - 10. Acetic Acid (0.1%) dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
 - 11. Formic acid -(greater than 96% purity or equivalent), store at room temperature.
 - a. Formic acid (aqueous, 0.1 M) dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years
 - b. Formic acid (aqueous, 0.3 M) dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years

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- c. Formic acid (aqueous, 5% v/v) mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years
- d. Formic acid (aqueous, 50% v/v) mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years
- e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years
- 12. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.
- 13. Solids reference matrix Ottawa or reagent-grade sand
- 14. 20 mM ammonium acetate solution in 95:5(v/v) Milli-Q water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL Milli-Q water and mix well to dissolve the ammonium acetate. Add 100 mL acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.
- 15.20 mM ammonium acetate solution in 90:10 acetonitrile/Milli-Q water Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 200 mL of Milli-Q water and mix well to dissolve the Ammonium Acetate. Add 1800 mL of acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.
- B. Standards:

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10 ml Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent. After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All stock, intermediate and spiking solutions are prepared using Methanol.

All initial calibration, initial calibration verification, and linear branched working standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS

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extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 04*.

If the compound purity is assayed to be <mark>98</mark>% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent, i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.

Attachment 05 - Native PFAS Intermediate A Attachment 06 - Native PFAS Intermediate B Attachment 07 - Native PFAS Intermediate M Attachment 08 - Extraction Standard Mix Attachment 09 - Internal Standard Mix Attachment 10 - Initial Calibration Standards Preparation Attachment 11 - Initial Calibration Standards Concentrations Attachment 12 - Bile Salts Stock Solutions Attachment 13 - Bile Salts Working Solution A Attachment 14 - Bile Salts Working Solution B Attachment 15 - PFAS Linear/Branched Mix A Attachment 16 - PFAS Linear/Branched Mix B Attachment 17 - Linear/Branched Bile Salts Solution Attachment 18 - ICV Working Standard

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Calibration

A. Initial Calibration

- A minimum of six calibration standards are required when using an average or linear curve fit. A
 minimum of seven calibration standards are required for a second-order curve fit(quadratic). In
 general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The
 calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and
 NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.
- 2. Analyze a Cal4 level standard that contains Bile Salts retention time marker and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

Example Initial Calibration Sequence:

- 1. Instrument Blank
- 2. Instrument Blank
- 3. Instrument Blank
- 4. CAL 1
- 5. CAL 2
- 6. CAL 3
- 7. CAL 4
- 8. CAL 5
- 9. CAL 6
- 10. CAL 7
- 11. ICB (Instrument Blank)
- 12. ICV

13. MDL

- 14. WDM (Linear Branched/Bile Salts standard)
- 3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 02* for additional information about compound relationships.

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	Louis a tra
3:3FTCA	
PFMPA	13C5-PFPeA
PFMBA	The second se
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
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	PFEESA			
	РҒНрА	13C4-PFHpA		
	PFOA	13C8-PFOA		
	PFNA	13C9-PFNA		
	PFDA	13C6-PFDA		
	PFUnA	13C7-PFUnA		
	PFDoA	13C2-PFDoA		
	PFTrDA	Avg 13C2- PFTeDA and 13C2-PFDoA		
18	PFTeDA	13C2-PFTeDA		
	PFBS	13C3-PFBS		
	PFPeS	1202 0511 0		
	PFHxS	13C3-PFHxS		
	PFHpS			
	PFOS			
	PFNS	13C8-PFOS		
	PFDS			
	PFDoS			
	4:2-FTS	13C2-4:2-FTS		
	6:2-FTS	13C2-6:2-FTS		
	8:2-FTS	13C2-8:2-FTS		
	PFOSA	13C8-PFOSA		
	NMeFOSA	D3-NMeFOSA		
	NEtFOSA	D5-NEtFOSA		
	NMeFOSAA	D3-NMeFOSAA		
	NEtFOSAA	D5-N-EtFOSAA		
	NMeFOSE	D7-NMeFOSE		

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D9-NEtFOSE

13C3-HFPO-DA

NEtFOSE

HFPO-DA

DONA 9CI-PF3ONS

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	11CI-PF3OUdS	

NOTE: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA.

4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic Acid (TCDCA), and Tauroursodeoxycholic Acid (TUDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

5. Fit the curve

- a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.
- b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for the compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.
- c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within $\pm 30\%$ of its true value.
- d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See *Attachment 04*.

6. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must be within $\pm 30\%$ of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

- B. Continuing calibration
 - 1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.
 - 2. Acceptance criteria
 - a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV

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recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

A. Sample Preparation

NOTE: Prior to weighing out samples, thoroughly mix each sample using a wooden tongue depressor or stainless steel spoon to ensure a homogeneous sample matrix. Stir from the bottom to the top in a circular motion along the sides of the jar, breaking particles to less than 1 mm by pressing against the side of the container. Remove rocks, invertebrates, and foreign objects. Vegetation can either be removed or cut into smaller pieces based on project requirements.

- On a calibrated, top-loading balance, accurately weigh 1.25 ± 0.10g (dry weight; note: maximum 10g sample aliquot) (0.5 g for biosolids(dry weight; note: maximum 1g sample aliquot)) of solid sample into a tared, labeled 50-mL centrifuge tube using a disposable polypropylene spatula. Record sample weight in the prep entry system.
- For each batch maximum 20 samples include the following quality control samples:
 a. Method Blank: Weigh 1.25 ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 g for biosolids) of reagent water
 - b. LCS: Fortify 1.25g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 mL for biosolids) reagent water and spiked with 200 μL of Native 1633 Mid-Level Spike Solution (PFAS Native M).
 - c. LLCS: Fortify 1.25g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 mL for biosolids) reagent water and spiked with 200 μL of Native 1633 Low-Level Spike Solution (PFAS Native B).
 - d. Matrix Spike/Matrix Spike Duplicate (MS/MSD): Fortify $1.25g \pm 0.10$ g(dry weight; note: maximum 10g sample aliquot) (0.5 g for biosolids(dry weight; note: maximum 1g sample aliquot)) of sample as specified in sample preparation log with 200 µL of Native 1633 Mid-Level Spike Solution (PFAS Native M).
- Add 25 µl working labeled extraction standard spike solution (PFAS EIS Mix) to each sample/QC tube.
- 4. Cap and vortex for approximately 30 seconds.
- 5. Allow samples/QC to equilibrate for at least 30 minutes.
- 6. Add 10 mL of 0.3% methanolic ammonium hydroxide to each centrifuge tube.
- 7. Cap and vortex
- 8. Shake for 30 minutes on a variable speed mixing table

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- Centrifuge @ 3000 rcf for 10 minutes and transfer supernatant to a clean 50 mL polypropylene centrifuge tube.
- 10. Add 15 mL of 0.3% methanolic ammonium hydroxide to the remaining solid sample in each centrifuge tube. Cap and vortex.
- 11. Shake for 30 minutes on a variable speed mixing table
- 12. Centrifuge @ 3000 rcf for 10 minutes and decant the supernatant from the second extraction into the centrifuge tube with the supernatant from the first extraction.
- 13. Add another 5 mL of 0.3% methanolic ammonium hydroxide to the remaining sample in each centrifuge tube.
- 14. Shake by hand to disperse.
- 15. Immediately decant the supernatant from the third extraction into the centrifuge tube with the supernatant from the first and second extraction.
- 16. Centrifuge for 10 minutes @ 3000 rcf.
- 17. Decant the extract from each tube into a clean, labeled, 125 ml HDPE bottle. Bring the 125mL HDPE container up to 125 mL with Milli-Q water.
- 18. Check that the pH is 4.0 ± 0.5 and adjust as necessary with 50% formic acid or with 5% formic acid. NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.
- 19. Invert container to thoroughly mix.
- B. Solid Phase Extraction (SPE) Manual Sample Extraction
 - 1. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.
 - 2. Condition each SPE cartridge with the following reagents in the following order:
 - a. 15 mL 1% methanolic ammonium hydroxide
 - b. 5 mL 0.3M formic acid
 - c. Discard conditioning eluent(s)
 - 3. Label each sample bottle, cap and reservoir with the same number to insure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).
 - 4. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges. Allow full volume to pass through each cartridge by gravity, if possible. Apply light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.
 - 5. After full volume has passed through the cartridges, dry the cartridges with vacuum no more than 15" of Hg for approximately five minutes. After five minutes, visually inspect the cartridge to determine if the sorbent is dry. This done by comparing the cartridge to a visual standard (an unused SPE cartridge). If the sorbent is not dry, continue to check at one minute intervals until the cartridge is dry.

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- 6. Discard the waste and rinse the waste reservoir with DI water. Wipe each needle with a Kimwipe/methanol.
- 7. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5ml of 1:1 0.1M formic acid/methanol and pass the rinses through the cartridge using vacuum. Dry the cartridge by pulling air through for 15 seconds. Discard the rinse solution.
- 8. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge.
- 9. Rinse the inside of the evaporation/concentrator tube using 5mL of 1% methanolic ammonium hydroxide.
- 10. Using a glass pipette, transfer the rinse to the SPE reservoirs, washing the walls of the reservoirs.
- 11. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.
- 12. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.
- 13. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix thoroughly.
- 14. Add 25 uL of Intenal Standard Spike Solution (PFAS NIS Mix) to each sample extract.
- 15. Bring each sample extract to final volume 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.
- 16. Cap and vortex to mix. Using a syringe, push \sim 3mL of MeOH through each filter and allow to dry
- 17. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 6 mL polypropylene syringe. Take the plunger out and carefully decant ~1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter ~1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.
- 18. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for dilution or reinjection if needed.
- C. Solid Phase Extraction (SPE) Automated Sample Extraction using Promochrom

This procedure can be used in place of the manual extraction described above.

- Verify pH of all samples/QC(needs to be between pH 4.0 ± 0.5). Modify with 50% Formic acid or 30% NH4OH if necessary. (Record the lot number of the acid/base used). NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.
- Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.
- 3. Check to make sure all reagent bottles are filled and that reagents are not expired.

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- Select "Clean sys-004" from the drop down menu to start the cleaning cycle prior to SPE. Make sure the "Clean" cartridges and bottles are attached for this cycle.
- 5. Remove "Clean" cartridges and attach the labeled cartridges containing graphitized carbon that will be used when running the samples. Disconnect the "Clean" bottles. Attach the sample bottle by twisting the bottle and not the cap. Place sample bottle upside down in corresponding numbered location on shaker. Poke two holes into the lip of each bottle. Load labeled centrifuge tubes in the moving tray beneath the cartridges.
- 6. Select "EPA 1633-004" from the drop down menu and press the green check mark to start the procedure. NOTE: N2 drydown should be 15 minutes long.
- 7. Once the cycle is complete, remove centrifuge tubes and discard the used cartridges and bottles. Reattach "Clean" bottles and cartridges, select "clean sys-004" from menu, select "start". This must be done between each batch and at the beginning and the end of the day.
- Add 25ul of Acetic acid to sample extracts and vortex to mix thoroughly before capping tubes.

9. Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to each sample extract.

- 10. Bring each sample extract to final volume 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.
- 11. Cap and vortex to mix. Using a syringe, push ~ 3mL of MeOH through each filter and allow to dry.
- 12. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 3 mL polypropylene syringe. Take the plunger out and carefully decant approximately 1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter approximately 1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.
- 13. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for reinjection if needed.
- D. LC/MS/MS Analysis
 - 1. Mass Calibration and Tuning
 - a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.
 - b. When masses fall outside of the ±0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.
 - 2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

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- 3. Example acquisition method: See Attachment 03. Mass Transitions: See Attachment 01.
- Instrument Sensitivity Check (ISC) and Instrument Blanks
 - a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within $\pm 30\%$ of their true value. The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
 - b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
- 5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.
- After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salts RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

Example Sample Sequence:

- Instrument blank
 Instrument blank
- 3. Instrument blank
- 4. Instrument Sensitivity Check (CCV0 _CAL1)
- 5. CCV 2_CAL4
- 6. Linear Branched/ Bile Salt marker (WDM)
- 7. Instrument Blank (ICB)
- 8. Method Blank (MB)
- 9. Low Level LCS (LLCS)
- 10. LCS
- 11. Sample (10 or less)
- 12. CCV 3 CAL4
- 13. Instrument Blank
- 7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

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Document number: PFAS-SOP63700	Solid Samples by LC-M3/M3 Using Drait Method 1033	Operating Procedure	
Old Reference:			
Version: 2		Organisation level: 5-Sub-BU	
Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS AII	Responsible: CLE PFAS All	

- 8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.
- 9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.
 - a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.
 - b. If LLCS and LCS recoveries are acceptable, proceed to sample quantitation.
 - c. If the LCS and LLCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS/LLCS must be reextracted.
 - d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.
- 10. Calculate the relative percent difference(RPD) between the sample and the matrix duplicate(DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data
- 11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), revial a fresh aliquot and reanalyze. If the extraction standard recovery(ies) are still outside the QC limit(s), reanalyze the sample at a dilution. If the extraction standard recovery(ies) are still outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) are still outside the QC limit(s), narrate with an NCM and report the data.
- 12. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.
- 13. Compare the retention times of all of the analytes, surrogates, and internals standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than
 - a. 0.4 minutes for isotopically-labeled compounds
 - b. 0.1 minutes from their analog for native compounds with an exact isotopicallylabeled compound
 - c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

Comparison of the second secon	Always check on-line for validity. Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633	Level: Standard Operating Procedure
2		5-Sub-BU
Approved by: HWD4 Effective Date; 20- NOV-2023	Document users: CLE PFAS AII	Responsible: CLE PFAS All

If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

- 14. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of 13C4-PFBA, 13C5-PFPeA, 13C4-PFHpA, 13C8-PFOA, 13C9-PFNA, 13C6-PFDA, 13C7-PFUnA, 13C2-PFDA, 13C2-PFDoDA 13C2-PFTeDA, 13C8-PFOSA, D3-NMePFOSA, D5-NEtFOSAA, D3-NMeFOSAA, D5-NEtPFOSA, D7-NMePFOSAE, D9-NEtPFOSAE, 13C3-PFBA, 13C4-PFOA, 13C5-PFNA, 13C2-PFOA, 18O2-PFHxS, PFBA, PFECA F(PFMPA), PFECA A(PFMBA), NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required in prepped samples. Ion ratios must be in control in calibration solutions. If they are outside of limits, stop the analysis and correct the issues.
- 15. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.
- 16. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of Internal Standard Spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring greater than a 10x dilution should be reextracted using a reduced aliquot.

Dilution Example 1/10: Mix 895 µl of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 µl of sample extract and 5 uL of Internal Standard Spike solution. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio

Peak Area Ratio = Analyte Response Labeled Analyte Response

2. On-Column Analyte Concentration using average RRF

On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

On-column Concentration = (peak area ratio - intercept) ÷ slope

4. Sample Concentration

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Document number: PFAS-SOP63700	Sond Samples by LC-MS/MS Using Drait Method 1055	Operating Procedure
Old Reference:		
Version: 2		Organisation level: 5-Sub-BU
Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS All	Responsible: CLE PFAS All

Sample concentration (ng/g) = (On-column concentration x Final Sample Volume x DF) \div Initial Sample Volume

5. Ion Ratio

ion ration = (peak area or height of quantifier)/(peak area or height of qualifier)

See cross-referenced SOPs for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are method defined.

QC parameter	Lower acceptance limit	High acceptance limit
Extracted Internal standard (EIS)	20%	150%
Non-extracted Internal Standard (NIS)	>30% of the average NIS from the initial calibration	200%
Analyte recoveries LCS/LLCS/MS/MSD	40%	150%
Sample/Duplicate	= 30%</td <td>NA</td>	NA

Note: lower acceptance limit for EIS cannot not be <20%, lower acceptance limit for analyte recovery cannot be <40%.

Records of the standard deviation of the percent recovery (SR) of EIS and NIS compounds from samples must be maintained and should be assessed periodically. It is recommended that this assessment is done quarterly. The assessment should be expressed as the average percent recovery +/- 2SR for each isotopically labeled compound, and evaluated each quarter for changes.

Historical data for MS/Ds, LCSs, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to the SOP for Determining Method Detection Limits and Limits of Quantitation for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank(sand), an LCS/LLCS (sand spiked with all compounds to be determined carried through the entire procedure), and a sample/Dup, must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

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Document number: PFAS-SOP63700		Operating Procedure
Old Reference:		0.5.
Version: 2		Organisation level: 5-Sub-BU
Approved by: HWD4 Effective Date: 20- NOV-2023	Document users: CLE PFAS AII	Responsible: CLE PFAS All

Attachment:
Attachment 01 - Mass Transitions (.doc)
Attachment 02 - Standard Relationships (.docx)
Attachment 03 - Acquisition Parameters (.pdf)
Attachment 04 - Example Certificate of Analysis (.pdf)
Attachment 05 - Native PFAS Intermediate A (.pdf)
Attachment 06 - Native PFAS Intermediate B (.pdf)
Attachment 07 - Native PFAS Intermediate M (.pdf)
Attachment 08 - Extraction Standard Mix (.pdf)
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Attachment 12 - Bile Salt Stock Solutions (.pdf)
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Attachment 14 - Bile Salts Working Solution B (.pdf)
Attachment 15 - PFAS Linear Branched Mix A (.pdf)
Attachment 16 - PFAS Linear Branched Mix B (.pdf)
Attachment 17 - Linear Branched Bile Salts Solution (.pdf)
Attachment 18 - ICV Working Standard (.pdf)

Attachment: Attachment 01 - Mass Transitions (doc)
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Attachment: Attachment 16 - PFAS Linear Branched Mix B (pdf)
Attachment: Attachment 17 - Linear Branched Bile Salts Solution (pdf)
Attachment: Attachment 18 - ICV Working Standard (pdf)

End of document

Version history

Version	Approval	Revision information
0	25.SEP.2023	
1	03.NOV.2023	
2	20.NOV.2023	

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Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1 =	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

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Compound	Parent Ion	Daughter Ion
13C8-PFOA	421.1	376.0
PFOA	413.0	369.0
PFOA (2)	413.0	169.0
13C4-PFOS	502.8	79.9
13C4-PFOS (2)	502.8	98.9
13C8-PFOS	507.1	79.9
13C8-PFOS (2)	507.1	98.9
PFOS	498.9	79.9
PFOS (2)	498.9	98.8
13C5-PFNA	468.0	423.0
13C9-PFNA	472.1	427.0
PFNA	463.0	419.0
PFNA (2)	463.0	219.0
13C8-PFOSA	506.1	77.8
PFOSA	498.1	77.9
PFOSA (2)	498.1	478.0
PFNS	548.8	79.9
PFNS (2)	548.8	98.8
13C2-PFDA	515.1	470.1
13C6-PFDA	519.1	474.1
PFDA	512.9	469.0
PFDA (2)	512.9	219.0
13C2-8:2-FTS	529.1	80.9
13C2-8:2-FTS (2)	529.1	509.0
8:2-FTS	527.1	507.0
8:2-FTS (2)	527.1	80.8
d7-NMePFOSAE	623.2	58.9
NMePFOSAE	616.1	58.9
d3-NMePFOSA	515.0	219.0
NMEPFOSA	511.9	219.0
NMEPFOSA (2)	511.9	169.0
d3-NMeFOSAA	573.2	419.0
NMeFOSAA	570.1	419.0
NMeFOSAA (2)	570.1	483.0
d9-NEtPFOSAE	639.2	58.9
NEtPFOSAE	630.0	58.9
d5-NETPFOSA	531.1	219.0
NEtPFOSA	526.0	219.0
NEtPFOSA (2)	526.0	169.0
PFDS	599.0	79.9

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Attachment "Attachment 02 - Standard Relationships" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples Page 24 of 84 by LC-MS/MS Using Draft Method 1633 "

Compound	Parent Ion	Daughter Ion
PFDS (2)	599.0	98.8
13C7-PFUnDA	570.0	525.1
PFUnDA	563.1	519.0
PFUnDA (2)	563.1	269.1
d5-NEtFOSAA	589.2	419.0
NEtFOSAA	584.2	419.1
NEtFOSAA (2)	584.2	526.0
13C2-PFDoDA	615.1	570.0
PFDoDA	613.1	569.0
PFDoDA (2)	613.1	319.0
PFDoS	699.1	79.9
PFDoS (2)	699.1	98.8
PFTrDA	663.0	619.0
PFTrDA (2)	663.0	168.9
13C2-PFTeDA	715.2	670.0
PFTeDA	713.1	669.0
PFTeDA (2)	713.1	168.9
13C3-HFPODA	286.9	168.9
13C3-HFPODA (2)	286.9	184.9
HFPODA	284.9	168.9
HFPODA (2)	284.9	184.9
DONA	376.9	250.9
DONA (2)	376.9	84.8
9CI-PF3ONS	530.8	351.0
9CI-PF3ONS (2)	532.8	353.0
11Cl-PF3OUdS	630.9	450.9
11CI-PF3OUdS (2)	632.9	452.9
PFECA B (NFDHA)	295.0	201.0
PFECA B(NFDHA) (2)	295.0	84.9
PFECA F (PFMPA)	229.0	84.9
3:3 FTCA	241.0	177.0
3:3 FTCA (2)	241.0	117.0
PFECA A (PFMBA)	279.0	85.1
PFEESA (PES)	314.8	134.9
PFEESA (PES) (2)	314.8	82.9
5:3 FTCA	341.0	237.1
5:3 FTCA (2)	341.0	217.0
7:3 FTCA	441.0	316.9
7:3 FTCA (2)	441.0	336.9

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

Inj Std	Internal Standard/Injection Standard
I13C3-PFBA	13C3-PFBA
II3C2-PFHxA	13C2-PFHxA
I13C4-PFOA	13C4-PFOA
I13C5-PFNA	13C5-PFNA
I13C2-PFDA	13C2-PFDA
I18O2-PFHxS	18O2-PFHxS
I13C4-PFOS	13C4-PFOS

Extraction Standards

Extraction Standard	Internal Standard			
E13C4-PFBA	13C3-PFBA			
E13C5-PFPeA				
E13C5-PFHxA				
E13C4-PFHpA	13C2-PFHxA			
E13C3-HFPO-DA				
E13C8-PFOA	13C4-PFOA			
E13C9-PFNA	13C5-PFNA			
E13C6-PFDA				
E13C7-PFUnA				
E13C2-PFDoA	13C2-PFDA			
E13C2-PFTeDA	1			
E13C3-PFBS				
E13C3-PFHxS				
E13C2-4:2-FTS	18O2-PFHxS			
E13C2-6:2-FTS]			
E13C2-8:2-FTS				

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Attachment "Attachment 04 - Example Certificate of Analysis" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Page 26 of 84 Samples by LC-MS/MS Using Draft Method 1633 "

Extraction Standard	Internal Standard
E13C8-PFOS	
E13C8-PFOSA	
Ed3-NMeFOSA	
Ed5-NEtFOSA	13C4-PFOS
Ed3-NMeFOSAA	1304-1103
Ed5-NEtFOSAA	
Ed7-NMeFOSE	$\alpha = 1$
Ed9-NEtFOSE	

Native PFAS Compounds

Native	Extraction Standard			
PFBA	13C4-PFBA			
PFPeA				
3:3FTCA	13C5-PFPeA			
PFMPA	ISCS-FFFEA			
PFMBA				
PFHxA				
NFDHA				
5:3FTCA	13C5-PFHxA			
7:3FTCA				
PFEESA				
PFHpA	13C4-PFHpA			
PFOA	13C8-PFOA			
PFNA	13C9-PFNA			
PFDA	13C6-PFDA			
PFUnA	13C7-PFUnA			
PFDoA	13C2-PFDoA			
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA			
PFTeDA	13C2-PFTeDA			
PFBS	13C3-PFBS			
PFPeS	12C2 DEU-S			
PFHxS	13C3-PFHxS			
PFHpS				
PFOS				
PFNS	1308-1708			
PFDS	7			

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Native	Extraction Standard		
PFDoS			
4:2-FTS	13C2-4:2-FTS		
6:2-FTS	13C2-6:2-FTS		
8:2-FTS	13C2-8:2-FTS		
PFOSA	13C8-PFOSA		
NMeFOSA	D3-NMeFOSA		
NEtFOSA	D5-NEtFOSA		
NMeFOSAA	D3-NMeFOSAA		
NEtFOSAA	D5-N-EtFOSAA		
NMeFOSE	D7-NMeFOSE		
NEtFOSE	D9-NEtFOSE		
HFPO-DA			
DONA			
9C1-PF3ONS	13C3-HFPO-DA		
11Cl-PF3OUdS	1		

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Attachment "Attachment 07 - Native PFAS Intermediate M" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 * Printed by Donnthy I now rd Mon 20 Nov 2023 15:26 FST

			JARM JARM ntegrated Valve C System Equilibrate Equilibrate Injection	Acquisition Method 633 ass Spec 12.500 min Period 12.500 min
(Da) <u>Q</u> 3 Mass (Da) 177.000	Q1 Mass (Da) Q3 Mass (Da) RT (n 212.800 168.900 2.71 Q1 Mass (Da) Q3 Mass (Da) RT (n 229.000 84.900 2.94	Scheduled MRM:YesPolarity:NkgativeScan Mode:NNAIon Source:Turbo SpraysMRM OLO3 Resolution:NoMRM detection window:60 secMRM detection window:60 secMRM detection of the sec0.6000 secResolution Q3:UhitIntensity Thres.:0.000 cpsSettling Time:0.0000 msecMR Pause:5.0070 msecMCA:NoStep Size:0.00 Da	ine: tion: tion: eriod: Pine: e: 11me: 1	Mass Spectrometer Method Properties
nin) Param	nin) Paran DF DF DF	e pray nsec s	5 8	rties
-25.00 -25. Stop: ID -50.00 -12.00 -12.	Start Stop ID -40.00 -40.00 PFBA CE -14.00-14.00 Start Stop ID -40.00 PFFFA F			

Q1 Mass (Da) 313.000 01 Mass (Da) 327.100 01 Mass (Da) 263.000 Q1 Mass (Da) 295.000 Q1 Mass (Da) 314.800 Q1 Mass (Da) 279.000 Q1 Mass (Da) 298.700 Q1 Mass (Da) Q3 Mass (Da) 03 Mass (Da) 269.000 03 Mass (Da) 307.000 Q3 Mass (Da) 219.000 Q3 Mass (Da) 201.000 <u>Q</u>3 Mass (Da) 134.900 Q3 Mass (Da) 85.100 Q3 Mass (Da) 79.900 RT (min) 3.55 RT (min) 3.41 RT (min) 3.52 RT (min) 3.67 RT (min) 3.21 RT (min) Paran Start Stop RT (min) 3.84 RT (min) 3.32 Param Start Stop ID DF -30.00 -30.00 PFHxA CE -15.00-15.00 Param Start Stop ID DF -110.00 -110.00 4:2-FTS CE -28.00 -28.00 Paran DF Paran DF Paran DF Paran DE Paran DE Start Stop ID -30.00 -30.00 PFECA B CE -25.00 -25.00 Start Stop ID -70.00 -70.00 PREESA CE -30.00-30.00 Start Stop ID -40.00 -40.00 PEECA A CE -20.00 -20.00 Start Stop ID -40.00 -40.00 PFPeA CE -14.00-14.00 Start Stop ID -90.00 -90.00 PFBS CE -65.00-65.00 Ĥ

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01 Mass (Da) 449.000	01 Mass (Da) 427.100	Q1 Mass (Da) 341.000	01 Mass (Da) 376.900	Q1 Mass (Da) 398.700	Q1 Mass (Da) 363.100	01 Mass (Da) 284.900	349.100 79.900 4.00
) Q3 Mass (Da) 79.900	 Q3 Mass (Da) 407.000) Q3 Mass (Da) 237.100) Q3 Mass (Da) 250.900) Q3 Mass (Da) 79.900) Q3 Mass (Da) 319.000) Q3 Mass (Da) 168.900	900 4.00 DF
 ») RT (min) 4.68 	a) RT (min) 3.98	a) RT (min) 3.75	a) RT (min) 3.96	a) RT (min) 4.3C	a) FT (min) 3.85	a) RT (min) 3.68	₩ -90.00
Paran	Paran DF	Paran DF	Paran Df	Partan DE	Paran DF	Paran DF	-90.00 -70.00
ar Start Stop ID -110.00 -110.00 PEHpS CE -90.00-90.00	arr Start Stop; ID -90.00 -90.00 6:2-FTS CE -35.00 -35.00	ar Start Stop ID -70.00 -70.00 5:3 FTCA CE -20.00-20.00	Mr Start Stop ID -40.00 -40.00 DOMA CE -20.00 -20.00	ar Start Stop ID -100.00 -100.00 PFHxS CE -80.00 -80.00	nr Start Stop ID -40.00 -40.00 PFHpA CE -15.00-15.00	ar Start Stop ID -20.00 -20.00 HEPODA CE -10.00-10.00	.00 PFFeS .00 -70.00

Q1 Mass (Da) 512.900 Q1 Mass (Da) 548.800 01 Mass (Da) 498.900 Q1 Mass (Da) 413.000 01 Mass (Da) 530.800 Q1 Mass (Da) 441.000 Q1 Mass (Da) 463.000 Q3 Mass (Da) 469.000 Q3 Mass (Da) 79.900 Q3 Mass (Da) 351.000 Q3 Mass (Da) 316.900 Q3 Mass (Da) 419.000 <u>Q</u>3 Mass (Da) 79.900 Q3 Mass (Da) 369.000 RT (min) 4.85 RT (min) 5.61 RT (min) 4.12 RT (min) 5.72 RT (min) 4.40 RT (min) 5.22 RT (min) 4.43 Paran Start Stop ID DF -60.00 -60.00 PFDA CE -18.00-18.00 Paran DF Paran DF Paran DF Paran DF Paran DE Paran Start Stop ID DF -100.00 -100.00 9Cl-PF3CNS CE -38.00 -38.00 Start Stop ID -100.00 -100.00 CE -110.00 Start Stop ID -90.00 -90.00 CE -110.00 Start Stop ID -80.00 -80.00 7:3 FICA CE -20.00 -20.00 Start Stop ID -50.00 -50.00 PENA CE -18.00-18.00 Start Stop ID -50.00 -50.00 PFOA CE -16.00-16.00 PENS -110.00 PFOS -110.00

Attachment "Attachment 11 - Initial Calibration Standard Concentrations" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalky! Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method Page 33 of 84 16:33

$ \begin{array}{c} (3) \ \mbox{Mess} \ \ (30) \ \ \mbox{Mess} \ \ \ (30) \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \$										
Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame -11000 -10000 CE -42.00 -42.0 Q3 Mass (Da) RT (min) Parame Start Stop: ID CE -42.00 -42.0 Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame Start Stop: ID Q3 Mass (Da) RT (min) Parame Start St	Q1 Mass 613,100	2	01 Mass 630.900	Q1 Mass 584.200	Q1 Mass 563.100	Q1 Mass 599.000	01 Mass 570,100	498.100	O1 Mass	Q1 Mass 527.100
	(Da)		(Da)	(Da)	(Da)	(Da)	(Da)			(Da)
Param Start -100.00 -100.00 cf ID -42.00 -42.0 -42.00 Param Start -110.00 -110.00 cf Stop -80.00 -80.00 -80.00 -80.00 cf ID -80.00 -80.00 -80.00 -80.00 -27.00 -27.00 -27.00 -27.00 -27.00 -27.00 cf Param Start -70.00 -70.00 cf Stop -130.00 -19.00 -90.00 -30.00 -90.00 cf ID -90.00 -90.00 -90.00 -90.00 cf Param Start -90.00 -90.00 cf Stop -130.00 -30. Param Start -90.00 -90.00 cf Stop -30.00 -30. Param Start -90.00 -90.00 cf Stop -43.00 -43. Param Start -60.00 -60.00 cf Stop -20.00 -20.				Q3 Mass (Da) 419.100		<u>0</u> 3 Mass (Da) 79,900				03 Mass 507.000
Start Stop ID -100.00 -100.00 CE -42.00 -42.0 -110.00 -110.00 CI -80.00 -80.00 CE -80.00 -80.00 CE -77.00 -27.00 -27. Start Stop ID -90.00 -90.00 CE -130.00 CE -130.00 CE -30.00 -90.00 CE -30.00 -30. Start Stop ID -90.00 -90.00 CE -30.00 -30. Start Stop ID -90.00 -90.00 CE -30.00 -20.00 CE -20.00 -20.00	RT (min) 5.87		RT (min) 6.49	RT (min) 5.04	RT (min) 5.39	RT (min) 6.16	RT (min) 4.79	6.14	RT (min)	FC (min) 4.58
Stop ID -42.00-42.0 -42.00-42.1 Stop ID -80.00-80.1 -80.00-80.1 -70.00-27.0 -70.00 -130.00 -130.00 -19.00-19. -30.00-30. -30.00-30. -43.00-43.0 -20.00-20.1	Paran DF		Paran DE	Paran Df	Paran DF	Paran DF	Paran DF	무	Paran	Paran DF
	Start Stop ID -60.00 -60.00 CE -20.00-20.		Start Stop ID -90.00 -90.00 CE -43.00 -43.	Start Stop ID -90.00 -90.00 CE -30.00 -30.	Start Stop ID -70.00 -70.00 CE -19.00-19	Start Stop ID -90.00 -90.00 CE -130.00	Start Stop ID -80.00 -80.00 CE -27.00-27.	-110.00 -110.00 CE -80.00 -80.	Start Stor	Start Stop ID -100.00-100.00 CE -42.00-42.

Q1 Mass (Da) 526.000 Q1 Mass (Da) 713.100 Q1 Mass (Da) 663.000 Q1 Mass (Da) 630.000 Q1 Mass (Da) 699.100 01 Mass (Da) 511.900 Q1 Mass (Da) 616.100 Q3 Mass (Da) 669.000 Q3 Mass (Da) 619.000 Q3 Mass (Da) 219.000 Q3 Mass (Da) 58.900 03 Mass (Da) 79.900 Q3 Mass (Da) 219.000 <u>Q</u>3 Mass (Da) 58.900 RT (min) 6.3C RT (min) 7.36 RT (min) 6.94 RT (min) Param Start Stop ID 6.71 DF -60.00 -60.00 PFTEDA CE -22.00-22.00 RCT (min) 7.24 RT (min) 7.08 RT (min) 6.94 Paran DF Paran Start Stop ID DF -60.00 -60.00 PFTrDA CE -21.00-21.00 Paran DF Param Start Stop ID DF -45.00 -45.00 NELPFOSAE CE -70.00-70.00 Paran DE Pairan DF Start Stop ID -100.00 -100.00 CE -150.00 Start Stop ID -90.00 -90.00 NELPFOSA CE -38.00 -38.00 Start Stop ID -90.00 -90.00 NMEPFOSA CE -37.00 -37.00 Start Stop ID -50.00 -50.00 NMEPFOSAE CE -70.00 -70.00 PEDoS -150.00

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Attachment "Attachment 13 - Bile Salts Working Solution A" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 = Printed by Dom/hv I over d Mon 20 Nov 2023 15-26 FST Page 35 of 84

01 Mass	01 Mass	Q1 Mass	01 Mass	Q1 Mass	01 Mass	01 Mass	01 Mass
398.700	363,100	284.900	349.100	313.000	327.100	295.000	298,700
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass (Da)
98.900	169,000	184.900	98,900	118.900	80.900	84,900	98.800
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	
RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (miri)	RT (min)	RT (min)
4.30	3.85	3.68	4.0C	3.55	3.41	3.52	3.66
Paran	Paran	Param	Paran	Paran	Paran	Paran	Param
Df	DF	DF	DF	DF	DF	DF	DF
Start Stop ID -90.00 -90.00 PFHxS 2	Start Stop ID -50.00 -50.00 PFHpA_2 CE -25.00-25.00	Start Stop ID -75.00 -75.00 HETODA_2 CE -10.00-10.00	Start Stop ID -110.00 -110.00 PFFes_2 CE -40.00 -40.00	Start Stop ID -40.00 -40.00 PFHxA_2 CE -31.00-31.00	Start Stop ID -100.00 -100.00 4:2 FTS_2 CE -50.00 -50.00	Start Stop ID -10.00 -10.00 PFECA B_2 CE -35.00-35.00	Start Stop ID -110.00 -110.00 PFBS 2 CE -35.00-35.00

Start Stop ID -100.00 -100.00 PENS 2 CE -90.00 -90.00	Paran DF	RT (min) 5.72	s (Da)	Q3 Mass 98.800	(Da)	Q1 Mass 548.800
Start Stop ID -50.00 -50.00 PENA_2 CE -30.00-30.00	Paran DF	RT (min) 4.43	s (Da) 0	Q3 Mass 219.000	(Da)	01 Mass 463.000
Start Stop ID -110.00-110.00 PFCS_2 CE -80.00-80.00	Paran DF	RT (min) 5.21	s (Da)	Q3 Mass 98.800	(Da)	Q1 Mass 498.900
Start Stop ID -50.00 -50.00 PROA_2 CE -26.00-26.00	Paran DF	RT (min) 4.12	s (Da) 0	Q3 Mass 169.000	(Da)	Q1 Mass 413.000
Start Stop ID -100.00-100.00 PETApS_2 CE -70.00-70.00	Paran Df	RT (min) 4.68	s (Da)	Q3 Mass 98.800	(Da)	Q1 Mass 449.000
Start Stop ID -120.00-120.00 6:2 FTS_2 CE -70.00-70.00	Paran DF	RT (min) 3.98	s (Da)	<u>0</u> 3 Mass 80.900	(Da)	01 Mass 427.100
Start Stop ID -80.00 -80.00 5:3 FTCA_2 CE -20.00-20.00	Paran DF	RT (min) 3.75	s (Da) C	Q3 Mass 217.000	(Da)	Q1 Mass 341.000
0 -70.00	-70.00	R				

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Q1 Mass (Da)	Q1 Mass (Q1 Mass (584.200	Q1 Mass (563.100	01 Mass (01 Mass (570.100	Q1 Mass (527.100	01 Mass (Da) 512,900	
	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)		
Q3 Mass (Da)	03 Mass (319.000	Q3 Mass (526.000	03 Mass (269,100	03 Mass (98.800	Q3 Mass (483.000	Q3 Mass (80,800	Q3 Mass (219.000	
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	
RT (min)	RT (min) 5.87	RT (min) 5.04	RT (min) 5.39	RT (min) 6.16	RT (min) 4.79	RT (min) 4.58	RT (min) 4.85	
Paran	Paran DF	Paran DF	Paran DF	Paran DE	Paran DF	Param DF	Paran D£	
Start Stop ID	Start Stor ID -60.00 -66.00 PETDOA 2 CE -38.00 -38.00	Start Stop ID -100.00 -100.00 NEtFOSAA_2 CE -30.00 -30.00	Start Stop: ID -70.00 -70.00 PEUNDA_2 CE -35.00 -35.00	Start Stop ID -100.00 -100.00 PFNS_2 CE -100.00 -100.00	Start Stop ID -90.00 -90.00 NMARTOSAA_2 CE -24.00 -24.00	Start Stop ID -100.00-100.00 8:2 FTS_2 CE -90.00-90.00	Start Stop ID -60.00 -60.00 PFDA_2 CE -31.00-31.00	

478.000 6.13 DF (Da) Q3 Mass (Da) RC (min) Param 169.000 7.08 DF	Q1 Mass 713.100Q3 Mass 168.900(Da)RT 6.71Param DFQ1 Mass 263.000(Da)Q3 Mass 68.900(Da)RT 	663.000 168.900 6.30 DÆ -60.00 CE -40.00
-130.00 -130.00 PFCSA_2 CE -35.00-35.00 Start Stop ID -110.00 IMMEPECSA_2 CE -37.00-37.00	Start Stop: ID -60.00 -60.00 PFTEDA_2 CE -40.00 -40.00 Start Stop: ID -40.00 -40.00 PFTEDA_2 CE -55.00 -55.00	PETEDA_2 -40.00

Attachment "Attachment 17 - Linear Branched Bile Salts Solution" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 Page 39 of 84 Printed by Darothy Love d Man 20 New 2023 15:26 EST

	Q1 Mass (Da) 216.800	Q1 Mass (Da) 216.000	01 Mass (Da) 699.100	<u>0</u> 1 Mass (Da) 314,800	Q1 Mass (Da) 441.000	Q1 Mass (Da) 241.000	Q1 Mass (Da) 632.900	
В) Q3 Mass (Da) 171.900) Q3 Mass (Da) 172.000) Q3 Mass (Da) 98.800) Q3 Mass (Da) 82.900) <u>0</u> 3 Mass (Da) 336,900) Q3 Mass (Da) 117.000) Q3 Mass (Da) 452.900	
	RT (min) 2.71	RT (min) 2.71	RT (min) 6.94	RT (min) 3.83	RT (min) 4.4C	RT (min) 2.98	RT (min) 6.48	
	Paran DF	Paran DF	Paran DE	Paran Df	Paran DF	Paran Df	Paran DE	
	Start Stop ID -40.00 -40.00 13C4-PFBA CE -14.00-14.00	Start Stop ID -40.00 -40.00 13C3-PFBA CE -14.00-14.00	Start Stop ID -90.00 -90.00 PEDoS 2 CE -140.00 -140.00	Start Stop ID -70.00 -70.00 PFEESA 2 CE -30.00-30.00	Start Stop ID -80.00 -80.00 7:3 FTCA_2 CE -20.00-20.00	Start Stop ID -50.00 -50.00 3:3 FICA_2 CE -45.00 -45.00	Start Stop ID -90.00 -90.00 11cl-PF300aS_2 CE -40.00 -40.00	

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Q1 Mass (Da) 402.100 Q1 Mass (Da) 367.100 Q1 Mass (Da) 318.000 Q1 Mass (Da) 286.900 Q1 Mass (Da) 315.100 Q1 Mass (Da) 329.100 01 Mass (Da) 302.100 01 Mass (Da) 268.300 Q3 Mass (Da) 79.900 03 Mass (Da) 322.000 <u>Q</u>3 Mass (Da) 270.000 Q3 Mass (Da) 168.900 Q3 Mass (Da) 273.000 <u>Q</u>3 Mass (Da) 80.900 Q3 Mass (Da) 79.900 Q3 Mass (Da) 223.000 RT (min) 3.85 RT (min) 3.66 RT (min) Param Start Stop ID 4.3C DF -100.00-100.00 13C3-PFHbS RT (min) 3.68 RT (min) 3.54 RT (min) 3.41 RT (min) 3.21 RT (min) 3.54 Paran DF Paran Paran DE Paran DF Paran DF Paran Paran DF 뜄 R Start Stop ID -40.00 -40.00 13C4-PF1pA CE -15.00 -15.00 Start Stor ID -20.00 -20.00 13C3-HEPODA CE -10.00-10.00 Start Stop ID -30.00 -30.00 13CS-PFHxA CE -15.00-15.00 Start Stop ID -30.00 -30.00 130 CE -15.00 -15.00 Start Stop ID -100.00 -100.00 13C2-4:2-FTS CE -28.00 -28.00 A -90.00 -90.00 Start Stop ID -40.00 -40.00 130 CE -14.00 -14.00 A -80.00-80.00 -65.00-65.00 13CS-PFPeA 13C2-PFHMA 13C3-PEBS

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Q1 Mass	01 Mass	Q1 Mass	01 Mass	Q1 Mass	<u>0</u> 1 Mass	Q1 Mass	
472.100	507.100	502.800	421.100	417.100	429.100	403.000	
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	
Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	Q3 Mass	
427.000	79.900	79.900	376.000	172.000	80.900	83,900	
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	
RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	
4.42	5.21	5.21	4.12	4.12	3.97	4.30	
Paran	Paran	Paran	Paran	Paran	Paran	Paran	
DF	DF	DF	DE	DE	DF	DF	
Start Stop ID	Start Stop ID	Start Stop ID	Start Stop ID	Start Stop ID	Start Stop ID	Start Stor ID	
-50.00 13C9-PPNA	-90.00 -90.00 1308-PFCS	-90.00 -90.00 13C4-PFCS	-50.00 13C8-PFCA	-50.00 -55.00 13C4-PFCA.	-90.00 -90.00 13C2-6:2-FTS	-100.00 -100.00 1802-PFHAcs	
CE -18.00 -18.00	CE -110.00 -110.00	CE -110.00 -110.00	CZ -16.00 -16.00	CE -16.00-16.00	CE -35.00-35.00	CE -80.00 -80.00	
					0		

Q1 Mass (Da) 589.200 01 Mass (Da) 570.000 Q1 Mass (Da) 573.200 Q1 Mass (Da) 506.100 01 Mass (Da) 529.100 Q1 Mass (Da) 515.100 Q1 Mass (Da) 519.100 Q1 Mass (Da) 468.000 Q3 Mass (Da) 419.000 03 Mass (Da) 525.100 03 Mass (Da) 80.900 03 Mass (Da) 474.100 Q3 Mass (Da) 419.000 03 Mass (Da) 470.100 Q3 Mass (Da) 77.800 Q3 Mass (Da) 423.000 RT (min) 5.02 RT (min) 4.78 RT (min) 4.85 RT (min) 5.38 RT (min) 6.13 RT (min) 4.58 RT (min) 4.85 RCT (min) 4.42 Paran Start Stop ID DF -90.00 -90.00 Paran Start Stor ID DF -70.00 -70.00 13C7-PFUnDA CE -19.00-19.00 Paran DF Paran DF Paran Paran DE Paran Df Paran DF Ŗ Start Stop ID -50.00 -50.00 13CS CE -18.00 -18.00 Start Stop ID -90.00 -90.00 d3-NMEFOSAA CE -27.00 -27.00 Start Stop ID -100.00 -100.00 13C2-8:2-FTS CE -42.00-42.00 Start Stop ID -60.00 -60.00 13C2-PFDA CE -18.00 -18.00 Start Stor ID -110.00 -110.00 13C8-PFOSA CE -80.00 -80.00 Start Stop ID -60.00 -60.00 130 CE -18.00 -18.00 13C6-PFDA 13CS-PFNA d5-NEtFOSAA

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01 Mass 318.000	01 Mass 715.200	01 Mass 531.100	Q1 Mass 639.200	Q1 Mass 515.000	01 Mass 623.200	Q1 Mass (Da) 615.100	
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	
Q3 Mass 120.300	Q3 Mass 670.000	03 Mass 219.000	03 Mass 58.900	03 Mass 219.000	Q3 Mass 58,900	<u>Q</u> 3 Mass 570.000	
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	
RT (min) 3.54	RT (min) 6.70	RT (min) 7.34	RT (min) 7.21	RT (min) 7.07	RT (min) 6.92	RT (min) 5.86	R
Paran DF	Paran DF	Paran DF	Paran Df	Paran DF	Param DF	Paran Df	-30.00
Start -40.00 CE	Start -60.00	Start -90.00	Start -45.00 CE	Start -90.00 CE	Start -50.00 CE	Start -60.00 Œ	0 -30.00
Stor ID 40.00 13 31.0031.00	Stop: ID -60.00 13 -22.00-22.00	Stop ID -90,00 d5 -38,00 -38,00	Stop: ID -45.00 d9 -70.00 -70.00	Stop: ID -90.00 d3 -37.00-37.00	Stop ID -50,00 d7 -70,00-70.00	Stop: ID -60.00 13 -20.00-20.00	0
1305-PFH24_2	1302-PFTEDA 2.00	d5-NELPFOSA	d9-Netprosae).00	d3-NMEPFOSA 1.00	d7-NMEPFOSAE).00	1362-FFDDDA 1.00	

Q1 Mass (1	01 Mass (1	Q1 Mass (1	01 Mass (1	01 Mass (1	Q1 Mass ()	01 Mass ()
286,900	529.100	429.100	329.100	507.100	402.100	302.100
	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)
Q3 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	Q3 Mass (De)	0,3 Mass (Da)	03 Mass (Da)
184,900	509.000	409.000	309.000	98,900	98.800	98.900
i) RT (min)	4.58 (mí.n)	a) RT (min)	a) RT (min)	a) RT (min)	3) RT (min)	a) RT (min)
3.68		3.97	3.41	5.20	4.29	3.66
) Paran) Paran) Paran	i) Pazan	i) Paran	i) Paran	ı) Paran
DF	DE	Df	DF	DF	DF	DF
Start Stop ID	Start Stop ID	Start Stop ID	Start Stop ID	Start Stop ID	r Start Stop ID	r Start Stop ID
-75.00 -75.00 13C3-HEPODA 2	-100.00 -100.00 1302-8:2-FTS 2	-100.00 13C2-6:2-FTS_2	-100.00 1302-4:2-FIS_2	-110.00 -110.00 13C8-PROS 2	-90.00 -90.00 13C3-PFHAS_2	-110.00 -110.00 13C3-PFBS 2
CE -10.00-10.00	CE -42.00 -42.00	CE -35.00-35.00	CE -28.00-28.00	CE -80.00 -80.00	CE -70.00-70.00	CE -35.00 -35.00

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	Parameter Table (Period 1 CUR: 35.00 IS: -3000.00 TEM: 450.00 CS2: 50.00 CAD: 10.00 EF -10.00	Q1 Mass (Da) 498.200	Q1 Mass (Da) 498.100	01 Mass (Da) 498.000	Q1 Mass (Da) 502.800	315.100 119.400 3.54
	11e (Period 35.00 -3000.00 450.00 40.00 50.00 50.00 -10.00 -10.00 -14.00	03 Mass 80.000	03 Mass 80,000	Q3 Mass 80,000	03 Mass (Da) 98,900	00 3.54
		(Da)	(Da)	(Da)	(Da)	DF
	Experiment	RT (min) 3.39	RT (min) 3.70	RT (min) 3.78	RT (min) 5.21	-40.00 Œ
	1):	Paran DF	Paran DF	Paran DF	Paran DF	-40.00 -31.00
		ran Start Stop ID -100.00 -100.00 TUDCA CE -100.00 -100.00	ram Start Stop ID -100.00-100.00 TCCCA CE -100.00 -100.00	ram Start Stop ID -100.00-100.00 TDCA CE -100.00 -100.00	carr Start Stop ID -110.00-110.00 13C4-PFOS_2 CE -80.00-80.00	10.00 13C2-PETHAA_2 31.00 -31.00
10						

Acquisition Method

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ass Spec 12.500 min Period 12.500 min -WRM ntegrated Valve C System Equilibrate Injection

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1 Mass (Da) 12.800 can Type: checluled MRM: olarity: eriod 1 Experiment 1: arget Cycle Time: eriods In File: uto-Equilibration: ast Modified: aquisition Method: tep Size: 9 R Pause: ettling Time: ntensity Thres.: esolution Q3: esolution Q1: arget Scan Time: RM detection window: MRM 0103 Resolution: on Source: can Mode: se target Cycle Time: cheduled Ionization: elative Start Time: ax. Dwell Time: cans in Period: eriod 1: oftware version aquisition Module: unber Of Scans: equisition Duration: annent: reated: xperiments in Period: ynchronization Mode: in. Dwell Time: Q3 Mass (Da) 168.900 RCI (min) 2.71 Paran 눴 A Start Stop ID -40.00 -40.00 LC Sync Off Unit 1250 3 ms Unit N/A 0.00 Da Š 5.0070 msec 0.0000 msec 0.00 cps 0.6000 sec Ş Turbo Spray N/A Negative Yes 0ff 0.00 msec 250 JTS Analyst 1.7.3 1250 12min30sec 1633.dam 60 sec MAM (MRM) Acquisition Method -14.00 - 14.00PFBA

Acquisition Information:

Wednesday June 14 2023 11:54:50 AM Wednesday May 24 2023 14:48:39 FM

1 Mass (27.100	1 Mass (95.000	1 Mass (14.800	1 Mass (79.000	1 Mass 98.700	1 Mass - 63.000	1 Mass 41,000	29,000
(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	(D2)
03 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	84.900
307.000	201.000	134.900	85,100	79,900	219.000	177.000	
RT (min)	RT (min)	RT (mún)	RT (min)	RT (min)	RT (min)	RT (min)	2.94
3.41	3,52	3.84	3.32	3.67	3.21	2.98	2.94
Paran	Paran	Paran	Paran	Paran	Paran	DF	DE
DF	DF	DE	DF	DF	DF	DF	
Start Stop ID	Start Stor ID	r Start Stop ID	r Start Stop ID	r Start Stop ID	r Start Stop ID	r Start Stop ID	- 3041 5004 10
-110.00 -110.00 4:2-FIS	-30.00 -30.00 PFECA B	-70.00 -70.00 PFEESA	-40.00 PFECA A	-90.00 -90.00 PFES	-40.00 -40.00 PFFEA	-50.00 -50.00 3:3 FTCA	-40.00 -40.00 PEECA F
CE -28.00 -28.00	CE -25.00-25.00	CE -30.00-30.00	CE -20.00-20.00	CE -65.00-65.00	CE -14.00-14.00	CE -12.00-12.00	CE -25.00-25.00

	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)
	41.000	76.900	98.700	63.100	84.900	49.100	13.000
Attachment "" to	03 Mass (Da)	03 Mass (Da)	<u>0</u> 3 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	03 Mass (Da)
	237,100	250,900	79,900	319.000	168.900	79,900	269.000
US Eurofins	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)
	3.75	3.96	4.30	3.85	3.68	4.00	3.55
Cleveland	DE DE	Paran DF	Paran DF	Paran	Paran DF	Paran DF	Paran DF
Attachment "" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (Start Stop ID	Start Stop ID	Start Stop ID	Start Stor ID	Start Stop ID	Start Stop ID	Start Stop ID
	-70.00 -70.00 5:3 FICA	-40.00 -40.00 DOWA	-100.00 -100.00 PFHbs	-40.00 -40.00 PFHpA	-20.00 -20.00 HEPODA	-90.00 -90.00 PFPeS	-30.00 -30.00 PEH&A
	CE -20.00 -20.00	CE -20.00 -20.00	CE -80.00 -80.00	CE -15.00-15.00	CE -10.00-10.00	CE -70.00-70.00	CE -15.00-15.00

1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)
48.800	30.800	41.000	63.000	98.900	13.000	49,000	27,100
Q3 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)
79,900	351.000	316.900	419.000	79.900	369,000	79,900	407.000
RT (min)	RT (min)	4.40	9 RT (min)	9 RT (min)) RT (min)) RT (min)) RT (min)
5,72	5.61		4.43	5.22	4.12	4.68	3.98
Paran	Paran	-	Paran)) Paran) Paran) Paran
DF	DF	DF	DF	DF	DF	DF	DF
aran Start Stop ID F -100.00-100.00 PFNS	Aran Start Stop ID x= -100.00 -901-PE3ONS CE -38.00-38.00	Aran Start Stop ID 32 -80.00 -80.00 7:3 FTCA CE -20.00-20.00	Aran Start Stop ID XE -50.00 PENN CE -18.00-18.00	Aram Start Stop ID AF -90.00 -90.00 PFCS CE -110.00 -110.00	aran Start Stor ID DE -50.00 -50.00 PECA CE -16.00-16.00	Paran Start Stop ID DF -110.00 -110.00 PERApS CE -90.00-90.00	Param Start Stop ID >F -90.00 -90.00 6:2-FTS CE -35.00-35.00

CE -110.00 -110.00	-110.00
1 Mass (Da) 0,3 Mass (Da) RT (min) Paran Sta 12.900 469.000 4.85 DF -60	Start Stop ID -60.00 -60.00 PFDA CE -18.00-18.00
(Da) Q3 Mass (Da) RT (min) Pararr Sta 507.000 4.58 DF -1	Start Stop ID -100.00 -100.00 8:2-FTS CE -42.00 -42.00
(Da) 0.3 Mass (Da) RT (min) Param St. 77.900 6.14 DF -1.	Start Stop ID -110.00 -110.00 PFCSA CE -80.00 -80.00
(Da) Q3 Mass (Da) RT (min) Paran Sta	Start Stor ID
419.000 4.79 DF -80	-80.00 -80.00 IMEFOSPA
CF	CE -27.00 -27.00
(Da) Q3 Mass (Da) RT (min) Paran Stu	Start Stop ID
79.900 6.16 DF -90	-90.00 -90.00 PFDS
CE	CE -130.00 -130.00
(Da) Q3 Mass (Da) RT (min) Param Sta	Start Stop ID
519.000 5.39 DF -70	-70.00 -70.00 PFUnDA
CE	CE -19.00-19.00
(Da) Q3 Mass (Da) RT (min) Param St.	Start Stor ID
419.100 5.04 DF -9	-90.00 -90.00 NELFOSAA
CE	CE -30.00 -30.00

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1 Mass 26.000		1 M	1 N 99.	11. 11.	11 N	1 P 13.	30.
	lass (Da) 000	1 Mass (Da) 30.000	1 Mass (Da) 99,100	1 Mass (Da) 11,900	1 Mass (Da) 16.100	1 Mass (Da) 13.100	1 Mass (Da) 30.900
03 Mass (Da)	03 Mass (Da) 219.000	03 Mass (Da) 58.900	Q3 Mass (Da) 79,900	Q3 Mass (Da) 219,000	03 Mass (Da) 58.900	03 Mass (Da) 569,000	0 Q3 Mass (Da) 450.900
PCT (min)	RT (min) 7.36	RT (min) 7.24	RT (min) 6.94	RT (min) 7.08	PT (min) 6.94	RT (min) 5,87	RT (min) 6.49
Paran	Paran DF	Paran DF	Paran DF	Paran DF	Param DF	Paran DF	Param
Start R	-90.00	Start -45.00	Start -100.0	Start -90,00	Start -50.00 CE	Start -60,00	Start -90.00
-38,00-38,00 Stop ID		õ	Start Stop ID -100.00 -100.00 PETos CE -150.00 -150.00	Stop ID -90.00 NMEPFOSA -37.00 -37.00	Stop ID -50.00 -70.00-70.	0	Stop ID 90.00 11C1-PF3CUds -43.00 -43.00
	FOSA	NELFFOSAE	. α 00	FOSA	NMEPFOSAE 00		- PF3QUdS

RT (min) Parar Start Stop: ID PECA B 2 3.52 DF -10.00 -10.00 PECA B 2 RT (min) Parar Start Stop: ID -30.00 -100.00 3.41 DF -100.00 -100.00 4: 2 FPS_2 RT (min) Parar Start Stop: ID 3.55 DF -40.00 -90.00 3.55 DF -40.00 -01.00 DF CF -31.00 PETAS_2 RT (min) Parar Start Stop: ID A00 DF -40.00 -91.00 Start Stop: ID -40.00 Start Stop: ID -75.00 Start CF -10.00 Start CF -10.00		95.000 84.9 1 Mass (Da) 03 M 27.100 80.9 1 Mass (Da) 03 M 13.000 118 49.100 98.9 1 Mass (Da) 03 M 49.100 98.9 1 Mass (Da) 118
Paran Start DF -10.00 Paran Start DF -100.0 Paran Start DF -40.00 DF -110.0 CE -110.0	RT (min) 3.52 RT (min) 3.41 RT (min) 3.55 RT (min) 4.00	
Paran Start DF -10.00 Paran Start DF -100.0 DF CE CE Paran Start DF -40.00 DF -110.0 CE	RT (min) 3.52 RT (min) 3.41 RT (min) 3.55 RT (min) 4.00	
Paran Start DF -10.00 Paran Start DF CE CE CE Paran Start DF -40.00 CE	(Da) RT (min) 3.52 (Da) RT (min) 3.41 (Da) RT (min) 3.55	
Paran Start DF -10.00 Earan Start DF -100.0 Paran CE Paran Start DF -40.00	(Da) PC (min) 3.52 (Da) PC (min) 3.41 (Da) PC (min) 3.55	
Paran Start DF -10.00 CE Paran Start DF -100.0	(Da) RT (min) 3.52 (Da) RT (min) 3.41	
Paran Start DF CE CE Paran Start DF -100.0	(Da) RT (min) 3.52 (Da) RT (min) 3.41	
Paran Start DF -10.00 CE	(Da) RT (min) 3.52	
Paran Start DF -10.00 CE	(Da) RT (min) 3.52	
RT (min) Param Start Stop ID 3.66 DF -110.00 -110.00 PFBS_2 CE -35.00 -35.00	0,3 Mass (Da) RT (mun) 96.800 3.66	1 Mass (Da) Q3 M 98.700 98.8
RT (min) Parar Start Stop ID 6.71 DF -60.00 -60.00 PETEDA CE -22.00 -22.00	03 Mass (Da) RT (min) 669.000 6.71	1 Mass (Da) 03 M 13.100 669.
-60.00 -60.00 PETELAA CE -21.00 -21.00	0E	05.000 BT3.000 B.30

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	1 Mass (Da) 96.900	1 Mass (De) 13.000	1 Mass (Da) 49.000	1 Mass (Da) 27.100	1 Mass (Da) 41.000	1 Mass (Da) 98.700	1 Mass (Da) 63,100	
* : · · · · · · · · · · · · · · · · · ·	Q3 Mass (Da) 98.800	Q3 Mass (Da) 169.000	Q3 Mass (Da) 98,800	03 Mass (Da) 80.900	Q3 Mass (Da) 217.000	Q3 Mass (Da) 98,900	03 Mass (Da) 169.000	
1	RT (min) 5.21	RT (min) 4.12	RT (min) 4,68	RT (min) 3.98	RT (min) 3.75	RT (min) 4+30	RT (min) 3.85	
	DF	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF	
	n Start Stor ID -110.06-110.00 PFCS_2 CE -80.00-80.00	rr Start Stop ID -50.00 -50.00 PFOA 2 CE -26.00 -26.00	rr Start Stop ID -100.00 -100.00 PFHpS_2 CE -70.00 -70.00	n Start Stop ID -120.00 -120.00 6:2 FTS 2 CE -70.00 -70.00	nr Start Stop ID -80.00 -80.00 5:3 FTCA_2 CE -20.00 -20.00	nr Start Stop ID -90.00 -90.00 PFHAS_2 CE -70.00 -70.00	n Start Stop ID -50.00 -50.00 PFHpA_2 CE -25.00-25.00	

1 Mass (Da) 84.200	1 Mass (Da) 63.100	1 Mass (Da) 99.000	1 Mass (Da) 70.100	1 Mass (Da) 27.100	1 Mass (Da) 12.900	1 Mass (Da) 48.800	1 Mass (Da) 63.000
Q3 Mass (Da) 526.000	Q3 Mass (Da) 269.100	Q3 Mass (Da) 98.800	03 Mass (Da) 483,000	03 Mass (Da) 80.800	Q3 Mass (Da) 219.000	03 Mass (Da) 98,800	Q3 Mass (Da) 219.000
RT (min) 5.04	RT (min) 5.39	RT (min) 6.16	RT (min) 4.79	RT (min) 4.58	RT (min) 4.85	RT (min) 5.72	RT (min) 4.43
Paran DF	Paran DE	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF
Start Stop ID -100.00 -100.00 NELEOSAA_2 CE -30.00 -30.00	Start Stop ID -70.00 -70.00 PPUnDA_2 CE -35.00 -35.00	Start Stop ID -100.00-100.00 PFDS 2 CE -100.00 -100.00	Start Stor ID -90.00 -90.00 NMEFOSAA_2 CE -24.00 -24.00	Start Stop ID -100.00 -100.00 8:2 FTS_2 CE -90.00-90.00	Start Stop ID 60.00 PFDA_2 CE31.00-31.00	Start Stop ID -100.00-100.00 PENS_2 CE -90.00-90.00	Start Stop ID -50.00 -50.00 PENA 2 CE -30.00-30.00

23 23	1 Mass (Da) 26.000	1 Mass (Da) 11,900	1 Mass (Da) 98.100	1 Mass (Da) 63.000	1 Mass (Da) 13,100	1 Mass (Da) 63.000	1 Mass (Da) 13,100	
•) Q3 Mass (Da) 169.000	Q3 Mass 169,000	03 Mass 478.000	03 Mass 68,900	Q3 Mass 168.900	 Q3 Mass (Da) 168,900 	a) Q3 Mass (Da) 319.000	
1		(Da)	(Da)	(Da)	(Da)	(Da)	(Da)	
1	RT (min) 7.35	RT (mi.r.) 7.08	RT (min) 6.13	RT (min) 3.21	RT (min) 6.71	RT (min) 6.30	RT (min) 5.87	
	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF	Paran DF	
	n Start Stop ID -90.00 -90.00 NELPECSA_2 CE -40.00 -40.00	rr Start Stop ID -110.00 -110.00 NMEPECSA_2 CE -37.00 -37.00	п Start Stop ID -130.00 -130.00 РРОЗА 2 CE -35.00 -35.00	n Start Stop ID -40.00 -40.00 PETeA_2 CE -55.00-55.00	rr Start Stop ID -60.00 -60.00 PETEDA_2 CE -40.00-40.00	m Start Stop ID -60.00 -60.00 PETEDA_2 CE -40.00 -40.00	MY Start Stop ID -60.00 -60.00 PFDDDA_2 CE -38.00-38.00	

03 Mass (Da) RT (min) Darran Start Stor
Q3 Mass (Da) RT (mún) Fararr Start Stop : 98.800 6.94 DF -90.00 -90.00 CE -140.00
03 Mass (Da) RT (min) Param Start Stop 1 82.900 3.83 DF -70.00 -70.00 CE -30.00
Q3 Mass (Da) RT (min) Paran Start Stop : 336.900 4.40 DF -80.00 -80.00 CE -20.00
03 Mass (Da) RT (min) Paran Start Stor 1 117.000 2.98 DF -50.00 -50.00 CE -45.00-
Q3 Mass (Da) RT (min) Pararr Start Stop 452.900 6.48 DF -90.00 -90.00 CE -40.00
Q3 Mass (Da) RT (min) Paran Start Stop ID 353.000 5.60 DF -100.00-100.00 9Cl-PF30NS_2 CE -38.00-38.00
84.800 3.95 DE -40.00 -40. CE -40.0

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1								
	1 Mass (Da) 86,900	1 Mass (Da) 18.000	1 Mass (Da) 15.100	1 Mass (Da) 29.100	1 Mass (Da) 02.100	1 Mass (De) 68.300	1 Mass (Da) 16.800	
	03 Mass (Da) 168.900	03 Mass (Da) 273.000	03 Mass (Da) 270.000	03 Mass (Da) 80.900	03 Mass (Da) 79,900	03 Mass (Da) 223.000	Q3 Mass (Da) 171.900	
. E	a) RT (min) 3.68	a) RT (min) 3.54	a) RT (min) 3.54	8) RT (min) 3.41	a) RT (min) 3.66	هد) RT (min) 3.21	3a) RT (min) 2.71	A
2 -	DF DF	Paran DF	Paran DF	Paran DF	Paran DF	Paran D£	Paran DF	-14.00
	Start Stop ID -20,00 -20.00 13C3-HEFODA CE -10.00-10.00	Start Stop ID -30.00 -30.00 13C5-PFHxA CE -15.00-15.00	Start Stop ID -30,00 -30.00 13C2-PFHxA CE -15.00-15.00	Start Stop ID -100.00 -100.00 13C2-4:2-FTS CE -28.00 -28.00	Start Stop ID -90.00 -90.00 13C3-PFBS Or -65.00 -65.00	Start Stor ID -40.00 -40.00 13C5-PFFeA CE -14.00 -14.00	Start Stor ID -40.00 -40.00 13C4-PFBA CE -14.00-14.00	00 -14.00
					• •			
					(4)			
-								1

	1 Mass (Da) Q3 Mass (Da) RT (min) Paran Start Stop ID	1 Mass (Da) Q3 Mass (Da) RT (min) Paran Start Stop ID 02.800 79.900 5.21 DF -90.00 -90.00 13C4-PFOS CE -110.00 -110.00	1 Mass (Da) 03 Mass (Da) RT (min) Paran Start Stop ID 21.100 376.000 4.12 DF -50.00 -50.00 13C8-PFCA CE -16.00 -16.00	1 Mass (Da) Q3 Mass (Da) RT (min) Param Start Stop ID 17.100 172.000 4.12 DF -50.00 -50.00 13C4-PFCA CE -16.00 -16.00	1 Mass (Da) Q3 Mass (Da) RT (min) Paran Start Stop ID 29.100 80.900 3.97 DF -90.00 -90.00 13C2-6:2-FTS CE -35.00 -35.00	1 Mass (Da) Q3 Mass (Da) RT (min) Paran Start Stor ID 03.000 83.900 4.30 DF -100.00 -100.00 1802-PFHbS CE -80.00 -80.00	1 Mass (Da) Q3 Mass (Da) RT (min) Paran Start Stop ID 02.100 79.900 4.30 DF -100.00-100.00 13C3-PFHxS CE -80.00-80.00	1 Mass (Da) Q3 Mass (Da) RT (min) Param Start Stop ID 67.100 322.000 3.85 DF -40.00 -40.00 13C4-PFHpA CE -15.00 -15.00
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1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	1 Mass (Da)	
73,200	06,100	29.100	15.100	19.100	68.000	72.100	
Q3 Mass (Da)	Q3 Mass (Da)) Q3 Mass (Da)) Q3 Mass (Da)	03 Mass (Da)) Q3 Mass (Da)) Q3 Mass (Da)	
419,000	77.800	80,900	470.100	474.100	423.000	427.000	
) RT (min)) RT (min)) RT (min)) RT (min)) RT (min)	1) RT (min)	1) RT (min)	R
4.78	6.13	4.58	4.85	4.85	4.42	4.42	
Paran	Param	Paran	Paran	Paran	Paran	Paran	-110
DF	DF	DF	DF	DF	DF	D₽	
Start Stop ID	Start Stop ID	Start Stop ID	Start Stop ID	Start Stop ID	Start Stor ID	Start Stop ID	-110.00 -110.00
-90,00 -90.00 d3-NMEFOSAA	-110.00-110.00 13C3-PFOSA	-100.00 -100.00 13C2-8:2-FTS	-60.00 -60.00 13C2-PFDA	-60.00 -60.00 13C6-PEDA	-50.00 -50.00 13CS-PFNA	-50.00 -50.00 13C3-PENA	
CE -27,00 -27.00	CE -80.00-80.00	CE -42.00 -42.00	CE -18.00-18.00	CE -18.00 -18.00	CE -18.00-18.00	CE -18.00-18.00	

ŝ 20 NOV 2023 15:26 EST Draft Method 1633 *

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1 Mass	1 Mass	1 Mass (Da)	1 Mass	1 Mass	1 Mass (Da)	1 Mass	1 Mass
29.100	29,100	29,100	07.100	02.100	02.100	18.000	15.200
(Da)	(Da)		(Da)	(Da)		(Da)	(Da)
Q3 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)	03 Mass (Da)	Q3 Mass (Da)
509_000	409.000	309,000	98.900	98.800	98.900	120,300	670.000
RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	RT (min)	6.70
4.58	3.97	3.41	5.20	4 29	3.66	3.54	
Paran	Paran	Paran	Paran	Paran	Paran	Paran	Paran
DF	DF	DF	Df	DF	DE	DF	D F
r Start Stop ID	r Start Stop ID	r Start Stop ID	r Start Stop ID	r Start Stop ID	r Start Stop ID	т Start Stop ID	rr Start Stop ID
-100.00-100.00 1302-8:2-FTS_2	-100.00-100.00 1302-6:2-FTS_2	-100.00 13C2-4:2-FTS_2	-110.00 -110.00 13C8-PFOS 2	-90.00 -90.00 13C3-PFHAS_2	-110.00 -110.00 13C3-PPBS 2	-40.00 -40.00 13C5-РЕНЖА 2	-60.00 -60.00 13C2-PFTEDA
CE -42.00-42.00	CE -35.00-35.00	CE -28.00-28.00	CE -80.00 -80.00	CE -70.00-70.00	CE -35.00 -35.00	CE -31.00-31.00	CE -22.00 -22.00

1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 13C3-HTPROA_2 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 13C3-HTPROA_2 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 13C2-FFHaA_2 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 13C2-FFHaA_2 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 13C4-FFROS_2 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 13C4-FFROS_2 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 100.00 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 100.00 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 100.00 1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID 100.0
a) RT (min) Parar Start 3.68 DF c 3.54 DF -75.00 3.54 DF c 5.21 DF -40.00 5.21 DF -40.00 5.21 DF -110.00 Farar Start 3.78 DF c CE c c a) RT (min) Parar Start CE c c c c c c c c c c c c c c c c c c
a) RT (min) Parar Start 3.68 DF -75.00 3.54 DF -75.00 S.154 DF -40.00 5.21 min) Parar Start 3.78 DF -110.00 FT (min) Parar Start 3.79 DF -100.00 Experiment 1): Experiment 1):
Paran Start DF - 75.00 Paran Start DF - 40.00 DF - 40.00 DF - 40.00 DF - 110.00 DF - 110.00 DF - 100.00 DF - 100.00 DF - 100.00 DF - 100.00 DF - 100.00 DF - 100.00 DF - 100.00
arr Start ce arr Start arr Start ce ce ce ce ce ce ce ce

Printed by Dorothy Love. d. Mon 20 Nov 2023 15:26 FST ing Draft Method 1633 *

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<punipb></punipb>	LPCF: cvcle:	A Mobile phase A	Mohile nhase	Set compressibility:	bile phase	ching	.5000	0.6500 5.0	0.5000 98.0	0.5000	0.5000 60.0	3.00 0.5000 60.0 0	0.5000 5.0	ענות תנות אין היייייייייייייייייייייייייייייייייי	B. (00	ient>	Pressure limits Maximum:	Pressure limits Minimum:	B.Curve:	B.Conc:	Time to reach the flow:	Flow:	Flow/Pressure:	<pre> General> </pre>	B.(221>	Mode:	Gradient start adjustment>	ton time:	ocle1:	:	dun	lume = 1	system Equlibration time =	C Method Properties	Total Time (min)	ntegrated Valve Method Properties alve Diverter
	Standard			No	port: A	valve>						_					6500 psi	1 psi	0	5.0 %	0.00 min	0.5000 mL/min	Flow			None		12 SUNARY GRADUENT				00 ul	0.00 min		Position	Ś

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	Wait for temperature equilibration before run (sample cooler): No	type: External on node: Before and after aspiration 0 Before and after aspiration 0 Rinse port on nethod: 2.0 y speed: 35 µL y volume: 500 µ 100 µ 100 µ	Cooler temperature:15 °CUse air gap volume:NoUse low dispersion injection:NoAcquisition cycle time optimization>NoPretreatment start timing:OffCut off loop:NoRinse settings>No	General> Yes Use Autosampler: Yes Sample plate settings> No Specify plate: No Specify needle stroke: No Injection settings> Sampling speed: Use cooler temperature: Yes	B.GEl flow: 0.0000 mL/min <execute after="" autopurge=""> Turn oven on after autopurge: No Turn pump on after autopurge: No Time program> utosampler odel: STL-40C XR</execute>	gs> urge before analysis: urge settings> pler purge: lacement> nc-replacement:	Anced An
--	--	--	---	---	---	---	--

	.01 Event 1	program> Command Value	Shutdown settings> Power off after shutdown: No	Event 2: No	al output settings>	visite visite visite visite	The successive Auto	Wait for temperature equilibration before run: Off	um Oven:	oven A>	aciel: A: CTO-40C	Ven	The program>	Pretreatment> Mode. Standard	

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS DOCUMENTATION

PRODUCT_CODE: COMPOUND:

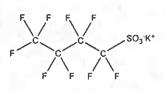
STRUCTURE:

L-PFBS Potassium perfluoro-1-butanesulfonate

LOT NUMBER: LPFBS1022

CAS #:

29420-49-3



MOLECULAR FORMULA: CONCENTRATION:

CHEMICAL PURITY: LAST TESTED: (mm/dd/yyy) EXPIRY DATE: (mm/dd/yyy) RECOMMENDED STORAGE: C₄F₉SO₄K 50.0 ± 2.5 μg/mL (K salt) 44.4 ± 2.2 μg/mL (PFBS acid) 44.2 ± 2.2 μg/mL (PFBS anion) >98% 10/04/2022 10/04/2027 Store ampoule in a cool, dark place

MOLECULAR WEIGHT: SOLVENT(S):

338.19 Methanol

DOCUMENTATION/ DATA ATTACHED:

Figure 1: LC/MS Data (Full Scan and Mass Spectrum) Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

See page 2 for further details.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Certified By:

B.G. Chittim, General Manager

Date: 10/14/2022

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA 519-822-2436 • Fax: 519-822-2849 • Info@well-labs.com

Form#:27. Issued 2004-11-10

LPFBS1022 (1 of 4)

Attachment ** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 Page 67 of 84

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compound it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products are compared to older lots in the same manner, which further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, $u_s(y)_s$ of a value y and the uncertainty of the independent parameters

$$u_{\varepsilon}(y(x_1, x_2, \dots, x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of $\pm 5\%$ (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

 x_1, x_2, \dots, x_n on which it depends is:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A1226), and ISO 17034 by ANSI National Accreditation Board (ANAB; AR-1523).





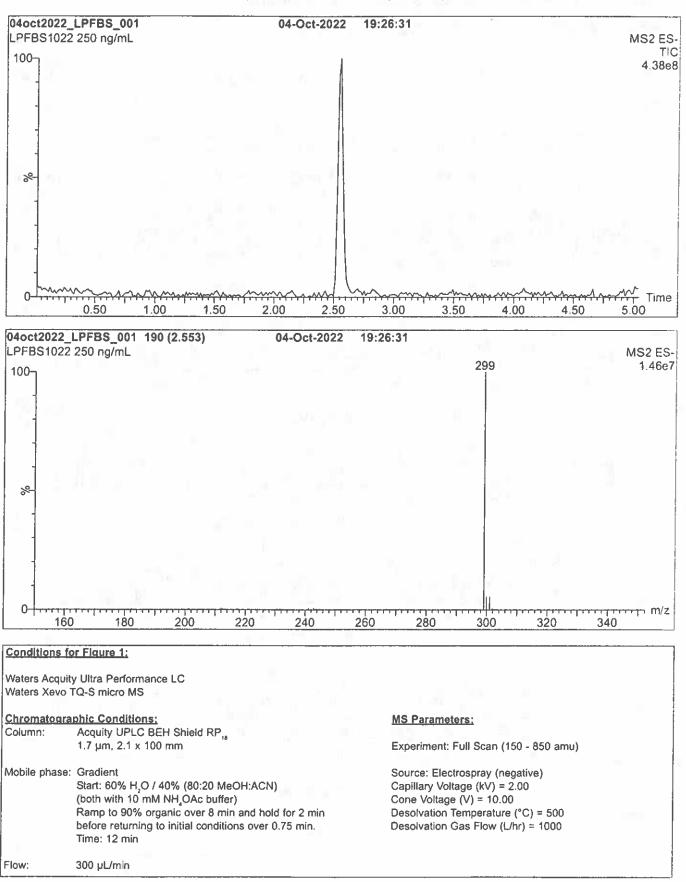
For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at <u>www.well-labs.com</u> or contact us directly at <u>info@well-labs.com</u>

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Attachment ** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 Page 68 of 84





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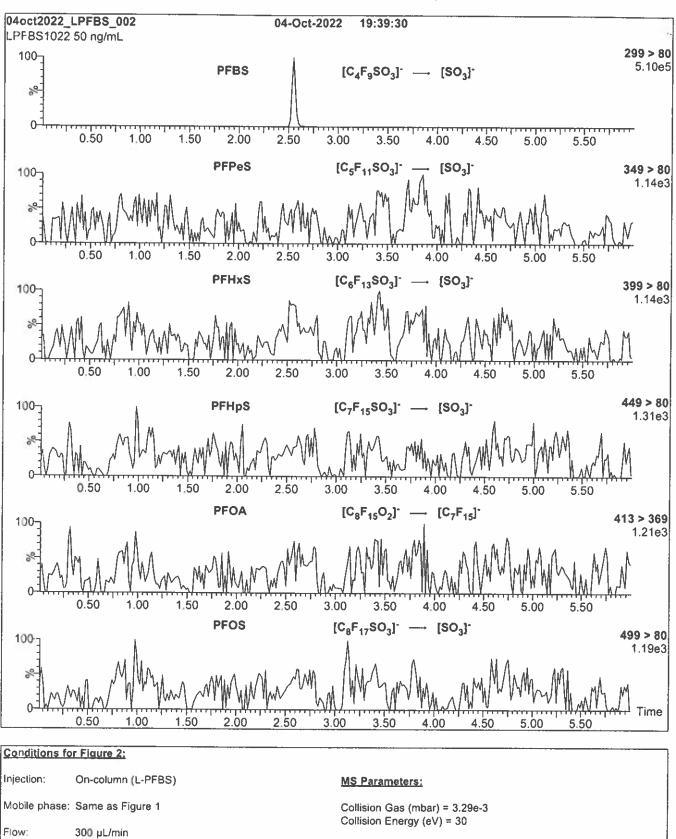


Figure 2: L-PFBS; LC/MS/MS Data (Selected MRM Transitions)

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Attachment *** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 Page 70 of 84

Attachment 5

		Native PFAS	i Intermediate	A				
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Native PFAS Intermediate A (ng/ml)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.1		500
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.05		250
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.025		125
Weilington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.025	100	125
Wellington	PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.025		125
Wellington	PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.025		125
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	= PFDA	50000	0.025		125
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000	0.025		125
Wellington	PFDoA	Perfluorododecanoic acid	307-55-1	PFDoA	50000	0.025	- CC	125
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.025	10 ²¹ H	125
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.025		125
Wellington	L-PFBS	Perfluorobutanesutfonic acid	375-73-5	PFBS	44200	0.025		111
Wellington	L-PFPeS	Perfluoropentansulfonic acid	2706-91-4	PFPeS	46900	0.025	1000	117
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.025		114
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.025		119
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.025		117
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.025	20 Eng	120
Wellington		Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.025		121
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400	0.025		121
Wellington	4;2 FTS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	46700	0.1	in fi	467
Wellington	6;2 FTS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	47400	0.1	10	474
Wellington	8;2 FTS	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	47900	0.1	10	479
Wellington	FOSA-I	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.025	1.00	125
Wellington	N-MeFOSA-M	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	50000	0.025		125
Wellington	N-EtFOSA-M	N-ethyt perfluorooctanesulfonamide	4151-50-2	NEIPFOSA	50000	0.025		125
Wellington	br-NMeFOSAA	N-methyl perfluorcoctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.025		125
Wellington	br-NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NETFOSAA	50000	0.025	1	125
Wellington	N-MeFOSE-M	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	50000	0.25		1250
Wellington	N-EtFOSE-M	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	50000	0.25		1250
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	50000	0.1	-	500
Wellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	47100	0.1	- L	471
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.05		250
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50000	0.05		250
Weilington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	50000	0.05		250
Wellington	9CI-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1- sulfonic acid	756426-58-1	9CI-PF3ONS	46600	0.1	L×	466
Wellington	11CI-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1- sulfonic acid	763051-92-9	11CI-PF3OUdS	47100	0.1		471
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.05		223
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000	0.625		3125
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000	0.625		3125

	Nati	ve PFAS Inter	mediate B				
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Native PFAS Intermediate B (ng/ml)
	Perfluorobutanoic acid	375-22-4	PFBA	500			5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			2.5
	Perfluorohexanoic acid	307-24-4	PFHxA	125			1.25
	Perfluoroheptanoic acid	375-85-9	PFHpA	125	1		1.25
	Perfluorooctanoic acid	335-67-1	PFOA	125	1		1.25
	Perfluorononanoic acid	375-95-1	PFNA	125	1		1.25
	Perfluorodecanoic acid	335-76-2	PFDA	125	1		1.25
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125	1		1.25
	Perfluorododecanoic acid	307-55-1	PFDoA	125	1		1.25
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125	1		1.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125	[1.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5	1		1.11
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117.25	1		1.17
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75	1		1.14
	Perftuoroheptanesulfonic acid	375-92-8	PFHpS	119	1		1.19
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5	1 i		1.17
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			1.20
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5	1		1.21
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121	1		1.21
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467	1		4.67
Native PFAS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474	0.1	10	4.74
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			4.79
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			1.25
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEIPFOSA	125			1.25
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			1.25
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			1.25
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			12.5
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			12.5
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			5
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			4.71
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			2.5
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			2.5
		250			2.5		
9	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9CI-PF3ONS	466			4.66
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	<u> </u>	11CI-PF3OUdS	471			4.71
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			2.23
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			6.25
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			31.3
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125		[31.3

Attachment ** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 Page 72 of 84

Solution Name	Analyte	CAS#	Acronym	Concentration (ng/mi)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Native PFAS Intermediate M (ng/mi)
1	Perfluorobutanoic acld	375-22-4	PFBA	500			62.5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			31.3
	Perfluorohexanoic acid	307-24-4	PFHxA	125	1.1		15.6
	Perfluoroheptanoic acid	375-85-9	PFHpA	125		29	15.6
	Perfluorooctanoic acid	335-67-1	PFOA	125	[15.6
	Perfluorononanoic acid	375-95-1	PFNA	125			15.6
	Perfluorodecanoic acid	335-76-2	PFDA	125		110	15.6
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125	100	a 	15.6
	Perfluorododecanoic acid	307-55-1	PFDoA	125	1.1		15.6
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125	100		15.6
	Perfluorotetradecanoic acld	376-06-7	PFTeDA	125	24		15.6
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			13.8
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117.25		- C.	14.7
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75			14.2
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119	1.00		14.9
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5	·		14.6
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120	1.5		15.0
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5	÷ .		15.1
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121	1.25 10		15.1
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			58.4
Native PFAS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474		10	59.3
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479		1.2	59.9
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			15.6
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125	11.52		15.6
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEIPFOSA	125			15.6
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			15.6
1	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125	100		15.6
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			156
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEIPFOSAE	1250		2.2	156
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500	1000	1 may 1	62.5
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471	- C		58.9
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			31.3
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			31.3
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			31.3
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9CI-PF3ONS	466			58.3
	11-Chloroeiccsafluoro-3-oxaundecane-1-sulfonic acid		11CI-PF3OUdS	471			58.9
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			27.8
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			78.1
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125	- 1		391
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			391

		Extractio	n Standard	Mix				
Vendor	Catalog Number	Analyte	CAS#	Acronym	Concentration (ng/mi)	Aliquot (mi)	Final Volume (mi) Methanol	Final Conc. Extraction Standard Mix (ng/mi)
Wellington	MPFBA	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	50000	0.1		500
Wellington	M5PFPeA	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	50000	0.05		250
Wellington	M5PFHxA	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	50000	0.025		125
Wellington	M4PFHpA	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	50000	0.025		125
Wellington	M8PFOA	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	50000	0.025		125
Wellington	M9PFNA	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	50000	0.0125		62.5
Wellington	M6PFDA	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	50000	0.0125		62.5
Wellington	M7PFUdA	Perfluoro-n-[1,2,3,4,5,6,7- 13C7]undecanoic acid	STL02580	13C7-PFUnA	50000	0.0125		62.5
Wellington	MPFDoA	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	50000	0.0125		62.5
Wellington	M2PFTeDA	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	50000	0.0125		62.5
Wellington	M3PFBS	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	46500	0.025		116
Wellington	M3PFHxS	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	47300	0.025		118
Wellington	M8PFOS	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	47800	0.025	10	120
Wellington	M8FOSA-I	Perfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	50000	0.025	10	125
Wellington	d3-N-MeFOSAA	N-methyl-d3-perfluoro-1- octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	50000	0.05		250
Wellington	d5-N-EtFOSAA	N-ethyl-d5-perfluoro-1- octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	50000	0.05	i	250
Wellington	M2-4;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2- 13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	46700	0.05		234
Wellington	M2-6;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2- 13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	47500	0.05	63	238
Wellington	M2-8;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2- <u>13</u> C2]decane sulfonic acid	STL02280	13C2-8:2FTS	47900	0.05		240
Wellington	M3HFPO-DA	Tetrafluoro-2-heptafluoropropoxy-13C3- propanoic acid	STL02255	13C3-HFPO-DA	50000	0.1		500
Wellington	d7-N-MeFOSE-M	N-methyl-d7- perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	50000	0.25		1250
Wellington	d9-N-EtFOSE-M	N-ethyl-d9- perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	50000	0.25		1250
Wellington	d-N-MeFOSA-M	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	50000	0.025		125
Wellington	d-N-EtFOSA-M	N-methyl-d3-perfluoro-1- octanesulfonamide	STL02705	D3-NMeFOSA	50000	0.025		125

		Interna	I Standard I	Aix				
Vendor	Catalog Number	Analyte	CAS#	Acronym	Concentration (ng/ml)	Aliquot (mi)	Final Volume (ml) Methanol	Final Conc. Internal Standard Mix (ng/ml)
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	50000	50	_	250
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	50000	25		125
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	50000	12.5		62.5
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	47800	25	10	120
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	50000	12.5		62.5
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	50000	25		125
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	1802-PFHxS	47300	25		118

	1633 Initial Calibration Standards Preparation											
Solution Name	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7					
Native PFAS Intermediate A (ml)	N/A	N/A	N/A	0.01	0.02	0.05	0.25					
Native PFAS Intermediate B (ml)	0.08	0.2	0.5	N/A	N/A	N/A	N/A					
Extraction Standard Mix (ml)	0.01	0.01	0.01	0.01	0.01	0.01	0.01					
Internal Standard Mix (ml)	0.01	0.01	0.01	0.01	0.01	0.01	0.01					
Final Volume (ml) (methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid)	2	2	2	2	2	2	2					

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	CAL1	CAL2	CAL3	Standard Concentra	CAL5	CAL6	CAL7
Compound Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	CAL4 Conc. (ng/mi)	CALS Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
PFpeA	0.100	0.250	0.625	1.25	2.50	6 25	31.3
PFHxA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFHpA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
FOA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFNA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFUnA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDoA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTrDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTeDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFBS	0.044	0.111	0.276	0.553	1.11	2.76	13.8
PFPeS	0.047	0.117	0.293	0.586	1.17	2.93	14.7
PFHxS	0.046	0.114	0.284	0.569	1.14	2.84	14.2
PFHpS	0.048	0.119	0.298	0.595	1.19	2.98	14.9
PFOS	0.047	0.117	0.291	0.583	1.17	2.91	14.6
PFNS	0.048	0.120	0.300	0.600	1.20	3.00	15.0
PFDS	0.048	0.121	0.301	0.603	1.21	3.01	15.1
PFDoS	0.048	0.121	0.303	0.605	1.21	3.03	15.1
1:2 FTS	0.187	0.467	1.17	2.34	4.67	<u>11.7</u> 11.9	58.4 59.3
3:2 FTS	0.190	0.479	1.19	2.37	4.74	11.9	59.3
PFOSA	0.192	0.479	0.313	0.625	4.79	3.13	15.6
MeFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
MeFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
MeFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
NETFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
IFPO-DA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
DONA	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFMPA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFMBA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
NFDHA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
CLPF3ONS	0.186	0.466	1.17	2.33	4.66	11.7	58.3
1CI-PF3OUdS	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFEESA	0.089	0.223	0.56	1.11	2.23	5.56	27.8
3:3 FTCA	0.250	0.625	1.56	3.13	6.25	15.6	78.1
3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
I3C4-PFBA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
3C5-PFPeA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
3C5-PFHxA 3C4-PFHpA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3C8-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3C9-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C6-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C7-PFUnA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C2-PFDoA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C2-PFTeDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C3-PFBS	0.581	0.581	0.581	0.581	0.581	0.581	0.581
3C3-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591
3C8-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
3C8-PFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3-NMeFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
5-NEtFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
3C2-4:2FTS	1.17	1.17	1,17	1.17	1.17	1.17	1.17
3C2-6:2FTS	1.19	1.19	1.19	1.19	1.19	1.19	1.19
3C2-8:2FTS	1.20	1.20	1.20	1.20	1.20	1.20	1,20
3C3-HFPO-DA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
7-NMeFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
9-NEIFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
5-NEtFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3-NMeFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3C3-PFBA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
3C4-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
3C2-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C4-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
3C5-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
3C2-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625

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	Bile Salts Stock Solutions											
Vendor	Catalog Number	Analyte	CAS#	Acronym	Amount (mg)	Aliquot (g)	Final Volume (ml) Methanol	Final Conc. Bile Salts Stock Solutions (ng/ml)				
Sigma Aldrich	T0557-500MG	Taurodeoxycholic acid	516-50-7	TDCA	500	0.05	50	1000000				
Sigma Aldrich	T6260-1G	Taurochenodeoxycholic acid	516-35-8	TCDCA	1000	0.05	50	1000000				
Sigma Aldrich	580549-1GM	Tauroursodeoxycholic acid	14605-22-2	TUDCA	1000	0.05	50	1000000				

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Attachment	13
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	Bile Salts Working Solution A										
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Bile Salts Working Solution A (ng/ml)				
TDCA Stock Solution	Taurodeoxycholic acid	516-50-7	TDCA	1000000	1	1.1.1	250000				
TCDCA Stock Solution	Taurochenodeoxycholic acid	516-35-8	TCDCA	1000000	1	4	250000				
TUDCA Stock Solution	Tauroursodeoxycholic acid	14605-22-2	TUDCA	1000000	1.1	7 - E (₁ - E)	250000				

	Bile Salts Working Solution B											
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Bile Salts Working Solution B (ng/ml)					
	Taurodeoxycholic acid	516-50-7	TDCA	250000			5000					
Bile Salts Working Solution A	Taurochenodeoxycholic acid	516-35-8	TCDCA	250000	0.1	5	5000					
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	250000			5000					

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		PFAS Linear	Branched M	lix A				
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. PFAS Linear/Branched Mix A (ng/ml)
Wellington	T-PFOA	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	50000	0.02		500
i volinigton		Perfluorooctanoic acid	335-67-1	PFOA	50000	0.01		500
Cambridge Isotope Laboratories		2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEIPFOSAE	50000	0.02		500
Cambridge Isotope Laboratories	ULM-11034-S	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	50000	0.01		500
Cambridge Isotope Laboratories	ULM-10780-S	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	100000	0.01	2	500
Cambridge Isotope Laboratories	ULM-10779-S	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	100000	0.02		500
Cambridge Isotope Laboratories	ULM-10977-S	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.02		500
Weilington	ipPFNA	Perfluoro-7-methylpoctanoic acid	15899-31-7	PF7MOA	50000	0.02		500
Wellington	PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.02		500

	PFAS	S Linear/Bra	nched Mix B				
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. PFAS Linear/Branched Mix B (ng/ml)
	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	500			125
	Perfluorooctanoic acid	335-67-1	PFOA	500			125
	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEIPFOSAE	500			125
PFAS	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500			125
Linear/Branched Mix A	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500	0.5	2	125
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			125
	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			125
	Perfluoro-7-methylpoctanoic acid	15899-31-7	PF7MOA	500			125
	Perfluorononanoic acid	375-95-1	PFNA	500			125

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Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (mi)	Final Volume* (ml)	Final Conc. 1633 Linear/Branched Bild Salts Solution (ng/mi
Bile Salts Working Solution B	Taurodeoxycholic acid	516-50-7	TDCA	5000			100
	Taurochenodeoxycholic acid	516-35-8	TCDCA	5000	0.1		100
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	5000		÷	100
	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	125	-	125	1.25
	Perfluorooctanoic acid	335-67-1	PFOA	125			1.25
	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	125	1 ° 1		1.25
PFAS	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	125	. H.	f	1.25
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	125	0 02		1.25
Vix B	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	125	1.1		1.25
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	Perfluoro-7-methylpoctanoic acid	15899-31-7	PF7MOA	125			1.25
	Perfluorononanoic acid	375-95-1	PFNA	125	1.000		1.25
· · · · · ·	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500	1 2 7		2.5
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250		2	1.25
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125			0.625
	Perfluoro-n-(13C9)nonanoic acid	STL02578	13C9-PFNA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	116			0.581
xtraction	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	118			0.591
Standard Mix	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	120	0.01		0.598
i	Perfluoro-1-[13C8]octanesuifonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acld	STL02118	D3-NMeFOSAA	250			1.25
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	250	1.1		1.25
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	234			1.17
	1H,1H,2H,2H-Perfluoro-1-{1,2-13C2}octane sulfonic acid	STL02279	13C2-6:2FTS	238			1.19
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	240	1.1		1.20
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	500			2.5
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	1250	1 mil		6.25
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250			6.25
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	125	1.1	1.1	0.625
	N-methyl-d3-perfluoro-1-octanesutfonamide	STL02705	D3-NMeFOSA	125	1.000	- 1	0.625
	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250		ľ	1.25
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
ternal	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	120	0.01		0.598
	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5	1.11		0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125		ŀ	0.625
	Perfluoro-1-hexane[1802]sulfonic acid	STL00994	1802-PFHxS	118			0.591

*Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

	10001014	Vorking Stand					
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume* (ml)	Final Conc. 1633 ICV Working Standard (ng/mi)
	Perfluorobutanoic acid	375-22-4	PFBA	500			2.50
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			1.25
	Perfluorohexanoic acid	307-24-4	PFHxA	125			0.625
	Perfuoroheptanoic acid	375-85-9	PFHpA	125			0.625
	Perfluorooctanoic acid	335-67-1	PFOA	125			0.625
	Perfluorononanoic acid Perfluorodecanoic acid	375-95-1	PFNA	125			0.625
	Perfluoroundecanoic acid	335-76-2 2058-94-8	PFDA PFUnA	125 125			0.625
	Perfluorododecanoic acid	307-55-1	PFDoA	125			0.625
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			0.625
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			0.625
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			0.553
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117			0.586
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	114			0.569
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119	1		0.595
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	117]		0.583
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			0.600
	Perfluorodecanesulfonic acid	335-77-3	PFDS	121			0.603
Native PFAS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			0.605
Intermediate	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467	0.01		2.34
4	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			2.37
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid Perfluorooctanesulfonamide	39108-34-4	8:2-FTS	479			2.40
	N-methyl perfluorooctanesulfonamide	754-91-6	PFOSA NMePFOSA	125 125			0.625
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEIPFOSA	125			0.625
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			0.625
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEIFOSAA	125		2	0.625
1	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			6.25
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEIPFOSAE	1250			6.25
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			2.50
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			2.36
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250]		1.25
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			1.25
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			1.25
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9CI-PF3ONS	466]		2.33
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9		471			2.36
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			1.11
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			3.13
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			15.6
	3-Perfluoroheptyl propanoic acid Perfluoro-n-[13C4]butanoic acid	812-70-4 STL00992	7:3 FTCA	3125 500	——		15.6
	Perfluoro-n-[13C5]pentanoic acid	STL00992	13C4-PFBA 13C5-PFPeA	250			2.5
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125			0.625
	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	116			0.581
Extraction	Perfluoro-1-(1,2,3-13C3)hexanesulfonic acid	STL02581	13C3-PFHxS	118	0.01		0.591
Standard Mix		STL01054	13C8-PFOS	120	0.0.		0.598
	Perfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250			1.25
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEIFOSAA	250			1.25
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	234			1.17
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279 STL02280	13C2-6:2FTS	238			1.19
	1H, 1H,2H,2H-Perfluoro-1-(1,2-13C2)decane sulfonic acid Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02280 STL02255	13C2-8:2FTS 13C3-HFPO-DA	240 500			1.20
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02255	D7-NMeFOSE	1250			6.25
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250			6.25
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL022704	D5-NEIFOSA	1250			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250			1.25
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
stamal	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
nternal Standard Mix	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	120	0.01		0.598
	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[1802]sulfonic acid	STL00994	18O2-PFHxS	118			0.591

*Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

Attachment ** to *US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 Page 84 of 84

APPENDIX D Health and Safety Plan





HALEY & ALDRICH, INC. SITE-SPECIFIC SAFETY PLAN

FOR

90-02 Queens Boulevard Site

Queens, New York

Project/File No. 208933



Prepared By: Matthew Forshay

Date: 10/9/2023

Approvals: The following signatures constitute approval of this Health & Safety Plan.

Juaran

Field Safety Manager: Brian Ferguson

Bennergu here

Project Manager: Cheryl Benmergui

Date: 11/6/2023

Date: 11/6/2023

HASP Valid Through: 12-31-2023



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STOP WORK AUTHORITY

In accordance with Haley & Aldrich (Haley & Aldrich) Stop Work Authority Operating Procedure (OP1035), any individual has the right to refuse to perform work that he or she believes to be unsafe without fear of retaliation. He or she also has the authority, obligation, and responsibility to stop others from working in an unsafe manner.

STOP Work Authority is the stop work policy for all personnel and subcontractors on the Site. When work has been stopped due to an unsafe condition, Haley & Aldrich site management (e.g., Project Manager [PM], Site Health & Safety Officer [SHSO], etc.) and the Haley & Aldrich Senior Project Manager (SPM) will be notified immediately.

Reasons for issuing a stop work order include, but are not limited to:

- The belief/perception that injury to personnel or accident causing significant damage to property or equipment is imminent.
- An Haley & Aldrich subcontractor is in breach of site safety requirements and/or their own site HASP.
- Identifying a substandard condition (e.g., severe weather) or activity that creates an unacceptable safety risk as determined by a qualified person.

Work will not resume until the unsafe act has been stopped OR sufficient safety precautions have been taken to remove or mitigate the risk to an acceptable degree. Stop work orders will be documented as part of an onsite stop work log, on daily field reports to include the activity/activities stopped, the duration, person stopping work, person in-charge of stopped activity/activities, and the corrective action agreed to and/or taken. Once work has been stopped, only the Haley & Aldrich SPM or SHSO can give the order to resume work. Haley & Aldrich senior management is committed to support anyone who exercises his or her "Stop Work" authority.



ISSUANCE AND COMPLIANCE

This HASP has been prepared in accordance with Occupational Safety and Health Administration (OSHA) regulations (CFR 29, Parts 1904, 1910, and 1926) if such are applicable.

The specific requirements of this HASP include precautions for hazards that exist during this project and may be revised as new information is received or as site conditions change.

- This HASP must be signed by all Haley & Aldrich personnel involved in implementation of the SOW (Section 2 of this HASP).
- This HASP, or a current signed copy, must be retained at all times when Haley & Aldrich staff are present.
- Revisions to this HASP must be outlined within the contents of the HASP. If immediate or minor changes are necessary, the Field Safety Manager (FSM), Haley & Aldrich, SSO and/or Project Manager (PM) may use Attachment 1 (HASP Amendment Form), presented at the end of this HASP. Any revision to the HASP requires employees and subcontractors to be informed of the changes so that they understand the requirements of the change.
- Deviations from this HASP are permitted with approval from the Haley & Aldrich FSM, PM, or Senior Health & Safety Manager (SHSM). Unauthorized deviations may constitute a violation of Haley & Aldrich company procedures/policies and may result in disciplinary action.
- This HASP will be relied upon by Haley & Aldrich's subcontractors and visitors to the site. Haley & Aldrich's subcontractors must have their own HASP which will address hazards specific to their trade that is not included in this HASP. This HASP will be made available for review to Haley & Aldrich's subcontractors and other interested parties (e.g. Facility personnel and regulatory agencies) to ensure that Haley & Aldrich has properly informed our subcontractors and others of the potential hazards associated with the implementation of the SOW to the extent that Haley & Aldrich is aware.

This site-specific HASP provides only site-specific descriptions and work procedures. General safety and health compliance programs in support of this HASP (e.g., injury reporting, medical surveillance, personal protective equipment (PPE) selection, etc.) are described in detail in the Haley &Aldrich Corporate Health and Safety Program Manual and within Haley & Aldrich's Standard Operating Procedures Both the manual and SOPs can be located on the Haley & Aldrich's Company Intranet. When appropriate, users of this HASP should always refer to these resources and incorporate to the extent possible. The manual and SOPs are available to clients and regulators upon request.



EMERGENCY EVENT PROCEDURES

1 - ASSESS THE SCENE

• STOP WORK

- Review the situation and ascertain if it's safe to enter the area.
- Evacuate the site if the conditions are unsafe.

2 - EVALUATE THE EMERGENCY

- Call 911, or designated emergency number, if required.
- Provide first aid for the victim if qualified and safe to do so.
 - o First aid will be addressed using the onsite first aid kit. *
 - If providing first aid, remember to use proper first aid universal precautions if blood or bodily fluids are present.
- If exposure to hazardous substance is suspected, immediately vacate the contaminated area.
 - o Remove any contaminated clothing and/or equipment.
 - o Wash any affected dermal/ocular area(s) with water for at least 15 minutes.
 - o Seek immediate medical assistance if any exposure symptoms are present.

*<u>Note</u>: Haley & Aldrich employees are not required or expected to administer first aid / CPR to any Haley & Aldrich staff member, Contractor, or Civilian personnel at any time; it is Haley & Aldrich's position that those who do are doing so on their own behalf and not as a function of their job.

3 - SECURE THE AREA

- Cordon off the incident area, if possible.
 - o Notify any security personnel, if required.
 - o Escort all non-essential personnel out of the area, if able.

4 - REPORT ON-SITE ACCIDENTS / INCIDENTS TO PM / SSO

- Notify the PM and SSO as soon as it is safe to do so.
 - o Assist PM and SSO in completing any additional tasks, as required.

5 - INVESTIGATE / REPORT THE INCIDENT

- Record details of the incident for input to the Gensuite.
 - o Complete any additional forms as requested by the PM and SSO.

6 - TAKE CORRECTIVE ACTION

- Implement corrective actions per the PM following root cause analysis.
 - o Complete Lessons Learned form.



Site Specific Health & Safety Plan 90-02 Queens Boulevard Site 10/9/2023

PROJECT INFORMATION AND CONTACTS

Project Name: 90-02 Queens Boulevard Site

Haley & Aldrich File No.: 208933

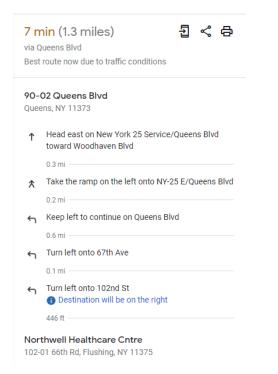
Location: 90-02 Queens Boulevard, Queens, New York

Location: 90-02 Queens Boulevard, Queens, Nev	VTORK
Client/Site Contact:	Selim Sabbagh
Phone Number:	Enter Phone Number
Haley & Aldrich Field Representative:	Matthew Forshay
Phone Number:	862-377-3587
Emergency Phone Number:	929-746-8015
Haley & Aldrich Project Manager:	Cheryl Benmergui
Phone Number:	332-240-1716
Emergency Phone Number:	929-746-8015
Field Safety Manager:	Brian Ferguson
Phone Number:	617-886-7439
Emergency Phone Number:	617-908-2761
Subcontractor Project Manager:	Tim Kelly
Phone Number:	631-524-6327
Nearest Hospital:	Northwell Healthcare Center
Address:	102-01 66 th Road
(see map on next page)	Flushing, Queens, NY
Phone Number:	Enter Phone Number
Nearest Occ. Health Clinic:	NYC Health + Hospitals/Gotham Health, Lefrak
http://www.talispoint.com/liberty/ext/	
Address:	59-17 Junction Boulevard
(see map on next page)	Queens, NY
Phone Number:	844-692-4692
Liberty Mutual Claim Policy	WC6-Z11-254100-033
WORKCARE Injury & Illness HOTLINE	1-888-449-7787
Emergency Response Number:	911
Other Local Emergency Response Number:	N/A
Other Ambulance, Fire, Police, or Environmental	
Emergency Resources:	



DIRECTIONS TO THE NEAREST HOSPITAL Liberty Mutual Medical Location Directory irget ビ Masjid Nur Allah 😡 495 (÷ LeFrak City ICPen PS 206 - The Horac adidas Store New York, Queen's Center Shop adidas Shoes 62nd Cr 0 natio Costco Wholesale ueens Boulevard O 63rd Ave STINRO 63rd Rd JHS 157 Ste kids Acade 495 A 7 min 1.3 miles Leading Edge Oral Surgery Elmhurst Fores Day Care Taco Be 86th St Ģ Northwell Healthcare Cntre Town Supe Resurrection Ascension Roman Catholic Church 0 Maria S. Sales DDS Owner of Gentle... Cheburechnaya e Flower Shop 1.5 m Dry Harbor Nursing Home &... • 11 Nursing Home he Great Escape Room Lil Pearls (1) Pediatric Dentistry Brainseed Te ego Park 0 Northwe Health-GoHealth œ Global medical WESTPOL The Marlboro 68th Rd 9 Luminary Tutoring 68th Dr Veterinary Care

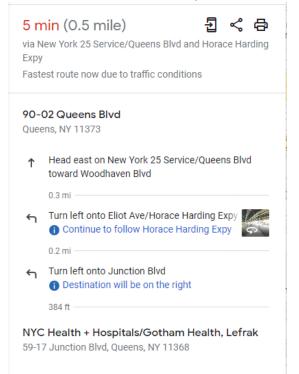
Directions to the Nearest Hospital:







Directions to the Nearest Occupational Clinic:



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WORK SCOPE

This Site-Specific Health and Safety Plan addresses the health and safety practices and procedures that will be exercised by all Haley & Aldrich employees participating in all work on the Project Site. This plan is based on an assessment of the site-specific health and safety risks available to Haley & Aldrich and Haley & Aldrich's experience with other similar project sites. The scope of work includes the following:

Work task will include: 1.) Drilling; and, .2) Soil, soil vapor, and groundwater sampling.

1.

Project Task Breakdown						
Task No.	Task Description		Employee(s) Assigned	Work Date(s) or Duration		
1	Drilling		Forshay, Matthew	4 Days Anticipated		
2	Soil, soil vapor, and gr	oundwater sampling	Forshay, Matthew	4 Days Anticipated		
	Subcontractor(s) Tasks					
	Firm Name	Work A	Activity	Work Date(s) or Duration		
Lakewood Environmental Drilling Services		Drilling		4 Days Anticipated		
Projected Start Date: 10/23/2023						
Projected	Projected Completion Date: 10/27/2023					



SITE OVERVIEW / DESCRIPTION

Site Classification

Residential and Commercial

2.

Site Description

The Site, identified as Block 2857, Lot 7501 on the New York City tax map in a R6 residential zoning area with a C1-2 commercial overlay in the Elmhurst neighborhood of Queens, New York, is 54,500 square feet (sf) in size and improved by a 6-story mixed use commercial and residential building, with a partial basement. The first floor is intended for medical facility use, while the partial basement proposed for commercial office space and gym facilities.

Background and Historic Site Usage

Historic uses of the Site include residential dwellings as early as 1902. Beginning in 1950's, the property was the former St. John's Hospital until 2015 when the building was renovated converting the first through fifth floors into residential use.

Site Status

Indicate current activity status and describe operations at the site:

Active

Residential dwellings are occupied.

Site Plan

Is a site plan or sketch available? Yes

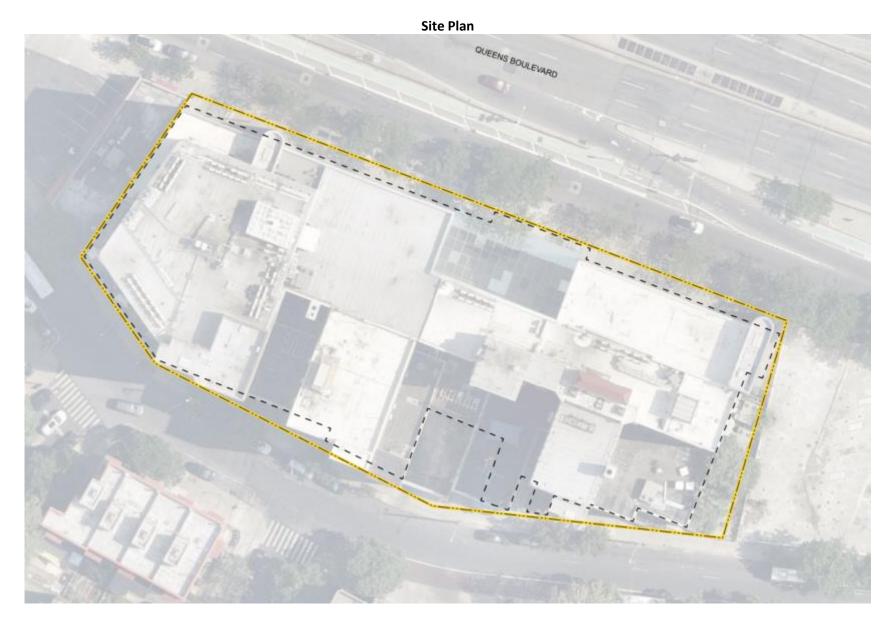
Work Areas

List and identify each specific work areas(s) on the job site and indicate its location(s) on the site plan:

Basement level



Site Specific Health & Safety Plan 90-02 Queens Boulevard Site 10/9/2023



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Note: This HASP has been developed for Haley & Aldrich purposes only and is not for use by others.



3. HAZARD ASSESSMENT

Indicate all hazards that may be present at the site and for each task. If any of these potential hazards are checked, it is the Project Manager's responsibility to determine how to eliminate / minimize the hazard to protect onsite personnel.

Site Chemical Hazards

Is this Site impacted with chemical contamination? Yes

Source of information about contaminants: Previous Investigation

Contaminant of Concern	Location/Media	Concentration	Units
1,1-Dichloroethane (1,1-DCA)	Groundwater	680	ug/L
1,1-Dichloroethylene (1,1- DCE)	Groundwater	560	ug/L
Tetrachloroethylene	Groundwater	6.8	ug/L
Vinyl Chloride	Groundwater	17	ug/L
1,1-Dichloroethylene (1,1- DCE)	Soil Vapor	120	ug/m3
Tetrachloroethylene	Soil Vapor	42	ug/m3
1,1,1-Trichloroethane	Soil Vapor	660	ug/m3

1,1-Dichloroethane (1,1-DCA): 1,1-Dichloroethane is a colorless, oily liquid with a sweet odor. It evaporates easily at room temperature and burns easily. It does not occur naturally in the environment. 1,1-Dichloroethane is used mostly as an intermediate in the manufacture of 1,1,1-trichloroethane (1,1,1-TCE). It is also used in limited amount as a solvent for cleaning and degreasing, and in the manufacture of plastic wrap, adhesives, and synthetic fiber

1,1-Dichloroethylene (1,1-DCE): Industrial chemical that is not found naturally in the environment. It is a colorless liquid with a mild, sweet smell. It is also called vinylidene chloride. **1,1-Dichloroethylene** is used to make certain plastics, such as flexible films like food wrap, and in packaging materials. It is also used to make flame retardant coatings for fiber and carpet backings, and in piping, coating for steel pipes, and in adhesive applications.

Tetrachloroethylene: is a colorless liquid with a sharp sweet odor. Tetrachloroethylene vapor is heavier than air and will be found in low lying areas.

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Trichloroethylene: is a nonflammable colorless liquid with a sweet odor. Trichloroethylene vapor is heavier than air and is found in low lying areas.

VOCs: include all organic compounds (substances made up of predominantly carbon and hydrogen) with boiling temperatures in the range of 50-260 degrees C, excluding pesticides. This means that they are likely to be present as a vapor or gas in normal ambient temperatures. Substances which are included in the VOC category include aliphatic hydrocarbons (such as hexane), aldehydes, aromatic hydrocarbons (such as benzene, toluene, and the xylenes or BTEX), and oxygenated compounds (such as acetone and similar ketones). The term VOC often is used in a legal or regulatory context and in such cases the precise definition is a matter of law.

VOCs are released from oil and gasoline refining, storage and combustion as well as from a wide range of industrial processes. Processes involving fuels, solvents, paints or the use of chemicals are the most significant sources. VOCs may also be emitted from cleaning products, degreasing products, fabrics, carpets, plastic products, glues, printed material, varnishes, wax, disinfectants, and cosmetics.

Typically, VOCs are present in gas or vapor and will enter the body by breathing contaminated air. Higher concentrations of VOCs may occur in areas of poor ventilation.

Site Hazards Checklist							
Weather							
Cold Temperatures	Hot Temperatures	High Winds	Select Hazard				
Cold Temperatures High Winds Select Hazard Cold Temperatures Cold stress may occur at any time work is being performed at low ambient temperatures and high velocity winds. Because cold stress is common and has potentially serious illnesses associated with outdoor work during cold seasons, regular monitoring and other preventative measures are vital. Refer to OP1003-Cold Stress for additional information and mitigation controls.							
Hot Temperatures Heat stress may occur at any time work is being performed at elevated ambient temperatures. Because heat stress is one of the most common and potentially serious illnesses associated with							



outdoor work during hot seasons, regular monitoring and other preventative measures are vital. Site workers must learn to recognize and treat the various forms of heat stress. The best approach is preventative heat stress management.

H&A employees and their subcontractors should be aware of potential health effects and/or physical hazards of working when there are hot temperatures or a high heat index. Refer OP1015-Heat Stress for a discussion on hot weather hazards.

High Winds

While high winds are commonly associated with severe thunderstorms and hurricanes they may also occur as a result of differences in air pressures, such as when a cold front passes across the area. They can cause downed trees and power lines, and flying debris (such as dust or larger debris), which adds additional risks and could lead to power outages, transportation disruptions, damage to buildings and vehicles, and serious injury.

Wind Advisory are issued for sustained winds 25 to 39 mph and/or gusts to 57 mph. High Wind warnings are issued by the National Weather Service when high wind speeds may pose a hazard or is life threatening. The criteria for this warning will varies by state. The Beaufort Wind Scale is a helpful tool to when dealing with high winds.

Biological						
Stinging Insects	Choose an item.	Choose an item.	Choose an item.			
Click + to Add Additiona	l Hazard Language					
	Location	n/Terrain				
Slip/Trip/FallsChoose an item.Choose an item.Choose an item.						

Slips, Trips & Falls

Slip and trip injuries are the most frequent injuries to workers. Statistics show most falls happen on the same level resulting from slips and trips. Both slips and trips result from unintended or unexpected change in the contact between the feet and the ground or walking surface. Good housekeeping, quality of walking surfaces (flooring), awareness of surroundings, selection of proper footwear, and appropriate pace of walking are critical for preventing fall accidents.

Site workers will be walking on a variety of irregular surfaces, that may affect their balance. Extra care



must be taken to walk cautiously near rivers because the bottom of the riverbed maybe slick and may not be visible. Rocks, gradient changes, sandy bottoms, and debris may be present but not observable.

Take your time and pay attention to where you are going. Adjust your stride to a pace that is suitable for the walking surface and the tasks you are doing. Check the work area to identify hazards - beware of trip hazards such as wet floors, slippery floors, and uneven surfaces or terrain. Establish and utilize a pathway free of slip and trip hazards. Choose a safer walking route. Carry loads you can see over. Keep work areas clean and free of clutter. Communicate hazards to on-site personnel and remove hazards as appropriate.

Miscellaneous						
Choose an item.	Choose an item.	Choose an item.	Choose an item.			
Click + to Add Additional Hazard Language						



Task Hazard Summary

Task 1 – Drilling

Drilling is conducted for a range of services that can include but are not limited to: soil characterization, environmental investigation, well installation, and ore exploration. Familiarity with basic drilling safety is an essential component of all drilling projects. Potential hazards related to drilling operations include, but are not limited to encountering underground or overhead utilities, traffic and heavy equipment, hoisting heavy tools, steel impacts, open rotation entanglement, and the planned or unexpected encountering of toxic or hazardous substances. While staff members do not operate drilling equipment, they may work in close proximity to operating drilling equipment and may be exposed to many of the same hazards as the drilling subcontractor. It is imperative that staff are aware of emergency stops and establish communication protocols with the drillers prior to the start of work.

See OP 1002 Drilling Safety for more information.

Task 2 – Soil Vapor

Soil gas sampling is employed as an indirect indicator of contamination in soil or groundwater particularly over and around landfill waste sites, or groundwater plumes. Soil gas sampling points can be installed manually using a slam bar or power driven mechanical devices (e.g., demolition hammer or Geoprobe) may be used based on site conditions (i.e., pavement, frozen ground, very dense clays, etc.). Soil gas samples can be drawn through the probe itself, or through Teflon tubing inserted through the probe and attached to the probe point. Samples are collected and analyzed as described below. Other field air monitoring devices, such as the Combustible Gas Indicator (CGI) and the Organic Vapor Analyzer (OVA), can also be used, depending on specific site conditions.

Because the sample is being drawn from underground, and no contamination is introduced into the breathing zone, soil gas sampling usually occurs in Level D. Nevertheless, ambient air should be constantly monitored to obtain background and breathing zone readings during the sampling procedure in the event the seal around the sampling point is breached. As long as the levels in ambient air do not rise above background, no upgrade of the level of protection is needed. Also, an underground utility search must be performed prior to sampling.

Task 2 – Soil Sampling

Soil sampling by H&A staff on active construction sites can be conducted in conjunction with a wide range activities such as building construction, earthwork and soil management related activities. These activities can include, but are not limited to: drill spoil characterization and management during building

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foundation element installation, characterization of excavated soils for management/disposal/reuse during earthwork activities, and as part of environmental remedial activities such as delineation and confirmation sampling. Familiarity with basic heavy construction safety, site conditions (geotechnical and environmental), and potential soil contaminants are essential components of soil sampling performed on active sites. Potential hazards related to soil sampling at construction sites include, but are not limited to: encountering site vehicle traffic and heavy equipment operations, manual lifting, generated waste, contact or exposure to impacted soil, and encountering unknown toxic or hazardous substances. Although soil sampling is commonly performed within active excavations, from stockpiles, or within trench excavations, sampling locations and situations will vary depending on site conditions. Care should be taken while entering and exiting excavations or trenches, and when accessing (climbing up or down) soil stockpiles, ensuring that the sampling area is not being actively accessed by construction equipment. Care should also be taken with handling of potentially environmentally impacted soil during sampling, with appropriate PPE identified and used. At no time during classification activities are personnel to reach for debris near machinery that is in operation, place any samples in their mouth, or come in contact with the soils without the use of gloves. Staff will have to carry and use a variety of sampling tools, equipment, containers, and potentially heavy sample bags. It is imperative that staff are aware of emergency / communication protocols with the Contractor prior to the start of work.

Task 2 – Water Sampling

Environmental water sampling could include activities such as groundwater sampling from permanent or temporary wells, or surface water sampling from streams, rivers, lakes, ponds, lagoons, and surface impoundments.

Sampling tasks could involve uncapping, purging (pumping water out of the well), and sampling, and/or monitoring, new or existing monitoring wells. A mechanical pump may be used to purge the wells and can be hand-, gas-, or electric-operated. Water samples taken from the wells are then placed in containers and shipped to an analytical laboratory for analysis. The physical hazards of these operations are primarily associated with the collection methods and procedures used.

When sampling bodies of water containing known or suspected hazardous substances, adequate precautions must be taken to ensure the safety of sampling personnel. The sampling team member collecting the sample should not get too close to the edge, where ground failure or slips, trips or falls may cause him/her to lose his/her balance. The person performing the sampling should have fall restraint or protection for the task. When conducting sampling from a boat in an impoundment or flowing waters, appropriate vessel safety procedures should be followed. Avoid lifting heavy coolers with back muscles; instead, use ergonomic lifting techniques, team lift or mechanical lifts. Wear proper gloves, such as when handling sample containers to avoid contacting any materials that may have spilled out of the sample containers.

Inhalation and absorption of COCs are the primary routes of entry associated with water sampling, due to the manipulation of sample media and equipment, manual transfer of media into sample containers, and proximity of operations to the breathing zone. During this project, several different groundwater sampling methodologies may be used based on equipment accessibility and the types of materials to be



sampled. These sampling methods may include hand or mechanical bailing. The primary hazards associated with these specific sampling procedures are not potentially serious; however, other operations in the area or the conditions under which samples must be collected may present chemical and physical hazards. The hazards directly associated with groundwater sampling procedures are generally limited to strains or sprains from hand bailing, and potential eye hazards. Exposure to water containing COCs is also possible. All tools and equipment that will be used at the site must be intrinsically safe (electronics and electrical equipment) and non-sparking or explosion-proof (hand tools).

Task Physical Hazards Checklist						
	Task 1	Task 2	Task 3	Task 4		
Potential Task Hazards	Drilling	Soil, Soil Vapor, and Groundwater Sampling	Task Name	Task Name		
Hand/Power Tools	\boxtimes	\boxtimes				
Heavy Equipment	\boxtimes	\boxtimes				
Underground Utilities	\boxtimes					
Noise	\boxtimes					
Ground Disturbance	\boxtimes					
Line of Fire	\boxtimes	\boxtimes				
Manual Lifting	\boxtimes	\boxtimes				
Sharp Objects	\boxtimes	\boxtimes				
Slippery Surfaces	\boxtimes	\boxtimes				
Rotating Equipment	\boxtimes					
Other: Specify						

Summary of Physical Hazards & Controls



Hand and Power Tools

Hand and power tools can expose staff to a wide range of hazards depending upon the tool used. Hazards can include but are not limited to: falling, flying, abrasive, and splashing objects, or harmful dusts, fumes, mists, vapors, or gases.

Serious accidents often occur before steps are taken to evaluate and avoid or eliminate tool-related hazards. Staff must recognize the hazards associated with the different types of tools and the safety precautions necessary to prevent those hazards.

See OP 1026 Hand and Power Tools for more information.

Controls

- Keep all tools in good condition with regular maintenance.
- Use the right tool for the job. Do not use a tool for a task which it was not designed for.
- Examine each tool for damage before use and do not use damaged tools.
- For tools that are damaged or defective, red tag the tool and take out of service.
- Operate tools per the manufacturers' instructions.
- Use the appropriate personal protective equipment.
- All electrically powered tools will be connected through a ground fault circuit interrupter (GFCI).
- All personnel must be trained on the use of the tool they are utilizing.

Heavy Equipment

Staff must be careful and alert when working around heavy equipment, failure or breakage and limited visibility can lead to accidents and worker injury. Heavy equipment such as cranes, drills, haul trucks, or other can fail during operation increasing chances of worker injury. Equipment of this nature shall be visually inspected and checked for proper working order prior to commencement of field work. Those operating heavy equipment must meet all requirements to operate the equipment. Haley & Aldrich, Inc. staff that supervise projects or are associated with high risk projects that involve digging or drilling should use due diligence when working with a construction firm.

See OP1052 Heavy Equipment for additional information.

Controls

- Only approach equipment once you have confirmed contact with the operator (e.g., operator places the bucket on the ground).
- Always maintain visual contact with operators and keep out of the strike zone whenever possible.
- Always be alert to the position of the equipment around you.
- Always approach heavy equipment with an awareness of the swing radius and traffic routes of all equipment and <u>never</u> go beneath a hoisted load.
- Avoid fumes created by heavy equipment exhaust.



Underground Utilities

Various forms of underground/overhead utility lines or conveyance pipes may be encountered during site activities. Prior to the start of intrusive operations, utility clearance is mandated, as well as obtaining authorization from all concerned public utility department offices. Should intrusive operations cause equipment to come into contact with utility lines, the SHSO, Project Manager, and Regional H&S Manager shall be notified immediately. Work will be suspended until the client and applicable utility agency is contacted and the appropriate actions for the situation can be addressed.

See OP1020 Work Near Utilities for complete information.

Controls

- Obtain as-built drawings for the areas being investigated from the property owner;
- Visually review each proposed soil boring locations with the property owner or knowledgeable site representative;
- Perform a geophysical survey to locate utilities;
- Hire a private line locating firm to determine location of utility lines that are present at the property;
- Identifying a no-drill or dig zone;
- Hand dig or use vacuum excavation in the proposed ground disturbance locations if insufficient data is unavailable to accurately determine the location of the utility lines.

Noise

Working around heavy equipment (drill rigs, excavators, etc.) often creates excessive noise. The effects of noise include physical damage to the ear, pain, and temporary and/or permanent hearing loss. Workers can also be startled, annoyed, or distracted by noise during critical activities. Noise monitoring data that indicates that working within 25 feet of operating heavy equipment result in exposure to hazardous levels of noise (levels greater than 85 dBA).

See OP 1031 Hearing Conservation for additional information.

Controls

- Personnel are required to use hearing protection (earplugs or earmuffs) within 25 feet of any operating piece of heavy equipment.
- Limit the amount of time spent at a noise source.
- Move to a quiet area to gain relief from hazardous noise sources.
- Increase the distance from the noise source to reduce exposure.

Ground Disturbance

Ground disturbance is defined as any activity disturbing the ground. Ground disturbance activities include, but are not limited to, excavating, trenching, drilling (either mechanically or by hand), digging, plowing, grading, tunneling and pounding posts or stakes.



Because of the potential hazards associated with striking an underground utility or structure, the operating procedure for underground utility clearance shall be followed prior to performing any ground disturbance activities.

See OP1020 Working Near Utilities

Controls

Prior to performing ground disturbance activities, the following requirements should be applied:

- Confirm all approvals and agreements (as applicable) either verbal or written have been obtained.
- Request for line location has been registered with the applicable One-Call or Dial Before You Dig organization, when applicable.
 - Whenever possible, ground disturbance areas should be adequately marked or staked prior to the utility locators site visit.
- Notification to underground facility operator/owner(s) that may not be associated with any known public notification systems such as the One-Call Program regarding the intent to cause ground disturbance within the search zone.
- Notifications to landowners and/or tenant, where deemed reasonable and practicable.
- Proximity and Common Right of Way Agreements shall be checked if the line locator information is inconclusive.

Line of Fire

Line of fire refers to the path an object will travel. Examples of line of fire situations typically observed on project sites include lifting/hoisting, lines under tension, objects that can fall or roll, pressurized objects or lines, springs or stored energy, work overhead, vehicles and heavy equipment.

Controls

- Never walk under a suspended load.
- Be aware and stay clear of tensioned lines such as cable, chain and rope.
- Be cautious of torque stresses that drilling equipment and truck augers can generate. Equipment can rotate unexpectedly long after applied torque force has been stopped.
- Springs and other items can release tremendous energy if compressed and suddenly released
- Items under tension and pressure can release tremendous energy if it is suddenly released.
- Not all objects may be overhead; be especially mindful of top-heavy items and items being transported by forklift or flatbed.
- Secure objects that can roll such as tools, cylinders, and pipes.
- Stay clear of soil cuttings or soil stockpiles generated during drilling operations and excavations, be aware that chunks of soil, rocks, and debris can fall or roll.

Manual Lifting/Moving

Most materials associated with investigation, remedial, or construction-related activities are moved by hand. The human body is subject to damage in the forms of back injury, muscle strains, and hernia if caution is not observed in the handling process.

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Controls

- Under no circumstances should any one person lift more than 49 pounds unassisted.
- Always push, not pull, the object when possible.
- Size up the load before lifting. If it is heavy or clumsy, get a mechanical aid or help from a worker.
- Bend the knees; it is the single most important aspect of lifting.
- When performing the lift:
 - Place your feet close to the object and center yourself over the load.
 - Get a good handhold.
 - Lift straight up, smoothly and let your legs do the work, not your back!
 - Avoid overreaching or stretching to pick up or set down a load.
 - Do not twist or turn your body once you have made the lift.
 - Make sure beforehand that you have a clear path to carry the load.
 - Set the load down properly.

Sharp Objects

Workers who handle sharp edged objects like sheets of steel or glass are at risk of cuts. Workers who handle sharp edged objects are also at risk of cuts. Injuries may occur to hands, fingers, or legs when they are in the way of the blade, when the blade slips, or if an open blade is handled unexpectedly. Other hazards at job sites include stepping on sharp objects (e.g. wooden boards with protruding nails, sharp work-tools, chisels, etc.) and colliding with sharp and/or protruding objects.

Controls

Always be alert when handling sharps. Never look away or become distracted while handling sharp objects. Use caution when working with tools; use right tool for the job. Keep tools sharp, dull blades are a safety hazard, requiring more force to make cuts which can lead to tool slippage. Wear appropriate PPE and do not handle sharp objects (i.e., broken glass) with bare hands. Use mechanical devices, when possible. Stay away from building debris; avoid handling site debris or placing your hand where you cannot see. Watch out for barbed wire and electrical fences; cover with a car mat or equivalent to cross or walk around; use the buddy system to avoid entanglement; wear gloves. Do not leave unprotected sharps unattended. Use protective shields, cases, styrofoam blocks, etc. Pass a sharp by handing it over carefully by the handle with the blade down or retracted. Fixed open blades are prohibited. Always cut away from the body, making several passes when cutting thicker materials. Make sure blades are fitted properly into the knife. Never cut items with a blade or other sharp object on your lap. Never try to catch a blade or cutting tool that is falling.

Slippery Surfaces

Both slips and trips result from unintended or unexpected change in the contact between the feet and ground or walking surface. Good housekeeping, quality of walking surfaces, selection of proper footwear, and appropriate pace of walking are critical for preventing fall accidents. Slips happen where there is too little friction or traction between the footwear and walking surface.



Common causes of slips are wet or oily surfaces, spills, weather hazards, loose unanchored rugs or mats and flooring or other walking surfaces that do not have same degree of traction in all areas.

Weather-related slips and falls become a serious hazard as winter conditions often make for wet or icy surfaces outdoors. Even wet organic material or mud can create hazardous walking conditions. Spills and leaks can also lead to slips and falls.

Controls

- Evaluate the work area to identify any conditions that may pose a slip hazard.
- Address any spills, drips or leaks immediately.
- Mark areas where slippery conditions exist.
- Select proper footwear or enhance traction with additional PPE.
- Where conditions are uncertain or environmental conditions result in slippery surfaces walk slowly, take small steps, and slide feet on wet or slippery surfaces.

Rotating Equipment

Exposure to rotating parts can occur when working near a drilling rig, or other similar equipment. All rotating parts should be covered with guards to prevent access by workers. When performing maintenance activities that require the rotating parts to be exposed, workers should not allow loose clothing, hands, or tools to approach the rotating parts. Energy isolation procedures must be followed, and guards must be replaced as soon as possible after completing the maintenance task.

Operation of drilling equipment also creates hazards associated with pinch points and rotating equipment. These are hazards where the body and extremities, especially the hands, can be caught in moving equipment and crushed.

Controls

- Evaluate work procedures to avoid placing the body and extremities in the path of rotating equipment and tools to avoid being struck by moving equipment, tools and machinery.
- Evaluate equipment and tool use to identify pinch points and develop procedures to avoid placing body parts in a position where they can be caught in moving equipment, tools and machinery.
- Follow energy isolation procedures if required
- Do not work near rotating equipment with long loose hair, loose clothing or jewelry.



PROTECTIVE MEASURES

The personal protective equipment and safety equipment (if listed) is specific to the associated task. The required PPE and equipment listed must be onsite during the task being performed. Work shall not commence unless the required PPE or Safety Equipment is present.

4.

Required Safety & Personal Protective Equipment					
Required Personal Protective	Task 1	Task 2	Task 3	Task 4	
Equipment (PPE)	Drilling	Soil, soil vapor, and groundwater sampling	Enter task description.	Enter task description.	
Hard hat	\boxtimes	\boxtimes			
Safety Glasses	\boxtimes	\boxtimes			
Safety Toed Shoes	\boxtimes	\boxtimes			
Class 2 Safety Vest	\boxtimes	\boxtimes			
Hearing Protection	\boxtimes				
Nitrile Gloves		\boxtimes			
Cut Resistant Gloves	\boxtimes	\boxtimes			
Level of protection required	D	D	Select	Select	
Required Safety Equipment					
First Aid Kit	\boxtimes	\boxtimes			



TRAINING REQUIREMENTS

The table below lists the training requirements staff must have respective to their assigned tasks and that are required to access the Site.

Site Specific Training Requirements

HAZWOPER - 40 Hour (Initial)

HAZWOPER - 8 Hour (Annual Refresher)

5.

Task Specific Training Requirements				
Required Training Type	Task 1	Task 2	Task 3	Task 4
	Enter task description.	Enter task description.	Enter task description.	Enter task description.
Choose an item.				



AIR MONITORING PLAN AND EQUIPMENT

Exposures to airborne substances shall be fully characterized throughout project operations to ensure that exposure controls are effectively selected and modified as needed.

Is air/exposure monitoring required at this work site for personal protection? Choose an item.

Is perimeter monitoring required for community protection? Choose an item.

Air monitoring plan not applicable Choose an item.

Air Monitoring/Screening Equipment Requirements

Photo-Ionization Detector (PID) 10.6eV

6.

The required equipment listed above must be on site. Work shall not commence unless the equipment is present and in working order.

Monitoring Plans

Select Monitoring Plan

*If chemical does not have an action level use TLV or REL, whichever is lowest, to be used as an action level. If TLV or REL are the same as PEL, cut the PEL in half for an action level.

Parameter/		Equipment	Action	Level*	Response Activity
Contaminant					
VOCs	PIC	0 10.6 eV	5 F	PM	Stop Work
Zone Location				Monitorin	ng Interval
Breathing + Exclusior	า	Choose an item.		15 Minute	25
Zone					



7. DECONTAMINATION & DISPOSAL METHODS

All possible and necessary steps shall be taken to reduce or minimize contact with chemicals and contaminated/impacted materials while performing field activities (e.g., avoid sitting or leaning on, walking through, dragging equipment through or over, tracking, or splashing potential or known contaminated/impacted materials.)

Personal Hygiene Safeguards

The following minimum personal hygiene safeguards shall be adhered to:

- 1. No smoking or tobacco products in any project work areas.
- 2. No eating or drinking in the exclusion zone.
- 3. It is required that personnel present on site wash hands before eating, smoking, taking medication, chewing gum/tobacco, using the restroom, or applying cosmetics and before leaving the site for the day.

It is recommended that personnel present on site shower or bathe at home at the end of each day of working on the site.

Decontamination Supplies

All decontamination should be conducted at the project site in designated zones or as dictated by Client requirements. Decontamination should not be performed on Haley & Aldrich owned or leased premises.

	Acetone	\boxtimes	Distilled Water		Polyethylene Sheeting
\boxtimes	Alconox Soap	\boxtimes	Drums		Pressure/Steam Cleaner
\boxtimes	Brushes		Hexane		Tap Water
	Disposal Bags		Methanol		Wash tubs
\boxtimes	5 Gallon Buckets	\boxtimes	Paper Towels		Other: Specify
	Location of Decontamination Station				
Des	Describe/Enter location of decontamination station or refer to a figure where it is shown.				

ALDRICH

Standard Personal Decontamination Procedures

Outer gloves and boots should be decontaminated periodically as necessary and at the end of the day. Brush off solids with a hard brush and clean with soap and water or other appropriate cleaner whenever possible. Remove inner gloves carefully by turning them inside out during removal. Wash hands and forearms frequently. It is good practice to wear work-designated clothing while on-site which can be removed as soon as possible. Non-disposable overalls and outer work clothing should be bagged onsite prior to laundering. If gross contamination is encountered on-site contact the Project Manager and Field Safety Manager to discuss proper decontamination procedures.

The steps required for decontamination will depend upon the degree and type of contamination but will generally follow the sequence below.

- 1. Remove and wipe clean hard hat
- 2. Rinse boots and gloves of gross contamination
- 3. Scrub boots and gloves clean
- 4. Rinse boots and gloves
- 5. Remove outer boots (if applicable)
- 6. Remove outer gloves (if applicable)
- 7. Remove Tyvek coverall (if applicable)
- 8. Remove respirator, wipe clean and store (if applicable)
- 9. Remove inner gloves (if outer gloves were used)

PPE that is not grossly contaminated can be bagged and disposed in regular trash receptacles.

Small Equipment Decontamination

Pretreatment of heavily contaminated equipment may be conducted as necessary:

- 1. Remove gross contamination using a brush or wiping with a paper towel
- 2. Soak in a solution of Alconox and water (if possible)
- 3. Wipe off excess contamination with a paper towel

Standard decontamination procedure:

- 4. Wash using a solution of Alconox and water
- 5. Rinse with potable water
- 6. Rinse with methanol (or equivalent)
- 7. Rinse with distilled/deionized water

Inspect the equipment for any remaining contamination and repeat as necessary.



Disposal Methods

Procedures for disposal of contaminated materials, decontamination waste, and single use personal protective equipment shall meet applicable client, locate, State, and Federal requirements.

Disposal of Single Use Personal Protective Equipment

PPE that is not grossly contaminated can be bagged and disposed in regular trash receptacles. PPE that is grossly contaminated must be bagged (sealed and field personnel should communicate with the Project Manager to determine proper disposal.

Select text from the drop down that applies to the project. Click + to add additional language from the drop down.





8. SITE CONTROL

The overall purpose of site control is to minimize potential contamination of workers, protect the public from the site's hazards, and prevent vandalism. Site control is especially important in emergency situations. The degree of site control necessary depends on site characteristics, site size, and the surrounding community. The following information identifies the elements used to control the activities and movements of people and equipment at the project site.

Communication

Internal

Haley & Aldrich site personnel will communicate with other Haley & Aldrich staff member and/or subcontractors or contractors with:

Face to Face Communication

External

H&S site personnel will use the following means to communicate with off-site personnel or emergency services.

Cellular Phones

Visitors

Project Site

Will visitors be required to check-in prior to accessing the project site?

Yes

Visitor Access

Authorized visitors that require access to the project site need to be provided with known information with respect to the site operations and hazards as applicable to the purpose of their site visit. Authorized visitors must have the required PPE and appropriate training to access the project site.

Site Safety Officer is responsible for facilitating authorized visitor access.

Zoning

Work Zone

The work zone will be clearly delineated to ensure that the general public or unauthorized worker access is prevented. The following will be used:

Cones



9. SITE SPECIFIC EMERGENCY RESPONSE PLAN

The Emergency Response Plan addresses potential emergencies at this site, procedures for responding to these emergencies, roles, responsibilities during emergency response, and training. This section also describes the provisions this project has made to coordinate its emergency response with other contractors onsite and with offsite emergency response organizations (as applicable).

During the development of this emergency response plan, local, state, and federal agency disaster, fire, and emergency response organizations were consulted (if required) to ensure that this plan is compatible and integrated with plans of those organizations. Documentation of the dates of these consultations are the names of individuals contacted is kept on file and available upon request.

The site has been evaluated for potential emergency occurrences, based on site hazards, and the major categories of emergencies that could occur during project work are:

- Fire(s)/Combustion
- Hazardous Material Event
- Medical Emergency
- Natural Disaster

A detailed list of emergency types and response actions are summarized in Table X below. Prior to the start of work, the SSO will update the table with any additional site-specific information regarding evacuations, muster points, or additional emergency procedures. The SSO will establish evacuation routes and assembly areas for the Site. All personnel entering the Site will be informed of these routes and assembly areas.

Pre-Emergency Planning

Before the start of field activities, the Project Manager will ensure preparation has been made in anticipation of emergencies. Preparatory actions include the following:

Meeting with the subcontractor/and or client concerning the emergency procedures in the event a person is injured. Appropriate actions for specific scenarios will be reviewed. These scenarios will be discussed, and responses determined before the sampling event commences. A form of emergency communication (i.e.; Cell phone, Air horn, etc.) between the Project Manager and subcontractor and/or client will be agreed on before the work commences.

A training session (i.e., "safety meeting") given by the Project Manager or their designee informing all field personnel of emergency procedures, locations of emergency equipment and their use, and proper evacuation procedures.

Ensuring field personnel are aware of the existence of the emergency response HASP and ensuring a copy of the HASP accompanies the field team(s).

Onsite Emergency Response Equipment

Emergency procedures may require specialized equipment to facilitate work rescue, contamination control and reduction or post-emergency cleanup. Emergency response equipment stocked



Table 9.1 Emergency Equipment and Emergency PPE				
Emergency Equipment	Specific Type	Quantity Stocked	Location Stored	
First Aid Kit	Enter text	Enter text	Brought to Site by H&A Field Staff	
Emergency PPE	Specific Type	Quantity Stocked	Location Stored	
Select	Enter text	Enter text	Enter text	

EVACUATION ALARM

Verbal Communication (Site Personnel are adjacent in work zone)

EVACUATION ROUTES

Will be given a map after site specific training

EVACUATION MUSTER POINT(S)/ SHELTER AREA(S)

Will be given a locations after site specific training

EVACUTION RESPONSE DRILLS

The Site relies on outside emergency responders and a drill is not required.



Table 9-2 – Emergency Planning

Emergency Type	Notification	Response Action	Evacuation Plan/Route
Chemical Exposure	Report event to SSO immediately	Refer to Safety Data Sheet for required actions	Remove personnel from work zone
Fire - Small	Notify SSO and contact 911	Use fire extinguisher if safe and qualified to do so	Mobilize to Muster Point
Fire – Large/Explosion	Notify SSO and contact 911	Evacuate immediately	Mobilize to Muster Point
Hazardous Material – Spill/Release	Notify SSO; SSO will contact PM to determine if additional agency notification is	If practicable don PPE and use spill kit and applicable procedures to contain the release	See Evacuation Map for route, move at least 100 ft upwind of spill location
Medical – Bloodborne Pathogen	Notify SSO	If qualified dispose in container or call client or city to notify for further instruction.	None Anticipated
Medical – First Aid	Notify SSO	If qualified perform first aid duties	None Anticipated
Medical – Trauma	If life threatening or transport is required call 911, immediately	Wait at site entrance for ambulance	Noe Anticipated
Security Threat	Notify SSO who will call 911 as warranted	Keep all valuables out of site and work zones delineated.	None Anticipated
Weather – Earthquake/Tsunami's	STOP WORK and evacuate Site upon any earthquake	Turn off equipment and evacuate as soon as is safe to do so	Mobilize to Shelter Location
Weather – Lightning Storm	STOP WORK	Work may resume 30 minutes after the last observed lightning.	None Anticipated
Weather – Tornadoes/Hurricanes	Monitor weather conditions STOP WORK and evacuate the site	Evacuate to shelter location or shelter in place immediately	Mobilize to Shelter Location
MUSTER POINT		SHELTER LOCATION	
Outside the building along Hoffman I	Drive	Inside the basement of 90-02 Queer	ns Boulevard



10. HASP ACKNOWLEDGEMENT FORM

All Haley & Aldrich employees onsite must sign this form prior to entering the site.

I hereby acknowledge receipt of, and briefing on, this HASP prior to the start of on-site work. I declare that I understand and agree to follow the provisions, processes, and procedures set forth herein at all times while working on this site.

Printed Name	Signature	Date



Site Specific Health & Safety Plan 90-02 Queens Boulevard Site 10/9/2023

ATTACHMENT A HASP AMENDMENT FORM

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HASP AMENDMENT FORM

This form is to be used whenever there is an immediate change in the project scope that will require an amendment to the HASP. For project scope changes associated with "add-on" tasks, the changes must be made in the body of the HASP. Before changes can be made, a review of the potential hazards must be initiated by the Haley & Aldrich Project Manager.

This original form must remain on site with the original HASP. If additional copies of this HASP have been distributed, it is the Project Manager's responsibility to forward a signed copy of this amendment to those who have copies.

Amendment No.	
Site Name	
Work Assignment No.	
Date	
Type of Amendment	
Reason for Amendment	
Alternate Safeguard Procedures	
Required Changes in PPE	

Project Manager Name (Print)	Project Manager Signature	Date
Health & Safety Approver Name (Print)	Health & Safety Approver Signature	Date
(Print)		



Site Specific Health & Safety Plan 90-02 Queens Boulevard Site 10/9/2023

ATTACHMENT B TRAINING REQUIREMENTS

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TRAINING REQUIREMENTS

Health and Safety Training Requirements

Personnel will not be permitted to supervise or participate in field activities until they have been trained to a level required by their job function and responsibility. Haley & Aldrich staff members, contractors, subcontractors, and consultants who have the potential to be exposed to contaminated materials or physical hazards must complete the training described in the following sections.

The Haley & Aldrich Project Manager/FSM will be responsible for maintaining and providing to the client/site manager documentation of Haley & Aldrich staff members' compliance with required training as requested. Records shall be maintained per OSHA requirements.

40-Hour Health and Safety Training

The 40-Hour Health and Safety Training course provides instruction on the nature of hazardous waste work, protective measures, proper use of personal protective equipment, recognition of signs and symptoms which might indicate exposure to hazardous substances, and decontamination procedures. It is required for all personnel working on-site, such as equipment operators, general laborers, and supervisors, who may be potentially exposed to hazardous substances, health hazards, or safety hazards consistent with 29 CFR 1910.120.

8-hour Annual Refresher Training

Personnel who complete the 40-hour health and safety training are subsequently required to attend an annual 8-hour refresher course to remain current in their training. When required, site personnel must be able to show proof of completion (i.e., certification) at an 8-hour refresher training course within the past 12 months.

8-Hour Supervisor Training

On-site managers and supervisors directly responsible for, or who supervise staff members engaged in hazardous waste operations, should have eight additional hours of Supervisor training in accordance with 29 CFR 1910.120. Supervisor Training includes, but is not limited to, accident reporting/investigation, regulatory compliance, work practice observations, auditing, and emergency response procedures.

Additional Training for Specific Projects

Haley & Aldrich personnel will ensure their personnel have received additional training on specific instrumentation, equipment, confined space entry, construction hazards, etc., as necessary to perform their duties. This specialized training will be provided to personnel before engaging in the specific work activities including:

- Client specific training or orientation
- Competent person excavations
- Confined space entry (entrant, supervisor, and attendant)
- Heavy equipment including aerial lifts and forklifts
- First aid/ CPR
- Use of fall protection
- Use of nuclear density gauges
- Asbestos awareness



ATTACHMENT C ROLES AND RESPONSIBILITIES



SITE ROLES AND RESPONSIBILITIES

Haley & Aldrich Personnel

Field Safety Manager (FSM)

The Haley & Aldrich FSM is a full-time Haley & Aldrich staff member, trained as a safety and health professional, who is responsible for the interpretation and approval of this Safety Plan. Modifications to this Safety Plan cannot be undertaken by the PM or the SSO without the approval of the FSM.

Specific duties of the FSM include:

- Approving and amending the Safety Plan for this project
- Advising the PM and SHSOs on matter relating to health and safety
- Recommending appropriate personal protective equipment (PPE) and air monitoring instrumentation
- Maintaining regular contact with the PM and SSO to evaluate the conditions at the property and new information which might require modifications to the HASP and
- Reviewing and approving JSAs developed for the site-specific hazards.

Project Manager (PM)

The Haley & Aldrich PM is responsible for ensuring that the requirements of this HASP are implemented at that project location. Some of the PM's specific responsibilities include:

- Assuring that all personnel to whom this HASP applies have received a copy of it;
- Providing the FSM with updated information regarding environmental conditions at the site and the scope of site work;
- Providing adequate authority and resources to the on-site SHSO to allow for the successful implementation of all necessary safety procedures;
- Supporting the decisions made by the SHSO;
- Maintaining regular communications with the SHSO and, if necessary, the FSM;
- Coordinating the activities of all subcontractors and ensuring that they are aware of the pertinent health and safety requirements for this project;
- Providing project scheduling and planning activities; and
- Providing guidance to field personnel in the development of appropriate Job Safety Analysis (JSA) relative to the site conditions and hazard assessment.

Site Health & Safety Officer (SHSO)

The SHSO is responsible for field implementation of this HASP and enforcement of safety rules and regulations. SHSO functions may include some or all of the following:

- Act as Haley & Aldrich's liaison for health and safety issues with client, staff, subcontractors, and agencies.
- Verify that utility clearance has been performed by Haley & Aldrich subcontractors.
- Oversee day-to-day implementation of the Safety Plan by Haley & Aldrich personnel on site.



- Interact with subcontractor project personnel on health and safety matters.
- Verify use of required PPE as outlined in the safety plan.
- Inspect and maintain Haley & Aldrich safety equipment, including calibration of air monitoring instrumentation used by Haley & Aldrich.
- Perform changes to HASP and document in Appendix A of the HASP as needed and notify appropriate persons of changes.
- Investigate and report on-site accidents and incidents involving Haley & Aldrich and its subcontractors.
- Verify that site personnel are familiar with site safety requirements (e.g., the hospital route and emergency contact numbers).
- Report accidents, injuries, and near misses to the Haley & Aldrich PM and FSM as needed.

The SHSO will conduct initial site safety orientations with site personnel (including subcontractors) and conduct toolbox and safety meetings thereafter with Haley & Aldrich employees and Haley & Aldrich subcontractors at regular intervals and in accordance with Haley & Aldrich policy and contractual obligations. The SHSO will track the attendance of site personnel at Haley & Aldrich orientations, toolbox talks, and safety meetings.

Field Personnel

Haley & Aldrich personnel are responsible for following the health and safety procedures specified in this HASP and for performing their work in a safe and responsible manner. Some of the specific responsibilities of the field personnel are as follows:

- Reading the HASP in its entirety prior to the start of on-site work;
- Submitting a completed Safety Plan Acceptance Form and documentation of medical surveillance and training to the SHSO prior to the start of work;
- Attending the pre-entry briefing prior to beginning on-site work;
- Bringing forth any questions or concerns regarding the content of the Safety Plan to the PM or the SHSO prior to the start of work;
- Stopping work when it is not believed it can be performed safely;
- Reporting all accidents, injuries and illnesses, regardless of their severity, to the SHSO;
- Complying with the requirements of this safety plan and the requests of the SHSO; and
- Reviewing the established JSAs for the site-specific hazards on a daily basis and prior to each shift change, if applicable.

Visitors

Authorized visitors (e.g., Client Representatives, Regulators, Haley & Aldrich management staff, etc.) requiring entry to any work location on the site will be briefed by the Site Supervisor on the hazards present at that location. Visitors will be escorted at all times at the work location and will be responsible for compliance with their employer's health and safety policies. In addition, this safety plan specifies the minimum acceptable qualifications, training and personal protective equipment which are required for entry to any controlled work area; visitors must comply with these



requirements at all times. Unauthorized visitors, and visitors not meeting the specified qualifications, will not be permitted within established controlled work areas.

SUBCONTRACTOR PERSONNEL

Subcontractor Site Representative

Each contractor and subcontractor shall designate a Contractor Site Representative. The Contractor Site Representative will interface directly with Insert Staff Name Here, the Subcontractor Site Safety Manager, with regards to all areas that relate to this safety plan and safety performance of work conducted by the contractor and/or subcontractor workforce. Contractor Site Representatives for this site are listed in the Contact Summary Table at the beginning of the Safety Plan.

Subcontractor Site Safety Manager

Each contractor / subcontractor will provide a qualified representative who will act as their Site Safety Manager (Sub-SSM). This person will be responsible for the planning, coordination, and safe execution of subcontractor tasks, including preparation of job hazard analyses (JHA), performing daily safety planning, and coordinating directly with the Haley & Aldrich SHSO for other site safety activities. This person will play a lead role in safety planning for Subcontractor tasks, and in ensuring that all their employees and lower tier subcontractors are in adherence with applicable local, state, and/or federal regulations, and/or industry and project specific safety standards or best management practices.

General contractors / subcontractors are responsible for preparing a site-specific HASP and/or other task specific safety documents (e.g., JHAs), which are, at a minimum, in compliance with local, state, and/or federal other regulations, and/or industry and project specific safety standards or best management practices. The contractor(s)/subcontractor(s) safety documentation will be at least as stringent as the health and safety requirements of the Haley & Aldrich Project specific HASP.

Safety requirements include, but are not limited to: legal requirements, contractual obligations and industry best practices. Contractors/subcontractors will identify a site safety representative during times when contractor/subcontractor personnel are on the Site. All contractor/subcontractor personnel will undergo a field safety orientation conducted by the Haley & Aldrich SHSO and/or PM prior to commencing site work activities. All contractors / subcontractors will participate in Haley & Aldrich site safety meetings and their personnel will be subject to training and monitoring requirements identified in this Safety Plan. If the contractors / subcontractors means and methods deviate from the scope of work described in Section 1 of this Safety Plan, the alternate means and methods must be submitted, reviewed and approved by the Haley & Aldrich SHSO and/or PM prior to the commencement of the work task. Once approved by the Haley & Aldrich SHSO and/or PM, the alternate means and methods submittal will be attached to this Safety Plan as an Addendum.



Site Specific Health & Safety Plan 90-02 Queens Boulevard Site 10/9/2023

ATTACHMENT D JOB SAFETY ANALYSES

Date printed: 11/13/2023 at 3:21 PM





90-02 QUEENS BOULEVARD SITE

KEY TASK ENTER TASK NUMBER.: ENTER TASK NAME.				
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Site Specific Health & Safety Plan 90-02 Queens Boulevard Site 10/9/2023 APPENDIX E NYSDEC DER-10 Appendix 1A/1B: NYSDOH Generic Community Health Plan/ Fugitive Dust and Particulate Monitoring



Appendix 1A New York State Department of Health Generic Community Air Monitoring Plan

Overview

A Community Air Monitoring Plan (CAMP) requires real-time monitoring for volatile organic compounds (VOCs) and particulates (i.e., dust) at the downwind perimeter of each designated work area when certain activities are in progress at contaminated sites. The CAMP is not intended for use in establishing action levels for worker respiratory protection. Rather, its intent is to provide a measure of protection for the downwind community (i.e., off-site receptors including residences and businesses and on-site workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative and remedial work activities. The action levels specified herein require increased monitoring, corrective actions to abate emissions, and/or work shutdown. Additionally, the CAMP helps to confirm that work activities did not spread contamination off-site through the air.

The generic CAMP presented below will be sufficient to cover many, if not most, sites. Specific requirements should be reviewed for each situation in consultation with NYSDOH to ensure proper applicability. In some cases, a separate site-specific CAMP or supplement may be required. Depending upon the nature of contamination, chemical- specific monitoring with appropriately-sensitive methods may be required. Depending upon the proximity of potentially exposed individuals, more stringent monitoring or response levels than those presented below may be required. Special requirements will be necessary for work within 20 feet of potentially exposed individuals or structures and for indoor work with co-located residences or facilities. These requirements should be determined in consultation with NYSDOH.

Reliance on the CAMP should not preclude simple, common-sense measures to keep VOCs, dust, and odors at a minimum around the work areas.

Community Air Monitoring Plan

Depending upon the nature of known or potential contaminants at each site, real-time air monitoring for VOCs and/or particulate levels at the perimeter of the exclusion zone or work area will be necessary. Most sites will involve VOC and particulate monitoring; sites known to be contaminated with heavy metals alone may only require particulate monitoring. If radiological contamination is a concern, additional monitoring requirements may be necessary per consultation with appropriate DEC/NYSDOH staff.

Continuous monitoring will be required for all <u>ground intrusive</u> activities and during the demolition of contaminated or potentially contaminated structures. Ground intrusive activities include, but are not limited to, soil/waste excavation and handling, test pitting or trenching, and the installation of soil borings or monitoring wells.

Periodic monitoring for VOCs will be required during <u>non-intrusive</u> activities such as the collection of soil and sediment samples or the collection of groundwater samples from existing monitoring wells. "Periodic" monitoring during sample collection might reasonably consist of taking a reading upon arrival at a sample location, monitoring while opening a well cap or

overturning soil, monitoring during well baling/purging, and taking a reading prior to leaving a sample location. In some instances, depending upon the proximity of potentially exposed individuals, continuous monitoring may be required during sampling activities. Examples of such situations include groundwater sampling at wells on the curb of a busy urban street, in the midst of a public park, or adjacent to a school or residence.

VOC Monitoring, Response Levels, and Actions

Volatile organic compounds (VOCs) must be monitored at the downwind perimeter of the immediate work area (i.e., the exclusion zone) on a continuous basis or as otherwise specified. Upwind concentrations should be measured at the start of each workday and periodically thereafter to establish background conditions, particularly if wind direction changes. The monitoring work should be performed using equipment appropriate to measure the types of contaminants known or suspected to be present. The equipment should be calibrated at least daily for the contaminant(s) of concern or for an appropriate surrogate. The equipment should be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

1. If the ambient air concentration of total organic vapors at the downwind perimeter of the work area or exclusion zone exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities must be temporarily halted and monitoring continued. If the total organic vapor level readily decreases (per instantaneous readings) below 5 ppm over background, work activities can resume with continued monitoring.

2. If total organic vapor levels at the downwind perimeter of the work area or exclusion zone persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities must be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities can resume provided that the total organic vapor level 200 feet downwind of the exclusion zone or half the distance to the nearest potential receptor or residential/commercial structure, whichever is less - but in no case less than 20 feet, is below 5 ppm over background for the 15-minute average.

3. If the organic vapor level is above 25 ppm at the perimeter of the work area, activities must be shutdown.

4. All 15-minute readings must be recorded and be available for State (DEC and NYSDOH) personnel to review. Instantaneous readings, if any, used for decision purposes should also be recorded.

Particulate Monitoring, Response Levels, and Actions

Particulate concentrations should be monitored continuously at the upwind and downwind perimeters of the exclusion zone at temporary particulate monitoring stations. The particulate monitoring should be performed using real-time monitoring equipment capable of measuring particulate matter less than 10 micrometers in size (PM-10) and capable of integrating over a period of 15 minutes (or less) for comparison to the airborne particulate action level. The equipment must be equipped with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration should be visually assessed during all work activities.

1. If the downwind PM-10 particulate level is 100 micrograms per cubic meter (mcg/m^3) greater than background (upwind perimeter) for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques must be employed. Work may continue with dust suppression techniques provided that downwind PM-10 particulate levels do not exceed 150 mcg/m³ above the upwind level and provided that no visible dust is migrating from the work area.

2. If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than 150 mcg/m³ above the upwind level, work must be stopped and a re-evaluation of activities initiated. Work can resume provided that dust suppression measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within 150 mcg/m³ of the upwind level and in preventing visible dust migration.

3. All readings must be recorded and be available for State (DEC and NYSDOH) and County Health personnel to review.

December 2009

Appendix 1B Fugitive Dust and Particulate Monitoring

A program for suppressing fugitive dust and particulate matter monitoring at hazardous waste sites is a responsibility on the remedial party performing the work. These procedures must be incorporated into appropriate intrusive work plans. The following fugitive dust suppression and particulate monitoring program should be employed at sites during construction and other intrusive activities which warrant its use:

1. Reasonable fugitive dust suppression techniques must be employed during all site activities which may generate fugitive dust.

2. Particulate monitoring must be employed during the handling of waste or contaminated soil or when activities on site may generate fugitive dust from exposed waste or contaminated soil. Remedial activities may also include the excavation, grading, or placement of clean fill. These control measures should not be considered necessary for these activities.

3. Particulate monitoring must be performed using real-time particulate monitors and shall monitor particulate matter less than ten microns (PM10) with the following minimum performance standards:

- (a) Objects to be measured: Dust, mists or aerosols;
- (b) Measurement Ranges: 0.001 to 400 mg/m3 (1 to 400,000 :ug/m3);

(c) Precision (2-sigma) at constant temperature: +/- 10 :g/m3 for one second averaging; and +/- 1.5 g/m3 for sixty second averaging;

(d) Accuracy: +/- 5% of reading +/- precision (Referred to gravimetric calibration with SAE fine test dust (mmd= 2 to 3 :m, g= 2.5, as aerosolized);

- (e) Resolution: 0.1% of reading or 1g/m3, whichever is larger;
- (f) Particle Size Range of Maximum Response: 0.1-10;
- (g) Total Number of Data Points in Memory: 10,000;

(h) Logged Data: Each data point with average concentration, time/date and data point number

(i) Run Summary: overall average, maximum concentrations, time/date of maximum, total number of logged points, start time/date, total elapsed time (run duration), STEL concentration and time/date occurrence, averaging (logging) period, calibration factor, and tag number;

(j) Alarm Averaging Time (user selectable): real-time (1-60 seconds) or STEL (15 minutes), alarms required;

(k) Operating Time: 48 hours (fully charged NiCd battery); continuously with charger;

(1) Operating Temperature: -10 to 50° C (14 to 122° F);

(m) Particulate levels will be monitored upwind and immediately downwind at the working site and integrated over a period not to exceed 15 minutes.

4. In order to ensure the validity of the fugitive dust measurements performed, there must be appropriate Quality Assurance/Quality Control (QA/QC). It is the responsibility of the remedial party to adequately supplement QA/QC Plans to include the following critical features: periodic instrument calibration, operator training, daily instrument performance (span) checks, and a record keeping plan.

5. The action level will be established at 150 ug/m3 (15 minutes average). While conservative,

this short-term interval will provide a real-time assessment of on-site air quality to assure both health and safety. If particulate levels are detected in excess of 150 ug/m3, the upwind background level must be confirmed immediately. If the working site particulate measurement is greater than 100 ug/m3 above the background level, additional dust suppression techniques must be implemented to reduce the generation of fugitive dust and corrective action taken to protect site personnel and reduce the potential for contaminant migration. Corrective measures may include increasing the level of personal protection for on-site personnel and implementing additional dust suppression techniques (see paragraph 7). Should the action level of 150 ug/m3 continue to be exceeded work must stop and DER must be notified as provided in the site design or remedial work plan. The notification shall include a description of the control measures implemented to prevent further exceedances.

6. It must be recognized that the generation of dust from waste or contaminated soil that migrates off-site, has the potential for transporting contaminants off-site. There may be situations when dust is being generated and leaving the site and the monitoring equipment does not measure PM10 at or above the action level. Since this situation has the potential to allow for the migration of contaminants off-site, it is unacceptable. While it is not practical to quantify total suspended particulates on a real-time basis, it is appropriate to rely on visual observation. If dust is observed leaving the working site, additional dust suppression techniques must be employed. Activities that have a high dusting potential-such as solidification and treatment involving materials like kiln dust and lime--will require the need for special measures to be considered.

7. The following techniques have been shown to be effective for the controlling of the generation and migration of dust during construction activities:

- (a) Applying water on haul roads;
- (b) Wetting equipment and excavation faces;
- (c) Spraying water on buckets during excavation and dumping;
- (d) Hauling materials in properly tarped or watertight containers;
- (e) Restricting vehicle speeds to 10 mph;
- (f) Covering excavated areas and material after excavation activity ceases; and
- (g) Reducing the excavation size and/or number of excavations.

Experience has shown that the chance of exceeding the 150ug/m3 action level is remote when the above-mentioned techniques are used. When techniques involving water application are used, care must be taken not to use excess water, which can result in unacceptably wet conditions. Using atomizing sprays will prevent overly wet conditions, conserve water, and provide an effective means of suppressing the fugitive dust.

8. The evaluation of weather conditions is necessary for proper fugitive dust control. When extreme wind conditions make dust control ineffective, as a last resort remedial actions may need to be suspended. There may be situations that require fugitive dust suppression and particulate monitoring requirements with action levels more stringent than those provided above. Under some circumstances, the contaminant concentration and/or toxicity may require additional monitoring to protect site personnel and the public. Additional integrated sampling and chemical analysis of the dust may also be in order. This must be evaluated when a health and safety plan is developed and when appropriate suppression and monitoring requirements are established for protection of health and the environment.