# 260 West 126<sup>th</sup> Street Remedial Investigation Work Plan

260 West 126<sup>th</sup> Street New York, New York Block 1931, Lot 56 BCP Site No. TBD

## Submitted to:

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# CERTIFICATION

I, Mohamed Ahmed, certify that I am currently a Qualif 6NYCRR Part 375 and that this Remedial Investigation V applicable statues and regulations and in substantial confo Site Investigation and Remediation (DER-10).	Vork Plan was prepared in accordance with all
DRAFT Mohamed Ahmed, P.G.	Date

#### 1.0 INTRODUCTION

On behalf of S & F 126 ST. LLC (the Applicant), Tenen Environmental, LLC (Tenen) has prepared this Remedial Investigation Work Plan (RIWP) for the property located at 260 West 126<sup>th</sup> Street, New York, New York (the Site). This RIWP is being submitted concurrently with a New York State Department of Environmental Conservation (NYSDEC) Brownfield Cleanup Program (BCP) Application. The Site location and layout are shown on Figures 1 and 2, respectively. This RIWP has been designed to meet the requirements of the BCP. The scope of work includes the collection of subsurface soil to assess conditions at the groundwater interface and proposed development depth; installation and sampling of four on-site groundwater monitoring wells to establish on-site groundwater conditions and groundwater flow direction; and installation and collection of six on-site soil vapor samples to assess vadose conditions at the groundwater interface. The results of this RI will be used to prepare a Remedial Investigation Report (RIR) and qualitative human health exposure assessment (QHHEA) and to support the development of a Remedial Action Work Plan (RAWP). This RIWP has been prepared in accordance with the New York State Department of Environmental Conservation (NYSDEC) Division of Environmental Remediation (DER) Technical Guidance for Site Investigation and Remediation (DER-10, May 3, 2010).

## 1.1 Work Plan Organization

This RIWP includes an introduction (Section 1), background information (Section 2), scope of work (Section 3), and proposed project schedule (Section 4). Quality assurance/quality control, health and safety (including community air monitoring), and project team information are addressed in separate appendices. Supporting tables and figures referenced throughout are included at the end and as attachments to this RIWP.

## 1.2 Summary of Previous Investigations

A Remedial Investigation was completed by Environmental Business Consultants (EBC) on behalf of Velocity Framers in November 2016 (Remedial Investigation Report dated December 2016) in accordance with the requirements of the New York City Mayor's Office of Environmental Remediation (NYC OER) E-Designation program and consisted of the installation of eight soil borings, the collection of 17 soil samples (including quality assurance/quality control [QA/QC] samples), the installation of four groundwater monitoring wells, the collection of four groundwater samples (including QA/QC samples), the installation of five soil vapor points, and the collection of five soil vapor samples. A summary of the previous RI findings is presented below. A detailed discussion of previous sampling events is included in Section 2.5 of this RIWP. Previous sample locations and sample results are depicted on Figures 3-6.

#### 1.2.1 Soil

Soil results were compared to NYSDEC Unrestricted Use (UU) and Restricted-Residential Use (RR) Soil Cleanup Objectives (SCOs) as listed in 6 NYCRR Part 375-6.8(a) and (b). UUSCOs are the most stringent cleanup standards and are used as comparison for site-specific proposed cleanup standards. The RRSCOs are consistent with the anticipated future use of the Site.

Soil sampling indicated the following:

- Several semivolatile organic compounds (SVOCs), most notably polycyclic aromatic hydrocarbons (PAHs), were detected in one shallow (0-2 feet below grade [ft-bg]) soil sample collected from the parking lot at concentrations exceeding the UUSCOs and RRSCOs.
- A variety of metals, including arsenic, barium, copper, chromium, lead, mercury, nickel, and zinc, were detected in shallow (0-2 ft-bg) and deep (10-12 ft-bg) soil samples across the Site in exceedance of UUSCOs. Of these, barium and lead were each detected in four shallow (0-2 ft-bg) and one deep (10-12 ft-bg) soil samples at concentrations exceeding their respective RRSCOs.

- Two pesticides, 4,4'-DDE and 4,4'-DDT, were detected in exceedance of their respective UUSCOs in three shallow (0-2 ft-bg) and one intermediate (2-4 ft-bg) soil samples, with the highest concentrations occurring in the sample collected from the northeastern corner of the Site. Pesticides were not detected in exceedance of RRSCOs in any soil samples.
- Volatile organic compounds (VOCs) and polychlorinated biphenyls were not detected in exceedance of UUSCOs or RRSCOs in any soil samples.

### 1.2.2 Groundwater

Groundwater results were compared to NYSDEC Technical and Operational Guidance Series (TOGS) 1.1.1 Ambient Water Quality Standards (AWQS) and Guidance Values (Class GA Standards).

Groundwater sampling indicated the following:

- A variety of SVOCs, specifically PAHs, were detected slightly in exceedance of Class GA Standards in groundwater samples collected from the southern portion of the Site.
- A variety of metals, including arsenic, barium, beryllium, cadmium, chromium, copper, iron, lead, magnesium, manganese, nickel, sodium, and thallium were detected in exceedance of Class GA Standards in unfiltered groundwater samples collected across the Site. Filtered groundwater samples were not analyzed as part of the 2016 RI.
- VOCs, pesticides, and PCBs were not detected in exceedance of Class GA Standards in any groundwater samples.

### 1.2.3 Soil Vapor

Soil vapor results were compared to the New York State Department of Health (NYSDOH) Soil Vapor Decision Matrices (Decision Matrices) as outlined in the October 2006 Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York (Soil Vapor Guidance) with May 2017 and February 2024 updates.

Soil vapor sampling indicated the following:

- Chlorinated VOCs (cVOCs) were detected in soil vapor across the Site, specifically tetrachloroethene (PCE), trichloroethene (TCE), carbon tetrachloride, and chloroform. In general, the highest concentrations of cVOCs were detected in the soil vapor sample collected from the southern portion of the parking lot, along the southern perimeter of the Site.
- A variety of petroleum-related VOCs were detected in soil vapor across the Site, including benzene, toluene, ethylbenzene, p/m-xylene, o-xylene, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene, 4-methyl-2-pentanone, 4-ethyltoluene, heptane, styrene, cyclohexane, and propylene. In general, the highest concentrations of petroleum-related VOCs were detected in the southern portions of the onsite building and parking lot.

### 1.3 Regulatory Interaction

One subsurface investigation has been completed to date to identify if impacts existed on-site due to historic site operations.

A Phase II Work Plan was prepared on behalf of Velocity Framers and submitted to NYC OER for the Site. A Remedial Investigation was subsequently conducted by EBC in November 2016 (Remedial Investigation Report dated December 2016) on behalf of Velocity Framers in accordance with NYC OER E-designation requirements.

Several years later, S & F 126 ST. LLC enrolled in the New York City Voluntary Cleanup Program (NYC VCP) to remediate a portion of Lot 56 on May 15, 2022 (NYC VCP #22CVCP053M). A hazardous materials (hazmat) Remedial Action Plan (RAP) was prepared by Tenen on behalf of Key Developers Inc. in March 2022 and approved by NYC OER. However, the proposed remediation of a portion of the Site under the VCP did not occur. The existing church building was excluded from the NYC VCP agreement, as the church building remained under the exclusive possession and control of the fee owner of the Site, Thomas Memorial Wesleyan Methodist Church, at that time. Additionally, only the eastern portion of the Site outside of the church building was contemplated for redevelopment at the time.

Following a change in conceptual redevelopment plans for the Site, a BCP Application was submitted for the entire Site (inclusive of the church building) to NYSDEC DER in conjunction with this RIWP. The work to be performed under this RIWP, as well as all future remedial work, will be performed in accordance with the requirements set forth in the forthcoming Brownfield Cleanup Agreement (BCA).

## 1.4 Work Plan Objectives

This RIWP has been developed to achieve the following BCP objectives:

- To fully characterize onsite subsurface soils, groundwater and soil vapor;
- To collect data sufficient to perform an onsite and offsite Qualitative Human Health Exposure Assessment; and,
- To produce data of sufficient quantity and quality to prepare a Remedial Action Work Plan (including alternatives analysis) to support the remediation of the Site.

#### 2.0 BACKGROUND

This section includes a description of the Site and surrounding uses, a summary of the proposed Site development, Site characteristics, and information regarding historical operations and regulatory interactions. Summaries of previous Site investigations are also provided.

## 2.1 Site Description and Surrounding Uses

The Site, located at 260 West 126<sup>th</sup> Street, New York, New York (Borough of Manhattan, Tax Block 1931, Lot 56), is a rectangular-shaped parcel located on the southern side of West 126<sup>th</sup> Street, between Frederick Douglass Boulevard (formerly Eighth Avenue) and Adam C. Powell Boulevard, in the Harlem section of Manhattan. The Site is approximately 12,490 square feet (SF) and has approximately 125 feet of frontage along West 126<sup>th</sup> Street and is approximately 100 feet deep.

The Site is developed with a two-story church building with a full cellar level covering approximately 2,725 SF of the western portion of the property. The church building is currently vacant and recently experienced a fire, rendering it unsafe to occupy. The remainder of the property is a paved, vacant parking lot.

The Site lot is zoned C4-4D, denoting a commercial contextual district that has bulk and density regulations and also allows for some residential uses. Site location, layout and usage are depicted on Figure 1.

## 2.2 Proposed Development

The Site will be redeveloped with a new 14-story mixed-use residential and community facility building with a full cellar that will encompass the entirety of the Site. Based on present design and plans, the redevelopment will include approximately 20-25 affordable residential units at 80% area median income (AMI) utilizing the Voluntary Inclusionary Housing (VIH) and 421-a programs. Demolition of the existing Site structure (two-story building with a full cellar) will be completed to facilitate site remediation as part of the redevelopment. The building design and plans are still being developed for the project; however, it is anticipated that excavation for development will extend approximately 30 feet below grade (ft-bg) across the Site. The water table was encountered at approximately 23 to 25 ft-bg and is expected to be encountered during excavation. The proposed development is consistent with the existing zoning and the recent development in this area of Manhattan.

#### 2.3 Site Characteristics

#### 2.3.1 Site Topography

Based on the U.S. Geological Survey (USGS 7.5 Minute Topographic Quadrangles: Central Park, NY) topographic map, the Site is located at an elevation of approximately 32 feet above mean sea level (msl) and is relatively flat.

### 2.3.2 Site Geology and Hydrogeology

The Site is underlain by a historic fill layer (sand, silt, ash, gravel, rock, and brick fragments) extending to at least 12 ft-bg. Soil below 12 ft-bg was not investigated as part of EBC's November 2016 RI and therefore the soil type at these depths is unknown.

Depth to groundwater at the Site was measured at depths ranging from 22.90 to 24.67 feet below grade. Groundwater flow is generally towards the south, likely due to the presence of sub-grade structures because the general groundwater flow direction would be expected to move in an easterly direction. The nearest subway line is located two blocks to the east of the Site along Malcom X Boulevard, and due to distance is not likely to impact groundwater flow direction.

## 2.4 Historical Operations

The Site lot has been developed since at least 1902. In 1902, the Site lot was divided into six separate tax parcels with six tightly grouped four-story residential buildings present on the north side of the Site, along West 126<sup>th</sup> Street. By 1951, the residential buildings had been demolished and the Site was mostly undeveloped except for the western portion of the Site, where a four-story storefront building with lumber storage remained. By 1962, the existing two-story church building with a full cellar level was present on the western portion of the Site. The Site has generally remained unchanged through current day with a two-story building on the western portion of the Site and licensed parking on the eastern portion of the Site.

#### 2.5 Previous Investigations

Previous assessments and investigations include an October 2016 Phase I Environmental Site Assessment (ESA) Report and a November 2016 Remedial Investigation, both conducted by EBC on behalf of Velocity Framers. A Remedial Investigation was completed by EBC in November 2016 in compliance with NYC OER E-designation requirements (Remedial Investigation Report dated December 2016). The Remedial Investigation Report, inclusive of analytical laboratory reports in their entirety, is included as Appendix A. The results of historical sampling are summarized on Figures 4-6, and a map depicting all previous sampling locations is included as Figure 3. A summary of previous analytical results are included as Tables 2 through 4. A detailed description of all previous assessments and investigations is included in the sections below. Based on the previous investigation conducted at the Site, the primary contaminants of concern for the Site are cVOCs, petroleum-related VOCs, PAHs, and metals (barium and lead).

# <u>2.5.1</u> Phase I Environmental Site Assessment Report, 260 West 126<sup>th</sup> Street, New York, NY, Environmental Business Consultants, October 2016.

A Phase I ESA was completed for the Site by EBC in October 2016 on behalf of Velocity Framers of Brooklyn, New York. The Phase I ESA did not identify any Recognized Environmental Conditions (RECs) in connection with the Site. However, EBC did identify three other environmental concerns (non-RECs), which are summarized as follows:

- The Site has an E-Designation (E-201) for Hazardous Materials, Noise, and Air as a result of the 125<sup>th</sup> Street Corridor Rezoning and Related Actions. The HazMat E-Designation required a Phase I and Phase II Testing Protocol. The Air E-Designation requires that any new residential and/or commercial development must endure that the heating, ventilation and air conditioning (HVAC) stacks are located at least 79 and 63 feet for No. 4 fuel oil and No. 2 fuel oil from the lot lines, respectively, or use natural gas as the type of fuel for space heating and hot water systems to avoid any potential significant adverse air quality impacts. The Noise E-Designation required a 40 dBA attenuation level for indoor spaces.
- Due to the age of the onsite building, the potential for the presence of asbestos-containing materials (ACMs) is considered likely. Specifically, asphaltic roofing materials, adhesives, plasters, and exterior use caulks commonly contain asbestos in older and newer buildings alike. EBC recommended conducting an ACM survey to identify building materials and components with asbestos content prior to demolition or disturbance.
- Due to the age of the onsite building, the potential for the presence of lead-based paint (LBP) is also considered likely. No significant damaged or peeling paints were identified; however, LBP may be present in hidden or inaccessible portions of the Site. Additionally, no children currently reside at the Site, which greatly reduces the risks associated with the potential presence of LBP. EBC recommended conducting a lead survey to evaluate the presence of LBP at the Site prior to demolition or disturbance.

# 2.5.2 Remedial Investigation Report, 260 West 126<sup>th</sup> Street, New York, NY, Environmental Business Consultants, December 2016.

The November 2016 RI was conducted by EBC on behalf of Velocity Framers in accordance with the requirements of the NYC OER E-Designation program and consisted of the installation of eight soil borings, the collection of 17 soil samples (including QA/QC samples), the installation of four groundwater monitoring wells, the collection of four groundwater samples (including QA/QC samples), the installation of five soil vapor points, and the collection of five soil vapor samples. All soil and groundwater samples collected during the November 2016 RI were analyzed for VOCs, SVOCs, target analyte list (TAL) metals (total metals only for groundwater), pesticides, and PCBs and all soil vapor samples collected during the RI were analyzed for TO-15 VOCs. The results of the November 2016 RI are detailed below:

#### Soil

Soil results were compared to NYSDEC UUSCOs and RRSCOs as listed in 6 NYCRR Part 375-6.8(a) and (b). UUSCOs are the most stringent cleanup standards and are used as comparison for site-specific proposed cleanup standards. The RRSCOs are consistent with the assumed future use of the Site.

Soil sampling indicated the following:

- Several SVOCs, most notably PAHs, were detected in one shallow (0-2 feet below grade [ft-bg]) soil sample collected from the parking lot at concentrations exceeding the UUSCOs and RRSCOs. Total SVOCs were detected at a max. concentration of 13.07 parts-per-million (ppm).
- A variety of metals, including arsenic, barium, copper, chromium, lead, mercury, nickel, and zinc, were detected in shallow (0-2 ft-bg) and deep (10-12 ft-bg) soil samples across the Site in exceedance of UUSCOs. Of these, barium (max. 2,760 ppm) and lead (max. 2,040 ppm) were each detected in four shallow (0-2 ft-bg) and one deep (10-12 ft-bg) soil samples at concentrations exceeding their respective RRSCOs.
- Two pesticides, 4,4'-DDE and 4,4'-DDT, were detected in exceedance of their respective UUSCOs in were detected in exceedance of their respective UUSCOs in three shallow (0-2 ft-bg) and one intermediate (2-4 ft-bg) soil samples, with the highest concentrations occurring in the shallow sample collected from the northeastern corner of the Site. 4,4'-DDE was detected at a max. concentration of 0.022 ppm and 4,4'-DDT was detected at a concentration of 0.025 ppm. Pesticides were not detected in exceedance of RRSCOs in any soil samples.
- Volatile organic compounds (VOCs) and polychlorinated biphenyls were not detected in exceedance of UUSCOs or RRSCOs in any soil samples.

#### Groundwater

Groundwater results were compared to NYSDEC Technical and Operational Guidance Series (TOGS) 1.1.1 Ambient Water Quality Standards (AWQS) and Guidance Values (Class GA Standards).

Groundwater sampling indicated the following:

- A variety of SVOCs, specifically PAHs, were detected slightly in exceedance of Class GA Standards in groundwater samples collected from the southern portion of the Site.
- A variety of metals, including arsenic, barium, beryllium, cadmium, chromium, copper, iron, lead, magnesium, manganese, nickel, sodium, and thallium were detected in exceedance of Class GA Standards in unfiltered groundwater samples collected across the Site. Filtered groundwater samples were not analyzed as part of the 2016 RI.

• VOCs, pesticides, and PCBs were not detected in exceedance of Class GA Standards in any groundwater samples.

## Soil Vapor

Soil vapor results were compared to the New York State Department of Health (NYSDOH) Soil Vapor Decision Matrices (Decision Matrices) as outlined in the October 2006 Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York (Soil Vapor Guidance) with May 2017 and February 2024 updates.

Soil vapor sampling indicated the following:

- Chlorinated VOCs (cVOCs) were detected in soil vapor across the Site, specifically tetrachloroethene (PCE), trichloroethene (TCE), carbon tetrachloride, and chloroform. PCE was detected at a max. concentration of 16.5 micrograms per cubic meter (ug/m3); TCE was detected at a max. concentration of 0.62 ug/m3; carbon tetrachloride was detected at a max. concentration of 0.52 ug/m3; and chloroform was detected at a max. concentration of 2.24 ug/m3. In general, the highest concentrations of cVOCs were detected in the soil vapor sample collected from the southern portion of the parking lot, along the southern perimeter of the Site.
- A variety of petroleum-related VOCs were detected in soil vapor across the Site, including benzene, toluene, ethylbenzene, p/m-xylene, o-xylene, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene, 4-methyl-2-pentanone, 4-ethyltoluene, heptane, styrene, cyclohexane, and propylene. Total BTEX concentrations ranged from 20.58 ug/m3 to 41.44 ug/m3. Of the BTEX compounds, the highest concentration was detected for toluene at 16.5 ug/m3. In general, the highest concentrations of petroleum-related VOCs were detected in the southern portions of the onsite building and parking lot.

#### 3.0 REMEDIAL INVESTIGATION

The Remedial Investigation (RI) proposed for the Site includes sampling of soil, soil vapor, ambient air and groundwater. The objectives of this RI are to fully characterize the soil, groundwater and soil vapor onsite and to provide data of sufficient quantity and quality to support development of a Remedial Investigation Report (RIR), Qualitative Human Health Exposure Assessment (QHHEA) and Remedial Action Alternatives Analysis.

This RIWP was developed to meet the following Site-specific objectives:

- Fully characterize onsite subsurface soil and groundwater, consistent with DER-10;
- Evaluate potential sources of contamination, the migration pathways, and actual or potential receptors of contaminants on or through soil, groundwater and soil vapor;
- Assess soil vapor conditions across the Site;
- Assess potential impacts to human health as a result of the release of contaminants at the Site.

## 3.1 Scope of Remedial Investigation

The scoping process, for the purpose of identifying and defining the RI tasks described below, included the following:

- Review of current and historical Site reports and data;
- Review of results from previous environmental assessments, work plans and reports; and
- Evaluation of DER-10 requirements and relevant State and Federal guidance documents.

The RI will begin following completion of the required 30-day public comment period. Following the NYSDEC approval of this RIWP, the RI will include the collection 18 soil samples from six soil borings, installation of four on-site groundwater monitoring wells, collection of four groundwater samples, installation of six temporary soil vapor points, collection of six soil vapor samples and collection of one outdoor ambient air sample. The type, location, and rationale for each exploration are detailed in the sections below and in summary table included in Section 3.6. Soil borings, monitoring wells, and soil vapor points are not proposed within the existing church building in the western portion of the Site due to safety concerns regarding the structural integrity of the building following a recent fire.

Installation of soil borings, groundwater monitoring wells, and soil vapor monitoring points will be completed in accordance with the sections below and the standard procedures included in the Quality Assurance Project Plan (QAPP) and Health and Safety Plan (HASP), included as Appendix B and C, respectively. Following the collection of this data, review and evaluation will be performed in order to determine if additional investigation is needed.

Proposed RI sample locations are shown on Figure 7. A summary table of proposed samples and sampling rationale is included in Section 3.6 of this Work Plan.

#### 3.2 Soil Sampling

A subsurface investigation will be performed to further characterize soil conditions onsite.

The following scope of work will be implemented:

• Advance six soil borings (SB-1 through SB-6) across the exterior portion of the Site to approximately two feet below the proposed development depth (30 ft-bg);

- Collect a minimum of 18 soil samples, or three from each soil boring. Soil samples will be collected
  from the grade to two ft-bg interval, from the two-foot interval directly above the groundwater
  interface, and from the two-foot interval directly below the proposed development depth (30-32 ftbg). If visual, olfactory, or PID evidence of contamination is noted in a boring, samples will also
  be collected from the two-foot interval of highest suspected contamination and the next apparent
  non-impacted interval; and,
- Analyze all soil samples for VOCs, SVOCs, pesticides, herbicides, PCBs, TAL metals (including total cyanide, tri- and hexavalent chromium), per- and polyfluoroalkyl substances (PFAS), and 1,4-dioxane.

## 3.2.1 Soil Sampling Methodology

A total of six soil borings will be advanced in the exterior portion of the Site as part of this RI; proposed soil sample locations are shown on Figure 7. Boring are not proposed within the existing church building in the western portion of the Site due to safety concerns regarding the structural integrity of the building following a recent fire. Based on field measurements and observations, boring locations may be moved or added. Coordination with NYSDEC will take place prior to modifications being made with regard to the above-described placement of proposed sampling locations.

The soil borings will be installed using a direct-push, track-mounted Geoprobe® unit. All soil borings will be advanced to approximately two feet below the proposed development depth (approximately 30-32 ft-bg). Soil samples will be collected from five-foot macrocores fitted with dedicated acetate liners. At each location, the liners for each interval will be opened and the soil screened for VOCs using a 10.6 electron-volt (EV) PID. The soil retrieved from each sampler will be described by Tenen field staff on boring logs using the Unified Soil Classification System. All observations regarding potential contamination such as odors, staining, etc. will be documented. Soil will be screened from grade to the terminal depth of each boring. All descriptions and observations will be documented in a field logbook.

A minimum of three soil samples will be collected from each soil boring. Soil samples will be collected from the grade to two ft-bg interval, from the two-foot interval directly above the groundwater interface, and from the two-foot interval directly below the proposed development depth (i.e., 30-32 ft-bg). Additional soil samples will be collected if PID, visual, or olfactory evidence of contamination is noted in the soil column. Samples will be collected from the two foot interval of highest suspected contamination and from the next apparent non-impacted two foot interval.

Soil samples selected for laboratory analysis will be collected directly from the acetate liner, placed in precleaned, pre-preserved laboratory-provided sample bottles or En Core samplers (En Novative Technologies), sealed and labeled, and placed in a cooler and chilled to 4°C for transport under chain-of custody procedures. Soil samples will be submitted to a NYSDOH ELAP-certified laboratory via courier service and analyzed for the respective analyte lists included in 6 NYCRR Part 375 SCOs. Laboratory analytical parameters and methods are outlined below. QA/QC procedures to be followed are described in the QAPP included as Appendix B.

A minimum of three soil samples collected from each soil boring will be analyzed for the following analytes on the Part 375 list with a Category B deliverable package:

- Target Compound List (TCL) VOCs by EPA Method 8260;
- SVOCs by EPA Method 8270;
- Pesticides by EPA Method 8081B;
- Herbicides by EPA Method 8151A;
- PCBs by EPA Method 8082A;
- Target Analyte List (TAL) Metals by EPA 6010C/7471B;
- Total Cyanide by EPA Method 9010C;

- Trivalent and Hexavalent Chromium by EPA Method 3060A;
- PFAS by USEPA Method 1633; and
- 1,4-Dioxane by USEPA Method 8270.

Emerging contaminants (e.g., PFAS and 1,4-Dioxane) will be sampled in accordance with the current NYSDEC guidance, *Guidelines for Sampling and Analysis of PFAS Under NYSDEC's Part 375 Remedial Programs* (April 2023).

## 3.3 Groundwater Sampling

The following scope of work is proposed to further characterize groundwater onsite:

- Four permanent monitoring wells will be installed to further assess and characterize groundwater conditions on-site. All wells will be advanced five feet into the groundwater table;
- Gauge and collect groundwater samples from four newly installed wells;
- Groundwater samples will be analyzed for VOCs, SVOCs, pesticides, herbicides, PCBs, total and dissolved TAL metals (including total cyanide, tri- and hexavalent chromium), PFAS and 1,4dioxane.
- Survey newly installed monitoring wells, collect one round of depth-to-groundwater measurements from all wells, evaluate groundwater elevations, and present updated groundwater contours.

## 3.3.1 Groundwater Well Sampling

Four newly installed permanent monitoring wells will be sampled; proposed monitoring well locations are depicted on Figure 7. All sampling equipment will be decontaminated prior to use. Prior to sampling, water levels will be measured using an electronic product-water level indicator. Sample collection will be accomplished by using low-flow procedures. Samples will not be collected until pH, temperature, and conductivity measurements stabilize and the turbidity reading is 50 Nephelometric Turbidity Units (NTU) or less, or stabilizes above 50 NTU.

Newly installed monitoring wells will be installed using a Geoprobe® direct-push rig. At each location, soil will be screened for VOCs using a PID. Newly installed monitoring wells will be constructed of two-inch inner diameter (ID) PVC casing and riser with a ten-foot PVC screen (0.020-inch slot). The ten-foot screen will straddle the groundwater interface (five feet above and five feet below). A filter pack of sand will be placed in the annular space around the screen of the monitoring wells (minimum 2-inches around the circumference of the screen) and will extend two feet above the screen. The annular area around the well casing above the sand pack will be sealed with hydrated bentonite pellets for an interval of two feet. The annular space above the bentonite pellets to one ft-bg will be backfilled with unimpacted drill cuttings or clean sand. Upon completion of the well, a locking well cap will be installed atop the PVC riser and a steel flush-mount roadbox and concrete apron will be installed at grade.

Groundwater well construction logs will be completed for all of the newly constructed wells, including description of the lithology, top of casing, and screening interval. Boring and well construction logs will be included in the RIR.

Newly installed wells will be developed on the day of installation by pumping, using a peristaltic pump and dedicated high-density polyethylene (HDPE) tubing. The wells will be developed until at least three well volumes have been evacuated, turbidity reaches 50 NTU or less or stabilizes above 50 NTU, and pH, temperature, and conductivity measurements stabilize. Stability is defined as variation between field measurements of 10 percent or less and no overall upward or downward trend in measurements. Previously installed wells to be sampled as part of this RI will be surged one week prior to sampling to remove potential blockages and promote the movement of fines and water through the well screens.

All newly installed wells will be surveyed to a common datum (likely North American Vertical Datum of 1988 [NAVD 88]).

Groundwater samples will be collected using low-flow techniques in accordance with EPA Region 1 Low-Stress (Low-Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells. (EQASOP-GW 001 Revision 3, dated July 30, 1996 with January 19, 2010 revision). All groundwater samples will be collected directly from dedicated tubing and placed in pre-cleaned, pre-preserved laboratory provided sample bottles, cooled to 4°C in the field, and transported under chain-of-custody command to the designated laboratory for analysis. Groundwater samples from newly installed wells will be collected at least one week after installation and analyzed for the following analytes on the Part 375 list and emerging contaminants with a Category B deliverable package:

- Target Compound List (TCL) VOCs by EPA Method 8260;
- SVOCs by EPA Method 8270;
- Pesticides by EPA Method 8081B;
- Herbicides by EPA Method 8151A;
- PCBs by EPA Method 8082A;
- Total and Dissolved TAL Metals by EPA 6010C/7471B;
- Total and Dissolved Cyanide by EPA Method 9010C;
- Total and Dissolved Trivalent and Hexavalent Chromium by EPA Method 3060A;
- PFAS by USEPA Method 1633; and
- 1,4-Dioxane by USEPA Method 8270.

Emerging contaminants (e.g., PFAS and 1,4-Dioxane) will be sampled in accordance with the current NYSDEC guidance, *Guidelines for Sampling and Analysis of PFAS Under NYSDEC's Part 375 Remedial Programs* (April 2023).

## 3.4 Soil Vapor and Ambient Air Sampling

The following scope of work is proposed to be implemented to determine soil vapor conditions at the site:

- Install six soil vapor points across the exterior portion of the Site. One soil vapor sample (SV-1) will be installed in the center of the Site to a depth of approximately three feet above the groundwater interface (approximately 23 to 25 ft-bg) to assess soil vapor conditions at the Site. Five soil vapor samples (SV-2 through SV-6) will be installed along the perimeters of the Site to the anticipated depth of the nearest building foundation to assess potential soil vapor migration offsite;
- One outdoor ambient air sample will be collected for each soil vapor sampling event; and,
- Analyze soil vapor and ambient air samples for EPA Method TO-15 VOCs.

### 3.4.1 Soil Vapor Sampling Methodology

A total of six soil vapor samples and one outdoor ambient air sample will be collected as part of this RI. Samples will be collected in accordance with the NYSDOH Soil Vapor Guidance.

A Geoprobe® direct push machine will be used to install the soil vapor sampling probes. At each soil vapor sampling location, access to the subsurface soil will be gained by drilling through the top surface material (concrete) using a drill bit. Upon penetration through the surface material, a disposable sampling probe consisting of a 1.5-inch long hardened point and a 6-inch long perforated vapor intake will be installed to a depth of approximately three feet above the groundwater interface (approximately 23 to 25 ft-bg) for SV-

1 or to the anticipated depth of the nearest building foundation for SV-2 through SV-6.

At the terminal depth of soil vapor locations, the sample probe will be attached to ¼-inch diameter Teflon® tubing and extended to the surface. The borehole above the sampling probe to grade will be sealed using an inert sealant to prevent ambient air mixing with the soil vapor. Ambient air will be purged from the boring hole by attaching the surface end of the ¼-inch diameter Teflon® tube to an air valve and then to a vacuum pump. The vacuum pump will remove no more than one to three volumes of air (volume of the sample probe and tube) prior to sample collection. The flow rate for both purging and sample collection will not exceed 0.2 liters per minute.

The soil vapor samples will be first screened for VOCs using a PID. A tracer gas (helium) will be used in accordance with the NYSDOH protocols to verify the integrity of the soil vapor probe seal. Helium will be used as the tracer gas and a bucket will serve to keep it in contact with the probe during testing. A portable monitoring device will be used to analyze a sample of soil vapor for the tracer prior to sampling. Although there is an allowable amount of tracer gas that can be detected as per the NYSDOH Soil Vapor Guidance, if the tracer sample results show any presence of the tracer gas, the probe seals will be adjusted to prevent infiltration which would result in the generation of inaccurate (likely biased low) results.

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.

Soil vapor samples will be collected in laboratory-supplied and batch-certified-clean 6-liter Summa canisters using two-hour regulators. All samples will be sealed, labeled, and placed in a secure container for delivery to a NYSDOH ELAP-certified analytical laboratory. All soil vapor samples will be analyzed for EPA Method TO-15 VOCs.

#### 3.4.2 Ambient Air Sampling Methodology

All samples will be collected in accordance with the NYSDOH Soil Vapor Guidance. Sample locations may be adjusted based on field observations or conditions.

One outdoor ambient air sample will be collected as part of this RI. The outdoor ambient air sample will be collected from breathing height (three to five feet above the ground surface) from an upgradient sampling location. The sampling flow rate will not exceed 0.2 liters per minute (L/min). Sampling will occur for a duration of eight hours. 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.

The ambient air sample will be collected in a laboratory-supplied and batch-certified-clean 6-liter Summa canister using an eight-hour regulator and will be sealed, labeled, and placed in a secure container for delivery to a NYSDOH ELAP-certified analytical laboratory. The sample will be analyzed for EPA Method TO-15 VOCs.

## 3.5 Quality Assurance / Quality Control (QA/QC)

Samples will be collected in accordance with the Quality Assurance Project Plan (QAPP) included as Appendix B.

Sample analysis will be performed by a NYSDOH ELAP-certified laboratory. The laboratory will report sample results on a 10-day turnaround time. An independent sub-consultant will validate sample results and prepare a Data Usability Summary Report (DUSR).

## 3.6 Summary Table of Proposed Sampling Locations

As required by Section 3.3(b)(3) of DER-10, a table describing all proposed sampling locations and QA/QC samples is presented below.

**Table 1. Proposed Sampling Locations and Analysis** 

<u>Soil</u>

Sample Location	Media	Sampling Intervals	Analytical Parameters	Sampling Method / Minimum Reporting Levels	Rationale	QA/QC Samples														
SB-1		From grade to two ft-bg, from the two-foot interval above the																		
SB-2		groundwater interface, and from the two-foot interval directly below		EPA 1633 / MDL less than 1 ug/kg for PFAS; EPA 8270 / MDL less than 0.1																
SB-3	Soil	samples will be PFAS, and	Part 375 Analytes, PFAS, and 1,4-	mg/kg for 1,4- Dioxane; EPA 8260C, 8270D, 8081B, 8082A,	Evaluate/fully characterize soil conditions across the	1 duplicate, 1 field blank, and 1 MS/MSD per 20 samples; 1 trip														
SB-4	Son		foot interval of highest suspected contamination	foot interval of highest suspected contamination	foot interval of highest suspected contamination	foot interval of highest suspected contamination	foot interval of highest suspected contamination	foot interval of highest suspected contamination	foot interval of highest suspected contamination	Dioxane	8151A, 7196A, 3050B, 7471B, 9010C/9012B/9014	Site.	blank per sample delivery group							
SB-5	SB-5 non-ir interva olfac			and 3060A/7196 / MDL less than Unrestricted Use SCOs																
SB-6		encountered in the soil column																		

## Groundwater

Sample Location	Media	Sampling Intervals	Analytical Parameters	Sampling Method / Minimum Reporting Levels	Rationale	QA/QC Samples
MW-1			Part 375	EPA 8260C, 8270D, 8270D- SIM Modified, 8081B,		
MW-2		10-ft screen across groundwater	analytes including total and	8151A, 7196A, 3050B, 7471B, 9010C/9012B/9014, 3060A/7196 and 1633 / MDL	Fully characterize	1 duplicate, 1 field blank, and 1 MS/MSD per 20
MW-3	Groundwater	interface (5 ft above and 5 ft below)	dissolved metals; 1,4- dioxane;	less than Class GA Standards for Part 375 analytes; MDL less than 0.28 ug/L for 1,4-	onsite groundwater	samples; 1 trip blank per sample delivery group
MW-4			PFAS	dioxane; MDL less than 2 nanograms per liter (ng/L) for PFAS		

## Soil Vapor and Outdoor Air

Sample Location	Media	Sampling Intervals	Analytical Parameters	Sampling Method / Minimum Reporting Levels	Rationale	QA/QC Samples
SV-1		3 ft above the groundwater interface (approximately 23 to 25 ft-bg)			Assess soil vapor conditions on-site at the groundwater interface	
SV-2						
SV-3	Soil			EPA TO-15 /	Assess soil vapor	
SV-4	Vapor	Anticipated depth of the nearest neighboring	TO-15 VOCs	MDL less than 1.00	conditions at the Site perimeters and the potential for soil	None
SV-5		building's foundation		ug/m3	vapor to migrate offsite	
SV-6	SV-6					
AA-1	Ambient Air	Breathing height (3-5 feet above the ground)			Assess ambient air conditions onsite	

## Quality Assurance / Quality Control

Sample Location	Media	Sampling Intervals	Analytical Parameters	Rationale
Trip Blanks Soil Duplicate Soil Blank Soil MS/MSD Groundwater Duplicate Groundwater Blank Groundwater MS/MSD	QA / QC		Part 375 analytes and/or 1,4-dioxane and PFAS, as necessary	Quality assurance and quality control

### MDL – Method Detection Limit

Reporting limits are laboratory- and sampling event-specific. The overall objective is to ensure that the minimum reporting levels are such that they can be used to evaluate potential sources, assess risk from detected compounds, and compare detected concentrations against applicable regulatory levels.

## 3.7 Qualitative Exposure Assessment

Following receipt of the laboratory analytical results, a QHHEA will be completed in accordance with Section 3.3(c)4 and Appendix B (NYSDOH guidance for preparing a qualitative human health exposure assessment) of DER-10. The QHHEA will utilize the results of the RI to evaluate and document potential exposure routes and identify and characterize potential current and future receptors. The results of the RI will be used to identify potential human exposure scenarios associated with contaminants in soil vapor, soil, and groundwater. The results of the QHHEA will be included in the RIR.

#### 3.8 Health and Safety Plan (HASP)

All work at the Site will be completed in accordance with the Health and Safety Plan (HASP) included in Appendix C.

## 3.9 Air Monitoring

The NYSDOH Generic Community Air Monitoring Plan (CAMP), included as Appendix 1A of DER-10, will be implemented during all ground-intrusive sampling activities. Special requirements will be implemented during any ground-intrusive work occurring within twenty feet of potentially exposed individuals or building openings (windows, vents, doors, etc.). Also, special requirements will be implemented during all work completed inside an occupied building. Details of the CAMP are included in Appendix D.

Daily CAMP reports will be sent to the NYSDOH and NYSDEC Project Managers via email. Daily reports will include a Site figure depicting Work Zones; activities; wind direction, in addition to CAMP monitor readings and CAMP station locations. Any exceedances of CAMP readings, corrective actions taken, and indication of whether the corrective actions reduced readings to below action levels will be communicated to the NYSDEC and the NYSDOH Project Managers as soon as practicable on the day of occurrence.

#### 3.10 Investigation-Derived Waste (IDW)

Following the completion of sampling, boreholes will be backfilled with clean cuttings or sand. If grossly contaminated soil cuttings are encountered or if excess soil cuttings are generated, they will be placed in 55-gallon drums. Purge water and other investigation-derived waste (IDW) will be containerized in 55-gallon drums. After the investigation is complete, the drum contents will be characterized for offsite disposal and, if analytical concentrations do not indicate the waste materials need to be disposed of as hazardous waste, a contained-in determination will be requested from NYSDEC.

## 3.11 Reporting

A RIR will be prepared in accordance with the requirements of DER-10. The report will include details of the sampling, tabulated sample results and an assessment of the data and conclusions. If warranted, recommendations for additional actions will be included.

Soil sample results will be compared to the Unrestricted Use and Restricted-Residential Use SCOs as included in Part 375-6.8. Groundwater sample results will be compared to the Class GA Standards, including February 2024 updates. Soil vapor sample results will be compared to the ambient air sample results. The results of all media will be evaluated comprehensively to determine the need for additional investigation and/or remediation.

The Remedial Investigation Report will also include the qualitative human health exposure assessment, CAMP results, laboratory data packages, DUSR, geologic logs, well construction diagrams, and well purging/sampling logs. All data will also be submitted electronically to NYSDEC via the Environmental Information Management System (EIMS) in EqUIS format.

# 4.0 SCHEDULE

The following project schedule has been developed for the work plan implementation.

Work Plan Implementation Schedule

Task	Task	Estimated Task	Total Duration
Number		Duration	(business days)
		(business days)	
0	Work Plan Approval	0	0
1	Mobilization	1	1
2	Soil Boring, Monitoring Well and Exterior Soil	2	4
	Vapor Point Installation / Soil Sampling	3	4
3	Groundwater and Soil Vapor Sampling and Well	1	5
3	Survey	1	3
4	Laboratory Analysis	10	15
5	Draft Report and Data Validation	30	45
6	Submittal of RI Report	1	46

Note: There will be a 30-day public comment period on the RIWP prior to work plan approval.

### 5.0 REFERENCES

New York State Department of Environmental Conservation, Division of Environmental Remediation. DER Technical Guidance for Site Investigation and Remediation (DER-10). NYSDEC 2010.

New York State Department of Environmental Conservation DEC Policy. Commissioner's Policy 51 – Soil Cleanup Guidance. October 21, 2010. NYSDEC 2010.

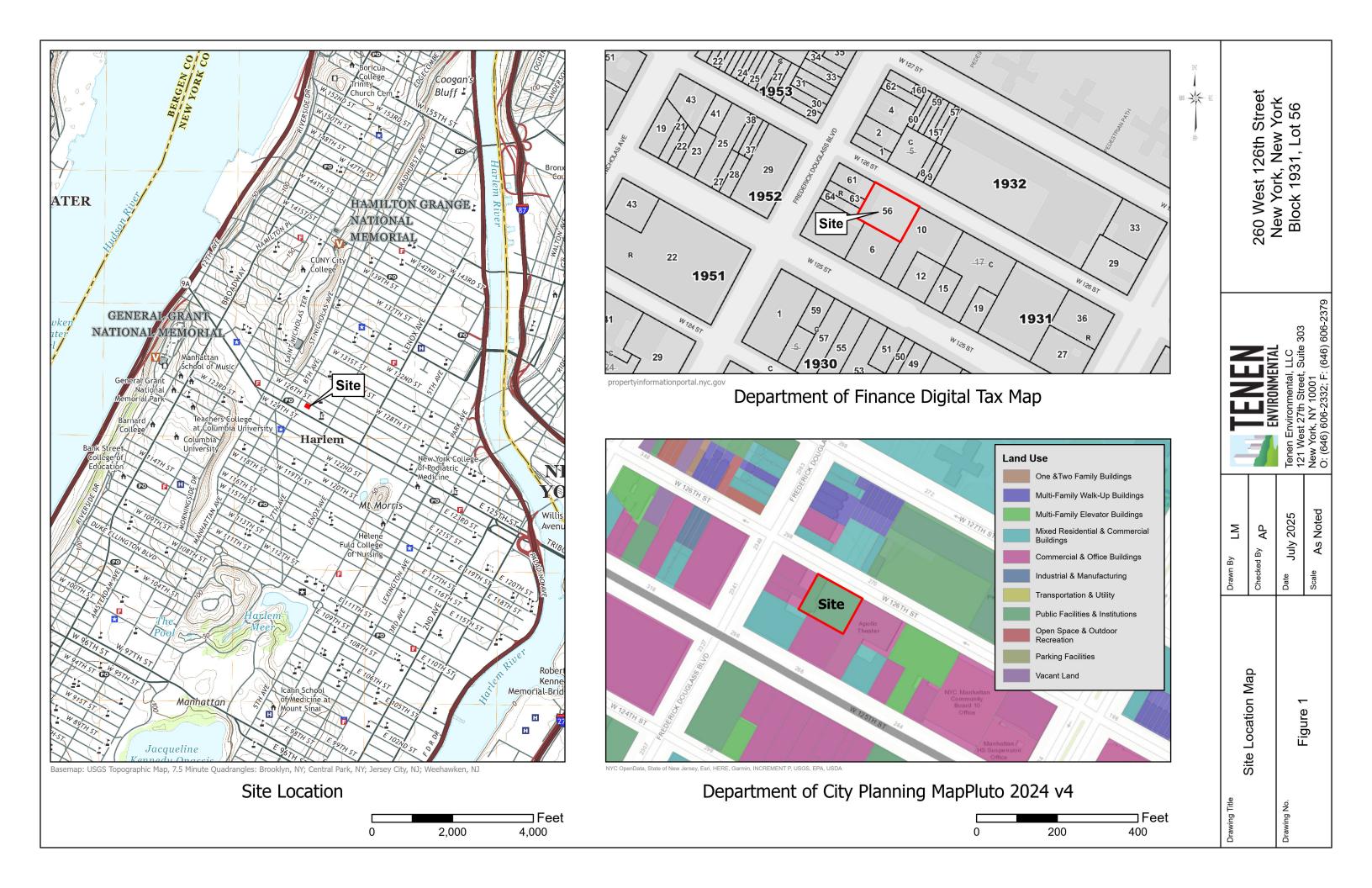
New York State Department of Environmental Conservation. Guidelines for Sampling and Analysis of Perand Polyfluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs. April 2023. NYSDEC 2023.

New York State Department of Health. Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York (NYSDOH, October 2006 with revisions).

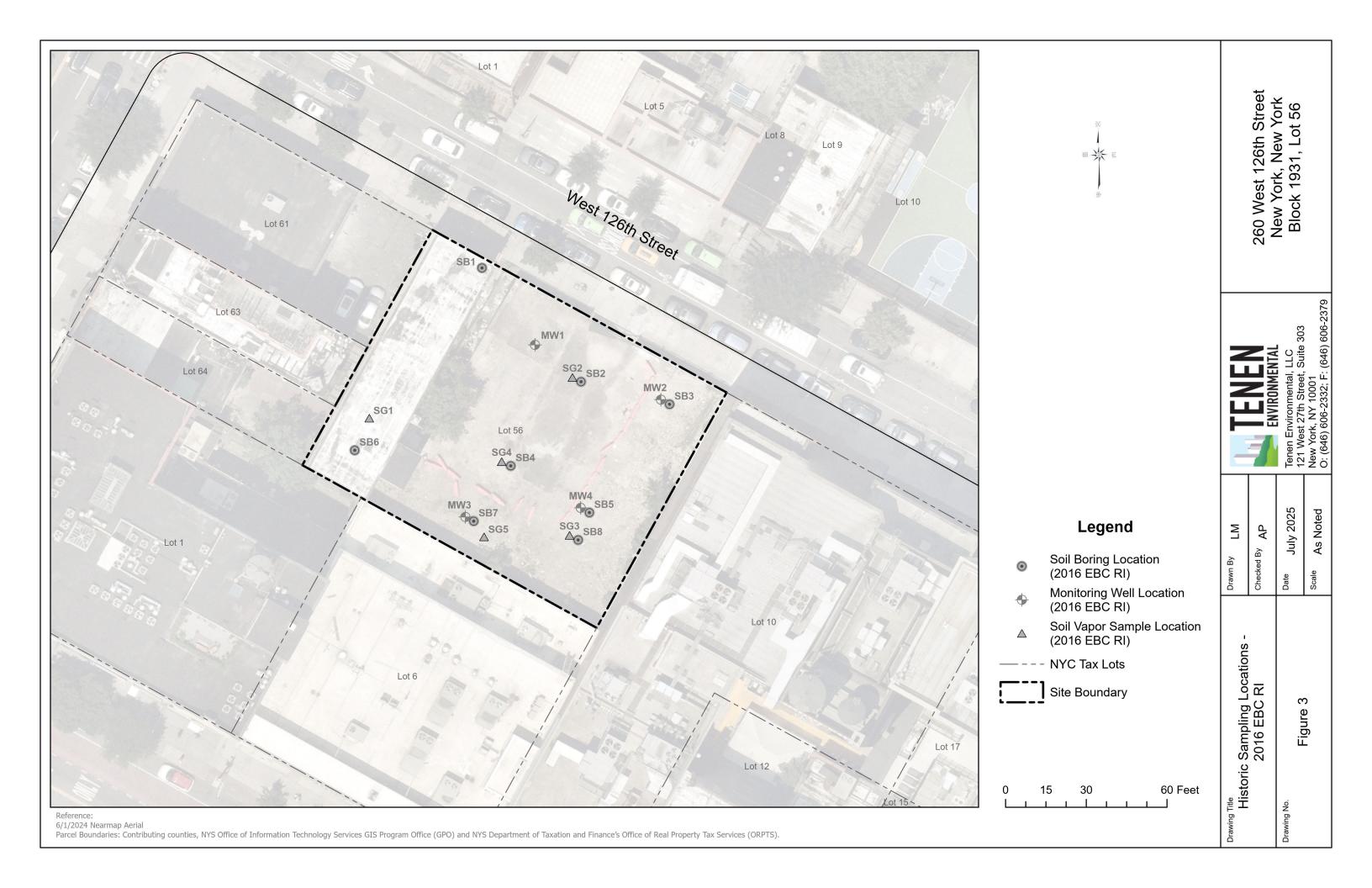
Phase I Environmental Site Assessment Report, 260 West 126<sup>th</sup> Street, New York, New York. Environmental Business Consultants. October 2016.

Remedial Investigation Report, 260 West 126<sup>th</sup> Street, New York, New York. Environmental Business Consultants. December 2016.

Figures











260 West 126th Street New York, New York Block 1931, Lot 56

2025

July

Date

Figure

 ${\sf Z}$ 

By

AP

Checked By

l Concentrations -6 EBC RI

Soil C 2016 I

Title Historic

As Noted

Analyte	NY-UNRES	NY-RESRR	
SVOCs	PI	om	
Benzo(a)anthracene	1	1	
Benzo(a)pyrene	1	1	
Benzo(b)fluoranthene	1	1	
Benzo(k)fluoranthene	0.8	3.9	
Chrysene	1	3.9	
Indeno(1,2,3-cd)pyrene	0.5	0.5	
Total Metals			
Arsenic	13	16	
Barium	350	400	
Chromium	30	180	
Copper	50	270	
Lead	63	400	
Mercury	0.18	0.81	
Nickel	30	310	
Zinc	109	2200	
Pesticides			
4,4'-DDE	0.0033	8.9	
4,4'-DDT	0.0033	7.9	

. Bold and shaded yellow value indicates concentration xceeds NY-UNRES SCOs

. Bold and shaded orange value indicates concentration ceeds NY-RESRR SCOs 3. NY-UNRES = 6 NYCRR Part 375 Unrestricted Use Soil

. NY-RESRR = 6 NYCRR Part 375 Restricted-Residential Us Soil Cleanup Objectives S. ND = Not detected

6. ppm = parts per million

# Legend

Soil Boring Location (2016 EBC RI)

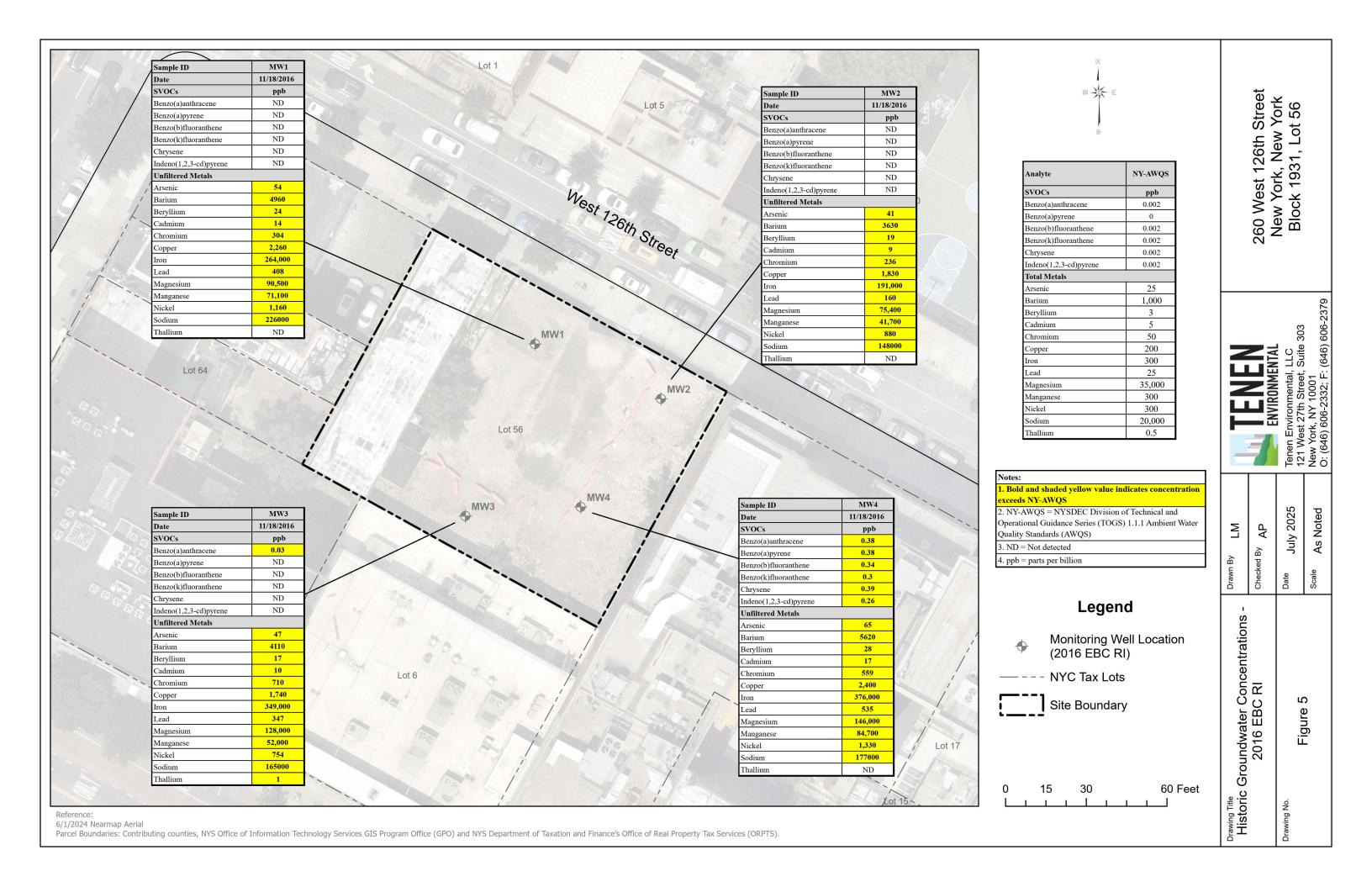
**NYC Tax Lots** 

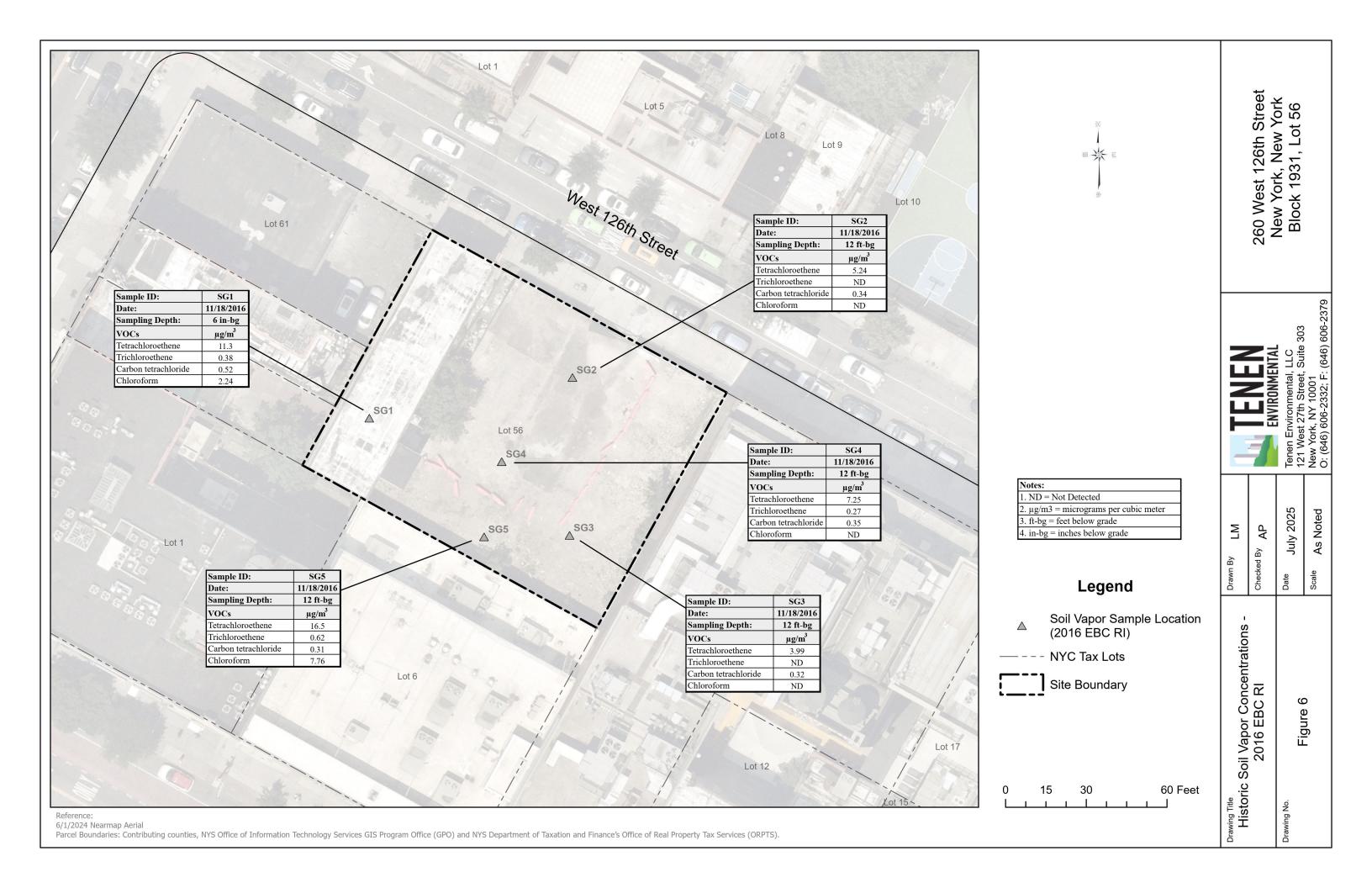
Site Boundary

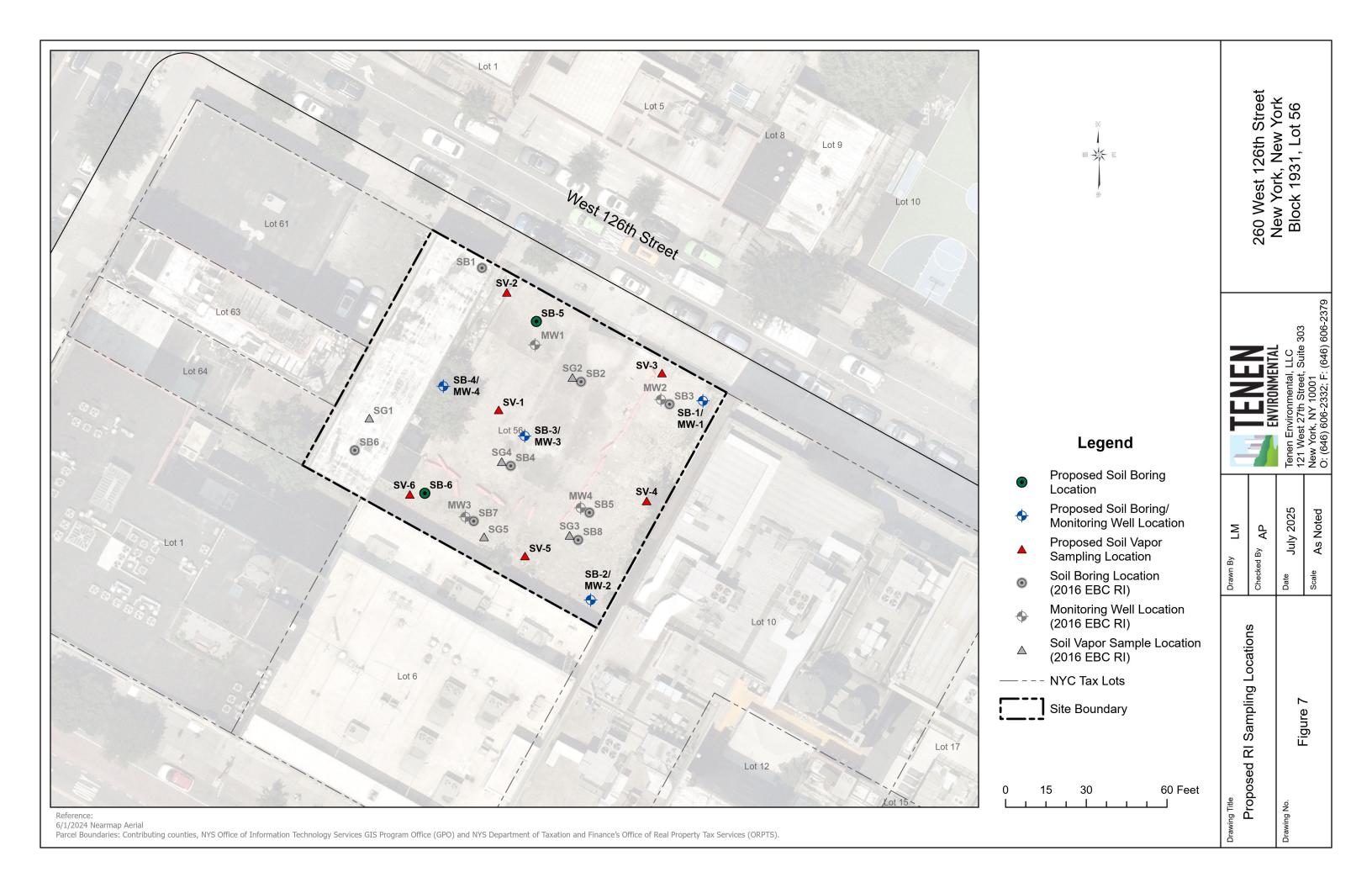
15 30

60 Feet

Parcel Boundaries: Contributing counties, NYS Office of Information Technology Services GIS Program Office (GPO) and NYS Department of Taxation and Finance's Office of Real Property Tax Services (ORPTS).







**Tables** 

## Table 2. Summary of Historic Soil Sample Exceedances 260 West 126th Street - New York, NY BCP Site No. TBD

LOCATION				SB-1	SB-1	SB-2	SB-2	SB-3
SAMPLE DEPTH (FT-BG)	1			0-2	2-4	0-2	10-12	0-2
SAMPLING DATE	NY-UNRES	NY-RESRR	Units	11/15/2016	11/15/2016	11/10/2016	11/10/2016	11/10/2016
LAB SAMPLE ID								
				Qual	Qual	Qual	Qual	Qual
Semivolatile Organic Compou	ınds							
Benzo(a)anthracene	1	1	ppm	ND	ND	0.18	ND	0.67
Benzo(a)pyrene	1	1	ppm	ND	ND	0.19	ND	0.68
Benzo(b)fluoranthene	1	1	ppm	ND	ND	0.2	ND	0.69
Benzo(k)fluoranthene	0.8	3.9	ppm	ND	ND	0.18	ND	0.65
Chrysene	1	3.9	ppm	ND	ND	0.26	0.15	0.83
Indeno(1,2,3-cd)pyrene	0.5	0.5	ppm	ND	ND	0.13	ND	0.49
<b>Total Metals</b>								
Arsenic	13	16	ppm	2.38	1.45	4.6	2.9	7.95
Barium	350	400	ppm	56.7	35.6	102	142	901
Chromium	30	180	ppm	15.4	12.8	14.1	15.6	28.1
Copper	50	270	ppm	19.9	14	76.4	47.8	67.4
Lead	63	400	ppm	38.3	23.4	138	189	562
Mercury	0.18	0.81	ppm	0.53	ND	0.02	0.48	0.25
Nickel	30	310	ppm	14.6	14.4	43.6	13.4	36.1
Zinc	109	2,200	ppm	30.5	16.8	167	75.3	685
Pesticides								
4,4'-DDE	0.0033	8.9	ppm	ND	ND	ND	ND	0.022
4,4'-DDT	0.0033	7.9	ppm	ND	ND	ND	ND	0.025

#### **Notes:**

**Bold and shaded yellow value indicates concentration exceeds NY-UNRES SCOs** 

**Bold and shaded orange value indicates concentration exceeds NY-RESRR SCOs** 

NY-UNRES = 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives

NY-RESRR = 6 NYCRR Part 375 Restricted-Residential Soil Cleanup Objectives

ND = Not detected

ppm = parts-per-million

Table 2. Summary of Historic Soil Sample Exceedances 260 West 126th Street - New York, NY BCP Site No. TBD

LOCATION				SB-3	SB-4	SB-4	SB-5	SB-5	SB-6
SAMPLE DEPTH (FT-BG)				10-12	0-2	10-12	0-2	10-12	0-2
SAMPLING DATE	NY-UNRES	NY-RESRR	Units	11/10/2016	11/10/2016	11/10/2016	11/10/2016	11/10/2016	11/15/2016
LAB SAMPLE ID									
				Qual	Qual	Qual	Qual	Qual	Qual
Semivolatile Organic Compou	ınds								
Benzo(a)anthracene	1	1	ppm	ND	0.28	0.51	1.1	ND	ND
Benzo(a)pyrene	1	1	ppm	ND	0.3	0.54	1.1	ND	ND
Benzo(b)fluoranthene	1	1	ppm	ND	0.25	0.55	1.1	ND	ND
Benzo(k)fluoranthene	0.8	3.9	ppm	ND	0.27	0.53	1.1	ND	ND
Chrysene	1	3.9	ppm	ND	0.31	0.65	1.3	ND	ND
Indeno(1,2,3-cd)pyrene	0.5	0.5	ppm	ND	0.18	0.45	0.81	ND	ND
Total Metals									
Arsenic	13	16	ppm	1.78	6.67	5.65	15.7	1.52	3.05
Barium	350	400	ppm	154	993	394	729	33	56.2
Chromium	30	180	ppm	16.1	44.1	30.2	14.7	19	8.48
Copper	50	270	ppm	26.1	36.2	45.1	28.4	26.8	14.1
Lead	63	400	ppm	134	642	247	2,040	4.1	235
Mercury	0.18	0.81	ppm	0.04	0.25	0.16	0.34	ND	0.45
Nickel	30	310	ppm	34.6	228	67.7	14.2	14.1	9.77
Zinc	109	2,200	ppm	81.5	664	260	690	29.2	92.8
Pesticides									
4,4'-DDE	0.0033	8.9	ppm	ND	ND	ND	ND	ND	0.012
4,4'-DDT	0.0033	7.9	ppm	ND	ND	ND	ND	ND	0.0039

#### **Notes:**

Bold and shaded yellow value indicates concentration exceeds NY-UNRES SCOs

**Bold and shaded orange value indicates concentration exceeds NY-RESRR SCOs** 

NY-UNRES = 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives

NY-RESRR = 6 NYCRR Part 375 Restricted-Residential Soil Cleanup Objectives

ND = Not detected

ppm = parts-per-million

### Table 2. Summary of Historic Soil Sample Exceedances 260 West 126th Street - New York, NY BCP Site No. TBD

LOCATION				SB-6	SB-7	SB-7	SB-8	SB-8
SAMPLE DEPTH (FT-BG)				2-4	0-2	10-12	0-2	10-12
SAMPLING DATE	NY-UNRES	NY-RESRR	Units	11/15/2016	11/10/2016	11/10/2016	11/10/2016	11/10/2016
LAB SAMPLE ID								
				Qual	Qual	Qual	Qual	Qual
Semivolatile Organic Compou	ınds							
Benzo(a)anthracene	1	1	ppm	ND	0.34	ND	0.14	0.66
Benzo(a)pyrene	1	1	ppm	ND	0.33	ND	0.12	0.65
Benzo(b)fluoranthene	1	1	ppm	ND	0.27	ND	ND	0.57
Benzo(k)fluoranthene	0.8	3.9	ppm	ND	0.28	ND	0.12	0.63
Chrysene	1	3.9	ppm	ND	0.36	ND	0.14	0.71
Indeno(1,2,3-cd)pyrene	0.5	0.5	ppm	ND	0.23	ND	ND	0.43
<b>Total Metals</b>								
Arsenic	13	16	ppm	2.26	8.51	2.71	1.91	7.13
Barium	350	400	ppm	122	2,760	131	73.6	703
Chromium	30	180	ppm	26.9	27.7	35.5	16.8	17.5
Copper	50	270	ppm	32.5	53.9	64.5	35.3	19.6
Lead	63	400	ppm	21.2	696	58.3	30.8	782
Mercury	0.18	0.81	ppm	0.09	0.26	0.42	0.23	0.75
Nickel	30	310	ppm	30	19.2	26.2	15.9	13.6
Zinc	109	2,200	ppm	38.9	1,960	83.5	37.5	400
Pesticides								
4,4'-DDE	0.0033	8.9	ppm	0.0038	ND	ND	ND	ND
4,4'-DDT	0.0033	7.9	ppm	0.013	0.0048	ND	ND	ND

### **Notes:**

**Bold and shaded yellow value indicates concentration exceeds NY-UNRES SCOs** 

**Bold and shaded orange value indicates concentration exceeds NY-RESRR SCOs** 

NY-UNRES = 6 NYCRR Part 375 Unrestricted Use Soil Cleanup Objectives

NY-RESRR = 6 NYCRR Part 375 Restricted-Residential Soil Cleanup Objectives

ND = Not detected

ppm = parts-per-million

Table 3. Summary of Historic Groundwater Sample Exceedances 260 West 126th Street - New York, NY BCP Site No. TBD

LOCATION			MW1	MW2	MW3	MW4
SAMPLING DATE	NIV AWOS	TI*4	11/18/2016	11/18/2016	11/18/2016	11/18/2016
LAB SAMPLE ID	NY-AWQS	Units	BV88459	BV88460	BV88461	BV88462
			Qual	Qual	Qual	Qual
Semivolatile Organic Com	pounds					
Benzo(a)anthracene	0.002	ppb	ND	ND	0.03	0.38
Benzo(a)pyrene	0	ppb	ND	ND	ND	0.38
Benzo(b)fluoranthene	0.002	ppb	ND	ND	ND	0.34
Benzo(k)fluoranthene	0.002	ppb	ND	ND	ND	0.3
Chrysene	0.002	ppb	ND	ND	ND	0.39
Indeno(1,2,3-cd)pyrene	0.002	ppb	ND	ND	ND	0.26
<b>Total Metals</b>						
Arsenic	25	ppb	54	41	47	65
Barium	1,000	ppb	4,960	3,630	4,110	5,620
Beryllium	3	ppb	24	19	17	28
Cadmium	5	ppb	14	9	10	17
Chromium	50	ppb	304	236	710	559
Copper	200	ppb	2,260	1,830	1,740	2,400
Iron	300	ppb	264,000	191,000	349,000	376,000
Lead	25	ppb	408.00	160	347	535
Magnesium	35,000	ppb	90,500	75,400	128,000	146,000
Manganese	300	ppb	71,100	41,700	52,000	84,700
Nickel	100	ppb	1,160	880	754	1,330
Sodium	20,000	ppb	226,000	148,000	165,000	177,000
Thallium	0.5	ppb	ND	ND	1	ND

### **Notes:**

### **Bold and shaded yellow value indicates concentration exceeds NY-AWQS**

NY-AWQS = NYSDEC Division of Technical and Operational Guidance Series (TOGS) 1.1.1 Ambient Water Quality Standards

ND = Not detected

ppb = parts-per-billion

Table 4. Summary of Volatile Organic Compounds in Soil Vapor 260 West 126th Street - New York, NY BCP Site No. TBD

LOCATION		SG1	SG2	SG3	SG4	SG5	
SAMPLING DATE	Units	11/18/2016	11/18/2016	11/18/2016	11/18/2016	11/18/2016	
LAB SAMPLE ID	Units	BV88458	BV88456	BV88457	BV88455	BV88454	
		Qual	Qual	Qual	Qual	Qual	
Volatile Organic Compoun	ıds						
Trichloroethene	ug/m <sup>3</sup>	0.38	ND	ND	0.27	0.62	
cis-1,2-Dichloroethene	ug/m <sup>3</sup>	ND	ND	ND	ND	ND	
1,1-Dichloroethene	ug/m <sup>3</sup>	ND	ND	ND	ND	ND	
Carbon tetrachloride	ug/m <sup>3</sup>	0.52	0.34	0.32	0.35	0.31	
Tetrachloroethene	ug/m <sup>3</sup>	11.3	5.24	3.99	7.25	16.5	
1,1,1-Trichloroethane	ug/m <sup>3</sup>	ND	ND	ND	ND	ND	
Methylene chloride	ug/m <sup>3</sup>	ND	ND	ND	ND	ND	
Vinyl chloride	ug/m <sup>3</sup>	ND	ND	ND	ND	ND	
Benzene	ug/m <sup>3</sup>	2.64	2.88	1.98	3.29	11.3	
Ethylbenzene	ug/m <sup>3</sup>	3.95	1.38	1.28	1.89	1.94	
Cyclohexane	ug/m <sup>3</sup>	15.2	ND	ND	ND	3.29	
1,2,4-Trimethylbenzene	ug/m <sup>3</sup>	4.69	1.39	ND	2.2	2.14	
1,3,5-Trimethylbenzene	ug/m <sup>3</sup>	1.29	ND	ND	ND	ND	
o-Xylene	ug/m <sup>3</sup>	4.95	1.73	1.64	2.37	2.48	
p/m-Xylene	ug/m <sup>3</sup>	13.6	5.81	5.12	6.64	6.68	
Heptane	ug/m <sup>3</sup>	2.58	1.05	ND	1.14	1.45	
Hexane	ug/m <sup>3</sup>	6.73	2.03	ND	1.68	16.1	
Toluene	ug/m <sup>3</sup>	16.3	8.78	16.5	15.1	15.3	

### **Notes:**

ND = Not detected

ug/m<sup>3</sup> = micrograms per cubic meter

# Appendix A Previous Reports (Submitted as Standalone PDFs)

# Appendix B Quality Assurance Project Plan

### **Quality Assurance Project Plan**

## for 260 West 126<sup>th</sup> Street Remedial Investigation Work Plan

260 West 126<sup>th</sup> Street New York, New York Block 1931, Lot No. 56 BCP Site # TBD

### Submitted to:

New York State Department of Environmental Conservation Division of Environmental Remediation Remedial Bureau B 625 Broadway, 12<sup>th</sup> Floor Albany, NY 12233-7016

### Prepared for:

S & F 126 ST. LLC 320 Roebling Street, Suite 749 Brooklyn, NY 11211

### Prepared by:



121 West 27<sup>th</sup> Street, Suite 303 New York, NY 10001

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### **Appendices**

Appendix A – Resumes

Appendix B – PFAS Field Sampling Guide

Appendix C – PFAS Compounds to be Analyzed and Associated MDLs

Appendix D – Laboratory Standard Operating Procedures for PFAS Analysis

### 1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been developed for the Remedial Investigation Work Plan (RIWP) prepared for the 260 West 126<sup>th</sup> Street property (the Site).

The Site, located at 260 West 126<sup>th</sup> Street, New York, New York (Tax Block 1931, Lot 56), is a rectangular shaped parcel located on the southern side of West 126<sup>th</sup> Street, between Frederick Douglass Boulevard (formerly Eighth Avenue) and Adam C. Powell Boulevard, in the Harlem section of Manhattan. The Site is approximately 12,490 square feet (SF) and has approximately 125 feet of frontage along West 126<sup>th</sup> Street and is approximately 100 feet deep.

The Site is developed with a two-story church building with a full cellar level covering approximately 2,725 SF of the western portion of the property. The church building is currently vacant and recently experienced a fire, rendering it unsafe to occupy. The remainder of the property is paved and utilized as a vacant parking lot.

### 1.1 Project Scope and QAPP Objective

The proposed scope of work includes the following:

- Advancement of soil borings, installation of permanent monitoring wells, and installation of temporary soil vapor points, and;
- Collection of soil, groundwater, soil vapor, and ambient air samples from soil borings, new permanent monitoring wells, and temporary soil vapor points.

The objective of the QAPP is to detail the policies, organization, objectives, functional activities and specific quality assurance/quality control activities designed to achieve the data quality goals or objectives of the Focused Remedial Investigation Work Plan. This QAPP addresses how the acquisition and handling of samples and the review and reporting of data will be documented for quality control (QC) purposes. Specifically, this QAPP addresses the following:

- The procedures to be used to collect, preserve, package, and transport samples;
- Field data collection and record keeping:
- Data management;
- Chain-of-custody procedures; and,
- Determination of precision, accuracy, completeness, representativeness, decision rules, comparability and level of quality control effort.

### 2.0 PROJECT ORGANIZATION

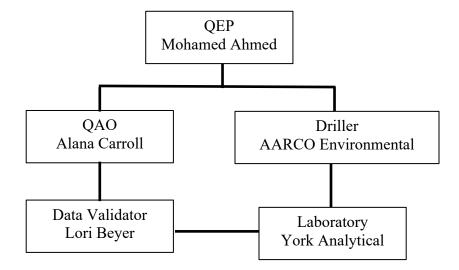
The personnel detailed are responsible for the implementation of the QAPP. Tenen Environmental, LLC (Tenen) will implement the RIWP on behalf of S & F 126 ST. LLC once it has been approved by the New York State Department of Environmental Conservation (NYSDEC).

The Project Manager and Qualified Environmental Professional (QEP) will be Mr. Mohamed Ahmed, Ph.D., CPG, principal at Tenen. Dr. Ahmed is a certified professional geologist with over 20 years of experience in the New York City metropolitan area. He has designed and implemented subsurface investigations and is proficient in groundwater modeling, design of groundwater treatment systems, and soil remediation. He has managed numerous projects focused on compliance with the requirements of the New York State Brownfield Cleanup Program and spills programs and the New York City E-designation program. Dr. Ahmed also has extensive experience in conducting regulatory negotiations with the New York State Department of Environmental Conservation, the New York City Department of Environmental Protection, the NYC Office of Housing Preservation and Development, and the Mayor's Office of Environmental Remediation. Dr. Ahmed holds advanced degrees in geology and Earth and Environmental Sciences from Brooklyn College and the Graduate Center of the City University of New York; his resume is included in Appendix A.

The Quality Assurance Officer will be Mrs. Alana Carroll, CPG, managing geologist at Tenen. Mrs. Carroll is a certified professional geologist with experience in all aspects of site assessment, development and implementation of remedial strategies. Her experience involves projects from inception through investigation, remediation and closure. Her expertise includes soil, soil vapor and groundwater remediation; remedial selection and design; field/health and safety oversight and preparation of work plans and reports to satisfy the requirements of various regulatory agencies. Mrs. Carroll received her BS in Geology from Hofstra University; her resume is included in Appendix A.

In addition, Tenen will utilize subcontractors for drilling (AARCO Environmental Services of Lindenhurst, NY), laboratory services (York Analytical Laboratories of Richmond Hill, NY) and data validation (L.A.B. Validation Corp. of East Northport, NY). The resume for the DUSR preparer, Ms. Lori Beyer, is included in Appendix A.

Contact Information Remedial Party (S & F 126 ST. LLC), Usher Goldman, 646.256.2188 Tenen Environmental, Mohamed Ahmed or Alana Carroll, 646.606.2332 An organization chart for the implementation of the Remedial Investigation Work Plan and QAPP is below.



### 3.0 SAMPLING AND DECONTAMINATION PROCEDURES

A detailed description of the procedures to be used during this program for collection of the soil, soil vapor, ambient air, and groundwater samples is provided below. Proposed sample locations are shown on Figure 7 of the Work Plan. An Analytical Methods/Quality Assurance Summary is provided in Table 1, included in Section 3.11.

### 3.1 Level of Effort for QC Samples

Field blank, trip blank, field duplicate and matrix spike (MS) / matrix spike duplicate (MSD) samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs. Each type of QC sample is discussed below.

- Field and trip blanks consisting of distilled water will be submitted to the analytical
  laboratories to provide the means to assess the quality of the data resulting from the fieldsampling program. Field (equipment) blank samples are analyzed to check for procedural
  chemical constituents that may cause sample contamination. Trip blanks are used to
  assess the potential for contamination of samples due to contaminant migration during
  sample shipment and storage.
- Duplicate samples are analyzed to check for sampling and analytical reproducibility.
- MS/MSD samples provide information about the effect of the sample matrix on the digestion and measurement methodology.

The general level of QC effort will be one field duplicate and one field blank (when non-dedicated equipment is used) for every 20 or fewer investigative samples of a given matrix. Additional sample volume will also be provided to the laboratory to allow one site-specific MS/MSD for every 20 or fewer investigative samples of a given matrix. One trip blank will be included along with each sample delivery group of volatile organic compound (VOC) samples.

The analytical laboratory, York Analytical Laboratories, is certified under the New York State Department of Health (NYSDOH) Environmental Laboratory Approval Program (ELAP) as Lab IDs 10854 and 12058. NYSDEC Analytical Services Protocol (ASP) Category B deliverables will be prepared by the laboratory.

### 3.2 Sample Handling

Samples will either be picked up by the laboratory, delivered to the laboratory in person by the sampler, or transported to the laboratory by overnight courier. All samples will be shipped to the laboratory to arrive within 48 hours after collection, and the laboratory will adhere to the analytical holding times for these analyses, as listed in the current version of the New York State ASP.

### 3.3 Custody Procedures

Sample custody will be controlled and maintained through the chain-of-custody procedures. The chain of custody is the means by which the possession and handling of samples is tracked from the site to the laboratory. Sample containers will be cleaned and preserved at the laboratory before shipment to the Site. The following sections (Sections 3.4 and 3.5) describe procedures for maintaining sample custody from the time samples are collected to the time they are received by the analytical laboratory.

### 3.4 Sample Storage

Samples will be stored in secure limited-access areas. Walk-in coolers or refrigerators will be maintained at 4°C, +/- 2°C, or as required by the applicable regulatory program. The temperatures of all refrigerated storage areas are monitored and recorded a minimum of once per day. Deviations of temperature from the applicable range require corrective action, including moving samples to another storage location, if necessary.

### 3.5 Sample Custody

Sample custody is defined by this QAPP as the following:

- The sample is in someone's actual possession;
- The sample is in someone's view after being in his or her physical possession;
- The sample was in someone's possession and then locked, sealed, or secured in a manner that prevents unsuspected tampering; or,
- The sample is placed in a designated and secured area.

Samples will be removed from storage areas by the sample custodian or laboratory personnel and transported to secure laboratory areas for analysis. Access to the laboratory and sample storage areas is restricted to laboratory personnel and escorted visitors only; all areas of the laboratory are therefore considered secure.

Laboratory documentation used to establish chain of custody and sample identification may include the following:

- Field chains of custody or other paperwork that arrives with the sample;
- Laboratory chain of custody;
- Sample labels or tags attached to each sample container;
- Sample custody seals;
- Sample preparation logs (i.e., extraction and digestion information) recorded in hardbound laboratory books, filled out in legible handwriting, and signed and dated by the chemist;
- Sample analysis logs (e.g., metals, GC/MS, etc.) information recorded in hardbound laboratory books that are filled out in legible handwriting, and signed and dated by the chemist;
- Sample storage log (same as the laboratory chain of custody); and,

• Sample disposition log, which documents sample disposal by a contracted waste disposal company.

### 3.6 Sample Tracking

All samples will be maintained in the appropriate coolers prior to and after analysis. Laboratory analysts will remove and return their samples, as needed. Samples that require internal chain of custody procedures will be relinquished to the analysts by the sample custodians. The analyst and sample custodian will sign the original chain of custody relinquishing custody of the samples from the sample custodian to the analyst. When the samples are returned, the analyst will sign the original chain of custody returning sample custody to the sample custodian. Sample extracts will be relinquished to the instrumentation analysts by the preparatory analysts. Each preparation department will track internal chain of custody through their logbooks/spreadsheets.

Any change in the sample during the time of custody will be noted on the chain of custody (e.g., sample breakage or depletion).

### 3.7 Soil Sampling

Soil borings will be advanced by a direct push Geoprobe©. In general, select soil intervals will be screened between grade and the terminal depth of the boring. All non-disposable sampling equipment will be decontaminated between borings, as described in Section 3.11.

Soil from grade to terminal depth of the boring will be scanned for volatile organic compounds (VOCs) using a photoionization detector (PID) and geologically described using the Unified Soil Classification System, including documentation of observations regarding potential contamination such as odors, staining, etc. All descriptions and observations will be documented in a field notebook.

A minimum of three soil samples will be analyzed from all borings installed onsite. Soil samples will be collected from grade to two feet below grade (ft-bg), from the two-foot interval directly above the groundwater interface, and from the two-foot interval directly below the proposed development depth (30 ft-bg). Aditional samples will be collected from any borings exhibiting evidence of contamination from the two-foot interval exhibiting the highest suspected contamination based on visual, olfactory, and PID readings, and the next apparent non-impacted two-foot interval.

All collected soil samples will be placed in pre-cleaned, pre-preserved laboratory provided sample bottles or En Core samplers (En Novative Technologies, Inc.), cooled to 4°C in the field, and transported under chain-of-custody command to the designated laboratory for analysis.

All soil samples will be analyzed for the following analytes on the Part 375 list with a Category B deliverable package:

- Target Compound List (TCL) VOCs by EPA Method 8260C;
- TCL SVOCs by EPA Method 8270D;
- Pesticides by EPA Method 8081B;

- Herbicides by EPA Method 8151A;
- Polychlorinated Biphenyls (PCBs) by EPA Method 8082A;
- Target Analyte List (TAL) Metals by EPA Method 6010C/7471B;
- Total Cyanide by EPA Method 9010C;
- Trivalent and Hexavalent Chromium by EPA Method 3060A;
- Per- and Polyfluoroalkyl Substances (PFAS) by USEPA Method 1633 [reporting limit of 0.5 parts per billion (ppb)], and;
- 1,4-Dioxane by USEPA Method 8270.

Field methods can impact the analysis of PFAS. PFAS samples will be collected using dedicated disposable sampling equipment when possible. If dedicated disposable sampling equipment cannot be used, samples will be collected with stainless steel tools (spoons, bowls, etc.) and decontaminated using detergent (Alconox) and clean, PFAS-free water. Sampling containers for PFAS will be made of high-density polyethylene (HDPE) with caps that will not be lined with polytetrafluoroethylene (PTFE). PFAS sampling containers will be kept in separate coolers from all other sampling containers and only regular ice will be used to cool the samples. Field staff conducting the sampling will not wear clothing that contains PTFE material or that has been waterproofed with PFAS material during sampling and all clothing worn will be laundered multiple times. Any bug spray or sunscreen utilized by field staff conducting the sampling will be PFAS free.

The following PFAS will be analyzed by EPA Method 1633:

- Perfluorobutanoic Acid (PFBA)
- Perfluoropentanoic Acid (PFPeA)
- Perfluorobutanesulfonic Acid (PFBS)
- 1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2FTS)
- Perfluorohexanoic Acid (PFHxA)
- Perfluoropentanesulfonic Acid (PFPeS)
- Perfluoroheptanoic Acid (PFHpA)
- Perfluorohexanesulfonic Acid (PFHxS)
- Perfluorooctanoic Acid (PFOA)
- 1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2FTS)
- Perfluoroheptanesulfonic Acid (PFHpS)
- Perfluorononanoic Acid (PFNA)
- Perfluorooctanesulfonic Acid (PFOS)
- Perfluorodecanoic Acid (PFDA)
- 1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2FTS)
- Perfluorononanesulfonic Acid (PFNS)
- N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSAA)
- Perfluoroundecanoic Acid (PFUnA)
- Perfluorodecanesulfonic Acid (PFDS)
- Perfluorooctanesulfonamide (FOSA)
- N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)
- Perfluorododecanoic Acid (PFDoA)

- Perfluorotridecanoic Acid (PFTrDA)
- Perfluorotetradecanoic Acid (PFTA)
- 2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-Propanoic Acid (HFPO-DA)
- 4,8-Dioxa-3h-Perfluorononanoic Acid (ADONA)
- Perfluorododecane Sulfonic Acid (PFDoDS)
- 9-Chlorohexadecafluoro-3-Oxanone-1-Sulfonic Acid (9Cl-PF3ONS)
- 11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11Cl-PF3OUdS)
- N-Methyl Perfluorooctane Sulfonamide (NMeFOSA)
- N-Ethyl Perfluorooctane Sulfonamide (NEtFOSA)
- N-Methyl Perfluorooctanesulfonamido Ethanol (NMeFOSE)
- N-Ethyl Perfluorooctanesulfonamido Ethanol (NEtFOSE)
- Perfluoro-3-Methoxypropanoic Acid (PFMPA)
- Perfluoro-4-Methoxybutanoic Acid (PFMBA)
- Perfluoro(2-Ethoxyethane)Sulfonic Acid (PFEESA)
- Nonafluoro-3,6-Dioxaheptanoic Acid (NFDHA)
- 3-Perfluoropropyl Propanoic Acid (3:3FTCA)
- 2H,2H,3H,3H-Perfluorooctanoic Acid (5:3FTCA)
- 3-Perfluoroheptyl Propanoic Acid (7:3FTCA)

A sampling guide is included in Appendix B. A list of PFAS compounds to be analyzed and associated MDLs is included in Appendix C. York Analytical, an ELAP-certified lab for PFAS by EPA Method 1633, will be performing all PFAS analyses of soil. The laboratory standard operating procedures for PFAS analysis is included in Appendix D.

### 3.8 Monitoring Well Installation and Development

Four newly installed permanent monitoring wells will be sampled. All monitoring wells will be installed using a Geoprobe® direct-push rig. All monitoring wells will consist of a two-inch inner diameter (ID) PVC casing and riser. A ten-foot PVC screen (0.020-inch slot) will be installed straddling the groundwater interface (five feet above and five feet below). A filter pack of sand will be placed in the annular space around the screen of the monitoring wells (minimum 2-inches around the circumference of the screen) and will extend two feet above the screen. The annular area around the well casing will be sealed with bentonite pellets for an interval of two feet above the filter pack. The annular space above the bentonite pellets to one foot below grade will be backfilled with unimpacted drilling cuttings or clean sand. The remaining one foot will be sealed with a concrete cap and well apron (expanding cement). A locking well cap will be installed upon completion of each well.

Following installation, at least three well volumes of the water column will be removed using a submersible pump. All permanent wells will be surveyed to a common site datum.

### 3.9 Groundwater Sampling

Prior to sample collection, static water levels will be measured and recorded from all previously-installed and newly-installed monitoring wells to be sampled. Monitoring wells will also be

gauged for the presence of non-aqueous phase liquid (NAPL). In the event that NAPL is detected, Tenen will record the thickness and will not collect a sample. If NAPL is not detected, Tenen will purge and sample monitoring wells using low-flow/minimal drawdown purge and sample collection procedures (peristaltic pump system). Prior to sample collection, groundwater will be evacuated from each well at a low-flow rate (typically less than 0.1 L/min). Field measurements for pH, temperature, turbidity, dissolved oxygen, specific conductance, oxidation-reduction potential and water level, as well as visual and olfactory field observations, will be periodically recorded and monitored for stabilization. Purging will be considered complete when pH, specific conductivity, dissolved oxygen and temperature stabilize and when turbidity measurements fall below 50 Nephelometric Turbidity Units (NTU) or become stable above 50 NTU.

Stability is defined as variation between field measurements of 10 percent or less and no overall upward or downward trend in the measurements. Upon stabilization of field parameters, groundwater samples will be collected and analyzed as discussed below.

Wells will be purged and sampled using dedicated high density polyethylene (HDPE) pump tubing following low-flow/minimal drawdown purge and sample collection procedures, as described above. The pump will be decontaminated between samples.

Groundwater samples will be collected for analysis through dedicated HDPE tubing. Prior to, and immediately following collection of groundwater samples, field measurements for pH, specific conductance, temperature, dissolved oxygen, turbidity and depth-to-water, as well as visual and olfactory field observations will be recorded. All collected groundwater samples will be placed in pre-cleaned, pre-preserved laboratory provided sample bottles, cooled to 4°C in the field, and transported under chain-of-custody command to the designated laboratory for analysis.

All groundwater samples will be analyzed with a Category B deliverable package, for the following:

- TCL VOCs by EPA Method 8260C;
- TCL SVOCs by EPA Method 8270D;
- Pesticides by EPA Method 8081B;
- Herbicides by EPA Method 8151A;
- PCBs by EPA Method 8082A;
- Total and Dissolved TAL Metals by EPA Method 6010C/7471B;
- Total and Dissolved Cyanide by EPA Method 9010C; and
- Total and Dissolved Trivalent and Hexavalent Chromium by EPA Method 3060A;
- 1,4-Dioxane by EPA Method 8270D-SIM Modified; and
- PFAS by EPA Method 1633 [reporting limit of 2 parts per trillion (ppb)].

Field methods can impact the analysis of PFAS. PFAS samples will be collected using dedicated disposable HDPE tubing. If dedicated disposable sampling equipment cannot be used, equipment will be decontaminated by standard two step decontamination using detergent (Alconox) and clean, PFAS-free water. All sources of water used for equipment decontamination will be verified in advance to be PFAS-free through laboratory analysis or certification. Sampling containers for PFAS will be made of HDPE with caps that will not be lined with PTFE. PFAS

sampling containers will be kept in separate coolers from all other sampling containers and only regular ice will be used to cool the samples. Field staff conducting the sampling will not wear clothing that contains PTFE material or that has been waterproofed with PFAS material during sampling and all clothing worn will be laundered multiple times. Any bug spray or sunscreen utilized by field staff conducting the sampling will be PFAS free.

The following PFAS will be analyzed by EPA Method 1633:

- PFBA
- PFPeA
- PFBS
- 4:2FTS
- PFHxA
- PFPeS
- PFHpA
- PFHxS
- PFOA
- 6:2FTS
- PFHpS
- PFNA
- PFOS
- PFDA
- 8:2FTS
- PFNS
- NMeFOSAA
- PFUnA
- PFDS
- FOSA
- NEtFOSAA
- PFDoA
- PFTrDA
- PFTA
- HFPO-DA
- ADONA
- PFDoDS
- 9C1-PF3ONS
- 11Cl-PF3OUdS
- NMeFOSA
- NEtFOSA
- NMeFOSE
- NEtFOSE
- PFMPA
- PFMBA
- PFEESA
- NFDH

- 3:3FTCA
- 5:3FTCA
- 7:3FTCA

A sampling guide is included in Appendix B. A list of PFAS compounds to be analyzed and associated MDLs is included in Appendix C. York Analytical, an ELAP-certified lab for PFAS by EPA Method 1633, will be performing all PFAS analyses of groundwater. The laboratory standard operating procedures for PFAS analysis is included in Appendix D.

### 3.10 Soil Vapor and Ambient Air Sampling

Soil vapor and ambient air samples will be collected in accordance with the NYSDOH *Guidance* for Evaluating Soil Vapor Intrusion In the State of New York, dated October 2006.

Six exterior soil vapor samples and one outdoor ambient air sample will be collected as part of the RI. One soil vapor sample (SV-1) will be installed in the center of the Site to a depth of approximately three feet above the groundwater interface (approximately 23 to 25 ft-bg) to assess soil vapor conditions at the Site. Five soil vapor samples (SV-2 through SV-6) will be installed along the perimeters of the Site to the anticipated depth of the nearest building foundation to assess potential soil vapor migration offsite.

Soil vapor samples will be collected using disposable points at the terminal depth of the sample. A Geoprobe® direct-push rig will be used to install the exterior soil vapor sampling points. Once the soil vapor sampling points have been driven to the desired depth, they will be attached to disposable tubing for sample collection.

The borehole above the sampling points to grade will be sealed using an inert sealant to prevent ambient air mixing with the soil vapor. Ambient air will be purged from the boring hole by attaching the surface end of the ¼-inch diameter Teflon® tube to an air valve and then to a vacuum pump. The vacuum pump will remove three volumes of air (volume of the sample probe and tube) prior to sample collection. The flow rate for both purging and sample collection will not exceed 0.2 liter per minute (L/min).

The soil vapor samples will be first screened for organic vapors using a PID. A tracer gas will be used in accordance with NYSDOH protocols to verify the integrity of the soil vapor probe seal. Helium will be used as the tracer gas and a bucket will serve to keep it in contact with the probe during testing. A portable monitoring device will be used to analyze a sample of soil vapor for the tracer prior to sampling. If the tracer sample results show a significant presence of the tracer, the probe seals will be adjusted to prevent infiltration.

The ambient air sample will be collected from breathing height (three to five feet above the floor). The sampling flow rate will not exceed 0.2 liters per minute (L/min).

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.

All soil vapor samples will be collected in laboratory-supplied and batch-certified 6-liter Summa canisters using two-hour regulators. The ambient air sample will be collected in a laboratory-supplied 6-liter Summa canister using an eight-hour regulator. All soil vapor samples will be analyzed for VOCs using EPA Method TO-15. The ambient air sample will be collected during soil vapor sample collection and will be analyzed for VOCs using EPA Method TO-15.

### 3.11 Analytical Methods/Quality Assurance Summary Table

A summary of the analytical methods and quality assurance methods are included in Table 1, below.

Table 1 Analytical Methods/Quality Assurance Summary

Matrix	Proposed		QA/C	QC Samp	oles	Total # Samples	Analytical	Methods	Preservative	Holding Times	Container
	Samples	TB	FB	DUP	MS/MSD		Parameters	Wichiods	1 Teservative		Container
	18	1	1	1	1/1	23	VOCs	8260C	Cool to 4°C, No Headspace	14 days to analysis	(3) Encore samplers, or; (1) 2-oz glass jar
	18	0	1	1	1 / 1	22	SVOCs	8270D	Cool to 4°C		
Soil	18	0	1	1	1 / 1	22	Pesticides, Herbicides	8081B, 8151A	Cool to 4°C		(2) 250 mL clear glass jar
	18	0	1	1	1 / 1	22	PCBs	8082A	Cool to 4°C		
	18	0	1	1	1/1	22	TAL Metals, Total Cyanide, Trivalent and Hexavalent Chromium	6010C/ 7471B, 9010C, 3060A	Cool to 4°C		
	18	0	1	1	1 / 1	22	PFAS	1633	Cool to 4°C		(1) 250 mL HDPE plastic bottle
	18	0	1	1	1 / 1	22	1,4-Dioxane	8270	Cool to 4°C		(2) 1-L amber glass bottle

Matrix	Proposed	QA/QC Samples				Total #	Analytical	Methods	Preservative	Holding	Container	
Matrix	Samples	ТВ	FB	DUP	MS/MSD	Samples	Parameters	Methods	Fieservative	Times		
	4	1	1	1	1 / 1	9	VOCs	8260C	Cool to 4°C, HCL		(3) 40 mL amber glass vials	
	4	0	1	1	1 / 1	8	SVOCs	8270D	Cool to 4°C			
	4	0	1	1	1 / 1	8	Pesticides, Herbicides	8081B, 8151A	Cool to 4°C	14 days to analysis	(6) 1 L amber glass bottles	
	4	0	1	1	1 / 1	8	PCBs	8082A	Cool to 4°C			
Groundwater	4	0	1	1	1 / 1	8	Total and Dissolved: TAL Metals, Cyanide, Trivalent and Hexavalent Chromium	6010C/ 7471B, 9010C, 3060A	Cool to 4°C		(1) plastic 500 mL HNO3 preserved bottle; Dissolved Metals: (1) plastic 500 mL	HNO3 preserved bottle; Dissolved Metals: (1) plastic 500 mL unpreserved
	4	0	1	1	1 / 1	8	1,4-Dioxane	8270D- SIM Modified	Cool to 4°C		(2) 1 L amber glass bottle	
	4	0	1	1	1 / 1	8	PFAS	1633	Cool to 4°C, Trizma		(1) 250 mL HDPE plastic bottle	
Soil Vapor	6	No QA/QC samples				6	Woo	TO-15	None	20.1	(1) 6-L	
Outdoor Air	1		INO QA	VQC san	npies	VOCs		10-13	inone	30 days	Summa	

TB – Trip Blank

260 West 126<sup>th</sup> Street, New York, NY BCP Site No. TBD

FB – Field Blank
DUP – Duplicate
°C – degrees Celsius
mL – milliliter
L – liter

### 3.12 **Decontamination**

Where possible, samples will be collected using new, dedicated sampling equipment so that decontamination is not required. All non-dedicated drilling tools and equipment will be decontaminated between boring locations using potable tap water and a phosphate-free detergent (e.g., Alconox) and/or a steam cleaner. All non-dedicated sampling equipment will also have a final rinse with deionized water. Decontamination water will be collected and disposed as investigation-derived waste (IDW).

### 3.13 Data Review and Reporting

The NYSDEC ASP Category B data package will be validated by an independent data validation subconsultant and a DUSR summarizing the results of the data validation process will be prepared. All reported analytical results will be qualified as necessary by the data validation and will be reviewed and compared against background concentrations and/or applicable New York State criteria:

Soil – Unrestricted Use, Protection of Groundwater, and Restricted-Residential Soil Cleanup Objectives (SCOs) as listed in 6NYCRR Part 375, and PFAS results will be compared to the proposed Unrestricted Use, Protection of Groundwater, and Restricted-Residential Use SCOs as listed in NYSDEC's April 2023 PFAS Guidelines;

Groundwater – Class GA groundwater standards and guidance values for groundwater as listed in NYSDEC Technical and Operations Guidance Series (TOGS) 1.1.1 with February 2023 updates; and,

Soil Vapor – Ambient air concentrations.

A report documenting the Remedial Investigation will be prepared, and will describe Site conditions and document applicable observations made during the sample collection. In addition, the report will include a description of the sampling procedures, tabulated sample results and an assessment of the data and conclusions. The laboratory data packages, DUSR, geologic logs, well construction diagrams, and field notes will be included in the report as appendices. All data will also be submitted electronically to NYSDEC via the Environmental Information Management System (EIMS) in EqUIS format.

Appendix A

Resumes

### Alana M. Carroll, PG Senior Project Manager

### **PROFESSIONAL PROFILE**

Ms. Alana Carroll is a professional geologist with experience managing a variety of environmental consulting projects in the New York metropolitan area and specializing in remedial investigations, conceptual site modeling, and remedial design and implementation. She provides analytical, technical, and regulatory guidance to clients, including developers and environmental attorneys, on a variety of projects in various stages of investigation, remediation, and redevelopment. Ms. Carroll has managed projects from inception through investigation, remediation, and closure in the New York State Brownfield Cleanup Program, the New York State Department of Environmental Conservation (NYSDEC) Spills and Voluntary Cleanup Programs, the New York State Superfund Program, and the New York City E-Designation Program.

### **CREDENTIALS AND PROFESSIONAL HONORS**

New York State Licensed Professional Geologist #000979 Adjunct Professor, Manhattan College, School of Engineering M.A., Earth and Environmental Sciences, Brooklyn College, New York B.S., Geology, Hofstra University, Uniondale, New York

### **CONTINUING EDUCATION AND TRAINING**

OSHA 10-Hour Construction Training (2015)
Hazardous Waste Operations and Emergency Response 40-Hour Certification
(2004; refreshers 2005, 2006, 2007, 2009, 2010, 2011, 2012, 2013, 2014, and 2015)
First Aid and CPR Certified (2012)
Amtrak Contractor Safety Training (2010 and 2011)

### PROFESSIONAL AFFILIATIONS

Member of Geologic Society of America Member of New Partners for Community Revitalization

### RELEVANT EXPERIENCE

New York State Brownfield Cleanup Program, Former West 18th Street MGP Site, Block 690, Lots 20 and 29, West Chelsea, Manhattan, New York—Successfully guided the client into the Brownfield Cleanup Program at the remediation stage. Prepared detailed remedial cost estimates for several redevelopment scenarios. Assisted in negotiating cleanup costs on behalf of the developer with the entity responsible for onsite contamination from former manufactured gas plant (MGP) operations. Designed and managed a pre-design investigation that delineated onsite coal tar impacts and differentiated petroleum impacts. Served on a team that designed an in situ stabilization treatability study. Prepared the Remedial Action Work Plan and Alternatives Analysis that included the excavation and removal of coal tar source material within two MGP gas holders and the encapsulation of residual coal tar. Performed an essential role on the construction and design team, coordinating with the structural, foundation, mechanical, and architectural contractors.

Alana M. Carroll, PG Page 2

New York State Brownfield Cleanup Program, 520 West 28th Street, West Chelsea, Manhattan, New York—Managed several investigations to address New York State Spills, New York City E-Designation, and New York State Brownfield Cleanup programs. Prepared scopes of work to address requirements of both State and City regulatory agencies. Served as an essential member of the construction and design team, coordinating with the structural, foundation, mechanical, and architectural contractors. Managed access with adjacent property owners for full-scale excavation. Coordinated with State and City agencies for the satisfaction of air, noise, and hazardous waste requirements. Coordinated and managed the characterization and disposal of over 35,000 tons of hazardous material and historic fill. Designed and managed the remedial action necessary to obtain a successful Track 1 Cleanup. Assisted in negotiating a nuanced approach to support excavation that allowed for a Track 1 Cleanup. Prepared the final engineering report that expedited the certificate of completion.

New York State Brownfield Cleanup Program, Teitelbaum Dry Cleaner, Long Island City, New York—Designed and managed multiple onsite and offsite investigations to address NYSDEC and New York State Department of Health (NYSDOH) regulatory requirements with respect to chlorinated solvent impacts to groundwater and soil vapor. Designed and managed chlorinated solvent plume delineation and remediation in both groundwater and soil vapor. Prepared a technical memorandum on the fate and transport of the onsite chlorinated solvent groundwater plume that established limited liability for downgradient impacts and identified a secondary source. Coordinated with multiple adjacent parties for access. Designed a remedial approach for the site building that included source removal, groundwater injection, and a retro-fitted subslab depressurization system (SSDS).

**Confidential Project, Steuben County, NY**—Performed a forensic review and analysis of environmental records associated with five parcels of land that the State deemed as illegal solid waste dumps. Prepared and presented two technical arguments to NYSDEC and NYSDOH detailing illegal dumping, historic fill material, human health exposure pathways, bioavailability of historic fill constituents and remedial alternatives. Designed a full-scale remedial investigation of soil, groundwater and sediments for five parcels of historically industrial land.

**New York City Voluntary Cleanup Program, Gallery Row, West Chelsea, Manhattan, New York**—Managed multiple investigations over five tax lots to address New York City E-Designation and Voluntary Cleanup Program requirements. Designed a remedial action that incorporates a phased and targeted excavation below Highline Park. Coordinated with State and City agencies for the satisfaction of air, noise, and hazardous waste requirements. Served as an integral part of the construction and design team.

New York State Brownfield Cleanup Program, Former Nu-Brite Dry Cleaner, 1299 First Avenue, East Side, Manhattan, New York—Designed and managed multiple investigations to address onsite chlorinated solvent impacts to soil, groundwater, and soil vapor. Site challenges included investigation and remedial action within existing, occupied building sites. Designed and managed a bedrock fracture investigation to address potential impacts to bedrock. Designed and managed offsite delineation of chlorinated solvent plume in soil vapor. Directed multiple offsite soil vapor investigations within adjacent properties; assisted in negotiating several nuanced access agreements. Managed an onsite interim remedial measure including the installation of a retro coat vapor barrier and retro-fitted SSDS within the site building.

New York State Brownfield Cleanup Program, 34th Street and 42nd Street, West Side, Manhattan, New York—Designed and managed multiple investigations to address New York State Spills and Brownfield Cleanup programs. Prepared scopes of work to address requirements of both state regulations and those agreed to by the former owner. Coordinated

Alana M. Carroll, PG Page 3

with NYSDEC to modify scopes based on field observations and limitations, which resulted in not having to mobilize for additional investigations. Coordinated with multiple entities for access to perform investigations, including Javits Convention Center, Amtrak, New York City Department of Transportation, Metropolitan Transit Authority, and their contractors. Developed a three-phase analysis plan with the laboratory to determine the minimum required extent of excavation next to an Amtrak line while limiting analytical costs, decreasing in the extent of excavation, and lowering disposal and structural support requirement costs.

New York State Brownfield Cleanup Program, 388 Bridge Street, Downtown Brooklyn, New York—Designed and managed all onsite and offsite investigations of soil, soil gas, groundwater, and indoor air, including coordination of staff and subcontractors. Prepared investigation reports for submittal to client, project team, NYSDEC, and NYSDOH. Participated in project team decision making with clients, lawyers, construction manager, and other consultants. Managed New York City Transit approvals for subsurface investigations near subway lines. Coordinated offsite access in residences, commercial spaces, and a private school. Participated in soil vapor extraction pilot test implementation and reporting. Assisted with implementation of an offsite SSDS in an existing building; activities included system design/layout, installation oversight, testing, and long-term operation and maintenance. Responsible for NYSDEC/NYSDOH coordination and reporting for all investigations. Tracked project activities for inclusion in NYSDEC/NYSDOH programmatic submittals, including monthly reports and remedial schedules.

New York Department of Environmental Remediation, Class 2 State Superfund, Laurel Hill Site, Queens, New York—Managed multiphase, multiparcel project involving design, installation, and ongoing operation, maintenance, and monitoring of six remedial caps. Site challenges included the division of the site into individual parcels that were independent of one another; subsequently, each parcel had a stormwater management design individual to the surrounding parcels. Other challenges included the site's position in a wetlands area fronting Newtown Creek, and working with the New York City Department of Transportation to facilitate its schedule for the adjacent Kosciusko Bridge restoration.

**New York State Brownfield Cleanup Program, Willets Point Development, Queens, New York**—Managed the Brownfield Cleanup Program application and Phase I environmental site assessment effort for 45 parcels of industrialized land. Coordinated with multiple interested parties, including New York City Department of Housing Preservation and Development and the Economic Development Corporation for access and contracting.

New York State Brownfield Cleanup Program, Uniforms for Industry, Queens, New York—Designed and managed an alternative approach to the offsite soil vapor intrusion investigation. Utilized soil vapor modeling to evaluate potential human health risks and migration probabilities. Provided support for the design of a retrofitted passive venting system.

New York State Spills Program, Gotham Center, Queens, New York—Responsible for proposal and budget development, subcontractor selection and coordination, negotiation, and preparation of subcontractor terms and agreements, budget, and invoice review for a comprehensive subsurface investigation. Prepared and implemented scope of work for delineation of soil contamination and calculation of contaminant mass estimates. Subsequent to interpretation of site data and subgrade characteristics, developed and presented remedial alternatives and associated costs for internal and client project teams. Prepared remedial investigation report in coordination with the New York City Economic Development Corporation and the client for submittal to state regulators.

# Matthew Carroll, P.E. Environmental Engineer/Principal

### **Experience Summary**

Matthew Carroll is an environmental engineer experienced in all aspects of site assessment and development and implementation of remedial strategies. He has managed projects from inception through investigation, remediation and closure. His expertise includes soil, soil gas, and groundwater remediation, preparation of cost estimates, remedial alternative selection and design, soil characterization for disposal, field safety oversight, and preparation of work plans and reports to satisfy New York and New Jersey state requirements, and New York City "e" designation and restrictive declarations. Mr. Carroll's project management experience includes past management of a New York City School Construction Authority hazardous materials contract. He is responsible for all engineering work performed by Tenen and is currently the project manager and remedial engineer for several New York State Brownfield Cleanup Program sites.

### **Selected Project Experience**

### 470 Kent Avenue, Brooklyn

As project manager, supported the client in due diligence and transactional activities, including a Phase I ESA, preliminary site investigation, and remedial cost estimate; preparation of BCP application and remedial investigation work plan. The former manufactured gas plant, sugar refinery and lumberyard will be developed as a mixed-use project with market rate and affordable housing and public waterfront access. As remedial engineer, will be responsible for development of remedial alternatives and oversight and certification of all remedial activities.

### **500 Exterior Street, Bronx**

Designed and implemented the investigation of this former lumberyard and auto repair shop that will be redeveloped as mixed use development with an affordable housing component; prepared BCP application and subsequent work plans and reports. Designed a remedial strategy incorporating both interim remedial measures (IRMs) and remediation during the development phase.

### Gateway Elton I and II, Brooklyn

Conducted soil disposal characterization, prepared Remedial Action Work Plans and designed methane mitigation systems for two phases of a nine-building residential development and commercial space; prepared and oversaw implementation of a Stormwater Pollution Prevention Plan during construction and prepared and certified the remedial closure reports for the project.

### Affordable Housing Development, Rve, NY

Consultant to the City of Rye on environmental issues pertaining to a county-owned development site slated for an afford senior housing; reviewed environmental documentation for the project and prepared summary memorandum for City Council review; recommended engineering controls to address potential exposure to petroleum constituents, presented report findings at public meetings and currently providing ongoing environmental support during project implementation.

### Queens West Development BCP Site, Long Island City, New York

Assistant Project Manager for two developers involved in the site.

- Responsible for oversight of remediation under the New York State Brownfield Cleanup Program
- Technical review of work plans and reports and coordination of the Applicant's investigation and oversight efforts
- Provided input for mass calculations and well placement for an in-situ oxidation remedy implemented on a proposed development parcel and within a City street
- Conducted technical review of work pertaining to a former refinery. Documents reviewed included work plans for characterization and contaminant delineation; pilot test (chemical oxidation); remediation (excavation and groundwater treatment). Managed field personnel conducting full time oversight and prepared progress summaries for distribution to project team
- Following implementation of remedial action, implemented the Site Management Plan and installation/design of engineering controls (SSDS, vapor barrier/concrete slab, NAPL recovery). Also responsible for coordination with NYSDEC

### Brownfield Cleanup Program Redevelopment Sites - West Side, New York City

Managed remediation of a development consisting of four parcels being addressed under one or more State and city regulatory programs (NYS Brownfield Cleanup Program, NYS Spills, and NYC "e" designation program). Remediation includes soil removal, screening and disposal; treatment of groundwater during construction dewatering and implementation of a worker health and safety plan and community air monitoring plan (HASP/CAMP)

Managed an additional BCP site, supported the Applicant in coordination with MTA to create station access for the planned No. 7 subway extension; also provided support the client in coordination with Amtrak to obtain access for remedial activities on the portion of the site that is within an Amtrak easement. The site will eventually be used for construction of a mixed-use high-rise building.

### BCP Site, Downtown Brooklyn, New York

Performed investigation on off-site properties and designed an SSDS for an adjacent building, retrofitting the system within the constraints of the existing structure; coordinated the installation of the indoor HVAC controls and vapor barrier; provided input to the design of a SVE system to address soil vapor issues on the site.

### West Chelsea Brownfield Cleanup Program Site

Designed an in-situ remediation program and sub-slab depressurization system to address contamination remaining under the High Line Viaduct; SSDS design included specification of sub-grade components, fan modeling and selection, identifying exhaust location within building constraints and performance modeling; prepared the Operations Maintenance and Monitoring Plan and Site Management Plan sections pertaining to the SSDS.

### Historic Creosote Spill Remediation - Queens, New York - New York State Voluntary Cleanup Program

Modeled contamination volume and extent and prepared mass estimates of historic fill constituents and creosote-related contamination; designed a soil vapor extraction (SVE) and dewatering system to address historic creosote release both above and below static

water table; coordinated with the Metropolitan Transit Authority and prepared drawings to secure approval to drill in the area of MTA subway tunnels.

### NYSDEC Spill Site- Far West Side, Manhattan

Provided support to client during negotiations with a major oil company regarding allocation of remedial costs. Worked with client's attorney to develop a regulatory strategy to address the client's obligations under the NYSDEC Spills Program and the New York City "e" designation requirements.

### Affordable Housing Site, Brooklyn, New York

Modified prior work plans for soil, soil vapor and groundwater investigation to address requirements for site entry into the New York City Brownfield Cleanup Program. Prepared technical basis for use of prior data previously disallowed by OER. Currently conducting site investigation.

### New York City School Construction Authority Hazardous Materials Contract

Provided work scopes and cost estimates, managed and implemented concurrent projects, including Phase I site assessments, Phase II soil, groundwater and soil gas investigations, review of contractor bid documents, preparation of SEQR documents, specifications and field oversight for above- and underground storage tank removal, and emergency response and spill control.

### Former Manufacturing Facility, Hoboken, New Jersey

Evaluated site investigation data to support a revision of the current property use to unrestricted; modified the John & Ettinger vapor intrusion model to apply the model to a site-specific, mixed use commercial/residential development; implemented a Remedial Action Work Plan that included the characterization, removal and separation of 9,500 cubic yards of historic fill; designed and implemented a groundwater characterization/delineation program using a real-time Triad approach; designed and implemented an innovative chemical oxidation technology for the property.

### Former Varnish Manufacturer - Newark, New Jersey

Prepared a Phase I environmental site assessment; implemented soil and groundwater sampling to assess presence of petroleum and chlorinated compounds; prepared alternate cost remediation scenarios for settlement purposes and implemented a groundwater investigation plan, including pump tests and piezometer installation to assess the effect of subsurface utilities and unique drainage pathways upon contaminant transport.

### **Education and Certifications**

Professional Engineer, New York

Bachelor of Engineering, Environmental; Stevens Institute of Technology, 2002

Bachelor of Science, Chemistry, New York University, 2002

Technical and Regulatory Training in Underground Storage Tanks, Cook College, Rutgers University, 2006

# Mohamed Ahmed, Ph.D., C.P.G. Sr. Geologist/Principal

### **Experience Summary**

Mohamed Ahmed is a certified professional geologist with nearly 23 years of experience in the New York City metropolitan area. He has designed and implemented subsurface investigations and is proficient in groundwater modeling, design of groundwater treatment systems and soil remediation. He has managed numerous projects focused on compliance with the New York State Brownfield Cleanup and Spills programs and the New York City "e" designation program. Dr. Ahmed also has extensive experience in conducting regulatory negotiations with the New York State Department of Environmental Conservation, the NYC Office of Housing Preservation and Development, and the Mayor's Office of Environmental Remediation.

### **Selected Project Experience**

### Willoughby Square, Downtown Brooklyn

As Project Manager, directs all regulatory interaction and investigation on this joint public-private sector redevelopment that will include a public park and four-level underground parking garage. Prepared the remedial investigation work plan and remedial action work plan, conducted investigation activities and waste characterization, and negotiated with the NYC Department of Environmental Protection and the Mayor's Office of Environmental Remediation to transition the site into the NYC Voluntary Cleanup Program.

### School Facility, Borough Park, Brooklyn

Managed all regulatory agency coordination, work plan and report preparation and remedial oversight; worked with OER to determine measures to retroactively address the hazardous materials and air quality E-designations on a previously constructed school building and prepared supporting documentation to justify the use of electrical units rather than natural gas.

### LGA Hotel Site, East Elmhurst, Queens

Project manager for all work conducted at this former gasoline service station which is being remediated under the NYS Brownfield Cleanup Program; technical oversight of work plans, reports, and design and implementation of field and soil disposal characterization.

### 436 10th Avenue, Manhattan

As project manager and technical lead, assisted client in developing remedial cost estimates used for property transaction, developed regulatory strategy to address NYS Spills and NYC E-designation requirements, and currently overseeing remedial activities which include removal and disposal of petroleum-contaminated bedrock and dewatering and disposal of impacted groundwater.

### Brownfield Cleanup Program Site, Downtown Brooklyn

Managed investigation and remediation under the BCP program for a proposed mixed-use development; designed the remedial investigation and prepared the remedial action work plan which includes an SVE system monitored natural attenuation. Prepared remedial cost

estimates for several scenarios. The project will include a 53-story mixed-use structure and parking garage.

### **Queens West Development, Long Island City**

Directed project team and subcontractors for soil investigation/remediation studies on multiple properties; provided technical support for negotiations with NYSDEC during investigation and remediation.

### Former Creosote Site, Long Island City

Designed and implemented a complex investigation to assess the nature and extent of historic creosote contamination at this former industrial site; conducted studies to optimize recovery of LNAPL and DNAPL and developed strategies using bioremediation and natural attenuation in conjunction with conventional remedial approaches. Performed pilot tests for soil vapor extraction system design and coordinated with NYSDEC and NYSDOH to implement sub-slab soil vapor sampling.

### NYSDEC Spill Site - Far West Side, Manhattan

Developed a detailed remedial cost estimate for to support client negotiations with a major oil company. The estimate included costs pertaining to: chipping, removal and disposal of petroleum-impacted bedrock; removal/disposal of recycled concrete; costs for dewatering and disposal of impacted groundwater during construction; and design and installation of a vapor barrier below the redevelopment.

### Active Industrial Facility, Newburgh, New York

Designed remedial investigation of soil and groundwater contaminated with trichloroethane; performed soil vapor pilot test and pump test to aid in design of soil and groundwater remediation alternatives; conducted sub-slab vapor sampling in accordance with NYSDOH guidance.

### Former Dry Cleaning Facility, New York City

Conducted soil and groundwater investigations, designed and installed a soil vapor extraction system and performed extensive testing of indoor air. Negotiated the scope of the RI and IRM with NYSDEC.

### Waterfront Redevelopment, Yonkers, NY

Designed and performed geophysics survey of six parcels to determine locations of subsurface features; supervised test pit excavation to confirm geophysics results and evaluate and classify soil conditions prior to development activities.

### Prince's Point, Staten Island, New York

Performed soil, groundwater and sediment sampling to delineate the extent of contamination; used field-screening techniques to control analytical costs and supervised soil excavation and disposal.

### **Apartment Complex, New York City, New York**

Coordinated with Con Edison, the owner of the adjacent property and NYSDEC to determine oil recovery protocol; assessed hydrogeological conditions and conducted pilot tests to design cost-effective recovery system; designed and supervised installation of recovery system.

### **Publications**

"Impact of Toxic Waste Dumping on the Submarine Environment: A Case Study from the New York Bight". Northeastern Geology and Environmental Sciences, V. 21, No. 12, p. 102-120. (With G. Friedman)

Metals Fluxes Across the Water/Sediment Interface and the Influence of pH. Northeastern Geology and Environmental Sciences, in press. (With G. Friedman)

"Water and Organic Waste Near Dumping Ground in the New York Bight". International Journal of Coal Geology, volume 43. (With G. Friedman)

### **Education and Certifications**

Ph.D., Earth and Environmental Sciences, Graduate Center of the City of New York (2001) M.Ph., Earth and Environmental Sciences, City University of New York (1998) M.A. Geology, Brooklyn College (1993) B.S. Geology, Alexandria University, Egypt (1982)

American Institute of Professional Geologists, Certified Professional Geologist, 1997-2015

### L.A.B. Validation Corp., 14 West Point Drive, East Northport, New York 11731

### Lori A. Beyer

SUMMARY:

General Manager/Laboratory Director with a solid technical background combined with Management experience in environmental testing industry. Outstanding organizational, leadership, communication and technical skills. Customer focused, quality oriented professional with consistently high marks in

customer/employee satisfaction.

### EXPERIENCE:

1998-Present

L.A.B. Validation Corporation, 14 West Point Drive, East Northport, NY

President

Perform Data Validation activities relating to laboratory generated Organic and Inorganic Environmental Data.

1998-Present American Analytical Laboratories, LLC. 56 Toledo Street, Farmingdale, NY

### Laboratory Director/Technical Director

- Plan, direct and control the operation, development and implementation of programs for the entire laboratory in order to meet AAL's financial and operational performance standards.
- Ensures that all operations are in compliance with AAL's QA manual and other appropriate regulatory requirements.
- Actively maintains a safe and healthy working environmental that is demanded by local laws/regulations.
- Monitors and manages group's performance with respect to data quality, on time delivery, safety, analyst development/goal achievement and any other key performance indices.
- Reviews work for accuracy and completeness prior to release of results to customers.

1996-1998 Nytest Environmental, Inc. (NEI) Port Washington, New York

### General Manager

- Responsible for controlling the operation of an 18,000 square foot facility to meet NEI's Inancial and operational performance standards.
- Management of 65 FTEs including Sales and Operations
- Ensure that all operations are in compliance with NEI's QA procedures
- Ensures that productivity indicators, staffing levels and other cost factors are held within established guidelines
- Maintains a quantified model of laboratory's capacity and uses this model as the basis for controlling the flow of work into and through the lab so as to ensure that customer requirements and lab's revenue and contribution targets are achieved.

1994-1996 Nytest Environmental, Inc. (NEI) Port Washington, New York

### Technical Project Manager

- Responsible for the coordination and implementation of environmental testing programs requirements between NEI and their customers
- Supervise Customer Service Department
- Assist in the development of major proposals
- Complete management of all Federal and State Contracts and assigned commercial contracts
- Provide technical assistance to the customer, including data validation and Interpretation
- Review and Implement Project specific QAPP's.

Nytest Environmental, Inc. (NEI) Port Washington, New York 1995-1996

### Corporate QA/QC Officer

- Responsible for the implementation of QA practices as required in the NJDEP and EPA Contracts
- Primary contact for NJDEP QA/QC issues including SOP preparation, review and approval
- Responsible for review, verification and adherence to the Contract requirements and NEI QA Plan

1992-1994 Nytest Environmental, Inc. (NEI) Port Washington, New York

### Data Review Manager

- Responsible for the accurate compilation, review and delivery of analytical data to the company's customers. Directly and effectively supervised a department of 22 personnel.
- Managed activities of the data processing software including method development, form creation, and production
- Implement new protocol requirements for report and data management formats
- Maintained control of data storage/archival areas as EPA/CLP document control officer

1987-1991 Nytest Environmental, Inc. (NEI) Port Washington, New York

### Data Review Specialist

- Responsible for the review of GC, GC/MS, Metals and Wei Chemistry data in accordance with regulatory requirements
- Proficient with USEPA, NYSDEC, NJDEP and NEESA requirements
- Review data generated in accordance with SW846, NYSDEC ASP, EPA/CLP and 40 CFR Methodologies

1986-1987 Nytest Environmental, Inc (NEI) Port Washington, New York GC/MS VOA Analyst

### **EDUCATION:**

1982-1985 State University of New York at Stony Brook, New York; BS Biology/Biochemistry

1981-1982 University of Delaware; Biology/Chemistry

Rutgers University; Mass Spectral Data Interpretation Course, GC/MS Training 5/91

8/92 Westchester Community College; Organic Data Validation Course

Westchester Community College; Inorganic Data Validation Course 9/93

Awards this Certificate of Achievement To

LORI BEYER

# for Successfully Completing

ORGANIC DATA VALIDATION COURSE (35 HOURS)

Dr. John Samuelian

Date AUGUST 1992

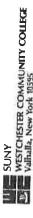
Assistant Dean

Professional Development Center

President



The Professional Development Center



# Westchester Community College Professional Development Center

Awards this Certificate of Achievement To

LORI BEYER

for Successfully Completing

INORGANIC DATA VALIDATION

Dale Boshart Instructor:

Date MARCH 1993

Assistant Dean

Professional Development Center



The Professional Development Center

SUNY
WESTCHESTER COMMUNITY COLLEGE
Valhalla, New York 10595

# New York State Department of Environmental Conservation 50 Wolf Road, Albany, New York 12233



July 8, 1992

Ms. Elaine Sall Program Coordinator Westchester Community College Valhalla, NY 10595-1698

Dear Elaine,

Thank you for your letter of June 29, 1992. I have reviewed the course outline for organic data validation, qualifications for teachers and qualifications for students. The course that you propose to offer would be deemed equivalent to that which is offered by EPA. The individuals who successfully complete the course and pass the final written exam would be acceptable to perform the task of organic data validation for the Department of Environmental Conservation, Division of Hazardous Waste Remediation.

As we have discussed in our conversation of July 7, 1992, you will forward to me prior to the August course deadline, the differences between the EPA SOW/90 and the NYSDEC ASP 12/91. You stated these differences will be compiled by Mr. John Samulian.

I strongly encourage you to offer an inorganic data validation course. I anticipate the same list of candidates would be interested in an inorganic validation course as well, since most of the data to be validated consists of both organic and inorganic data.

Thank you for you efforts and please contact me if I can be of any further assistance.

Sincerely,

maureen t

Maureen P. Serafini

Environmental Chemist II Division of Hazardous Waste Remediation

(V)





The Professional Development Center
AT WESTCHESTER COMMUNITY COLLEGE

October 2, 1992

Ms. Lori Beyer 3 sparkill Drive East Northport, NY 11731

Dear Ms. Beyer:

Congratulations upon successful completion of the Organic Data Validation course held August 17 - 21, 1992, through Westchester Community College, Professional Development Center. This course has been deemed by New York State Department of Environmental Conservation as equivalent to EPA's Organic Data Validation Course.

Enclosed is your Certificate. Holders of this Certificate are deemed competent to perform organic data validation for the New York State DEC Division of Hazardous Waste Remediation.

The Professional Development Center at Westchester Community College plans to continue to offer courses and seminars which will be valuable to environmental engineers, chemists and related personnel. Current plans include a TCLP seminar on November 17th and a conference on Environmental Monitoring Regulations on November 18th.

We look forward to seeing you again soon at another environmental program or event. Again, congratulations.

Very truly yours,

Passing Grade is 70% Your Grade is 99%

Elaine Sall Program Coordinator

ES/bf





The Professional Development Center
AT WESTCHESTER COMMUNITY COLLEGE

June 21, 1993

Dear Ms. Beyer:

Enclosed is your graded final examination in the Inorganic Data Validation course you completed this past March. A score of 70% was required in order to receive a certificate of satisfactory completion. Persons holding this certificate are deemed acceptable to perform Inorganic Data Validation for the New York State Department of Environmental Conservation, Division of Hazardous Waste Remediation.

I am also enclosing a course evaluation for you to complete if you have not already done so. The information you provide will greatly aid us in structuring further courses. We wish to make these course offerings as relevant, targeted and comprehensive as possible. Your evaluation is vital to that end.

Congratulations on your achievement. I look forward to seeing you again at another professional conference or course. We will be co-sponsoring an environmental monitoring conference on October 21, 1993 with the New York Water Pollution Control Association, Lower Hudson Chapter, at IBM's Yorktown Heights, NY site. Information regarding this event will be going out in August.

Very truly yours,

Elaine Sall

Program Coordinator

ES/bf

Enclosures



## Appendix B

PFAS Field Sampling Guidelines



# PFAS Field Sampling Guidelines for Groundwater and Soil

For Waters: 2 x 125 mL Bottles per sample filled to the neck of the bottle

#### PLEASE READ THESE INSTRUCTIONS PRIOR TO CONDUCTING SAMPLING

Sampling for PFAS for determination using EPA 537m can be challenging due to the prevalence of these compounds in consumer products. The following guidelines reflect current knowledge and are recommended when conducting sampling.

#### **Consider Sampling for PFAS First...**

Sample containers for other methods may have PFAS present on their sampling containers which could cross-contaminate your sample(s). We are analyzing down to the low parts-per-trillion (ppt) range so cross-contamination prevention is an important consideration.

#### **SAMPLING**

All Sampling done with Nitrile Gloves, provided by YORK

#### SAMPLE CONTAINERS

All sample containers - PP ONLY (Target list of 21 PFAS) Caps are unlined and made of PP (no Teflon® lined caps) Bottles are Batch Certified to be Target PFAS-free (< Reporting Limit)

#### **FIELD EQUIPMENT**

- -Must not contain Teflon® (aka PTFE) or LDPE materials
- -All sampling materials must be made from stainless steel, HDPE, acetate, silicone, or polypropylene
- -No waterproof field notebooks can be used
- -No plastic clipboards, binders, or the like
- -No adhesives (e.g.Post-It® Notes, Duct tape) can be used
- -Sharpies and permanent markers not allowed; regular ball point pens are acceptable
- -Aluminum foil must not be used
- -Keep PFAS samples in separate cooler, away from sampling containers that may contain PFAS
- -Coolers filled with regular ice only Do not use chemical (blue) ice packs

#### **EQUIPMENT DECON**

- -"PFAS-free" water (e.g. Poland Spring\*)-on-site for decontamination
- -Only Alconox and Liquinox can be used for decontamination
- $^\star$  Poland Spring has been demonstrated to be PFAS -free when freshly opened

#### FIELD SAMPLING CLOTHING CONSIDERATIONS

Do not use fabric softener on clothing to be worn in field

Do not used cosmetics, moisturizers, hand cream, or other related products the morning of sampling

Do not use sunscreen or insect repellants

No materials containing Tyvek®

All safety boots made from polyurethane and PVC

No clothing or boots containing Gore-Tex®

Wet weather gear made of polyurethane and PVC only

#### **FOOD CONSIDERATIONS**

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

#### SAMPLE CONTAINER HANDLING

-For **AQUEOUS** Samples: Each sample set contains 2 x 125

mL containers. Fill each container to the neck

-For **SOILS**-1 x 250 mL container, FILL HALF WAY ONLY

- -No preservative is necessary for this appliction at this time.
- -Place closed, labeled Sample bottles into ZipLock bag.
- -Dispose of Nitrile gloves in provided waste bag.
- -Place in separate cooler from other samples, WET ICE only
- -Follow instructions on next page for more detail.
- -If you have a Quality Assurance Project Plan follow that guidance



## **PFAS -Recommended Field Sampling Guidelines**

#### PLEASE READ INSTRUCTIONS ENTIRELY PRIOR TO SAMPLING EVENT

Sampler should wash hands before wearing nitrile gloves in order to limit contamination during sampling. Each sample set\* requires a set of containers to comply with the method as indicated below. \*Sample set is composed of samples collected from the same sample site and at the same time. A pair of Nitrile gloves is included with each sample Zip-lock bag/bottle set. One Field Blank set per day is provided.

Note: PP is Polypropylene

Sample Containers	Bottle Type	Preservation
2 Sampling Containers - Empty- per sample-Waters	125 mL PP container-Waters	None, Cool <6C
SOILS- 1 Container-fill only half way	250 mL PP for Soils	·
1 PP Bottle with PFAS-free Water for Field Blank	125 mL PP container	None, Cool <6C
1 Field Blank (FRB) - Empty-per sampling day	125 mL PP container	None, Cool <6C
2 - Empty PP bottles for MS/DUP when needed (Soils MS/DUP come from same Bottle)	125 mL PP container	None, Cool <6C

NOTE: Sampling containers for WATERS <u>must be filled to the neck</u>. **SOILS, fill bottle only 1/2 full** 

#### FIELD BLANK and MS/DUP Bottles are labeled with <u>NEON GREEN LABELS</u>

Field blanks are required per sampling event day and the containers have been provided. Follow the instructions below.

#### **Field Blank Instructions:**

- 1. Locate the PFAS Field Blank bottle (empty, labeled) supplied The PFAS Field Blank Water container is pre-filled at YORK with PFAS-free water to transfer to the empty PFAS Field Blank bottle.
- Locate the empty container labeled "Field Blank" with Neon green labels
- 3. Open both containers and proceed to transfer contents of the "PFAS FIELD BLANK WATER" container into the "PFAS FIELD BLANK" Bottle
- 4. Field Blanks to be analyzed must be listed on the Chain-of-Custody.
- 5. Both the <u>empty</u> Field Blank water container and the <u>filled</u> Field Blank container must be returned to YORK along with the samples taken.

### Matrix Spike/ Matrix Dup Instructions: Note: Soil MS/MSD can be from the same container as the sample

- 1. Locate the PFAS MS and DUP bottles (empty, labeled-NEON GREEN) supplied -normally 1 set per 20 field samples
- 2. Transfer chosen Field MS /Dup as a normal sample and indicate sample ID on container and on Chain-of-Custody

### Sampling Instructions: ALL SAMPLE BOTTLES HAVE NEON YELLOW LABELS

- 1. Do not overfill or rinse the container. Any sample(s) for Matrix Spike and Matrix Duplicates are treated similarly.
- 2. Close containers securely. Label legibly and place containers in ZipLoc® bags, and in a separate cooler (no other container types).
- 3. Ensure Chain-of-Custody and all sample labels contain required information. Place all samples in separate coolers (separate from other samples for different parameters). Place wet ice (bagged) on samples for return to YORK.

  Samples should be kept at 4°C ±2. Samples must not exceed 10°C during first 48 hours after collection. Hold time is 14 days.

## **Appendix C**

PFAS Compounds to be Analyzed and Associated MDLs

Appendix C
PFAS Compounds to be Analyzed and Associated MDLs

Analyte	Soil MDL (ng/g)	Groundwater MDL (ng/l)
Perfluorobutanoic Acid (PFBA)	0.0504	0.512
Perfluoropentanoic Acid (PFPeA)	0.056	0.428
Perfluorobutanesulfonic Acid (PFBS)	0.0432	0.268
1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2FTS)	0.0808	0.836
Perfluorohexanoic Acid (PFHxA)	0.0464	0.236
Perfluoropentanesulfonic Acid (PFPeS)	0.0232	0.14
Perfluoroheptanoic Acid (PFHpA)	0.0232	0.16
Perfluorohexanesulfonic Acid (PFHxS)	0.0592	0.192
Perfluorooctanoic Acid (PFOA)	0.052	0.348
1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2FTS)	0.28	1.08
Perfluoroheptanesulfonic Acid (PFHpS)	0.0368	0.216
Perfluorononanoic Acid (PFNA)	0.0784	0.252
Perfluorooctanesulfonic Acid (PFOS)	0.0792	0.364
Perfluorodecanoic Acid (PFDA)	0.0752	0.324
1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2FTS)	0.3872	1.244
Perfluorononanesulfonic Acid (PFNS)	0.0424	0.248
N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSAA)	0.1	0.436
Perfluoroundecanoic Acid (PFUnA)	0.0512	0.348
Perfluorodecanesulfonic Acid (PFDS)	0.032	0.184
Perfluorooctanesulfonamide (FOSA)	0.0432	0.216
N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	0.0824	0.432
Perfluorododecanoic Acid (PFDoA)	0.0408	0.368
Perfluorotridecanoic Acid (PFTrDA)	0.0528	0.3
Perfluorotetradecanoic Acid (PFTA)	0.1064	0.212
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-Propanoic Acid (HFPO-DA)	0.0984	0.448
4,8-Dioxa-3h-Perfluorononanoic Acid (ADONA)	0.1464	0.504
Perfluorododecane Sulfonic Acid (PFDoDS)	0.0384	0.304
9-Chlorohexadecafluoro-3-Oxanone-1-Sulfonic Acid (9Cl-PF3ONS)	0.196	0.66
11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11Cl-PF3OUdS)	0.1672	0.66
N-Methyl Perfluorooctane Sulfonamide (NMeFOSA)	0.1	0.348
N-Ethyl Perfluorooctane Sulfonamide (NEtFOSA)	0.112	0.368
N-Methyl Perfluorooctanesulfonamido Ethanol (NMeFOSE)	0.2504	1.88
N-Ethyl Perfluorooctanesulfonamido Ethanol (NEtFOSE)	0.5104	0.98
Perfluoro-3-Methoxypropanoic Acid (PFMPA)	0.0408	0.228
Perfluoro-4-Methoxybutanoic Acid (PFMBA)	0.0312	0.212
Perfluoro(2-Ethoxyethane)Sulfonic Acid (PFEESA)	0.0832	0.176
Nonafluoro-3,6-Dioxaheptanoic Acid (NFDHA)	0.0952	0.944
3-Perfluoropropyl Propanoic Acid (3:3FTCA)	0.144	1.32
2H,2H,3H,3H-Perfluorooctanoic Acid (5:3FTCA)	0.5048	4.68
3-Perfluoroheptyl Propanoic Acid (7:3FTCA)	1.76	3.156

## Appendix D

Laboratory Standard Operating Procedures for PFAS Analysis

Revision 1.2

Effective Date: 06/23/2023

## **Standard Operating Procedure**

## **Determination of Target Per- and Polyfluoroalkyl Substances** (PFAS) in Aqueous and Solid matrices by Isotope Dilution **Analysis by HPLC/MS-MS** According to EPA Method 1633 Draft 3

Krys Trafalski
Scott R. Hall Scott Hall

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#### 1. **SCOPE AND APPLICATION**

This method is used to identify and quantitate specific PFAS compounds in extracts of non-potable water and solid (soil/sediment) samples using HPLC/MS-MS (High Pressure Liquid Chromatography/Tandem Mass Spectrometry). Currently the compounds (40) that are measured by this methodology are listed in Table 1.0 below.

Table 1 N-Target DEAS

Table 1.0-Target PFAS					
Perfluoroalkyl carboxylic acids					
Perfluorobutanoic acid	PFBA	375-22-4			
Perfluoropentanoic acid	PFPeA	2706-90-3			
Perfluorohexanoic acid	PFHxA	307-24-4			
Perfluoroheptanoic acid	PFHpA	375-85-9			
Perfluorooctanoic acid	PFOA	335-67-1			
Perfluorononanoic acid	PFNA	375-95-1			
Perfluorodecanoic acid	PFDA	335-76-2			
Perfluoroundecanoic acid	PFUnA	2058-94-8			
Perfluorododecanoic acid	PFDoA	307-55-1			
Perfluorotridecanoic acid	PFTrDA	72629-94-8			
Perfluorotetradecanoic acid	PFTeDA	376-06-7			
Perfluoroalkyl sulfonic acids Acid Form					
Perfluorobutanesulfonic acid	PFBS	375-73-5			
Perfluoropentansulfonic acid	PFPeS	2706-91-4			
Perfluorohexanesulfonic acid	PFHxS	355-46-4			
Perfluoroheptanesulfonic acid	PFHpS	375-92-8			
Perfluorooctanesulfonic acid	PFOS	1763-23-1			
Perfluorononanesulfonic acid	PFNS	68259-12-1			
Perfluorodecanesulfonic acid	PFDS	335-77-3			
Perfluorododecanesulfonic acid	PFDoS	79780-39-5			
Fluorotelomer sulfonic acids					
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4			
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2			
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4			
Perfluorooctane sulfonamides					
Perfluorooctanesulfonamide	PFOSA	754-91-6			
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8			
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2			
Perfluorooctane sulfonamidoacetic acids					
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9			
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6			
Perfluorooctane sulfonamide ethanols					
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7			
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2			
Per- and Polyfluoroether carboxylic acids					
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6			
4,8-Dioxa-3 <i>H</i> -perfluorononanoic acid	ADONA	919005-14-4			
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1			
Perfluoro-4-methoxybutanoic acid PFMBA 863090-89-5					
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6			
Ether sulfonic acids					
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9CI-PF3ONS	756426-58-1			
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9			
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7			

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Fluorotelomer carboxylic acids
3-Perfluoropropyl propanoic acid 3:3FTCA 356-02-5
2H,2H,3H,3H-Perfluorooctanoic acid 5:3FTCA 914637-49-3
3-Perfluoroheptyl propanoic acid 7:3FTCA 812-70-4

The estimated reporting limits (MRL) based upon the preparation/analysis parameters herein at the time of this revision are approximately 2.0 - 25.0 ng/L (ppt) for aqueous samples and 0.5 - 5.0 µg/kg for solids. The linear range for these PFAS can be extended by dilution. These MRLs are based upon a volume of 250 mL - 500 mL extracted for aqueous samples and 2 - 5 g for solids. Please refer to *Appendix 1: Target Compound Reporting Limits* for limits of detection and quantitation based on a 5 g extract and 500 mL extract for soils and waters, respectively. Please note, MDL/LOD values are based on the initial November 2022 study and are subject to change based on ongoing study data. The most up-to-date study data will be kept on file by the Quality Department and readily available in the test code section(s) of Element LIMS.

This method is "performance-based," which means that modifications may be made without additional EPA review to improve performance (e.g., overcome interferences, or improve the sensitivity, accuracy, or precision of the results) provided that all performance criteria in this method are met. Requirements for establishing equivalency are in Section 9.1.2 and include 9.1.2.2c. For Clean Water Act (CWA) uses, additional flexibility is described at 40 CFR 136.6. Changes in performance, sensitivity, selectivity, precision, recovery, etc., that result from modifications within the scope of 40 CFR Part 136.6, and Section 9.0 of this method must be documented, as well as how these modifications compare to the specifications in this method. Changes outside the scope of 40 CFR Part 136.6 and Section 9.0 of this method may require prior review or approval.

#### 2. SUMMARY

Environmental samples are prepared and extracted using method-specific procedures. Sample extracts are subjected to cleanup procedures designed to remove interferences. Analyses of the sample extracts are conducted by HPLC-MS/MS in the multiple reaction monitoring (MRM) mode. Sample concentrations are determined by isotope dilution or extracted internal standard quantification (see Section 10.3) using isotopically labeled compounds added to the samples before extraction.

#### 2.1 Extraction

**2.1.1** Aqueous samples are spiked with isotopically labeled standards, extracted using solid-phase extraction (SPE) cartridges and undergo cleanup using carbon before analysis.

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**2.1.2** Solid samples are spiked with isotopically labeled standards, extracted into basic methanol, and cleaned up by carbon and SPE cartridges before analysis.

#### 2.2 Analysis

- **2.2.1** Extracts are analyzed by HPLC-MS/MS in MRM mode. Extracts contain Non-extracted Internal Standards (NIS) to monitor instrument performance and are used for quantitative analysis.
- **2.2.2** Individual PFAS analytes are identified through peak analysis of the quantification and confirmation ions (Precursor and product ions) where applicable.
- **2.2.3** The concentration of each analyte is calculated using the isotope dilution technique. This approach corrects the target analyte concentration based on recovery of isotopic analogues and essentially behave like extracted internal standards (EIS). For QC purposes, the percent recoveries of the isotope dilution analogues are calculated using the integrated peak areas of isotope performance standards, which are added to the final extract and function as traditional internal standards (non-extracted internal standards), exclusively applied to the isotope dilution analogues.

#### 3. **DEFINITIONS**

- 3.1 ANALYTICAL SEQUENCE A set of samples that are analyzed on the same instrument continuously over a period, typically a 24 hours. An analytical sequence may consist of multiple extraction batches and various matrices and is bracketed by method specific quality control analyses.
- 3.2 CALIBRATION STANDARD (CAL) A solution of the method analytes, isotope dilution analogues, and isotope performance standards (Internal standards) prepared from primary dilution and stock standards. The calibration standards are used to calibrate the instrument response with respect to analyte concentration.
- 3.3 CONTINUING CALIBRATION VERIFICATION (CCV) A calibration standard containing target compounds, isotopic analogues, and internal standards. The CCV is analyzed periodically to verify the accuracy of an existing calibration.
- 3.4 EXTRACTION BATCH A set of up to 20 Field Samples (not including quality control samples) extracted together by the same person(s) during a workday using the same lot of standards and reagents.
- 3.5 FIELD DUPLICATES Separate samples collected at the same time and sampling location, shipped, and stored under identical conditions. Method precision, including

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the contribution from sample collection procedures, is estimated from the analysis of Field Duplicates. Field Duplicates may be used to prepare duplicate QC samples.

- 3.6 FIELD BLANK (FB) An aliquot of reagent water free of constituents of concern that is placed in a sample container in the during field sampling and treated as a sample in all respects. This includes shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are introduced to the sample from shipping, storage, and the field environment.
- 3.7 ISOTOPE DILUTION ANALOGUES Isotopically labeled analogues of the method analytes that are added to the sample prior to extraction in a known amount. Note: Not all target PFAS currently have an isotopically labeled analogue. In these cases, an alternate isotopically labeled analogue is used as detailed in this SOP and reference method.
- 3.8 ISOTOPE DILUTION TECHNIQUE An analytical technique for measuring analyte concentration using the ratio of the peak area of a native analyte to that of an isotopically labeled analogue. The analogue is added to a sample in a known amount and carried through the entire analytical procedure. The ration is used to determine a correction factor that is applied to the native compound's result.
- 3.9 ISOTOPE PERFORMANCE STANDARDS (Internal Standards) Quality control compounds that are added to all standard solutions and extracts in a known amount and used to measure the relative response of the isotopically labelled analogues that are components of the same solution. For this method, the isotope performance standards are three isotopically labeled analogues of the method analytes. The isotope performance standards are indicators of instrument performance and are used to calculate the recovery of the isotope dilution analogues through the extraction procedure. In this method, the isotope performance standards are not used in the calculation of the recovery of the native analytes.
- 3.10 METHOD BLANK An aliquot of reagent water to which known quantities of the method analytes and isotope dilution analogues are added. The results of the MBLK verify method performance in the absence of sample matrix.
- 3.11 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) Aliquots of field samples that have been fortified with a known concentration of target compounds, prior to sample preparation and extraction, and analyzed to measure the effect of matrix interferences. The use of MS/MSD samples is generally not required in isotope dilution methods because the labeled compounds added to every sample provide more performance data than spiking a single sample in each preparation batch.

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3.11 SAMPLE DUPLICATE (DUP) – A duplicate aliquot of a field sample analyzed in the same manner as a parent sample to measure reproducibility.

- 3.12 LIMIT OF QUANTITATION (LOQ) The smallest concentration that produces a quantitative result with known and recorded precision and bias. The LOQ shall be set at or above the concentration of the lowest initial calibration standard (the lowest calibration standard must fall within the linear range). Determined by matrix through the entire preparation and analysis process.
- 3.13 METHOD DETECTION LIMIT (MDL) The minimum measured concentration of a substance that can be reported with 99% confidence that the measured analyte concentration is distinguishable from method blank results (40 CFR 136, Appendix B).
- 3.14 MINIMUM LEVEL OF QUANTITATION (ML) The lowest level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. The ML represents the lowest concentration at which an analyte can be measured with a known level of confidence. It may be equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed. Alternatively, the ML may be established by multiplying the MDL (pooled or not pooled, as appropriate) by 3.18 and rounding the result to the number nearest to 1, 2, or 5 x 10n, where n is zero or an integer (see 68 FR 11770).
- 3.15 PRECURSOR ION For the purpose of this method, the precursor ion is the deprotonated molecule ([M-H]-) of the method analyte (except for HFPO-DA, in which the precursor ion is formed by decarboxylation). In MS/MS, the precursor ion is mass selected and fragmented by collisionally activated dissociation to produce distinctive product ions of smaller m/z.
- 3.16 PRIMARY DILUTION STANDARD (PDS) SOLUTION A solution containing the analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 3.17 PRODUCT ION For the purpose of this method, a product ion is one of the fragment ions produced in MS/MS by collisionally activated dissociation of the precursor ion.
- 3.18 INITIAL CALIBRATION VERIFICATION (ICV) A calibration standard prepared independently from the primary calibration solutions. For this method, the ICV is a repeat of the entire dilution scheme starting with the same stock materials (neat compounds or purchased stock solutions) used to prepare the primary calibration solutions. Independent sources and separate lots of the starting materials are not required, provided the laboratory has obtained the purest form of the starting materials

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calibration standards.

3.19 QUANTITATIVE STANDARD - A quantitative standard of assayed concentration

and purity traceable to a Certificate of Analysis.

3.20 STOCK STANDARD SOLUTION - A concentrated solution containing one or more

method analytes prepared in the laboratory using assayed reference materials or

purchased from a reputable commercial source with a Certificate of Analysis.

3.21 TECHNICAL GRADE STANDARD – As defined for this method, a technical-grade

standard includes a mixture of the branched and linear isomers of a method analyte. For

the purposes of this method, technical-grade standards are used to identify retention

times of branched and linear isomers of method analytes.

3.22 ANALYTE – A PFAS compound included in this method. The analytes are listed in

Table 1.

3.23 CALIBRATION STANDARD (CS) – A solution prepared from a secondary standard

and/or stock solutions and used to calibrate the response of the LC-MS/MS instrument.

3.24 CONTINUING CALIBRATION VERIFICATION (CCV) STANDARD — The mid-point

calibration standard that is used to verify calibration.

3.25 CFR – Code of Federal Regulations

3.26 EXTRACTED INTERNAL STANDARD (EIS) QUANTIFICATION – The response of the

target compound is compared to the response of the labeled analog of another

compound in the same LOC.

3.27 INSTRUMENT SENSITIVITY CHECK – solution used to check the sensitivity of the instrument. The solution contains the native compounds at the concentration of the

LOQ.

3.28 IPR – INITIAL PRECISION AND RECOVERY; four aliquots of a reference matrix

spiked with the analytes of interest and labeled compounds and analyzed to establish

the ability of the laboratory to generate acceptable precision and recovery. An IPR is performed prior to the first time this method is used and any time the method or

instrumentation is modified.

3.29 OPR - ONGOING PRECISION AND RECOVERY- – Ongoing precision and recovery

standard (OPR); a method blank spiked with known quantities of analytes. The OPR is

analyzed exactly like a sample. Its purpose is to ensure that the results produced by the

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laboratory remain within the limits specified in this method for precision and recovery. Applies to OPR and LLOPR (low level OPR at **2x** the LOQ level).

3.30 SPE – SOLID PHASE EXTRACTION; a technique in which an analyte is extracted from an aqueous solution or a solid extract by passage over or through a material capable of reversibly adsorbing an analyte. Also termed liquid-solid extraction.

#### 4. INTERFERENCES

LC-MS/MS data from blanks, samples, and spikes must be evaluated for interferences. If any interferences are present, take corrective action if necessary. Do not use aluminum foil because PFAAs can be potentially transferred from the aluminum foil to the glassware. Only aluminum foil rinsed with LC/MS grade methanol can be used where necessary.

- 4.1 PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed in the Reagents section.
- 4.2 Method interferences may be caused by contaminants in solvents, reagents (including DI water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. All items such as these must be routinely demonstrated to be free from interferences (less than 1/2 the Reporting Limit), under the conditions of the analysis by analyzing Method Blanks. Subtracting blank values from sample results is not permitted.
- 4.3 PTFE products can be a source of PFAS (PFOA) contamination. The use of PTFE in the procedure should be avoided. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFOA contamination.
  - 4.3.1 Standards and samples are injected from polypropylene autosampler vials with polypropylene or polyolefin snap caps, once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.
  - 4.3.2 Random evaporation losses have been observed with the polypropylene caps causing high Internal Std. recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence, then recap with polyolefin caps for storage.

4.3.2 Teflon-lined screw caps have detected PFAS at low concentrations. Repeated injection from the same Teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene snap caps.

- 4.3.3 Aqueous samples should not come in contact with any glass containers or pipettes as PFAS analytes can potentially adsorb to glass surfaces. Standards dissolved in organic solvent may be purchased in glass ampoules. These standards in organic solvent are acceptable and subsequent transfers may be performed using glass syringes and pipets. Following extraction, the eluate must be collected in a polypropylene tube prior to concentration to dryness. Concentration to dryness in glass tubes may cause poor recovery.
- 4.4 LC/MS grade methanol must be used for all steps where methanol is used in this method. HPLC grade methanol has been demonstrated to be acceptable if tested prior to use.
- 4.5 Matrix interferences may be caused by contaminants that are coextracted from a sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.
  - 4.5.1 Co-extracted Organic Material Under normal HPLC conditions matrix effects due to co-extracted organic material enhanced the ionization of 4:2 FTS appreciably. Total organic carbon (TOC) is a good indicator of humic content of the sample.
  - 4.5.2 Solid phase extraction cartridges may be a source of interferences. The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. SPE cartridges should be sealed while in storage to prevent ambient contamination of the SPE sorbent.
- 4.6 Contamination by carryover can occur whenever a high-concentration and low concentration samples are sequentially analyzed. To reduce carryover, the sample syringe in automatically rinsed with solvent between injections. These operations are programmed into the LC multisampler system.

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4.7 Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

4.8 Both branched and linear PFAS isomers can potentially be found in the environment. Linear and branched isomers are known to exist for PFOS, PFOA, PFHxS, and PFBS, based upon scientific literature. We have also seen branched isomers for PFHpA, NMeFOSAA, NEtFOSAA and PFNA. If multiple isomers are present for one of these PFAS they likely are adjacent peaks that completely resolve or not, but usually with a deflection point resolved during peak integration. The later of these peaks matches the retention time of its labeled linear analog. In general, earlier peaks are the branched isomers and are not the result of peak splitting.

Currently, all these species are available as linear isomers. Some available branched and linear reference standards of the technical mixtures for these specific PFAS are used to ensure that all appropriate peaks are included during peak integration. These species currently include PFOA, PFHxS, NMeFOSAA, and NEtFOSAA. These branched isomers elute before the linear isomer and are integrated and reported as total for those species. Others are also included at this time such as those listed in section 7.3.4.

4.9 In an attempt to reduce PFOS bias, it is required that m/z 499>80 transition be used as the quantitation transition.

#### 5. SAMPLE HANDLING

- 5.1 Aqueous Samples samples are collected by our clients in 250 or 500ml HDPE bottles with unlined HDPE or polypropylene caps and filled to the neck. Each sample submitted should be submitted in triplicate-with one used for determination of Suspended solids and possible pre-screening. Sub-sampling should be avoided whenever possible. When historical data are available indicating high levels of PFAS, sub-sampling may be an advisable option.
- 5.2 **Soil Samples** samples are collected in wide mouth 125- or 250-mL HDPE bottles with PP unlined caps.
- 5.3 SAMPLE SHIPMENT AND STORAGE/HOLDING TIMES Maintain all aqueous samples protected from light at 0 6 °C from the time of collection until shipped to the laboratory. Samples must be shipped as soon as practical with sufficient ice to maintain the sample temperature below 6 °C during transport. Samples are to be received by the

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laboratory within 48 hours of collection. The laboratory must confirm that the sample temperature is 0 - 6  $^{\circ}$ C upon receipt. Once received by the laboratory, the samples may be stored at  $\leq$  -20  $^{\circ}$ C, or at 0 - 6  $^{\circ}$ C, until sample preparation. However, the allowable holding time for samples

depends on the storage temperature, as described below:

**5.3.1** Aqueous samples may be held in the laboratory for up to 90 days from collection, when stored at ≤ -20 °C and protected from the light. When stored at 0 - 6 °C and protected from the light, aqueous samples may be held for up to **28 days**, with the caveat that issues were observed with certain perfluorooctane sulfonamide ethanols and perfluorooctane sulfonamidoacetic acids after **7 days**. These issues are more likely to elevate the observed concentrations of other PFAS compounds via the transformation of these precursors if they are present in the sample.

- **5.3.2** Solid samples (soils and sediments) may be held for up to 90 days, if stored in the dark at either 0 6 °C or ≤ -20 °C, with the caveat that samples may need to be extracted as soon as possible (within 7 days) if NFDHA is designated as an important analyte by the customer.
- 5.4 **SAMPLE EXTRACT HOLDING TIMES** Store sample extracts in the dark at less than 0 4 °C until analyzed. If stored in the dark at less than 0 4 °C, sample extracts may be stored for up to 90 days, with the caveat that issues were observed for some ether sulfonates after 28 days. These issues may elevate the observed concentrations of the ether sulfonates in the extract over time. Samples may need to be extracted as soon as possible if NFDHA is an important analyte.

#### **6. APPARATUS AND MATERIALS** (as listed or demonstrated equivalents)

- 6.1 250 500 mL polypropylene bottles with polypropylene caps. VWR Scientific or equivalent: Part no. 414004-125, 12 pk. Alternate: White PP unlined lid L238WH and 16oz. clarified PP single wall jar 70-400 neck, item J066-Containers and Packaging.com or equivalent.
- 6.2 Transport Tube: Virgin Polypropylene, White, Plastic, 10 mL Capacity, 16 mm OD, 93 mm Overall Lg, Self-Standing, 250 PK, Item 710Z420, Gamut.com (Grainger), with PP cap or equivalent.
- 6.3 Graduated cylinders, 50, 100, 250, 500 and 1000mL, Polypropylene, VWR Scientific or equivalent

- 6.4 Analytical Balance, 0.0001g., checked for accuracy each day of use with Class S weights, certified annually by an outside service.
- 6.5 Extract concentrators: Organomation Model N-EVAP 112, 24 position concentrator with water batch control and nitrogen supply controls or equivalent
- 6.6 3.1 Micron in-line filters, Promochrom only
- 6.7 1.0-2.0 mL polypropylene snap cap vials, Agilent part no. 5182-0567 or equivalent
- 6.8 Snap caps, polypropylene or olefin, 11 mm, 11/9k, Agilent Part no. 5182-0542
- 6.9 Solid Phase Extraction Tubes: for EPA 1633: WAX (weak anion exchange mixed mode polymeric sorbent - Phenomenex No. 8B-S038-HCH 200 mg or Waters Oasis 150 mg Cat. # 186002493. Must have a pKa > 8 to remain positively charged during the extraction. Alternate is Agilent Bond Elute WAX 200 mg-cat. No. 5610-2151
- 6.10 Syringes, Hamilton or equivalent 5.0 μL, 10 μL 25 μL, 100 μL, 250 μL, 500 μL, Teflon free
- 6.11 Solid Phase Extraction System-automated-Promochrom 8 position autosampler system for 6 mL capacity SPE tubes. System retrofit to remove all PTFE components and replaced with PEEK tubing or PFAS free tubing. Automated bottle rinsing feature required with 3.1 um in line PP filters.
- 6.12 Nitrogen Evaporation System- TurboVap nitrogen evaporation system operated at less than 55C.
- LC/MS-MS system- Agilent 1260 or 1290 HPLC system interfaced to an Agilent 6.13 6470A or 6460C Triple Quadrupole system. The instrument control and qualitative/quantitative software is Mass Hunter versions B.8.0 and B.9.0 or later.
  - 6.13.1 HPLC System-Agilent 1260 or 1290 Infinity II
    - 6.13.1.1 The Agilent 1260 or 1290 Infinity II HPLC system is configured with temperature-controlled column oven compartment. 4 column configuration, temperature controlled (refrigerated) auto sampler

compartments, injection valve, proportioning valves, variable flow controls and variable injection capabilities.

- 6.13.1.2 The delay column (PFAS and other interference removal) is an Agilent Eclipse Plus C18, 4.6mm x 50 mm, 3.5 um-Part no. 959943-902 or equivalent.
- 6.13.1.3 The analytical column is a Restek Raptor C18 part no. 9304252 50mm x 2.1 mm ID, 1.8 u particle size or equivalent.

#### 6.13.2 Agilent LC/MS-MS- Agilent 6470AAR/6460C

6.14.2.1 Agilent model 6470AAR/6460C triple Quadrupole system with Agilent Jet Stream ESI source. UHP nitrogen is used as cell gas and High purity nitrogen is delivered for the sheath gas from a Peak Scientific nitrogen generator system.

- 6.14 Vortex Mixer- Benchmark Industries or equivalent
- 6.15 Variable Speed shaker table, 18" x 12"- Orbital Shaker- Jiangau Tenlin Instr. Co., Ltd., Model no. TLSK-III 20-230 RPM, 0-999 min, or equivalent
- 6.16 Centrifuge, 50 mL, Premiere Model XC-2450 Series Centrifuge 6 x 50 mL, 3500 RPM max., or equivalent
- 6.17 Mechanical Pipettors- 10-100  $\mu$ L; 100-1000  $\mu$ L; 1000-5000  $\mu$ L-4 E'S Scientific or equivalent, calibrated quarterly.
- 6.18 Vortex Mixer- Benchmark Industries or equivalent
- 6.19 pH paper, short range 6-8 and full range with 0.5 pH readability- VWR Scientific or equivalent
- 6.20 15 mL PP or HDPE Centrifuge tubes, Corning Part no. 430791
- 6.21 3 mL Disposable Transfer pipets, PE, VWR part no. 16001-176
- 6.22 1.0 mL polypropylene snap cap vials, Agilent part no. 5182-0567
- 6.23 Snap caps, polypropylene, 11 mm, 11/9k, Agilent Part no. 5182-0542
- 6.24 2mL self-standing PP microcentrifuge snap cap tubes, SKS Scientific part no. 0747-17
- 6.25 Collection tubes, 15 mL graduated PP or HDPE Centrifuge tubes, Corning Part no. 430791
- 6.26 Disposable 10 mg scoops, PP
- 6.27 Ultrasonic mixer
- 6.28 10 mL disposable syringes, PP or HDPE, luer fitting
- 6.29 13mm or 25 mm 0.2 um Nylon membrane filters, PALL Acrodisc or equivalent

#### 7. REAGENTS AND STANDARDS (as listed or demonstrated equivalents)

7.1 ALL REAGENTS and STANDARDS MUST BE LOGGED INTO THE ELEMENT LIMS SYSTEM. This includes lot numbers, expiration, open and prepared dates, receipt date, Certification/traceability documents from supplier(s) if provided and preparer.

- 7.2 SOLVENTS and REAGENTS-all as listed or equivalents
  - 7.2.1 Methanol, hypergrade for LC/MS. (Merck) from Sigma Aldrich Part no. 1060354000 or equivalent (HPLC Plus grade is an acceptable alternate)
  - 7.2.2 Water, hypergrade for LC/MS. (Merck) from Sigma Aldrich Part no. 1153334000 or equivalent (HPLC plus grade is an acceptable alternate). Alternatively, York PFAS free water demonstrated ion and PFAS free can be used.
  - 7.2.3 Acetic Acid, glacial. ACS grade or equivalent.
  - 7.2.4 Ammonium Hydroxide, conc. Cert. ACS grade, 28-30% in water, Sigma Aldrich part no.1054231000, or equivalent
  - 7.2.5 Methanolic Potassium Hydroxide (0.05 M) add 3.3 g of KOH to 1L MeOH.
  - 7.2.6 Sodium Hydroxide, pellets, ACS grade- Sigma Aldrich part no. 221465-500G, or equivalent
  - 7.2.7 Potassium Hydroxide, pellets, ACS grade
  - 7.2.8 Ammonium Acetate ACS grade or better, Ammonium Acetate, HPLC or cert. ACS grade. Sigma Aldrich Part no. 73594-100-G-F or equivalent.
  - 7.2.9 Ammonium Acetate 5 mM for HPLC in aqueous solution: HPLC gradient A--Weigh 0.3854 g (+ 0.0005) Ammonium Acetate and add to 1-liter hypergrade Water. Mix until dissolved then sonicate for 5 mins. To remove air bubbles. Stability 2 weeks.
  - 7.2.10 Methanolic Ammonium Hydroxide 0.3 % take 2.5 mL of conc. ammonium hydroxide into 247 mL MeOH (measure the 247 mL in a PP graduated cylinder-they are under QQ1 somewhere). Use a mechanical pippetor to add the 2.5 mL (not strictly quantitative FYI)-Make 4 bottles of this. Used for soil extractions. 1 month shelf life.

- 7.2.11 Methanolic Ammonium Hydroxide 1.0 % take 8.25 mL of conc. ammonium hydroxide into 242 mL MeOH (measure the 242 mL in a PP graduated cylinder-they are under QQ1 somewhere). Use a mechanical pipet to add the 8.25 mL (not strictly quantitative FYI)- Make 4 bottles of this -used in Promochrom-1 month life.
- 7.2.12 **Aqueous Ammonium Hydroxide 3%** take 24.8 mL of ammonium hydroxide and add 242 mL PFAS free water. 3-month life- <u>used for pH adjustment</u>
- 7.2.13 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. This solution is used to prepare the instrument blank, calibration standards, and is used to dilute the extracts of samples that exceed the calibration range.
- 7.2.14 **Formic Acid 0.1M-aqueous** add 873 µL formic acid into 250 mL PFAS free water- Make 2 bottles of this-used to prepare 7.2.16 below. 2-year life
- 7.2.15 **Formic Acid, 0.3M**-aqueous- add 2.62 mL (2619  $\mu$ L) into 250 mL PFAS free water- **Make 4 bottles of this** *-used in Promochrom-*2 year life
- 7.2.16 Formic Acid methanolic 1:1, 0.1M formic acid- mix equal volumes of Methanol and 0.1 M formic acid- Make 4 bottles of this -used in Promochrom-2 year life
- 7.2.17 **Formic Acid 5% aqueous** add 12.5 mL Formic acid into 250 mL PFAS free water. *Used for pH adjustment*. 2-year shelf life

#### 7.3 Stock Standards

Stock Standards are purchased in mid to high concentration levels from Wellington Laboratories, Inc. Guelph, ONT, CA. Currently, Wellington is the preferred supplier of these materials. As a second source verification, prepare a mid-level from the stock independently from the preparation used for initial calibration. Document this preparation in Element. See Attachments 1,2, and 3 for detailed information.

7.3.1 Internal Standards (7-Non-Extracted –NIS)) used for the method are MPFOA, MPFOS, M3PFBA, MPFDA, MPFHxA, MPFHxS and MPFNA. These are purchased at 250 - 1000 ng/mL depending upon the ISTD in a

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mixture. This mixture is purchased from Wellington Labs in 1.2 mL volumes with the following **part no.: MPFAC-HIF-IS**. Stored at 4C or less unopened this solution has a 5-year shelf life. Once opened, the life is one year from open date.

- 7.3.2 Isotopic Surrogate Analogs (24 isotopes) are purchased for the method described from Wellington Labs at 250-5000 ng/mL levels, depending upon the isotope. Part no. is **MPFAC-HIF-ES**.
- 7.3.3 Stock Standard mixtures of both linear and branched isomers of the EPA 1633 40 compound list are purchased from Wellington Labs at varying concentrations in 5 different mixtures under part nos. PFAC-MXJ, PFAS-MXI, PFAC-MXH, PFAC-MXG, PFAC-MXF.
- 7.3.4 Second source standard mixture of both linear and branched isomers of the EPA 1633 40 compound list is purchased from Absolute Standards at 2 µg/mL under part no. 65735.
- 7.3.5 Qualitative branched isomers mix- individual available branched and linear mixes for the following PFAS are used daily to allow for qualitative knowledge of the PFAS branched isomers so they are integrated/included in quantitative analysis: T-PFOA, lp-PFNA, br-FOSA, br-NEtFOSA, br-NMeFOSA, br-NEtFOSE and br-NMeFOSE. These are purchased at 50,000 ng/mL levels from Wellington Labs-the names above are the Catalogue nos. These have a 5-year life at stock concentrations.

Make a 100 ng/mL Intermediate mix by adding 2.0  $\mu$ L of the individual stocks up to 1.0 mL with MeOH.

Make a working solution by taking 200  $\mu$ L of the 100 ng/mL intermediate into 750  $\mu$ L of cal matrix solution (7.2.13) and add 50  $\mu$ L of 1:10 EIS mix.

Transfer 300  $\mu$ L to an autosampler vials, add 3  $\mu$ L of ISTD working mix, cap vortex, and store until needed. Shelf life is 1 year.

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The summary below details the procurement requirements for this method - All from Wellington Laboratories, Inc. except the second source from Absolute Standards:

Description	Part nos.	Comes in
40 Compound Target 1633 list targets	PFAC-MXJ	4 Days – 1.2 mL
	PFAS-MXI	
	PFAC-MXH	
	PFAC-MXG	
	PFAC-MXF	
Isotopic Surrogates-24	MPFAC-HIF-ES	4 Days – 1.2 mL
EPA 1633 – 7 Internal Std.	MPFAC-HIF-IS	4 Days – 1.2 mL
Method 1633 PFCs – Cal Std.	65735	2 days – 1.2 mL

### 7.4 Preparation of Standards

#### 7.4.1 Preparation of Working Standards and Intermediates from STOCK Materials

All stock standards are prepared by the vendor in methanol containing a bit of sodium hydroxide to prevent losses of target PFAS compounds due to potential esterification in methanolic solution. The stocks come prepared with 4 molar equivalents (a 3x excess) of sodium hydroxide for stocks at the  $50~\mu g/mL$  levels. This insures their stability with respect to potential loss due to esterification. The basic solution ensures that any acidic sites on the glass ampules or acidic impurities in the methanol are neutralized to prevent ester formation and forms the sodium salt of the PFAS to stabilize it.

When preparing any intermediate level standards, the dilution must be prepared in alkaline methanol to prevent the above from occurring.

To do this, prepare a 5.0 mM NaOH in Hypergrade Methanol (or LC/MSMS grade) by dissolving 0.02 g. of sodium hydroxide into 100 mL of MeOH. This has a 2-week life.

For intermediate standards that are made to 10 mL final volume, add 100  $\mu$ L of 5.0 mM NaOH/MeOH as part of the preparation. This results in a final concentration of NaOH at 0.05 mM.

For intermediate standards prepared to a final volume of 1.0 mL. add 10  $\mu$ L of the 5.0 mM NaOH/MeOH.

For working calibration standards/CCV/SCV made to  $500 \mu L$  final volume, using the mixture detailed in section 7.1.13 (MeOH/Water/acetic acid/ammonium hydroxide). This approximates the matrix of the final extracts for analysis.

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#### 7.4.2 Storage and Handling of Standards

All <u>working standards</u> should be stored at either room temperature or 4 °C provided the containers are sealed properly.

<u>Stock Standards</u> may be stored at 4 - 10 °C but before using must sit to allow equilibration to room temperature followed by either vigorous vortex mixing or sonication for 3-5 mins.

#### 7.4.3 <u>Detailed Standards Preparation Procedure-EPA 1633</u>

#### 7.4.4 Internal Standards-See Attachment 1

Internal Standards are purchased as a **stock mixture** at 250-1000 ng/mL.

These as transferred to a snap cap vial that has been pre-rinsed with 5 mM NaOH/MeOH then allowed to dry.

7.4.4.1 Working level of Non-Extracted Internal Standard (NIS) — make a 1:1 dilution of the stock by taking 500  $\mu$ L of the Stock and adding 500  $\mu$ L MeOH.

Use as is by adding 3  $\mu$ L to 300  $\mu$ L volumes for QC samples, or calibration.

### 7.4.5 Isotopic Surrogates (Extracted Internal Standards)- See Attachment 2

7.4.5.1 Stock Surrogates are purchased as a mixture at 250-5000 ng/mL. These are transferred to a snap cap vial that has been pre-rinsed with 5 mM NaOH/MeOH then allowed to dry.

Option 1- Use Stock as received and add 25 μL to all samples/QC to be extracted.

Option 2- Prepare **2** mL of Working EIS by preparing a 1:2 dilution to yield 125-2500 ng/mL for use as follows:

Take 1000  $\mu$ L of the Surrogate Stock, plus 25  $\mu$ L of 5 mM NaOH/MeOH and 975  $\mu$ L MeOH to give 2.0 mL final volume. **50 \muL are added to ALL preparation blanks, samples, and QC**. This is sufficient for approx. 40 x 50  $\mu$ L additions to all blanks, QC, and samples.

This corresponds to adding 5 to 100 ng of EIS compounds to the initial samples and QC. The final volume of extractions will typically be 5.0 ml so this yields 1-20 ng/mL of the isotope EISs in the final extract for analysis.

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For calibration, the Stock mix at 250-5000 ng/mL is used by adding 100 uL up to 1.0 mL final volume to yield 25/500 ng/mL in each calibration level as directed in the calibration section 7.4.7.1.

#### 7.4.6 Target Analytes- EPA 1633- See Attachment 3

The target analytes for this method are purchased commercially from Wellington Labs using the five part numbers described in Section 7.3.3. They contain the method target analytes only at varying concentrations. These mixtures are transferred from their glass ampules to snap cap vials that have been pre-rinsed with 5 mM NaOH/MeOH then allowed to dry. Again, these are the nominal concentrations and the actual anion concentrations for those present as salts are listed in the documentation and are reflected in both Mass Hunter and Element.

Preparation of a 1.0 mL volume of a 10x intermediate of each of the 5 mixes for Calibration. Some of the higher levels on the curve use aliquots of the stock as shown in Figure 2.

Scale the volume accordingly if less is desired. Note that the EPA 1633 mixes come 1.2 mL per vial so this recipe may consume one vial quickly.

**7.4.6.1 OPR and LLOPR** - these are a mid-level blank spike and low-level blank spike (at 2x the LOQ). These are prepared as follows from the EPA 1633 Target mixtures (5 components) by taking 200  $\mu$ L of each STOCK into a snap cap vial giving 1.0 mL final volume.

- 1. Element ID Y22B199 PFAC-MXF mix 200 μL.
- 2. Element ID Y22B200 PFAC-MXG mix 200 μL.
- 3. Element ID Y22B201  $\,$  PFAC-MXH mix 200  $\mu$ L.
- 4. Element ID Y22B204 PFAC-MXI mix 200 μL.
- 5. Element ID Y22B205 PFAC-MXJ mix 200 μL.

For OPR (BS) at mid-level add 100  $\mu$ L to each matrix for the batch OPR and for the **LLOPR** add 20  $\mu$ L of the spike mix and process through all steps of the specific matrix preparation.

#### 7.4.7 Second Source Standard

The Second Source intermediate standard is prepared in a similar manner to Target Analytes detailed in the previous section whereby a 10x intermediate is prepared by diluting 100  $\mu$ L of stock obtained from Absolute Standards (2  $\mu$ g/mL) in 1.0 mL of solvent. The final concentration of this standard is nominally 200 ng/mL.

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#### 7.4.8 Calibration

Calibration of the LC-MSMS systems is done by an eight-level calibration covering the range 0.2 to 1650 ng/mL, nominal. Various PFAS species are present as salts and at differing concentrations and these are reflected in Mass Hunter and Element as their actual concentrations. Six to eight levels are prepared depending upon the analyte. These levels are prepared as directed below using the internal standards, surrogates, and target analytes from above.

This is made to a final volume of 1000  $\mu$ L in the matrix described in section 7.1.13 (MeOH/Water/acetic acid/ammonium hydroxide)

This preparation excludes the ISTD in the initial preparation. After preparation as directed, withdraw 300  $\mu$ L of each level into a 500  $\mu$ L PP vial and add 3  $\mu$ L of ISTD before analysis, cap, and vortex to mix.

These are stored at <10C and are stable for 6 months when prepared as directed.

7.4.7.1 Calibration Curve Preparation - Based upon a final volume of 1.0 mL in CAL Matrix Solution\*

See Attachment 4 for details.

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#### For Final volume of 1.0 mL

Recipe uses both a 1:10 intermediate for some levels AND the Stock for other points as indicated
All standards in Stds refrig. Adjacent to QQQ1 N2 generator in box labeled EPA 1633 standards- all are opened, labeled and good to use.

	Stock: Y22B201	Stock: Y22B200	Stock: Y22B199	Stock: Y22B204	Stock: Y22B205	Stock: Y22B198
	1633 MXH Targets	1633 MXG Targets	1633 MXF Targets	1633 MXI Targets	1633 MXJ Targets	1633 EIS isotope Mix
	Intermediate @10x *	Intermediate at 10x*	Intermediate at 10x*	Intermediate at 10x*	Intermediate at 10x*	Intermediate at 10x
Level	uL of MXH 10x Interm.	ul of MXG interm.	ul of MXF interm.	ul of MXI interm.	ul of MXJ interm.	uL of EIS Interm.
1	2	2	4	2	2.5	50
2	5	5	10	5	6.25	50
3	12.5	12.5	25	12.5	15.6	50
4	25	25	50	25	31.3	50
5	50	50	100	50	62.5	50
6	125	125	250	125	15.6 of Stock	50
7	25 of Stock	25 of Stock	50 of Stock	25 of Stock	31.2 of Stock	50
8	62.5 of STOCK	62.5 of STOCK	125 of STOCK	62.5 of STOCK	78.0 of Stock	50

<sup>\* 100</sup> uL up to 1 mL in MeOH

Amount of CAL Matrix to make up to 1.0 mL Final volumes:

<sup>\*</sup>CAL MATRIX: Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid — Prepared by adding 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. This solution is used to prepare the instrument blank and is used to dilute the extracts of samples that exceed the calibration range.

CAL LEVEL	μL of CAL Matrix
1	937.5
2	918.8
3	871.9
4 *	793.7
5	637.5
6	309.0
7	843.8
8	609.5

INTERNAL STANDARD MIX (non-extracted IS-NIS). Mix 500  $\mu$ L of STOCK ISTD at 250-1000 ng/mL with 500  $\mu$ L of Methanol. This results in 125-500 ng/mL Intermediate ISTD. See 7.4.4.1.

Add 3.0  $\mu$ L to 300  $\mu$ L of each level 1-8 in a 500  $\mu$ L PP autosampler vials and cap with polyolefin cap, vortex to mix and run. Add 3  $\mu$ L to 300  $\mu$ L of all sample/QC extracts before analysis.

\*Level 4 is also used as the CCV for each analysis sequence run initially, then after every 10 samples and at the end of the sequence. Multiple vials should be prepared for this level.

#### 7.4.9 Checking the Efficacy of the Surrogate/Spike Mixes

On a monthly basis the surrogate (EIS) and spike mixes from the vials used for spiking are assayed to ensure stability. These are prepared for the analysis by taking 3.0  $\mu$ L of the surrogate (EIS) mix and 3  $\mu$ L of the Spike mix into 294  $\mu$ L MeOH/Water/Acetic Acid/Ammonium hydroxide from 7.1.13, then add 3  $\mu$ L of NIS (ISTD). This yields a 1:100 dilution of the EIS and Spike mixes. Use 100 as the dilution factor in the Mass Hunter worklist.

#### 7.4.10 Second Source - Initial Calibration Verification (ICV)

The initial calibration verification is prepared in a similar manner to calibration standards whereby 50  $\mu$ L of intermediate stock is diluted in 1.0 mL of solvent. The final concentration of this standard is nominally 10 ng/mL.

#### 8. PROCEDURE

#### 8.1 Preventative and Routine Maintenance

HPLC/MS/MS Preventative Maintenance		
As Needed:	Daily (When in use)	
Change pump seals.	Check solvent reservoirs for sufficient level of	
Change in-line filters in autosampler (HPLC).	solvent.	
Check/replace in-line frit if excessive pressure or poor performance.	Verify that pump is primed, operating pulse free. (ripple < 1%)	
Replace column if no change following in- line	Check needle wash reservoir for sufficient solvent.	
frit change.	Verify capillary heater temperature functioning.	
Clean needle.	Verify vaporizer heater temperature.	
Replace or clean Capillary	Verify rough pump oil levels. Verify	
Replace fused silica tube in ESI interface. Clean	turbo-pump functioning.	
lenses.	Verify nitrogen pressure for auxiliary and sheath	
Clean skimmer.	gasses.	
Ballast rough pump 30 minutes.	Possible Checktune	
Check Nozzle flow pattern		
<u>Semi-Annually</u>	Annually	
Replace oil mist and odor elements. Replace	Vacuum system components including fans and	
activated alumina filter if applicable	fan covers.	
	Clean/replace fan filters, if applicable.	

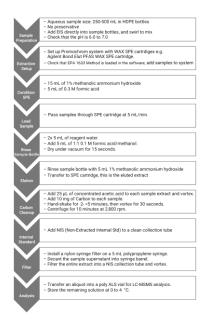
# 8.2 Sample Preparation (Extraction, Clean-up and Concentration)Aqueous Matrices

A summary of the steps for the steps related to aqueous samples are shown in Figure 1.0 and in the summary below.

- 1. Determine % Suspended Solids 10.0 mL <u>+</u> 0.02 mL through a tared 0.2 um PP filter. Dry filter > 12 hours @ 105C, cool in desiccator. Calc % TSS.
- 2. Check pH with short range pH paper to insure pH =  $6.5 \pm 0.5$ . Adjust as necessary with either 5% aqueous formic acid to lower pH or with 3% aqueous ammonium hydroxide to raise pH.
- 3. Weigh sample bottle as is to  $\pm$  0.1 g.-remove cap first since that will not be weighed later since autosampler caps are used.
- 4. Homogenize sample by inversion 3-4 x-place full volume on Promochrom System using WAX SPE cartridges.
- 5. Set up MBLK, OPR at 2x LOQ (low LCS) and mid-level OPR (mid-level LCS)-spike with 10  $\mu$ L of Spike mix for LLOPR and 100  $\mu$ L of spike mix for mid-OPR.
- 6. Spike all with 25 μL EIS solution (isotopic surrogates)

- 7. Follow Promochrom method for EPA 1633
- 8. Initiate SPE program EPA1633AQ on the Promochrom system
- 9. Once the program is finished there will be 5 ml in the collection tube. If less, make up to exactly 5.0 mL with MeOH.
- 10. Remove the sample bottle from the Promochrom system and weigh the empty bottle. That will determine the weight (volume for water) assume 1 g = 1.0 mL. Enter this value into the element bench sheet and the initial volume.
- 11. Add 25 µL of concentrated acetic acid to each collection tube and vortex to mix.
- 12. Add 10 mg of activated carbon to all samples and QC. Hand mix and vortex mix for no more than 2 minutes.
- 13. Centrifuge at 2800 rpm for approx. 10 minutes.
- 14. Filter the final volume through 0.2 um nylon filter using a syringe.
- 15. If the client provides only 250 mL of sample, in order to meet reporting limits, it may be required to concentrate the unfiltered extract by a factor of at least 2 on a TurboVap at 1.2 Liters/min with nitrogen at <55 °C. For example, if the final volume is 5.0 mL, concentrate to 2.0 mL final volume (2.5 x concentration). If 500 mL is provided, skip this step.
- 16. Enter the final volume achieved into the bench sheet in Element.
- 17. Transfer a portion of the final extract to a 2 mL snap cap, labeled.
- 18. Take a 300  $\mu$ L portion of the extract into a 500  $\mu$ L PP autosampler vial, add 3  $\mu$ L of NIS (non-extracted internal std.). Cap, vortex, store at <6 °C.
- 19. Sample is ready for analysis.

Figure 1.0 Aqueous Sample Preparation Steps



- 8.2.1 To measure sample initial volume for aqueous samples, remove the cap and weight the bottle and record the weight in the sample weight. For MBLK, LLOPR and OPR use 250-500 mL volumes). After SPE processing, be sure the empty bottle is dry and weight to determine the amount of sample in grams (essentially equal to volume in mL). Use that number for the initial volume in Element LIMS.
- 8.2.2 For every 20 field samples (Field blanks are considered field samples in as they are treated as such), a blank (MBLK), blank spikes, (2 levels-LLOPR and OPR as BS1 and BS2 respectively. A matrix spike is not necessary since isotope dilution is used. If an MS/MSD is required by a specific project, spike 100  $\mu$ L of the mid-level BS mix (OPR).
- 8.2.3 All polypropylene equipment including graduated cylinders and sample transfer lines/reservoirs should be washed prior to using with extraction solvent (Methanol).
  - 8.2.4 Add 25  $\mu$ L of EIS (isotopic surrogates) (250/5000 ng/mL) to each sample and QC sample, recap, and invert to mix well.
  - 8.2.5 Add, 20 µl (low level spike), 100 µL (mid-level spike)
  - 8.2.6 Using the Promochrom automated system, run a cleaning run. Be sure the reservoirs of LC/MS grade methanol and HPLC plus grade water or equivalent are full. Prime all lines and align all components.
  - 8.2.7. Load in the EPA1633 method and adjust the sample volume to 10 mL more than the highest volume container measured by visual comparison to a calibrated bottle of the same size. Please refer to Appendix 2: Promochrom Method detailing the automated SPE extraction method on the Promochrom system.
  - 8.2.8 The SPE method solvents for extractions are as follows:
    - Solvent 1 = MeOH
    - Solvent  $2 = H_2O$
    - Solvent 3 = 0.3 M Formic acid,
    - Solvent 4 = 1:1 0.1M Formic Acid/MeOH,
    - Solvent 5 = MeOH with 1% ammonium hydroxide ("Basic MeOH")
       W1 = Aqueous waste, W2 = Organic waste
  - 8.2.9 Place labeled 15 mL graduated collection vessels in the sample collection tray and use Element labels to identify the vials at this point. Print 2 sets of labels for each since they will be used after the concentration step as well. These are graduated.

8.2.10 Connect the bottles to the automated system.

8.2.12 Initiate the EPA1633Aq SPE Extraction Program. Each run is approximately 1 hour 45 minutes.

#### 8.2.13 **Evaporation Options** - Aqueous Samples

#### **N-EVAP** systems

- 8.2.13.1 The resulting 5 mL extracts are not further concentrated unless Work Plan reporting limits need to be lower than standard RLs. When this is required by the Work Plan, the extracts and QC are transferred to the N-EVAP concentrator systems operated at 50-55 degrees C (never more than 55C) in their original collection vials. The nitrogen flow is initiated at 1.2 ml/min and adjusted on each individual sample to provide a gentle stream causing a slight disturbance at the surface of the methanol extracts.
- 8.2.13.2 As this evaporation proceeds the walls of each vessel are rinsed with methanol when the volume is approximately 2.5 mL and then again when the volume is reduced to just below 2.0 mL. Then Bring up the final volume to 2.5 mL. This is a 2x concentration when needed.
- 8.2.14 Swirl final extract, make up to 2.0 mL with methanol. Using a disposable polypropylene pipet, carefully transfer to a 2 mL PP snap cap vial.
- 8.2.15 Withdraw an aliquot of 300  $\mu$ L into a 500  $\mu$ L autosampler vial (PP) and add 3.0  $\mu$ L of ISTD (NIS) mix.
- 8.2.16 Cap with polyolefin flexible caps and vortex to mix.
- 8.2.17 Store Extracts at <6°C until analysis.

# 8.3 Sample Preparation (Extraction, Clean-up, and Concentration) - Soil Matrices

- 1. Determine % solids: use 5 grams; dry at 110C > 12 hours.
- 2. Mix sample with a stainless-steel spatula to homogenize-exclude Sticks, vegetation, rocks and the like.
- 3. Remove 5.0 g. from the homogenized sample container. Add to a tared 50 mL centrifuge tube. Determine the weight + 0.01 g.
- 4. Prepare QC using clean matrix (Ottawa Sand) wetted with 1 mL PFAS free water in 50 mL centrifuge tubes.
- 5. For all samples, QC blanks and LCSs (LLOPR and ML OPR) and a 25  $\mu$ L aliquot of EIS onto the soil. The current Element standard ID is Y22J305. For the OPRs add

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appropriate amount of spike solution (20  $\mu$ L for LLOPR and 100  $\mu$ L for OPR. The current Element Std ID is Y22J304.

- 6. Swirl the samples to mix then let sit for 30 minutes.
- 7. Add 10 mL of 0.3% methanolic ammonium hydroxide to each centrifuge tube.
- 8. Vortex to mix then shake on the shaker table for 30 minutes.
- 9. Next, centrifuge at 3500 rpm for 5 minutes or 2800 rpm for 10 minutes.
- 10. Transfer the supernatant liquid to a clean 50 mL centrifuge tube
- 11. Add 15 mL of 0.3% methanolic ammonium hydroxide to each of the original centrifuge tubes.
- 12. Vortex to mix then shake on the shaker table for 30 minutes.
- 13. Next, centrifuge at 3500 rpm for 5 minutes or 2800 rpm for 10 minutes.
- 14. Transfer the supernatant liquid to the centrifuge tubes from 10.0 above.
- 15. Add another 5 mL of 0.3% methanolic ammonium hydroxide to each of the original centrifuge tubes.
- 16. Vortex to mix then shake on shaker table for 30 minutes.
- 17. Next, centrifuge at 3500 rpm for 5 minutes or 2800 rpm for 10 minutes.
- 18. Transfer the supernatant liquid to the centrifuge tubes from 10.0 above.
- 19. Add 10 mg of activated carbon to the combined extract using a 10 mg scoop and hand swirl for 2 minutes (never more than 5 minutes of losses of Target PFAS will occur)
- 20. Centrifuge at 3500 rpm for 5 minutes or 2800 rpm for 10 minutes
- 21. Immediately Decant into a 50 mL centrifuge tube.
- 22. Place in TurboVap or on the N-EVAP system and concentrate at 55 deg. C to a final volume of approx. 7 mL at a nitrogen flow of 1.2 mL/min.
- 23. Add 35-40 mL of PFAS free water to the tube and vortex to mix.
- 24. Check the pH=  $6.5 \pm 0.5$  if not adjust accordingly using 5% formic acid to lower pH or 3% aqueous ammonium hydroxide to raise pH to within this range.
- 25. Set up the soil EPA 1633 method on the Promochrom be sure volume is set to 50 ml for sample size. Please refer to *Appendix 2: Promochrom Method* detailing the automated SPE extraction method on the Promochrom system.
- 26. Place samples and QC centrifuge tubes on the autosampler
- 27. Once the program is finished, note the final volume and use that in the Element bench sheet as final volume. Should be 5.0 mL. If less make up to 5.0 mL with MeOH.
- 28. Add 25 µL of concentrated acetic acid to each collection tube and vortex to mix.
- 29. Add 10 mg of carbon to all samples and QC and mix for 2 minutes (no more than 5 minutes).
- 30. Immediately centrifuge at 2800 rpm for 10 minutes.

- 31. Filter the extract through a 0.2 um nylon membrane using a syringe and filter into a 2 mL snap cap vial.
- 32. When ready for analysis, remove 300  $\mu$ L of extract and transfer to a 500  $\mu$ L autosampler vial. Add 3  $\mu$ L of NIS (internal standard), vortex to mix. Cap with polyolefin flexible caps and vortex to mix.
- 33. Store Extracts at <6°C until analysis
- 34. Samples/QC are now ready for analysis.

#### 8.4 Sample Analysis--Running Samples/QC - Acquisition Method

The acquisition method is detailed in Attachment 4 (HPLC) and Attachment 5 (MS/MS) of this SOP. The method is an HPLC with dynamic MRM method with precursor and product ions with specific acquisition parameters to maximize sensitivity and specificity. This list may be modified to add other PFAS target analytes as necessary.

- 8.3.1 The triple Quadrupole (QQQ) system must be optimized for each target analyte (including surrogates and internal standards) using the Mass Hunter Optimizer program. This program determines the most abundant precursor and product ions for each compound and their abundances. These data are then used to build an MRM (multiple reaction monitor) method for acquisition. This is done initially or after any major maintenance procedures are performed to the triple quadrupole system. A high-level standard is used for this in the [M-H]<sup>-</sup> mode or M-COOH for HFPO-DA.
- 8.3.2 The MS/MS is checked for tuning on a weekly basis (if necessary) before analysis using the Tune context by selecting the CHECKTUNE radio button. This is done only in negative ion mode since that is what we are operating under. If the Checktune fails, run the Autotune program-note: this takes approx. 45 mins. in negative mode. After autotune or any tuning adjustment, a re-calibration of the instrument is required.
  - 8.3.3 Before any QC or samples can be run, the HPLC must be allowed to purge for at least thirty minutes. This purge must be done using the initial mobile phase conditions used in the method must be allowed to run for 15 minutes or until pressure has stabilized (ripple must be < 1%)
  - 8.3.4 An instrument sequence (Worklist) is then made. It should begin with a blank, a primer (5 ng/mL) followed by a blank with ISTD to establish system cleanliness.
  - 8.3.5 After a successful initial calibration has been completed, the analytical sequence for a batch of samples analyzed during the same time period is as follows. Standards and sample extracts must be brought to room temperature and

vortexed prior to aliquoting into an instrument vial in order to ensure homogeneity of the extract.

# 8.3.6 Analysis Sequence

- 1. Instrument Blank \*
- 2. Instrument Sensitivity Check -LOQ Standard Level (SEQ-CAL 1) S/N > 3:1
- 3. Calibration Verification Standard (CCV)
- 4. Qualitative Identification Standards —Branched PFAS PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NEtFOSE, and NMeFOSE.
- 5. Instrument Blank (SEQ-CCB) \*
- 6. Method Blank (Batchxxxx-BLK1)
- 7. Low-level OPR (LLOPR) (Batchxxxx-BS1)
- 8. OPR (Batchxxxx-BS2)
- 9. Field Samples (10 or fewer)
- 10. Calibration Verification Standard (SEQ-CCVn)
- 11. Instrument Blank (SEQ-CCBn) \*
- 12. Field Samples (10 or fewer)
- 13. Calibration Verification Standard (SEQ-CCVn)
- 14. Instrument Blank (SEQ-CCBn) \*

8.3.7 The run can end with a script to put the instrument into standby mode.

# 8.4 Daily Sample Preparation/Analysis Sequence

- Prepare extracts for analysis by placing a 300  $\mu$ l aliquot of sample extract containing 3  $\mu$ L of internal standards into a PP auto-sampler vial. Apply Polyolefin cap.
- Confirm that the samples loaded on the auto-sampler were entered correctly in the injection log. Make any necessary corrections.
- Run instrument CCV checks at the RL (0.25-0.5 ng/mL), then at a mid-level and high level rotating every ten samples (5, 25 ng/mL) and ending with a mid-level CCV.
- Enter the Worklist (<u>injection sequence</u>) into the instrument software and load samples onto the auto-sampler in the order shown above in Section 8.3.6

# 8.5 Data Review

The Agilent Mass Hunter Quantitation program is used to review all data. All identifications are based upon retention time (RT) of the transition of the precursor to product ion represented by a peak. Retention times should not vary

<sup>\*</sup> Contains solvent system for calibration, NIS, and EIS

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more than  $\pm$  0.5 minutes as compared to the initial CCV analyzed for an analytical sequence. All positive detections of target PFAS must be less than the high point concentration of the calibration curve.

- 8.5.1 Since certain PFAS species are manufactured by different processes the presence of branched as well as linear isomers may be found. To properly quantitate these species, the analyst must sum the related branched and linear isomers. This affects the following species: PFOS, PFHxS, PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NEtFOSE, and NMeFOSE.
- 8.5.2 Any detection greater than the upper limit of the calibration curve requires dilution into the upper half of the curve, where possible.

# 9. CALIBRATION

# 9.1 Initial Calibration

The initial calibration covers the range 0.20 ng/mL to 1560 ng/mL nominal conc. or higher depending upon the linearity of the PFAS species. After acquisition, the data are quantitated in Mass Hunter and the default calibration model for target compounds is generated using Quadratic regression, FORCED through the origin where applicable. All same level species (EIS) used average response factor model. Depending upon the response and accuracy at each level as shown in the Mass Hunter program, use Linear, Forced, weighted (1/x) or quadratic, Forced, with or without weighting to achieve the best fit which is based upon the best accuracy on a compound-by-compound basis. In any case, the correlation coefficient must be greater than 0.990. Average Response Factor (RF) or Relative Standard Deviation (RSD) should be  $\leq$  20% where used.

9.1.1 The calibration levels as shown in Section 7.4.7 use 8 levels. All points are included in the calibration with the exception of some species that saturate at levels 7 and 8.

# 9.2 Calibration Verification

9.2.1 Relative Standard Error (RSE): For calibrations using an average RF curve fit, the relative standard deviation (RSD) is the measure of relative error. However, if a quadratic regression is used RSE should be calculated for calibration curve. RSE is calculated by the Mass Hunter software. The RSE for all method analytes should be ≤ 20% to establish instrument linearity.

9.2.2 An independently prepared Initial Calibration Verification must be run immediately following initial calibration. The concentration of this standard should be in the middle of the calibration range (e.g., 5.0 ng/mL) and prepared from a separate preparation as that of the calibration. Unless project-specific data quality objectives are required, the values from the second-source check should be + 30% of the expected concentration.

9.2.2.1 **Corrective Action**: Quantitative sample analyses should not proceed for a failing ICV. Recalibrate and re-run the ICV if necessary.

An independently prepared Initial Calibration Verification must be run immediately following initial calibration. The concentration of this standard should be in the middle of the calibration range (e.g., 5.0 ng/mL) and prepared from a separate preparation as that of the calibration. Unless project-specific data quality objectives are required, the values from the second-source check should be <u>+</u> 30% of the expected concentration.

**Corrective Action**: Quantitative sample analyses should not proceed for a failing ICV. Recalibrate and re-run the ICV if necessary.

# 9.3 Continuing Calibration Verification

The first CCV is at a mid-level and analyzed every 10 client samples including a closing CCV.

The mid-Level CCV must be + 30% of the true value.

**Corrective Action**: If any of the required calibration check criteria fail, the system must be evaluated, and any appropriate instrument repair or maintenance must be performed. Sample data are unacceptable and must be rerun. Reinjection of the standard may be done. If the calibration check standard still fails, the system must be recalibrated.

# 10. Quality Control

10.1 Initial Demonstration of Capability (IDOC)

10.1.1 The initial demonstration requirement of EPA 1633 must be acceptable before analysis of samples may begin. To establish the ability to generate acceptable precision and recovery, the laboratory must perform the following operations for each sample matrix type to which the method will be applied by that laboratory.

The IDOC includes the following key elements:

- Initial Demonstration of Precision and Recovery (IPR)
- MDL determination

# 10.1.2 Initial Demonstration of Precision and Recovery-IPR

- Extract, concentrate, and analyze four aliquots of aqueous and soil matrices spiked with 100  $\mu$ L of the native spike solution OPR Mix Y22J304, 50  $\mu$ L of the EIS solution no. Y22J305. At least one method blank, matching the matrix being analyzed, must be prepared with the IPR batches by matrix. All sample processing steps that are used for processing samples, including preparation and extractions, cleanup and concentration, must be included in this test.
- Using results of the set of four analyses, compute the average percent recovery (R) of the extracts and the relative standard deviation (RSD) of the concentration for each target and EIS compound.
- For each native and isotopically labeled compound, compare RSD and % recovery with the corresponding limits for initial precision and recovery in Table 5. If RSD and R for all compounds meet the acceptance criteria, system performance is acceptable, and analysis of blanks and samples may begin. Note these acceptance criteria are not finalized and are based upon a single lab validation. Data for this table is derived from the single-laboratory validation study and are only provided as examples for this draft method. The data will be updated to reflect the inter-laboratory study results in a subsequent revision. Therefore, these criteria will change after inter-laboratory validation. Several sections of this method state that Table 5 criteria are required, this is standard language that will be applicable when the method is finalized.

# 10.1.3 MDL Determination

<u>MDL Determination</u> –In order to perform the MDL study, 7 total extractions are performed on 3 different days (Extraction Day 1= 3 LRBs and 3 LFBs, Extraction Day 2 is 2 of each, and Extraction Day 3 is also 2 of each).

The levels extracted represent approx. 3-5 x the expected LOQ.

Once extracted, the analyses are conducted on 3 separate days the MDL is determined according to the EPA MDL protocol defined in Definition and

Procedure of the Determination of the Method Detection Limit, Revision 2 Dec. 2016 as detailed below:

Make all computations as specified in the analytical method and express the final results in the method-specified reporting units.

Calculate the sample standard deviation (SD) of the replicate spiked sample measurements and the sample standard deviation of the replicate method blank measurements from all instruments to which the MDL will be applied.

Compute the MDLs (the MDL based on spiked samples) as follows:

# $MDL_s = 3.143 \times SD$ (for seven replicates; SD = Standard Deviation)

Compute the MDL<sub>b</sub> (MDL based on method blanks-LRBs) as follows:

- If none of the blanks give numerical results, then the MDL<sub>b</sub> does not apply.
- If only some of the blanks (but not all) give a result, set the MDLb to the highest result found.
- If ALL method blanks show a detection, then use the following calculation to determine MDL<sub>b</sub>:

# MDL<sub>b</sub> = Average of Blank Detections + (3.143 x Std. Dev.)

Calculate the final MDL by selecting the greater of MDL<sub>s</sub> or MDL<sub>b</sub>.

# 10.2 On-going QC Requirements

Preparation Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence.

The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch may contain a matrix spike/matrix spike duplicate (MS/MSD), two laboratory control samples (LCS-LLOPR and OPR) and a method blank. Laboratory generated QC samples (Blank, LLOPR, OPR, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate.

10.2.1 <u>METHOD BLANK</u> - One method blank must be extracted with every prep batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples the matrix is Lab reagent water. For Soils the method blank matrix is

## Ottawa sand. Criteria:

- The method blank must not contain any analyte at or above 1/2 the LOQ (Reporting Limit).
- Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- 10.2.2 <u>LABORATORY CONTROL SAMPLES</u> (LCS- also called OPR and LLOPR) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g., water for aqueous spiked with analytes of known identity and concentration and isotopic surrogate analogs. The OPRs must be processed in the same manner and at the same time as the associated samples. Recovery for Aqueous low level OPR target analytes are 40-150% until more data are derived. For all other Aqueous OPR levels recovery targets are 50-150%. These data are based upon EPA 1633 draft ranges that will change and are not used for acceptance/rejection but are reported until such time that fully validated acceptance ranges are provided in the final version of the method.
- 10.2.3 Matrix spike/Matrix spike duplicate (MS/MSD or MS/MSD). These are not typically required since each sample contains isotopic PFAS analogues that correct for any matrix effects. If the client requests them, then they are processed accordingly but are not a requirement of this method. If done they are by matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the Laboratory control limits are flagged accordingly. Until enough statistical data per matrix is available, no criteria are offered. If a specific QA Project Plan has required limits, this is preempted. Any outliers must be qualified accordingly.
- 10.2.4 <u>Initial calibration verification (ICV)</u> —A second source standard is not required for this method. A second independently prepared mid-level standard is prepared and used for this purpose and analyzed after the ICAL. The concentration should be at the mid-range of the curve and must recover within 70-130 % of expected value.

Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.

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- Evaluate the initial calibration standards.
- Rerun the initial calibration.

10.2.5 <u>Internal Standard</u>- The Non-extracted Internal Standard (NIS) is added to each field and QC sample prior to analysis. The IS response (peak area) must not deviate by more than 50-200% from the mean response (peak area) of the initial calibration. If the areas are low for all the field samples and QC samples in the batch, it suggests a loss of instrument sensitivity, while low areas in only some field or QC samples suggests a possible bad injection.

## Corrective action includes:

- Reinject the questionable samples.
- Verifying the CCV NIS areas are compliant with the range, if so, this suggests either matrix effects or may require a small dilution to mitigate interference if only some of the NIS compounds are affected.
- Qualify affected data.

# 10.3 Initial Demonstration of Capability (IDC)

Initial Demonstration of Capability involves the following processes listed ion Table 1.0 as follows.

Table 1.0 - Initial Demonstration of Capability (IDC)

Requirement	Specification and Frequency	Acceptance Criteria
Initial Demonstration of Precision and Recovery (IPR)	Extract, concentrate, and analyze four aliquots of the matrix (aqueous and soil) spiked with target native standard solution, EIS solution and finally the NIS (ISTD). Extract a method blank of each matrix with each matrix IPR batch. All steps that are used for processing samples, including preparation and extraction must be included.	Using results of the set of four analyses, compute the average percent recovery (R) of the extracts and the relative standard deviation (RSD) of the concentration for each target and EIS compound.
		For each native and isotopically labeled compound, compare RSD and % recovery with the corresponding limits for initial precision and recovery in Table 5. If RSD and R for all compounds meet the acceptance criteria, system performance is acceptable, and analysis of blanks and samples may begin.
Method Detection Limit (MDL)	Method detection limit (MDL) - Each laboratory must also establish MDLs for all the analytes using the MDL procedure at 40 CFR Part 136, Appendix B. An MDL determination must be performed for all target compounds.	The minimum level of quantification (ML) can be calculated by multiplying the MDL by 3.18 and rounding to the nearest integer

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Calibration	Analyze a mid-level ICV, each time a	Results must be 70-130% of true value.
Verification (ICV	new calibration is performed or at a	
or SCV)	minimum, quarterly.	
Section 9.1.5	The ICV must be an independent	
	dilution beginning with the common	
	starting materials used for ICAL. No	
	2 <sup>nd</sup> source is required due to	
	availability.	

# 10.4 QC Requirements

Ongoing QC requirements are detailed in Table 3.0 as follows.

**Table 3.0 QC Requirements** 

Summary of Quality Control		
Method Reference	Requirement	Specification and Frequency
Section 10.1	Mass Calibration	Annually and on as-needed basis
Section 10.1.7	Mass Calibration Verification	After mass calibration
Section 10.3	Initial Calibration (ICAL)	Minimum 6 calibration standards for linear models and 7 calibration standards for non-linear models.
Sections 10.2.2, 14.4	Retention Time (RT) window	After ICAL and at the beginning of analytical sequence
Sections 7.3.1, 9.4	Extracted Internal Standard (EIS) Analytes	All CAL standards, batch QC and field samples
Sections 7.3.2	Non-extracted Internal Standards (NIS)	All CAL standards, batch QC and field samples
Sections 7.3.4, 10.3.1, 13.3	Instrument Sensitivity Check (ISC)	Daily, prior to analysis
Section 14.2	Calibration Verification (CV) (CCV)	At the beginning and every 10 samples and at the end
Section 14.6	Instrument Blank	Daily prior to analysis and after high standards
Sections 9.1.3, 9.5, 14.7	Method Blank (MB)	One per preparation batch
Section 14.5	Ongoing Precision Recovery (OPR)	One per preparation batch
Section 11.0	Limit of Quantitation Verification (LLOPR)	Prior to analyzing samples
Section 11.0	Matrix Spike (MS/MSD)	One per preparation batch (if required) Normally not needed, since Isotope dilution is employed

# 11.0 DATA REVIEW, CALCULATIONS AND REPORTING

Samples concentrations are determined using either linear regression or quadratic regression FORCED through the origin. Weighted  $(1/x \text{ or } 1/x^2)$  may assist with low level accuracy and is recommended where necessary. All calibration curves have greater than 6 points. Any target analyte exceeding the calibration range will require dilution.

# 11.1 Data interpretation

All sample data calculations are performed by the Agilent Mass Hunter software in ng/mL and then final data are calculated considering final extract volumes and the initial sample volumes extracted which are entered into the Element bench sheet.

- 11.2 Linear and Branched Isomers are addressed in Section 8.5 and are reported for the noted species as Total which is a sum of the linear and branched isomers for affected species.
- 11.3 All Data are uploaded into Element LIMS and all final concentration calculations and associated recoveries are detailed. All pdfs of Mass Hunter Quant reports are uploaded to the Element Raw Data drive for association with ICALs and all batch and analysis sequence runs. Data are set to Analyzed status once uploaded and initially reviewed, then locked.
- 11.4 The Data is then evaluated using the York Qualinator <sup>TM</sup> data review tool which evaluates all data CCVs, QC, ISTDS, Recoveries, etc. and automatically assigns outlier qualifiers for review and acceptance by the reviewer. The accepted data are then uploaded to Element and final reviewed in Laboratory Data Entry/Review module. Once reviewed, the status is set to Reviewed indicating the data are ready to be Reported by the Reporting Group.

# 12. HEALTH AND SAFETY

12.1 General safety considerations and requirements are detailed in the York Laboratory Safety and Health Standard Operating Procedure No. Safety011600.

Specific safety rules applying to the conduct of this analysis requiring the following:

- When handling standards and samples, latex gloves are required.
- Also, when handling neat materials, a fume hood and safety glasses are required.
- When handling samples, gloves and glasses are required.
- Highly odorous samples must be handled in a fume hood.
- Refer to SDSs for specific safety/health information.
- 12.2 The analysts must exercise normal care and be supervised and trained to work in an analytical chemistry laboratory. The analysts will be handling fragile glassware, needles, syringes, volatile and flammable chemicals, toxic chemicals, and corrosive chemicals.

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- No smoking or open flames are allowed.
- No food or food products may be brought into the laboratory.

Solvents should not be left uncovered on the laboratory benches. All solvent transfers should be done in the hoods.

Hood doors must be kept in a position which yields approx. 100 fpm face velocity. Solvent evaporation must be done in the hood with exhaust elevated and in the rear.

Waste containers that had solvents must be vented to a hood until all solvents have evaporated.

Safety glasses are provided and must always be worn in the laboratory. Gloves are provided and must be worn when working with chemicals. Laboratory coats are provided and should be worn to protect the analysts' clothes. Syringes and needles must be kept in their original cases when not in use. Care must be exercised in using and handling syringes to avoid injury. Report any sticking with a needle immediately to your supervisor.

# 12.3 Specific Safety Concerns

- 12.3.1 Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS must be handled in the laboratory as hazardous and toxic chemicals.
- 12.3.2 Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.
- 12.3.3 Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries.
- 12.3.4 Eye protection, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.
- 12.3.5 Perfluorocarboxylic acids are acids and are not compatible

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with strong bases.

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

Methanol	-Flammable Poison -Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Acetic Acid, Glacial	-Flammable liquid and vapor. -Irritation	10 ppm TWA; 25 mg/m3 TWA	Eye: Causes severe eye irritation. Contact with liquid or vapor causes severe burns and possible irreversible eye damage.  Skin: Causes skin burns. May be harmful if absorbed through the skin. Contact with the skin may cause blackening and hyperkeratosis of the skin of the hands.  Ingestion: May cause severe and permanent damage to the digestive tract. Causes severe pain, nausea, vomiting, diarrhea, and shock. May cause polyuria, oliguria (excretion of a diminished amount of urine in relation to the fluid intake) and anuria (complete suppression of urination). Rapidly absorbed from the gastrointestinal  tract.  Inhalation: Effects may be delayed. Causes chemical burns to the respiratory tract. Exposure may lead to bronchitis, pharyngitis, and dental erosion. May be absorbed through the lungs.  Chronic: Chronic exposure to acetic acid may cause erosion of dental enamel, bronchitis, eye irritation, darkening of the skin, and chronic inflammation of the respiratory tract. Acetic acid can cause occupational asthma. One case of a delayed asthmatic response to glacial acetic acid has been reported in a person with bronchial asthma. Skin sensitization to acetic acid is rare, but has
Ammonium Hydroxide, conc. 28-30%	- Inhalation hazard - Skin Corrosion -Eye Damage and Irritation	OSHA PEL: 35 mg/m3 ; 50 ppm OSHA TWA: 18 mg/m3; 25 ppm	Ammonia is an irritant and corrosive to the skin, eyes, respiratory tract and mucous membranes. May cause severe chemical burns to the eyes, lungs and skin. Skin and respiratory related diseases could be aggravated by exposure. The extent of injury produced by exposure to ammonia depends on the duration of the exposure, the concentration of the liquid or vapor and the depth of inhalation.  Exposure Routes: Inhalation (vapors), skin and/or eye contact (vapors, liquid), ingestion (liquid).

Title: PFAS\_LCMSMS1633

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Formic Acid,	-Flammable	OSHA TWA:	Formic acid is an irritant and corrosive to the skin, eyes,
conc.	liquid and vapor -Harmful if swallowed -Causes severe skin burns and eye damage	5 ppm or 9 mg/m3 OSHA PEL: 10 ppm	respiratory tract and mucous membranes. May cause severe chemical burns to the eyes, lungs and skin. Skin and respiratory related diseases could be aggravated by exposure. The extent of injury produced by exposure to ammonia depends on the duration of the exposure, the concentration of the liquid or vapor and the depth of inhalation. Exposure Routes: Inhalation (vapors), skin and/or eye contact
	-Toxic if inhaled -May cause respiratory irritation		(vapors, liquid), ingestion (liquid).

# 13. WASTE MANAGEMENT/POLLUTION PREVENTION

# **Neat Materials**

Waste management procedures require the prudent use of neat materials. The ordering of neat standards and materials must be done to minimize unused material which would result in storage or handling of excess material. Quantities ordered should be sufficient to provide for necessary standards with consideration to shelf life. When ordering a unique material for a standard, be sure to order the smallest practical quantity.

# <u>Solvents</u>

The solvents used at York for this procedure include isopropanol and Methanol. These solvents are used for sample extraction or LC cleanup, all amounts are either consumed during concentration or placed in one-liter amber jars in the hood areas for evaporation. Any remaining solvent/water is transferred to a drum designated for solvent waste.

# Acids and Bases

The acids and bases used for this procedure include Acetic Acid and Formic Acid. The bases used are Ammonium hydroxide, sodium hydroxide and potassium hydroxide. Store concentrated base and acids separately whether waste or neat material.

# Samples

Unused or remaining water samples are returned to the sample control room for continued storage for proper disposal by the sample control group.

# 14. REFERENCES

 EPA METHOD 1633 Draft 3 December, 2022- Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS; EPA 821-D-22-001

Title: PFAS\_LCMSMS1633 Revision 1.2

Effective Date: 06/23/2023

## 15. REVISION HISTORY

Revision 1.0 10/24/2022

Revision 1.1 02/10/2023 Modified LLOPR in Section 7.4.6.1 to reflect 2x the MRL
Revision 1.2 06/23/2023 Added Appendix 1: Target Compound Reporting Limits.
Updated Section 9.2 to add acceptable limits for ICAL RSE.
Described the Promochrom extraction method in Sections

First issue.

Described the Promochrom extraction method in Sections 8.2, 8.3, and added *Appendix 2: Promochrom Method*. Redefined analytical batch as analytical sequence.

Updated Reference to EPA 1633 Draft 3.

Added reference to and preparation procedures for a second source standard in Section 7.

Revised qualitative identification requirements for analytes in Section 8.
Minor formatting edits.

Title: PFAS\_LCMSMS1633 Revision 1.2

Effective Date: 06/23/2023

# **Appendix 1 – Target Compound Reporting Limits**

Appendix 1 Taiget	Soil Water			
Target Compoud	MDL/LOD	RL/LOQ	MDL/LOD	RL/LOQ
Gerra Pers	ng/L	ng/L	μg/kg	μg/kg
Perfluorobutanesulfonic acid (PFBS)	0.111	0.177	0.47	1.77
Perfluorohexanoic acid (PFHxA)	0.053	0.200	0.35	2.00
Perfluoroheptanoic acid (PFHpA)	0.105	0.200	0.71	2.00
Perfluorohexanesulfonic acid (PFHxS)	0.179	0.183	0.68	1.83
Perfluorooctanoic acid (PFOA)	0.172	0.200	0.42	2.00
Perfluorooctanesulfonic acid (PFOS)	0.167	0.186	0.82	1.86
Perfluorononanoic acid (PFNA)	0.189	0.200	0.52	2.00
Perfluorodecanoic acid (PFDA)	0.191	0.200	0.75	2.00
Perfluoroundecanoic acid (PFUnA)	0.198	0.200	1.13	2.00
Perfluorododecanoic acid (PFDoA)	0.163	0.200	0.88	2.00
Perfluorotridecanoic acid (PFTrDA)	0.125	0.200	0.74	2.00
Perfluorotetradecanoic acid (PFTA)	0.103	0.200	0.69	2.00
N-MeFOSAA	0.148	0.200	0.79	2.00
N-EtFOSAA	0.194	0.200	1.03	2.00
Perfluoropentanoic acid (PFPeA)	0.109	0.400	0.23	4.00
Perfluoro-1-octanesulfonamide (FOSA)	0.146	0.200	0.88	2.00
Perfluoro-1-heptanesulfonic acid (PFHpS)	0.155	0.200	0.91	1.91
Perfluoro-1-decanesulfonic acid (PFDS)	0.191	0.193	1.32	1.93
1H,1H,2H,2H-Perfluorooctanesulfonic acid (6:2 FTS)	0.595	0.760	1.06	7.60
1H,1H,2H,2H-Perfluorodecanesulfonic acid (8:2 FTS)	0.755	0.768	2.05	7.68
Perfluoro-n-butanoic acid (PFBA)	0.109	0.800	0.33	8.00
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	0.139	0.356	0.50	3.56
Perfluoro-3,6-dioxaheptanoic acid (NFDHA)	0.193	0.400	2.14	4.00
Perfluoro-4-oxapentanoic acid (PFMPA)	0.062	0.400	0.25	4.00
Perfluoro-5-oxahexanoic acid (PFMBA)	0.096	0.400	0.37	4.00
Perfluoro-1-pentanesulfonate (PFPeS)	0.157	0.188	0.76	1.88
1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS)	0.595	0.750	1.79	7.50
HFPO-DA (Gen-X)	0.608	0.800	3.23	8.00
11CL-PF3OUdS	0.311	0.756	1.38	7.56
9CL-PF3ONS	0.246	0.748	0.70	7.48
ADONA	0.174	0.756	0.53	7.56
Perfluorododecanesulfonic acid (PFDoS)	0.169	0.194	0.93	1.94
Perfluoro-1-nonanesulfonic acid (PFNS)	0.124	0.192	0.86	1.92
3-Perfluoropropyl propanoic acid (FPrPA or 3:3FTCA)	0.634	1.000	2.03	5.00
3-Perfluoropentyl propanoic acid (FPePA or 5:3 FTCA)	2.098	5.000	7.33	25.00
3-Perfluoroheptyl propanoic acid (FHpPA or 7:3FTCA)	1.500	5.000	9.47	25.00
N-MeFOSE	0.611	2.000	3.99	20.00
N-MeFOSA	0.180	0.200	1.58	2.00
N-EtFOSE	0.697	2.000	3.99	20.00
N-EtFOSA	0.198	0.200	1.80	2.00

# **Appendix 2: Promochrom Method**

Step	Action	Inlet 1	Inlet 2	Flow (mL/min)	Volume (mL)	Target
1	Elute	1% Basic Methanol	-	8	15	Waste
2	Elute	0.3M Formic Acid	-	8	5	Waste
3	Add Sample	Sample Inlet	-	10	60 or 510*	Waste
4	Rinse Sample	Water	Air (20%)	70	2.5	Sample Container
5	Add Sample	Sample Inlet	-	5	5	Waste
6	Rinse Sample	Water	Air (20%)	70	5	Sample Container
7	Add Sample	Sample Inlet	-	5	5	Waste
8	Rinse Sample	Water	Air (20%)	70	5	Sample Container
9	Add Sample	Sample Inlet	-	5	5	Waste
10	Rinse Sample	1:1 0.1M Formic Acid/Methanol	Air (20%)	70	1.3	Sample Container
11	Add Sample	Sample Inlet	-	5	3	Waste
12	Rinse Sample	1:1 0.1M Formic Acid/Methanol	Air (20%)	70	5	Sample Container
13	Add Sample	Sample Inlet	-	5	5	Waste
14	Air Purge	SPE Cartridge	-	5	5	Minutes
15	Add Sample	Sample Inlet	-	5	5	Waste
16	Blow N2	Sample Inlet	-	5	1	. Minute
17	Rinse Sample	1% Basic Methanol	Air (20%)	70	1.3	Sample Container
18	Collect	Sample Inlet	-	5	3	Centrifuge Tube
19	Rinse Sample	1% Basic Methanol	Air (20%)	70	5	Sample Container
20	Collect	Sample Inlet	-	5	5	Centrifuge Tube
21	Collect	Sample Inlet	-	5	5	Centrifuge Tube

<sup>\*60</sup> mL is used for soil matrices and 510 mL is used for aqueous matrices.

Revision 1.2 Effective Date: 06/23/2023

# Attachment 1 – Non-Extracted Internal Standards (NIS)



# **Analytical Standard Record**

Y22B197

Description:	MPFAC-HIF-IS-EPA 1633 ISTD STOCK	Prepared:	02/16/2022
Standard Type:	Other	Expires:	09/07/2026
Solvent	Methanol/Water (<1%)	Prepared By:	Robert Q. Bradley
Final Volume (mls):	1	Department	<b>PFAS</b>
Vials:	1	Lot No.:	MPFACHIFIS0921

Standard ID:

Vendor: Wellington Laboratories

Comments:	Stock ISTD for EPA method 1633

Analyte	CAS Number	Concentration	Units
мзргва		1.	ug/mL
MPFDA		0.25	ug/mL
MPFHxA		0.5	ug/mL
MPFHxS		0.474	ug/mL
MPFNA		0.25	ug/mL
MPFOA		0.5	ug/mL
MPFOS		0.479	ug/mL



# CERTIFICATE OF ANALYSIS DOCUMENTATION

## MPFAC-HIF-IS

Mass-Labelled Perfluoroalkyl Substance Injection Standard Solution/Mixture

PRODUCT CODE:

LOT NUMBER: SOLVENT(S):

DATE PREPARED: (mm/dd/yyyy)

LAST TESTED: (mm/dd/yyyy)

EXPIRY DATE: (mm/dd/yyyy)
RECOMMENDED STORAGE:

MPFAC-HIF-IS

MPFACHIFIS0921

Methanol/Water (<1%)

09/07/2021

09/07/2021 09/07/2026

Store ampoule in a cool, dark place

## **DESCRIPTION:**

MPFAC-HIF-IS is a solution/mixture of five mass-labelled ( $^{13}$ C) perfluoroalkylcarboxylic acids (C<sub>a</sub>, C<sub>b</sub>, C<sub>a</sub>-C<sub>10</sub>) and two mass-labelled ( $^{18}$ O and  $^{13}$ C) perfluoroalkanesulfonates (C<sub>b</sub> and C<sub>b</sub>). The components and their concentrations are given in Table A.

The individual mass-labelled perfluoroalkylcarboxylic acids and mass-labelled perfluoroalkanesulfonates all have chemical purities of >98% and isotopic purities of ≥99% per ¹³C or >94% per ¹°O.

## **DOCUMENTATION/ DATA ATTACHED:**

Table A: Components and Concentrations of the Solution/Mixture

Figure 1: LC/MS Data (SIR)

Figure 2: LC/MS/MS Data (Selected MRM Transitions)

## **ADDITIONAL INFORMATION:**

- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

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Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 MPFACHIFIS0921 (1 of 5) rev1

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#### 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 compounds 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/IV/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 45% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This 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

$$\mathbf{x_{_1}},\,\mathbf{x_{_2}},...\mathbf{x_{_n}} \text{ on which it depends is:} \qquad \qquad u_c(y(x_1,x_2,...x_n)) = \sqrt{\sum_{i=1}^n u(y,x_i)^2}$$

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 ±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 <a href="https://www.well-labs.com">www.well-labs.com</a> or contact us directly at <a href="mailto:info@well-labs.com">info@well-labs.com</a> or contact us directly at <a href="mailto:info@well-labs.co

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 MPFACHIFIS0921 (2 of 5)

# Table A: MPFAC-HIF-IS; Components and Concentrations (ng/mL, ± 5% in methanol/water (<1%))

Compound	Acronym		ntration mL)	Peak Assignment in Figure 1
Perfluoro-n-(2,3,4-13C <sub>3</sub> )butanoic acid	M3PFBA	10	00	1
Perfluoro-n-(1,2-13C <sub>2</sub> )hexanoic acid	MPFHxA	50	00	2
Perfluoro-n-(1,2,3,4-13C <sub>a</sub> )octanoic acid	MPFOA	50	500	
Perfluoro-n-(1,2,3,4,5-13C <sub>8</sub> )nonanoic acid	MPFNA	2	250	
Perfluoro-n-(1,2-13C <sub>2</sub> )decanoic acid	MPFDA	2	250	
Compound	Acronym	Concentration* (ng/mL)		Peak Assignment
Compound		as the salt	as the acid	in Figure 1
Sodium perfluoro-1-hexane(18O <sub>2</sub> )sulfonate	MPFHxS	500	474	3
Sodium perfluoro-1-(1,2,3,4-13C <sub>a</sub> )octanesulfonate	MPFOS	500	479	6

<sup>\*</sup> Concentrations have been rounded to three significant figures.

Certified By: \_

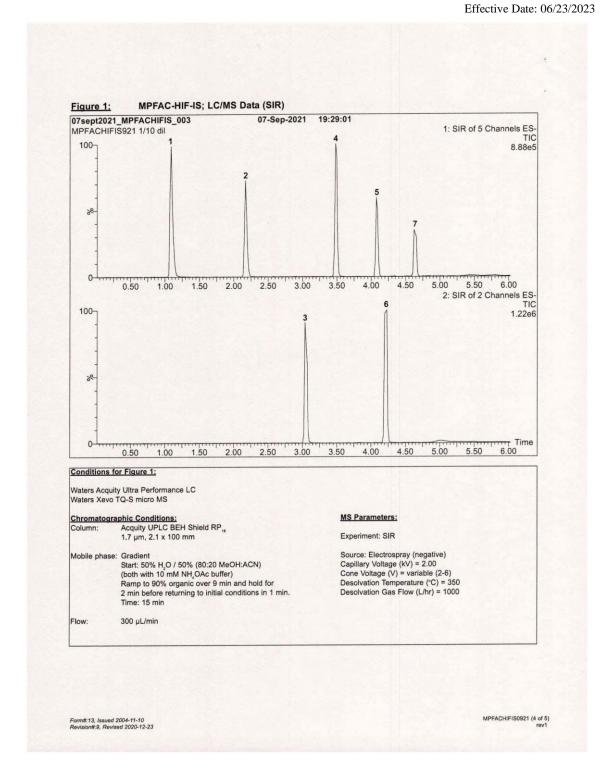
B.G. Chittim, General Manager

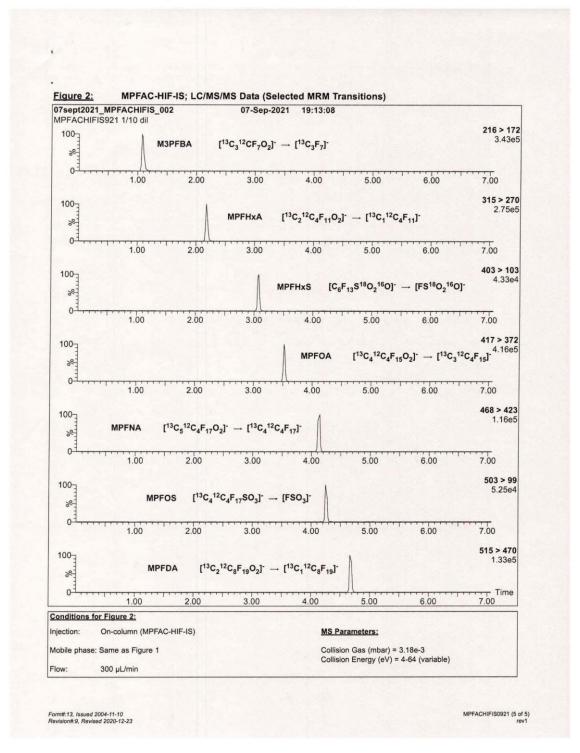
Date: 10/13/2021

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Revision 1.2





# Attachment 2 – Extracted Internal Standards (EIS)



# **Analytical Standard Record**

Standard ID: Y22B198

MPFAC-HIF-ES-EPA 1633 STOCK EIS mix 02/17/2022 Description: Prepared: Standard Type: Other Expires: 08/06/2024 MeOH/IPA/1% H2O Solvent Prepared By: Robert Q. Bradley Final Volume (mls): Department 1 PFAS Vials: Lot No.: MPFACHIFES0821 Wellington Laboratories

Comments:

Analyte	CAS Number	Concentration	Units
d3-N-MeFOSAA		1	ug/mL
d5-N-EtFOSAA		1	ug/mL
d7-N-MeFOSE		5	ug/mL
d9-N-EtFOSE		5	ug/mL
d-N-EtFOSA		0.5	ug/mL
d-N-MeFOSA		0.5	ug/mL
M2-4:2FTS		0.938	ug/mL
M2-6:2FTS		0.951	ug/mL
M2-8:2FTS		0.96	ug/mL
M2PFTeDA		0.25	ug/mL
M3HFPO-DA		2	ug/mL
M3PFBS		0.466	ug/mL
M3PFHxS		0.474	ug/mL
M4PFHpA		0.5	ug/mL
M5PFHxA		0.5	ug/mL
M5PFPeA		1	ug/mL
M6PFDA		0.25	ug/mL
M7PFUdA		0.25	ug/mL
M8FOSA		0.5	ug/mL
M8PFOA		0.5	ug/mL
M8PFOS		0.479	ug/mL
M9PFNA		0.25	ug/mL
MPFBA		2	ug/mL
MPFDoA		0.25	ug/mL



# CERTIFICATE OF ANALYSIS DOCUMENTATION

# MPFAC-HIF-ES

Mass-Labelled Per- and Poly-fluoroalkyl Substance **Extraction Standard Solution/Mixture** 

PRODUCT CODE:

MPFAC-HIF-ES

LOT NUMBER:

MPFACHIFES0821

SOLVENT(S):

Methanol/Isopropanol (1%)/Water (<1%)

DATE PREPARED: (mm/dd/yyyy) LAST TESTED: (mm/dd/yyyy)

08/05/2021

08/16/2021

EXPIRY DATE: (mm/dd/yyyy)

08/16/2024

RECOMMENDED STORAGE:

Refrigerate ampoule

#### **DESCRIPTION:**

MPFAC-HIF-ES is a solution/mixture of ten mass-labelled (13C) perfluoroalkylcarboxylic acids (C4-C12, C14), three mass-labelled (13C) perfluoroalkanesulfonates (C<sub>s</sub>, C<sub>s</sub>, and C<sub>s</sub>), three mass-labelled (one 13C and two 2H) perfluoro-1-octanesulfonamides, three mass-labelled (13C) fluorotelomer sulfonates (4:2, 6:2, and 8:2), two mass-labelled (2H) perfluorooctanesulfonamidoacetic acids, two mass-labelled (2H) perfluorooctanesulfonamidoethanols, and mass-labelled (13C) hexafluoropropylene oxide dimer acid. The components and their concentrations are given in Table A.

The individual mass-labelled perfluoroalkylcarboxylic acids, mass-labelled perfluoroalkanesulfonates, masslabelled fluorotelomer sulfonates, perfluoro-1-(13C8) octanesulfonamide, and mass-labelled hexafluoropropylene oxide dimer acid all have chemical purities of >98% and isotopic purities of ≥99%. The individual mass-labelled perfluorooctanesulfonamidoacetic acids, mass-labelled perfluorooctanesulfonamidoethanols, and two mass-labelled (2H) perfluoro-1-octanesulfonamides all have chemical purities of >98% and isotopic purities of ≥98%.

#### **DOCUMENTATION/ DATA ATTACHED:**

Table A: Components and Concentrations of the Solution/Mixture

Figure 1: LC/MS Data (SIR)

Figure 2: LC/MS/MS Data (Selected MRM Transitions)

# **ADDITIONAL INFORMATION:**

- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

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MPFACHIFES0821 (1 of 7)

Title: PFAS\_LCMSMS1633 Revision 1.2

Effective Date: 06/23/2023

#### 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 compounds 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 tot 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, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This 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

$$\mathbf{x_{_{f}}}, \mathbf{x_{_{2}}}...\mathbf{x_{_{n}}} \text{ on which it depends is:} \\ u_{_{c}}(y(x_{_{1}}, x_{_{2}},...x_{_{n}})) = \sqrt{\sum_{_{i=1}}^{n} u(y, x_{_{i}})^{2}}$$

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 ±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).

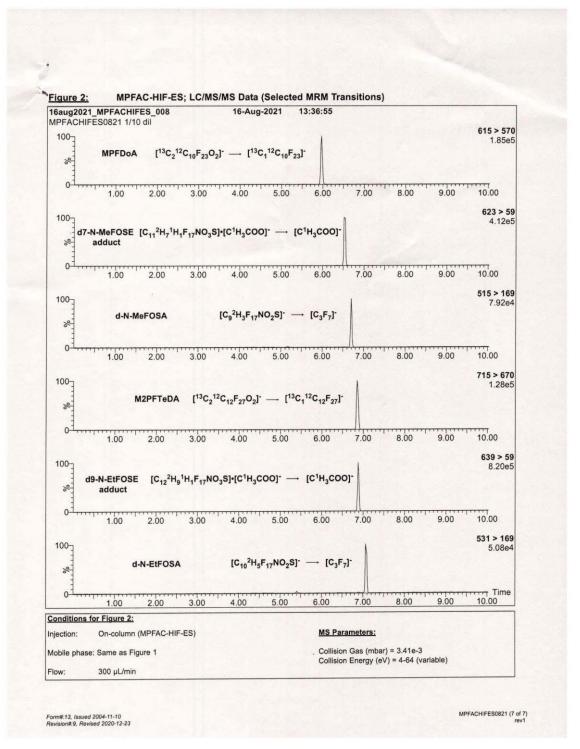




\*\*For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at www.well-labs.com or contact us directly at info@well-labs.com\*\*

MPFACHIFES0821 (2 of 7)

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23



MPFAC-HIF-ES; Components and Concentrations (ng/mL, ± 5% in Methanol/Isopropanol (1%)/Water (<1%)) Table A:

Compound	Acronym	Concentration (ng/mL)		Peak Assignment in Figure 1
Perfluoro-n-(¹³C₄)butanoic acid	MPFBA	2000		1
Perfluoro-n-(13C <sub>s</sub> )pentanoic acid	M5PFPeA	1000		2
Perfluoro-n-(1,2,3,4,6-13C <sub>5</sub> )hexanoic acid	M5PFHxA	500		5
Perfluoro-n-(1,2,3,4-¹³C₄)heptanoic acid	M4PFHpA	500		7
Perfluoro-n-(13C <sub>s</sub> )octanoic acid	M8PFOA	500		10
Perfluoro-n-(13C <sub>p</sub> )nonanoic acid	M9PFNA	250		11
Perfluoro-n-(1,2,3,4,5,6-13C <sub>e</sub> )decanoic acid	M6PFDA	250		14
Perfluoro-n-(1,2,3,4,5,6,7-13C,)undecanoic acid	M7PFUdA	250		17
Perfluoro-n-(1,2-13C <sub>2</sub> )dodecanoic acid	MPFDoA	250		19
Perfluoro-n-(1,2-13C <sub>2</sub> )tetradecanoic acid	M2PFTeDA	250		22
Perfluoro-1-(13C <sub>a</sub> )octanesulfonamide	M8FOSA	500		18
N-methyl-d <sub>3</sub> -perfluoro-1-octanesulfonamide	d-N-MeFOSA	500		21
N-ethyl-d <sub>s</sub> -perfluoro-1-octanesulfonamide	d-N-EtFOSA	500		24
N-methyl-d <sub>3</sub> -perfluoro-1-octanesulfonamidoacetic acid	d3-N-MeFOSAA	1000		15
N-ethyl-d <sub>s</sub> -perfluoro-1-octanesulfonamidoacetic acid	d5-N-EtFOSAA	1000		16
2-(N-methyl-d <sub>3</sub> -perfluoro-1-octanesulfonamido)ethan-d <sub>4</sub> -ol	d7-N-MeFOSE	5000		20
2-(N-ethyl-d <sub>s</sub> -perfluoro-1-octanesulfonamido)ethan-d <sub>4</sub> -ol	d9-N-EtFOSE	5000		23
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)(13C <sub>3</sub> )propanoic acid	M3HFPO-DA	2000		6
Compound	Acronym	Concentration* (ng/mL)		Peak Assignment
		as the salt	as the acid	in Figure 1
Sodium perfluoro-1-(2,3,4-15C <sub>3</sub> )butanesulfonate	M3PFBS	500	466	3
Sodium perfluoro-1-(1,2,3-13C <sub>3</sub> )hexanesulfonate	M3PFHxS	500	474	8
Sodium perfluoro-1-(13C <sub>s</sub> )octanesulfonate	M8PFOS	500	479	12
Sodium 1H,1H,2H,2H-perfluoro-(1,2-13C <sub>2</sub> )hexanesulfonate	M2-4:2FTS	1000	938	4
Sodium 1H,1H,2H,2H-perfluoro-(1,2-13C <sub>2</sub> )octanesulfonate	M2-6:2FTS	1000	951	9
Sodium 1H,1H,2H,2H-perfluoro-(1,2-13C <sub>2</sub> )decanesulfonate	M2-8:2FTS	1000	960	13

<sup>\*</sup> Concentrations have been rounded to three significant figures.

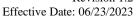
Certified By:

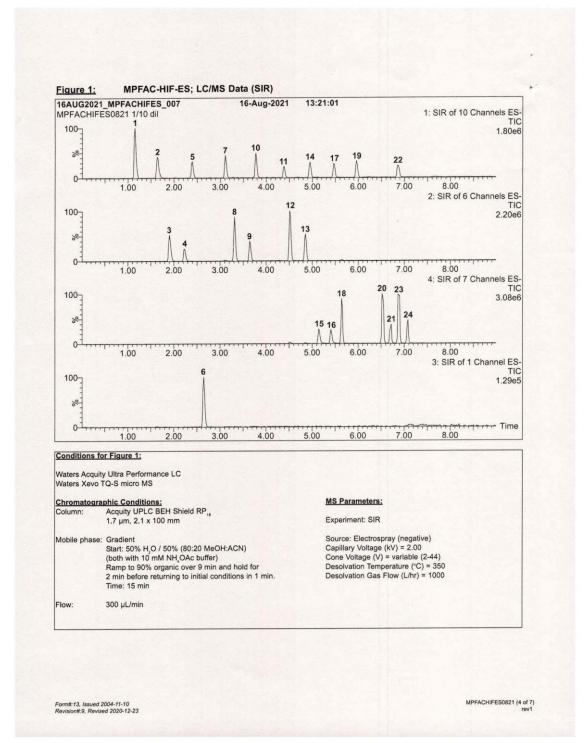
Date: 10/13/2021

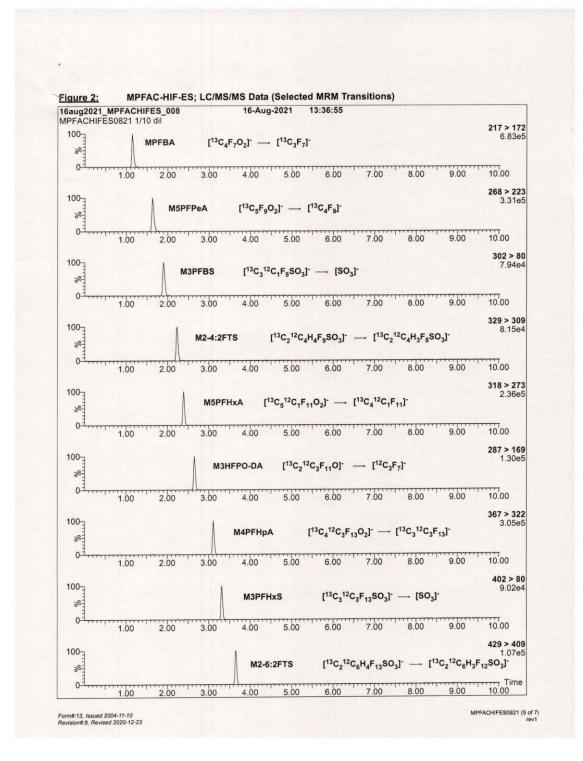
Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23

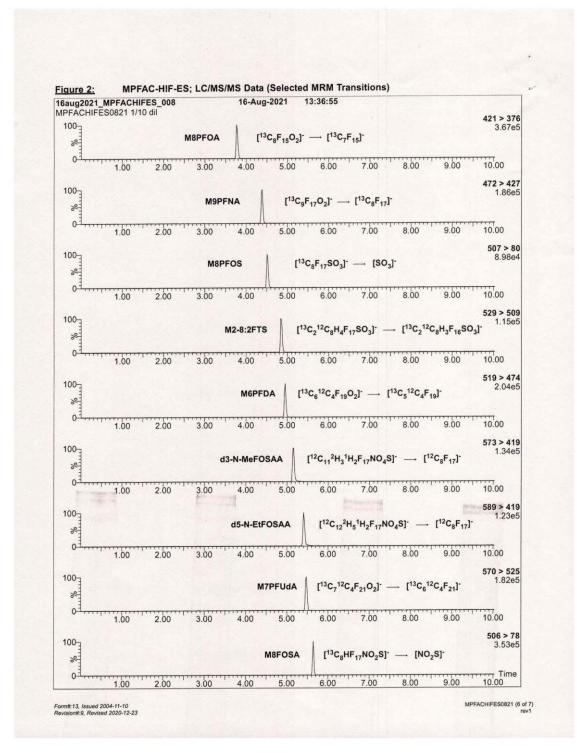
MPFACHIFES0821 (3 of 7) rev1

Revision 1.2





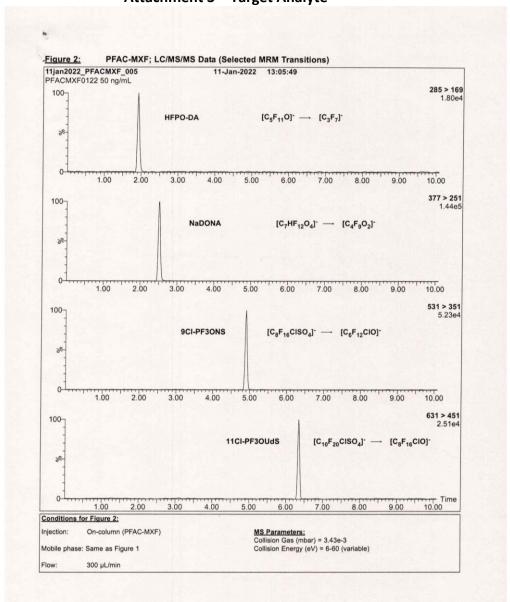




Revision 1.2

Effective Date: 06/23/2023

# Attachment 3 - Target Analyte



Title: PFAS\_LCMSMS1633 Revision 1.2

Effective Date: 06/23/2023

#### 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 compounds 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:

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## **UNCERTAINTY:**

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

The combined relative standard uncertainty,  $u_{\mu}(y)$ , of a value y and the uncertainty of the independent parameters

$$x_i, \ x_2, \dots x_n \text{ on which it depends is:} \qquad u_c(y(x_1, x_2, \dots x_n)) = \sqrt{\sum_{i=1}^n u(y, x_i)^2}$$

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.

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#### EXPIRY DATE / PERIOD OF VALIDITY:

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#### LIMITED WARRANTY:

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#### QUALITY MANAGEMENT:

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**CONFIDENTIAL DOCUMENT** Page 58 of 102

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXF0122 (2 of 5) rev0

# -Table A: PFAC-MXF; Components and Concentrations (ng/mL; ± 5% in Methanol/Water (<1%))

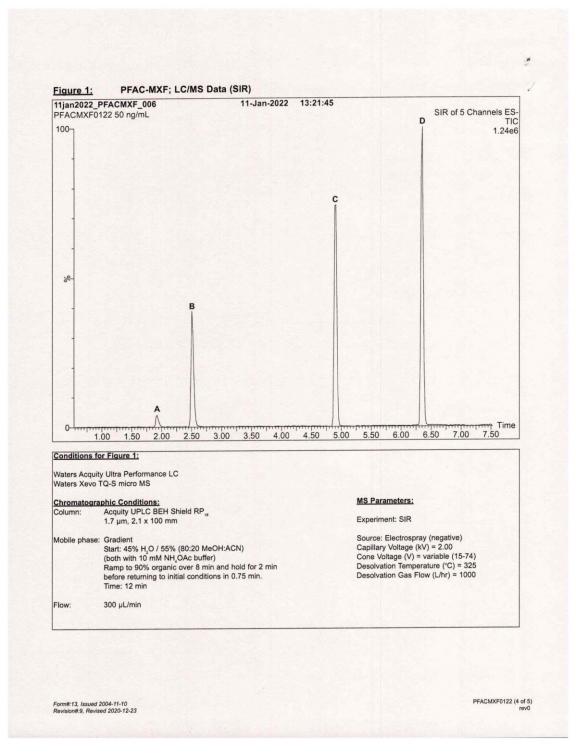
Compound	Acronym	Concentration* (ng/ml)		Peak Assignment in Figure 1
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid	HFPO-DA			Α
Compound	Acronym	Concentration* (ng/mL)		Peak
		as the salt	as the acid	Assignment in Figure 1
Sodium dodecafluoro-3H-4,8-dioxanonanoate	NaDONA	2000	1890	В
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	9CI-PF3ONS	2000	1870	С
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	11CI-PF3OUdS	2000	1890	D

<sup>\*</sup> Concentrations have been rounded to three significant figures.

Certified By:

Date: 01/12/2022

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXF0122 (3 of 5) rev0





# **Analytical Standard Record**

Standard ID: Y22B199

Description: PFAC-MXF-Native Repl.STOCK EPA 1633 PFAS Prepared: 02/17/2022 Standard Type: Expires: Other 01/11/2025 MeOH/H20 Prepared By: Robert Q. Bradley Final Volume (mls): Department: PFAS 1 Lot No.: PFACMXF0122

Vendor: Wellington Laboratories

Comments:

Analyte	CAS Number	Concentration	Units
11CL-PF3OUdS	763051-92-9	1.89	ug/mL
9CL-PF3ONS	756426-58-1	1.87	ug/mL
ADONA	919005-14-4	1.89	ug/mL
HFPO-DA (Gen-X)	13252-13-6	2	ug/mL

Reviewed By Date

Page 1 of 1



# CERTIFICATE OF ANALYSIS DOCUMENTATION

# PFAC-MXF

Native Replacement PFAS Solution/Mixture

PRODUCT CODE:

LOT NUMBER: SOLVENT(S):

DATE PREPARED: (mm/dd/yyyy)

LAST TESTED: (mm/dd/yyyy)

EXPIRY DATE: (mm/dd/yyyy)

RECOMMENDED STORAGE:

PFAC-MXF

PFACMXF0122

Methanol / Water (<1%)

01/10/2022

01/11/2022

01/11/2025

Refrigerate ampoule

#### **DESCRIPTION:**

PFAC-MXF is a solution/mixture of sodium dodecafluoro-3H-4,8-dioxanonanoate (NaDONA), the major and minor components of F-53B (9CI-PF3ONS and 11CI-PF3OUdS), and GenX (HFPO-DA). The components and their concentrations are given in Table A.

The individual native components of this mixture all have chemical purities of >98%.

# DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture

Figure 1: LC/MS Data (SIR)

Figure 2: LC/MS/MS Data (Selected MRM Transitions)

# ADDITIONAL INFORMATION:

- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acid to the methyl ester.

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Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXF0122 (1 of 5) rev0

#### PFAC-MXI; Components and Concentrations (µg/mL; ± 5% in methanol) Table A:

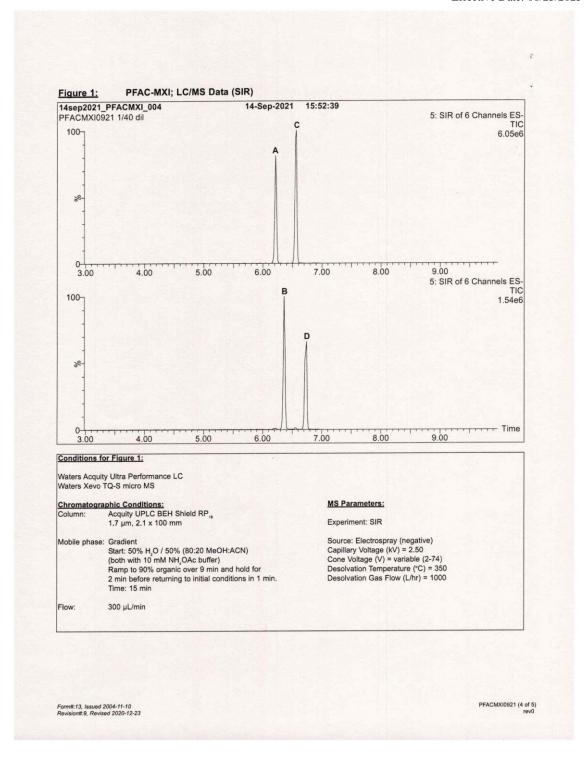
Compound	Acronym	Concentration (μg/mL)	Peak Assignment in Figure 1
N-methylperfluoro-1-octanesulfonamide	N-MeFOSA	1.00	В
N-ethylperfluoro-1-octanesulfonamide	N-EtFOSA	1.00	D
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	N-MeFOSE	10.0	Α
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	N-EtFOSE	10.0	С

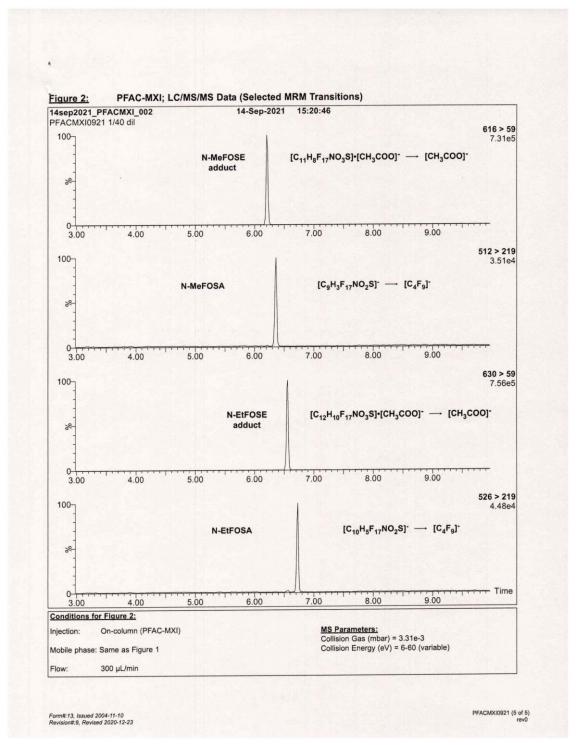
Certified By:

Date: 09/23/2021

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23

PFACMXI0921 (3 of 5) rev0





Revision 1.2 Effective Date: 06/23/2023



# **Analytical Standard Record**

Standard ID:	Y22B20

Description: PFAC-MXI-EPA 1633 Stock Prepared: 02/17/2022 Standard Type: Other Expires: 02/17/2023 Methanol Prepared By: Robert Q. Bradley Final Volume (mls): Department: PFAS 1 Lot No.: PFACMXI0921

Vendor: Wellington Laboratories

Comments:

Analyte	CAS Number	Concentration	Units
N-EtFOSA	4151-50-2	1	ug/mL
N-EtFOSE	1691-99-2	10	ug/mL
N-MeFOSA	31506-32-8	1	ug/mL
N-MeFOSE	24448-09-7	10	ug/mL

Reviewed By Date

Page 1 of 1



# CERTIFICATE OF ANALYSIS DOCUMENTATION

## PFAC-MXI

Native Perfluorooctanesulfonamide and Perfluorooctanesulfonamidoethanol Solution/Mixture

 PRODUCT CODE:
 PFAC-MXI

 LOT NUMBER:
 PFACMXI0921

 SOLVENT(S):
 Methanol

 DATE PREPARED: (mm/dd/yyyr)
 09/08/2021

 LAST TESTED: (mm/dd/yyyr)
 09/14/2021

 EXPIRY DATE: (mm/dd/yyyr)
 09/14/2026

RECOMMENDED STORAGE: Store ampoule in a cool, dark place

# DESCRIPTION:

PFAC-MXI is a solution/mixture of two native perfluorooctanesulfonamides (FOSAs) and two native perfluorooctanesulfonamidoethanols (FOSEs). The components and their concentrations are given in Table A.

The individual components have a chemical purity of >98%.

## **DOCUMENTATION/ DATA ATTACHED:**

Table A: Components and Concentrations of the Solution/Mixture

Figure 1: LC/MS Data (SIR)

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

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Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXI0921 (1 of 5) rev0

Title: PFAS\_LCMSMS1633 Revision 1.2

Effective Date: 06/23/2023

#### 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 compounds it contains.

#### HANDLING:

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## SYNTHESIS / CHARACTERIZATION:

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#### 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 55% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This 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<sub>x</sub>(y), of a value y and the uncertainty of the independent parameters

$$x_i, x_2,...x_n$$
 on which it depends is: 
$$u_{\epsilon}(y(x_1, x_2,...x_n)) = \sqrt{\sum_{i=1}^n u(y,x_i)^2}$$

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

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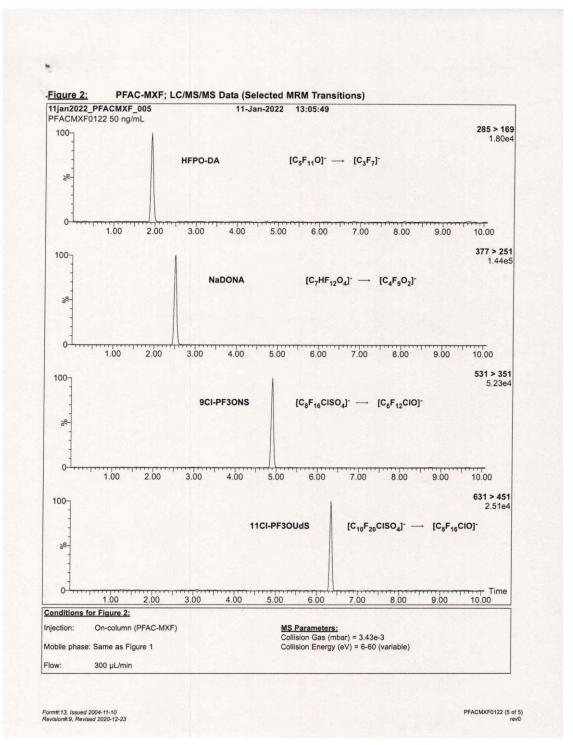




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PFACMXI0921 (2 of 5)

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23



Title: PFAS\_LCMSMS1633 Revision 1.2

Effective Date: 06/23/2023

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Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXF0122 (2 of 5) rev0

## -Table A: PFAC-MXF; Components and Concentrations (ng/mL; ± 5% in Methanol/Water (<1%))

Compound	Acronym		Concentration* (ng/ml)		
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid	HFPO-DA	20			000 A
Compound		Concentration* (ng/mL)		Peak	
Compound	Acronym	as the salt	as the acid	Assignment in Figure 1	
Sodium dodecafluoro-3H-4,8-dioxanonanoate	NaDONA	2000	1890	В	
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	9CI-PF3ONS	2000	1870	С	
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	11CI-PF3OUdS	2000	1890	D	

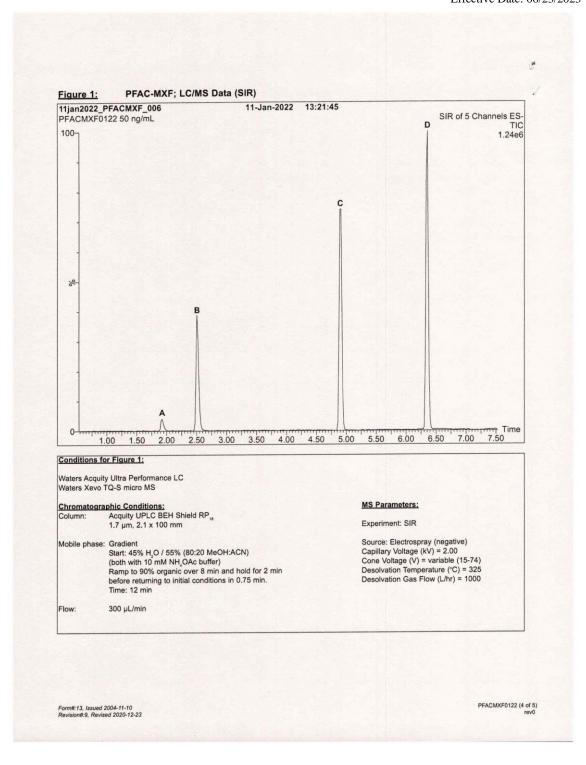
<sup>\*</sup> Concentrations have been rounded to three significant figures.

Certified By:

Date: 01/12/2022

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXF0122 (3 of 5) rev0

Revision 1.2 Effective Date: 06/23/2023





# **Analytical Standard Record**

Standard ID: Y22	В	1	9	١
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PFAC-MXF-Native Repl.STOCK EPA 1633 PFAS Prepared: Description: 02/17/2022 Standard Type: Expires: Other 01/11/2025 MeOH/H20 Prepared By: Robert Q. Bradley Final Volume (mls): Department: PFAS Lot No.: PFACMXF0122 Vendor: Wellington Laboratories

Comments:

Analyte	CAS Number	Concentration	Units
11CL-PF3OUdS	763051-92-9	1.89	ug/mL
9CL-PF3ONS	756426-58-1	1.87	ug/mL
ADONA	919005-14-4	1.89	ug/mL
HFPO-DA (Gen-X)	13252-13-6	2	ug/mL

Reviewed By Date

Page 1 of 1



# CERTIFICATE OF ANALYSIS DOCUMENTATION

## PFAC-MXF

Native Replacement PFAS Solution/Mixture

PRODUCT CODE:

LOT NUMBER: SOLVENT(S):

DATE PREPARED: (mm/dd/yyyy)

LAST TESTED: (mm/dd/yyyy)

EXPIRY DATE: (mm/dd/yyyy)

RECOMMENDED STORAGE:

PFAC-MXF

PFACMXF0122

Methanol / Water (<1%)

01/10/2022

01/11/2022

01/11/2025

Refrigerate ampoule

#### **DESCRIPTION:**

PFAC-MXF is a solution/mixture of sodium dodecafluoro-3H-4,8-dioxanonanoate (NaDONA), the major and minor components of F-53B (9CI-PF3ONS and 11CI-PF3OUdS), and GenX (HFPO-DA). The components and their concentrations are given in Table A.

The individual native components of this mixture all have chemical purities of >98%.

## DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture

Figure 1: LC/MS Data (SIR)

Figure 2: LC/MS/MS Data (Selected MRM Transitions)

## ADDITIONAL INFORMATION:

- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acid to the methyl ester.

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Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXF0122 (1 of 5) rev0

# Table A: PFAC-MXJ; Components and Concentrations (µg/mL; ± 5% in methanol)

Compound	Acronym	Concentration (μg/mL)
3-Perfluoropropyl propanoic acid	FPrPA	4.00
3-Perfluoropentyl propanoic acid	FPePA	20.0
3-Perfluoroheptyl propanoic acid	FHpPA	20.0

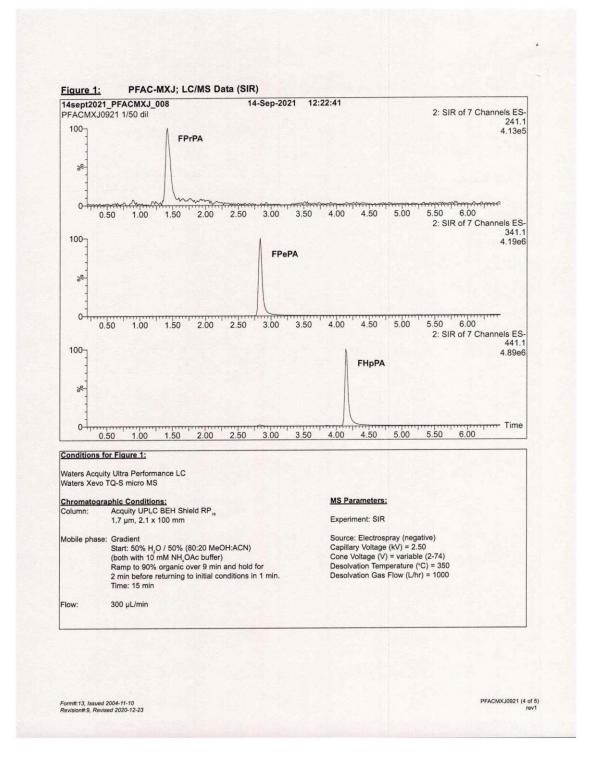
Certified By:

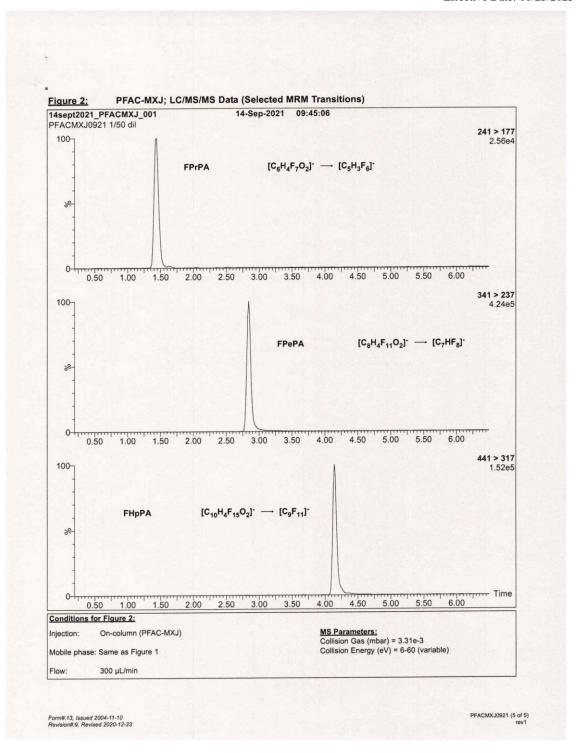
B.G. Chittim, General Manager

Date: 10/02/2021

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXJ0921 (3 of 5) rev1

Revision 1.2





Revision 1.2

Effective Date: 06/23/2023



## **Analytical Standard Record**

Standard ID:

Y22B205

Description: PFAC-MXJ-EPA 1633 Stock mix Prepared: 02/17/2022 Standard Type: Other Expires: 09/14/2026 Solvent Methanol Prepared By: Robert Q. Bradley Final Volume (mls): Department PFAS Vials: Lot No.: PFACMXJ0921 Wellington Laboratories

Comments:

Analyte	CAS Number	Concentration	Units
3-Perfluoroheptyl propanoic acid (FHpPA	812-70-4	20	ug/mL
3-Perfluoropentyl propanoic acid (FPePA)	914637-49-3	20	ug/mL
3-Perfluoropropyl propanoic acid (FPrPA)	356-02-2	4	ug/mL

Reviewed By

Page 1 of 1



# CERTIFICATE OF ANALYSIS DOCUMENTATION

#### PFAC-MXJ

Native X:3 Fluorotelomer Carboxylic Acid Solution/Mixture

 PRODUCT CODE:
 PFAC-MXJ

 LOT NUMBER:
 PFACMXJ0921

 SOLVENT(S):
 Methanol

 DATE PREPARED: (mm\dd\yyy)
 09/08/2021

 LAST TESTED: (mm\dd\yyy)
 09/14/2021

 EXPIRY DATE: (mm\dd\yyy)
 09/14/2026

RECOMMENDED STORAGE: Store ampoule in a cool, dark place

#### **DESCRIPTION:**

PFAC-MXJ is a solution/mixture of three native X:3 fluorotelomer carboxylic acids. The components and their concentrations are given in Table A.

The individual components have a chemical purity of >98%.

## DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture

Figure 1: LC/MS Data (SIR)

Figure 2: LC/MS/MS Data (Selected MRM Transitions)

## ADDITIONAL INFORMATION:

See page 2 for further details.

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Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXJ0921 (1 of 5) rev1

#### 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 compounds 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/IUV/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, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This 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

$$\mathbf{x_{j}},\,\mathbf{x_{2}},...\mathbf{x_{n}}\text{ on which it depends is:} \qquad \qquad u_{c}(y(x_{1},x_{2},...x_{n})) = \sqrt{\sum_{i=1}^{n}u(y,x_{i})^{2}}$$

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).



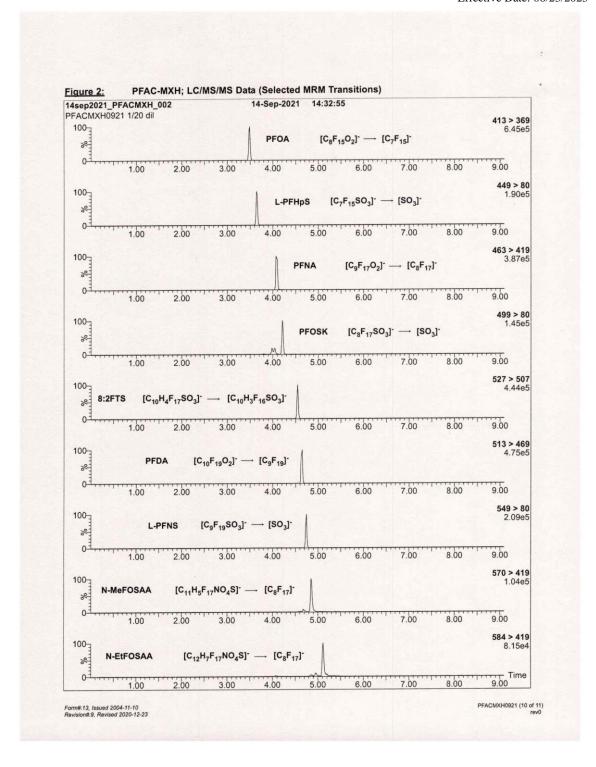


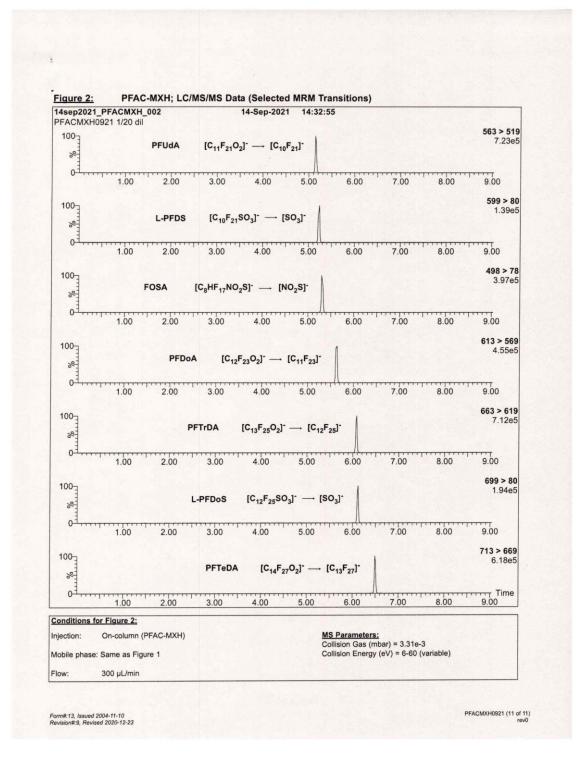
\*\*For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at <a href="www.well-labs.com">www.well-labs.com</a> or contact us directly at <a href="mailto:info@well-labs.com">info@well-labs.com</a> or <a href="mailto:info@well-labs.com">info@well-labs.com</a> or co

PFACMXJ0921 (2 of 5)

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23

Revision 1.2 Effective Date: 06/23/2023





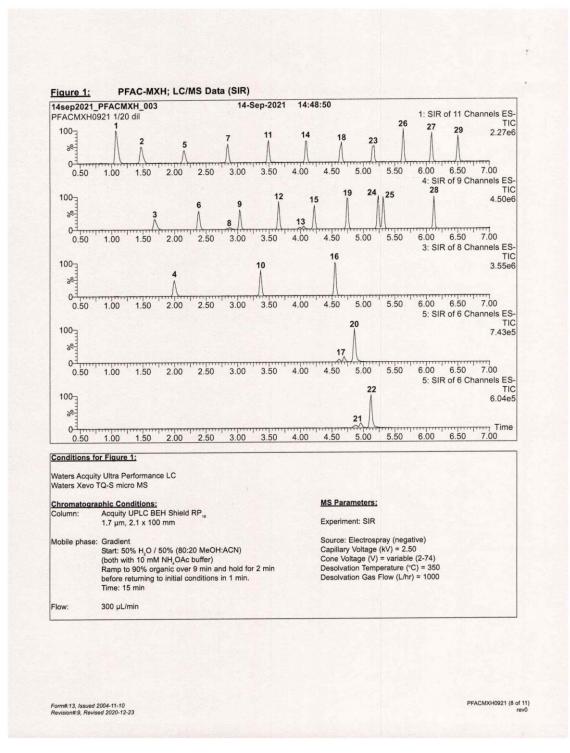
#### PFOSK; Isomeric Components and Percent Composition (by 19F-NMR)\* Table E:

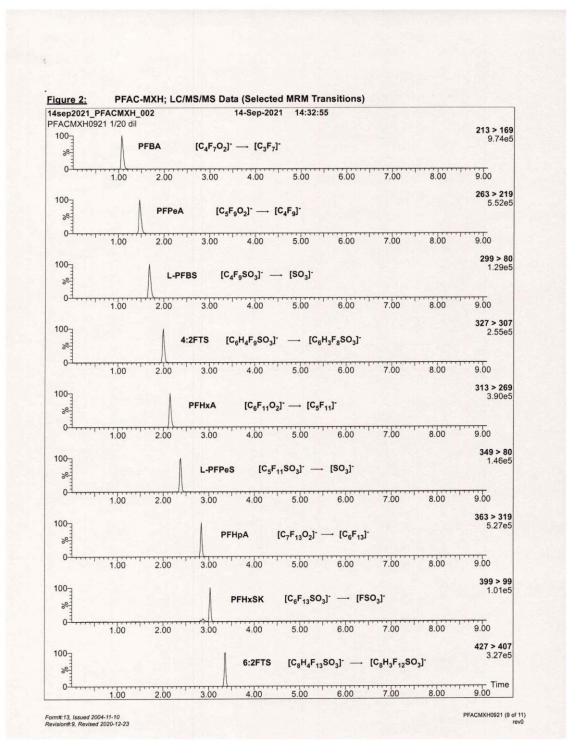
somer	Compound	Structure	Comp	cent osition -NMR
1	Potassium perfluoro-1-octanesulfonate	CF <sub>3</sub> CF <sub>2</sub> SO <sub>3</sub> ·K*	78.8	78.8
2	Potassium 1-trifluoromethylperfluoroheptanesulfonate**	CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CFSO <sub>3</sub> ·K+ CF <sub>3</sub>	1.2	
3	Potassium 2-trifluoromethylperfluoroheptanesulfonate	CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CFCF <sub>2</sub> SO <sub>3</sub> ·K* CF <sub>3</sub>	0.6	
4	Potassium 3-trifluoromethylperfluoroheptanesulfonate	CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K <sup>+</sup> CF <sub>3</sub>	1.9	
5	Potassium 4-trifluoromethylperfluoroheptanesulfonate	CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> CFCF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K <sup>+</sup> CF <sub>3</sub>	2.2	
6	Potassium 5-trifluoromethylperfluoroheptanesulfonate	CF <sub>3</sub> CF <sub>2</sub> CFCF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> -K* CF <sub>3</sub>	4.5	21.
7	Potassium 6-trifluoromethylperfluoroheptanesulfonate	CF <sub>3</sub> CFCF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K* CF <sub>3</sub>	10.0	21.
8	Potassium 5,5-di(trifluoromethyl)perfluorohexanesulfonate	CF <sub>3</sub> CF <sub>3</sub> CCF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> -K+ CF <sub>3</sub>	0.2	
9	Potassium 4,4-di(trifluoromethyl)perfluorohexanesulfonate	CF <sub>3</sub> CF <sub>3</sub> CF <sub>2</sub> CCF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K <sup>+</sup> CF <sub>3</sub>	0.03	
10	Potassium 4,5-di(trifluoromethyl)perfluorohexanesulfonate	CF <sub>3</sub> CF <sub>3</sub> CFCFCF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K <sup>+</sup> CF <sub>3</sub>	0.4	
11	Potassium 3,5-di(trifluoromethyl)perfluorohexanesulfonate	CF <sub>3</sub> CF <sub>3</sub> CFCF <sub>2</sub> CFCF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K+ CF <sub>3</sub>	0.07	

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23

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<sup>\*</sup> Percent of total perfluorooctanesulfonate isomers only.
\*\* Systematic Name: Potassium perfluorooctane-2-sulfonate.





# Table B: br-NMeFOSAA; Isomeric Components and Percent Composition (by ¹9F-NMR)\*

somer	Compound	Structure	Comp	cent osition -NMR
1	N-methylperfluoro-1-octanesulfonamidoacetic acid	CF <sub>3</sub> (CF <sub>2</sub> ) <sub>7</sub> SO <sub>2</sub> NCH <sub>2</sub> CO <sub>2</sub> H CH <sub>3</sub>	76.0	76.0
2	N-methylperfluoro-3-methylheptanesulfonamidoacetic acid	CF <sub>3</sub> (CF <sub>2</sub> ) <sub>3</sub> CF(CF <sub>2</sub> ) <sub>2</sub> SO <sub>2</sub> NCH <sub>2</sub> CO <sub>2</sub> H CF <sub>3</sub> CH <sub>3</sub>	0.7	
3	N-methylperfluoro-4-methylheptanesulfonamidoacetic acid	CF <sub>3</sub> (CF <sub>2</sub> ) <sub>2</sub> CF(CF <sub>2</sub> ) <sub>3</sub> SO <sub>2</sub> NCH <sub>2</sub> CO <sub>2</sub> H CF <sub>3</sub> CH <sub>3</sub>	2.0	
4	N-methylperfluoro-5-methylheptanesulfonamidoacetic acid	CF <sub>3</sub> CF <sub>2</sub> CF(CF <sub>2</sub> ) <sub>4</sub> SO <sub>2</sub> NCH <sub>2</sub> CO <sub>2</sub> H CF <sub>3</sub> CH <sub>3</sub>	6.0	24.0
5	N-methylperfluoro-6-methylheptanesulfonamidoacetic acid	CF <sub>3</sub> CF(CF <sub>2</sub> ) <sub>6</sub> SO <sub>2</sub> NCH <sub>2</sub> CO <sub>2</sub> H CF <sub>3</sub> CH <sub>3</sub>	14.0	
6	N-methylperfluoro-5,5-dimethylhexanesulfonamidoacetic acid	CF <sub>3</sub> CF <sub>3</sub> C(CF <sub>2</sub> ) <sub>4</sub> SO <sub>2</sub> NCH <sub>2</sub> CO <sub>2</sub> H CF <sub>3</sub> CH <sub>3</sub>	0.2	
7	Other Unidentified Isomers		1.1	

<sup>\*</sup> Percent of total N-methylperfluorooctanesulfonamidoacetic acid isomers only.

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXH0921 (4 of 11) rev0

# Table C: br-NEtFOSAA; Isomeric Components and Percent Composition (by 19F-NMR)\*

somer	Compound	Structure	Comp	cent osition -NMR
1	N-ethylperfluoro-1-octanesulfonamidoacetic acid	$\mathrm{CF_3(CF_2)_7SO_2NCH_2CO_2H}$ $\mathrm{C_2H_5}$	77.5	77.5
2	N-ethylperfluoro-3-methylheptanesulfonamidoacetic acid	CF <sub>3</sub> (CF <sub>2</sub> ) <sub>3</sub> CF(CF <sub>2</sub> ) <sub>2</sub> SO <sub>2</sub> NCH <sub>2</sub> CO <sub>2</sub> H CF <sub>3</sub> C <sub>2</sub> H <sub>5</sub>	2.3	
3	N-ethylperfluoro-4-methylheptanesulfonamidoacetic acid	$\begin{array}{ccc} {\rm CF_3(CF_2)_2CF(CF_2)_3SO_2NCH_2CO_2H} \\ {\rm CF_3} & {\rm C_2H_5} \end{array}$	2.2	
4	N-ethylperfluoro-5-methylheptanesulfonamidoacetic acid	$\begin{array}{ccc} \mathrm{CF_3CF_2CF(CF_2)_4SO_2NCH_2CO_2H} \\ \mathrm{CF_3} & \mathrm{C_2H_5} \end{array}$	5.4	
5	N-ethylperfluoro-6-methylheptanesulfonamidoacetic acid	$\begin{array}{ccc} \operatorname{CF_3CF(CF_2)_5SO_2NCH_2CO_2H} \\ \operatorname{CF_3} & \operatorname{C_2H_5} \end{array}$	10.4	22.5
6	N-ethylperfluoro-5,5-dimethylhexanesulfonamidoacetic acid	$\begin{array}{c}CF_3\\CF_3C(CF_2)_4SO_2NCH_2CO_2H\\CF_3\\C_2H_5\end{array}$	0.3	22.5
7	N-ethylperfluoro-4,5-dimethylhexanesulfonamidoacetic acid	$\begin{array}{c} CF_3\\ CF_3CFCF(CF_2)_3SO_2NCH_2CO_2H\\ CF_3\\ CF_3\\ C_2H_5 \end{array}$	0.3	
8	N-ethylperfluoro-3,5-dimethylhexanesulfonamidoacetic acid	$\begin{array}{c} CF_3\\ CF_3CFCF_2CF(CF_2)_2SO_2NCH_2CO_2H\\ CF_3 & C_2H_5 \end{array}$	0.3	
9	Other Unidentified Isomers		1.3	T.

<sup>\*</sup> Percent of total N-ethylperfluorooctanesulfonamidoacetic acid isomers only.

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXH0921 (5 of 11) rev0

#### PFHxSK; Isomeric Components and Percent Composition (by 19F-NMR)\* Table D:

somer	Compound	Structure	Comp	cent osition F-NMR
1	Potassium perfluoro-1-hexanesulfonate	CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> 'K <sup>+</sup>	81.1	81.1
2	Potassium 1-trifluoromethylperfluoropentanesulfonate**	CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> CFSO <sub>3</sub> ·K* CF <sub>3</sub>	2.9	
3	Potassium 2-trifluoromethylperfluoropentanesulfonate	CF <sub>3</sub> CF <sub>2</sub> CF <sub>2</sub> CFCF <sub>2</sub> SO <sub>3</sub> ·K*	1.4	
4	Potassium 3-trifluoromethylperfluoropentanesulfonate	CF <sub>3</sub> CF <sub>2</sub> CFCF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K*	5.0	18.9
5	Potassium 4-trifluoromethylperfluoropentanesulfonate	CF <sub>3</sub> CFCF <sub>2</sub> CF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K*	8.9	10.0
6	Potassium 3,3-di(trifluoromethyl)perfluorobutanesulfonate	CF <sub>3</sub> CF <sub>3</sub> CCF <sub>2</sub> CF <sub>2</sub> SO <sub>3</sub> ·K <sup>+</sup> CF <sub>3</sub>	0.2	
7	Other Unidentified Isomers		0.5	

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23

PFACMXH0921 (6 of 11) rev0

Percent of total perfluorohexanesulfonate isomers only. Systematic Name: Potassium perfluorohexane-2-sulfonate.



# CERTIFICATE OF ANALYSIS DOCUMENTATION

### **PFAC-MXH**

Native Per- and Poly-fluoroalkyl Substance Solution/Mixture

PRODUCT CODE:

PFAC-MXH

LOT NUMBER:

PFACMXH0921

SOLVENT(S):

Methanol / Isopropanol (2%) / Water (<1%)

DATE PREPARED: (mm/dd/yyyy)

09/09/2021

LAST TESTED: (mm/dd/yyyy)

09/14/2021

EXPIRY DATE: (mm/dd/yyyy)

09/14/2026

RECOMMENDED STORAGE:

Refrigerate ampoule

#### **DESCRIPTION:**

PFAC-MXH is a solution/mixture of eleven native linear perfluoroalkylcarboxylic acids ( $C_4$ - $C_{14}$ ), eight native perfluoroalkanesulfonates ( $C_4$ ,  $C_5$ ,  $C_7$ ,  $C_9$ ,  $C_{10}$  and  $C_{12}$  linear;  $C_6$  and  $C_8$  linear and branched), three native fluorotelomer sulfonates (4:2, 6:2, and 8:2), two native linear and branched perfluoroctanesulfonamidoacetic acids, and perfluoro-1-octanesulfonamide (FOSA). The components and their concentrations are given in Table  $\frac{1}{2}$ 

The individual components of this mixture all have chemical purities of >98%.

## **DOCUMENTATION/ DATA ATTACHED:**

Table A: Components and Concentrations of the Solution/Mixture

Table B: Isomeric Components and Percent Composition of br-NMeFOSAA

Table C: Isomeric Components and Percent Composition of br-NEtFOSAA

Table D: Isomeric Components and Percent Composition of PFHxSK

Table E: Isomeric Components and Percent Composition of PFOSK

Figure 1: LC/MS Data (SIR)

Figure 2: LC/MS/MS Data (Selected MRM Transitions)

#### ADDITIONAL INFORMATION:

- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

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Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23 PFACMXH0921 (1 of 11) rev0

,010

Title: PFAS\_LCMSMS1633 Revision 1.2

Effective Date: 06/23/2023

PFACMXH0921 (2 of 11)

#### 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 compounds 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/LV/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 45% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This 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

$$\mathbf{x}_{i},\,\mathbf{x}_{2},...\mathbf{x}_{n}\,\text{on which it depends is:} \qquad \qquad u_{e}(y(x_{1},x_{2},...x_{n})) = \sqrt{\sum_{i=1}^{n}u(y,x_{i})^{2}}$$

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:

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23

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 <a href="https://www.well-labs.com">www.well-labs.com</a> or contact us directly at <a href="mailto:info@well-labs.com">info@well-labs.com</a> or contact us directly at <a href="mailto:info@well-labs.co

### PFAC-MXH; Components and Concentrations (μg/mL, ± 5% in methanol / isopropanol (2%) / water (<1%)) Table A:

Compound	Acronym	Concer (µg/	ntration* mL)	Peak Assignment in Figure 1		
Perfluoro-n-butanoic acid	PFBA	4.0	00	1		
Perfluoro-n-pentanoic acid	PFPeA	2.0	00	2		
Perfluoro-n-hexanoic acid	PFHxA	1.00		5		
Perfluoro-n-heptanoic acid	PFHpA	1.00		7		
Perfluoro-n-octanoic acid	PFOA	1.0	00	11		
Perfluoro-n-nonanoic acid	PFNA	1.0	00	14		
Perfluoro-n-decanoic acid	PFDA	1.0	00	18		
Perfluoro-n-undecanoic acid	PFUdA	1.0	00	23		
Perfluoro-n-dodecanoic acid	PFDoA	1.0	00	26		
Perfluoro-n-tridecanoic acid	PFTrDA	1.0	00	27		
Perfluoro-n-tetradecanoic acid	PFTeDA	1.0	00	29		
Perfluoro-1-octanesulfonamide	FOSA	1.0	00	25		
	N-MeFOSAA: linear isomer	0.760 s 0.240		0.760		20
N-methylperfluorooctanesulfonamidoacetic acid *	N-MeFOSAA: ∑ branched isomers			17		
	N-EtFOSAA: linear isomer	0.775		22		
N-ethylperfluorooctanesulfonamidoacetic acid *	N-EtFOSAA: ∑ branched isomers	0.225		21		
	Acronym	Concentration* (µg/mL)		Peak Assignmen		
Compound	Actonym	as the salt	as the acid	in Figure 1		
Potassium perfluoro-1-butanesulfonate	L-PFBS	1.00	0.887	3		
Sodium perfluoro-1-pentanesulfonate	L-PFPeS	1.00	0.941	6		
	PFHxSK: linear isomer	0.811	0.741	9		
Potassium perfluorohexanesulfonate <sup>c</sup>	PFHxSK: ∑ branched isomers	0.189	0.173	8		
Sodium perfluoro-1-heptanesulfonate	L-PFHpS	1.00	0.953	12		
	PFOSK: linear isomer	0.788	0.732	15		
Potassium perfluorooctanesulfonate <sup>6</sup>	PFOSK: ∑ branched isomers	0.211	0.196	13		
Sodium perfluoro-1-nonanesulfonate	L-PFNS	1.00	0.962	19		
Sodium perfluoro-1-decanesulfonate	L-PFDS	1.00	0.965	24		
Sodium perfluoro-1-dodecanesulfonate	L-PFDoS	1.00	0.970	28		
Sodium 1H,1H,2H,2H-perfluorohexanesulfonate	4:2FTS	4.00	3.75	4		
Sodium 1H,1H,2H,2H-perfluorooctanesulfonate	6:2FTS	4.00	3.80	10		
Sodium 1H,1H,2H,2H-perfluorodecanesulfonate	8:2FTS	4.00	3.84	16		

See Table B for percent composition of linear and branched N-MeFOSAA isomers.

See Table C for percent composition of linear and branched N-EtFOSAA isomers.

See Table D for percent composition of linear and branched PFHxSK isomers.

See Table E for percent composition of linear and branched PFOSK isomers.

Certified By:

Date: 09/23/2021

Form#:13, Issued 2004-11-10 Revision#:9, Revised 2020-12-23

PFACMXH0921 (3 of 11)

<sup>\*</sup> Concentrations have been rounded to three significant figures.



# Analytical Standard Record

Standard ID: Y22B201

Description: PFAC-MXH STOCK PFAS EPA 1633 Prepared: 02/17/2022 Standard Type: Other Expires: 09/14/2026 MeOH/IPA/H2O Prepared By: Robert Q. Bradley Final Volume (mls): Department: PFAS Lot No.: PFACMXH0921

Vendor: Wellington Laboratories

Comments:

Analyte	CAS Number	Concentration	Units	
1H,1H,2H,2H-Perfluorodecanesulfonic acid	39108-34-4	3.84	ug/mL	
1H,1H,2H,2H-Perfluorohexanesulfonic acid	757124-72-4	3.75	ug/mL	
1H,1H,2H,2H-Perfluorooctanesulfonic acid	27619-97-2	3.8	ug/mL	
N-EtFOSAA	2991-50-6	1	ug/mL	
N-MeFOSAA	2355-31-9	1	ug/mL	
Perfluoro-1-decanesulfonic acid (PFDS)	335-77-3	0.965	ug/mL	
Perfluoro-1-heptanesulfonic acid (PFHpS)	375-92-8	0.953	ug/mL	
Perfluoro-1-nonanesulfonic acid (PFNS)	68259-12-1	0.962	ug/mL	
Perfluoro-1-octanesulfonamide (FOSA)	754-91-6	1	ug/mL	
Perfluoro-1-pentanesulfonate (PFPeS)	2706-91-4	0.941	ug/mL	
Perfluorobutanesulfonic acid (PFBS)	375-73-5	0.887	ug/mL	
Perfluorodecanesulfonic acid(PFDS)	335-77-3	0.965	ug/mL	
Perfluorodecanoic acid (PFDA)	335-76-2	1	ug/mL	
Perfluorododecanoic acid (PFDoA)	307-55-1	1	ug/mL	
Perfluoroheptanoic acid (PFHpA)	375-85-9	1	ug/mL	
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	0.914	ug/mL	
Perfluorohexanoic acid (PFHxA)	307-24-4	1	ug/mL	
Perfluoro-n-butanoic acid (PFBA)	375-22-4	4	ug/mL	
Perfluorononanoic acid (PFNA)	375-95-1	1	ug/mL	
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	0.928	ug/mL	
Perfluorooctanoic acid (PFOA)	335-67-1	1	ug/mL	
Perfluoropentanoic acid (PFPeA)	2706-90-3	1	ug/mL	
Perfluorotetradecanoic acid (PFTA)	376-06-7	1	ug/mL	
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	1	ug/mL	
Perfluoroundecanoic acid (PFUnA)	2058-94-8	1	ug/mL	

Reviewed By Date

Page 1 of 1

Revision 1.2

Effective Date: 06/23/2023

# Attachment 4 – Calibration Concentrations, nominal

Calibration So	olutions (ng/m I							
CS1 (LOQ)	CS2 Perfluor	oalkyl carboxyli	c	CS3	CS4 (CV1)	CS5	CS6	CS7 <sup>2</sup>
acids								
PFBA		0.8	2	5	10	20	50	250
PFPeA		0.4	1	2.5	5	10	25	125
PFHxA		0.2	0.5	1.25	2.5	5	12.5	62.5
PFHpA		0.2	0.5	1.25	2.5	5	12.5	62.5
PFOA		0.2	0.5	1.25	2.5	5	12.5	62.5
PFNA		0.2	0.5	1.25	2.5	5	12.5	62.5
PFDA		0.2	0.5	1.25	2.5	5	12.5	62.5
PFUnA		0.2	0.5	1.25	2.5	5	12.5	62.5
PFDoA		0.2	0.5	1.25	2.5	5	12.5	62.5
PFTrDA		0.2	0.5	1.25	2.5	5	12.5	62.5
PFTeDA		0.2	0.5	1.25	2.5	5	12.5	62.5
Perfluoroalky	l sulfonic acids							
PFBS		0.2	0.5	1.25	2.5	5	12.5	62.5
PFPeS		0.2	0.5	1.25	2.5	5	12.5	62.5
PFHxS		0.2	0.5	1.25	2.5	5	12.5	62.5
PFHpS		0.2	0.5	1.25	2.5	5	12.5	62.5
PFOS		0.2	0.5	1.25	2.5	5	12.5	62.5
PFNS		0.2	0.5	1.25	2.5	5	12.5	62.5
PFDS		0.2	0.5	1.25	2.5	5	12.5	62.5
PFDoS		0.2	0.5	1.25	2.5	5	12.5	62.5
Fluorotelome	· sulfonic acids							
4:2FTS		0.8	2	5	10	20	50	NA
6:2FTS		0.8	2	5	10	20	50	NA
8:2FTS		0.8	2	5	10	20	50	NA
Perfluoroocta	ne sulfonamide	s						
PFOSA		0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSA		0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSA		0.2	0.5	1.25	2.5	5	12.5	62.5
Perfluoroocta	ne sulfonamido	acetic acids						
NMeFOSAA		0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSAA		0.2	0.5	1.25	2.5	5	12.5	62.5
Perfluoroocta	ne sulfonamide	ethanols						
NMeFOSE		2	5	12.5	25	50	125	625
NEtFOSE		2	5	12.5	25	50	125	625
Per- and polyl	luoroether car	boxylic acids						
HFPO-DA		0.8	2	5	10	20	50	250
ADONA		0.8	2	5	10	20	50	250
PFMPA		0.4	1	2.5	5	10	25	125
PFMBA		0.4	1	2.5	5	10	25	125
NFDHA		0.4	1	2.5	5	10	25	125
Ether sulfonic	acids							
9Cl-PF3ONS		0.8	2	5	10	20	50	250
11Cl-PF3OUd	S	0.8	2	5	10	20	50	250
PFEESA		0.4	1	2.5	5	10	25	125

#### Calibration Solutions (ng/mL) Compound

Calibration Solutions (ng	<del></del>		GGA	COL (CITE)	00.	Lone	- CC=2
	rotelomer carboxyli	ic	CS3	CS4 (CV <sup>1</sup> )	CS5	CS6	CS7 <sup>2</sup>
acids							
3:3FTCA	1.0	2.5	6.26	12.5	25	62.4	312
5:3FTCA	5.0	12.5	31.3	62.5	125	312	1560
7:3FTCA	5.0	12.5	31.3	62.5	125	312	1560
Extracted Internal Stands	ard (EIS) Analytes						
<sup>13</sup> C <sub>4</sub> -PFBA	10	10	10	10	10	10	10
<sup>13</sup> C <sub>5</sub> -PFPeA	5	5	5	5	5	5	5
<sup>13</sup> C <sub>5</sub> -PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>4</sub> -PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>8</sub> -PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>9</sub> -PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
<sup>13</sup> C <sub>6</sub> -PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
<sup>13</sup> C <sub>7</sub> -PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
<sup>13</sup> C <sub>2</sub> -PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
<sup>13</sup> C <sub>2</sub> -PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
<sup>13</sup> C <sub>3</sub> -PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>3</sub> -PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>8</sub> -PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>2</sub> -4:2FTS	5	5	5	5	5	5	5
<sup>13</sup> C <sub>2</sub> -6:2FTS	5	5	5	5	5	5	5
<sup>13</sup> C <sub>2</sub> -8:2FTS	5	5	5	5	5	5	5
¹³C₀-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D <sub>3</sub> -NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D <sub>3</sub> -NMeFOSAA	5	5	5	5	5	5	5
D5-NEtFOSAA	5	5	5	5	5	5	5
D7-NMeFOSE	25	25	25	25	25	25	25
D <sub>9</sub> -NEtFOSE	25	25	25	25	25	25	25
¹³C₃-HFPO-DA	10	10	10	10	10	10	10
Non-extracted Internal St	andard (NIS) Anal	ytes		•		•	•
<sup>13</sup> C <sub>3</sub> -PFBA	5	5	5	5	5	5	5
<sup>13</sup> C <sub>2</sub> -PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>4</sub> -PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>5</sub> -PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
<sup>13</sup> C <sub>2</sub> -PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
<sup>18</sup> O <sub>2</sub> -PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
<sup>13</sup> C <sub>4</sub> -PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5

<sup>&</sup>lt;sup>1</sup>This calibration point is used as the calibration verification (CV)

<sup>&</sup>lt;sup>2</sup> A minimum of six contiguous calibrations standards are required for linear models and a minimum of seven calibration standards are required for second-order models.

Injection

 $3.00~\mu L$ 

Injection with needle wash

Effective Date: 06/23/2023

## **Attachment 5 - HPLC Method Parameters**

# **HPLC Acquisition Method Report**

70 10e-6/bar

90 10e-6/bar

Time set

10.00 min

Time set

1.50 min

Compressibility Value Set

Compressibility Value Set



Stroke A

Automatic Stroke Calculation A

Compress A

Compressibility Mode A Compressibility A

Compress B

Compressibility Mode B Compressibility B

Stop Time

Stoptime Mode Stoptime Post Time

> Posttime Mode Posttime

> > ammonium acetate

Methanol

**Solvent Composition** Channel Name 1

Name 2 Selected Used Percent 90.00 % 10.00 % Ch. 1 Yes

Tim	e	ta	h	lρ

	Time	Α	В	Flow
1	3.50 min	50.00 %	50.00 %	0.400 mL/min
2	8.00 min	10.00 %	90.00 %	0.400 mL/min
3	8.50 min	90.00 %	10.00 %	0.400 mL/min

Name: Column Comp. Module: G1316C

Left Temperature Control

Temperature Control Mode Temperature Set Temperature 50.0 °C **Enable Analysis Left Temperature** 

Enable Analysis Left Temperature On Yes 0.8 °C **Enable Analysis Left Temperature Value** 

**Right Temperature Control** 

**Right temperature Control Mode** Temperature Set Right temperature 50.0 °C

**Enable Analysis Right Temperature** 

**Enable Analysis Right Temperature On** Yes **Enable Analysis Right Temperature Value** 0.8 °C

Stop Time

Stoptime Mode As pump/injector Post Time

Posttime Mode Off

Timetable

Valve Position Position 1 (Port 1 -> 2)

Ready when front door open Yes

Revision 1.2

Effective Date: 06/23/2023

# **Attachment 6 - Triple Quadrupole Acquisition Method**

# **Acquisition Method Report**



Agilent Technologies

			_			
Αc	an	isiti	on l	Vieth	hor	Info

Method Name Method Path

PFAS1633\_ACQ\_092922.m

Method Description

D:\MassHunter\methods\PFAS1633\_ACQ\_092922.m EPA 1633\_Target PFAS Isotope Dilution\_Acquisition

aaa

HiP Sampler Binary Pump Column Comp.

# MS QQQ Mass Spectrometer

Ion Source AJS ESI Tune File \atunes.TUNE.XML

Stop Mode No Limit/As Pump Stop Time (min) Time Filter Width (min) 0.07

Time Filter LC->Waste Pre Row LC->Waste Post Row

Time Segment	s												
Index		Time 5c (min)	an Type	ion Mo	de	Div Valve	Delta EMW	5tore		le Time (ms)	Triggered?	MRM R	epeats
1		,	namicMRM	ESI+Agiler Strear		To MS	350	Yes		550	Yes		3
Time Segment	1												
Scan Segments	ı												
Cpd Name	ISTD?	Prec lon	MS1 Res	Prod Ion	MS2 Res	Primary	Trigger	Frag (V)	CE(V)	Cell Acc (∀)	Ret Time (min)	Ret Window	Polarity
11-CF PF30UdS	No	631	Unit/Enh (6490)	451	Unit/Enh (6490)	Yes	No	170	33	. 4	7.62	3	Negative
1H,1H,2H, 2H- perfluoro-1	No	527	Unit/Enh (6490)	507	Unit/Enh (6490)	Yes	No	170	28	4	7.14	3	Negative
decanesulf onate (8 2F TS) 1H,1H,2H,	No	527	Unit/Enh	80.9	Unit/Enh	Yes	No	170	40	4	7.14	3	Negative
2H- perfluoro-1			(6490)		(6490)								-
decanesulf onate (8 2F TS) 1H,1H,2H, 2H- perfluoro-1	No	327	U nit/Enh (6490)	307	Unit/Enh (6490)	Yes	No	162	20	4	4.788	3	Negative
hexanesulf onate (4 2F TS) 1H,1H,2H, 2H- perfluoro-1	No	327	Unit/Enh (6490)	80.9	Unit/Enh (6490)	Yes	No	162	36	4	4.788	3	Negative
hexanesulf onate (4 2F TS) 1H,1H,2H, 2H- perfluoro-1	No	427	U nit/Enh (6490)	407	Unit/Enh (6490)	Yes	No	162	24	4	6.168	3	Negative
octanes ulf onate (6 2F TS) 1H,1H,2H, 2H- perfluoro-1	No	427	Unit/Enh (6490)	79.7	Unit/Enh (6490)	Yes	No	162	48	4	6.168	3	Negative
octanesulf onate (6 2F TS)													
3:3FTCA	No	241	U nit/Enh (6490)	177	Unit/Enh (6490)	Yes	No	74	4	4	3.4	3	Negative
3-3FTCA	No	241	Unit/Enh (6490)	117	Unit/Enh (6490)	Yes	No	74	44	4	3.4	3	Negative

Report generation date: 18-Oct-2022 09:01:43 AM

Page 1 of 8

# YORK ANALYTICAL LABORATORIES, Inc.

Title: PFAS\_LCMSMS1633

Revision 1.2

Revision 1.2

Effective Date: 06/23/2023

# **Acquisition Method Report**



# Agilent Technologies

D 1 11													
Cpd Name	ISTD?		MS1 Res		MS2 Res	Pri mary	Trigger	Frag (V)	CE(V)	Cell A∞ (V)	Ret Time (min)	Ret Window	Polarity
5-3FTCA	No	341	U nit/Enh (6490)	237	Unit/Enh (6490)	Yes	Νo	84	12	4	5.73	3	Negative
5-3FTCA	Νo	341	Ù nit/Enh (6490)	217	Unit/Enh (6490)	Yes	Νo	84	24	4	5.73	3	Negative
7-3FTCA	No	441	Unit/Enh (6490)	337	Unit/Enh (6490)	Yes	Νo	76	12	4	6.7	3	Negative
7-3FTCA	No	441	Unit/Enh	317	Unit/Enh	Yes	No	76	24	4	6.7	3	Negative
9- CI-	No	531	(6490) U nit/Enh	351	(6490) Unit/Enh	Yes	No	175	29	4	6.89	3	Negative
PF3ONS ADONA	No	377	(6490) U nit/Enh	251	(6490) Unit/Enh	Yes	No	103	9	4	5.62	3	Negative
ADONA	No	377	(6490) Unit/Enh	85	(6490) Unit/Enh	Yes	No	103	37	4	5.62	3	Negative
d3-	No	515	(6490) U nit/Enh	219	(6490) Unit/Enh	Yes	No	134	20	4	7.17	3	Negative
NMeFOSA			(6490)		(6490)					4			
d3-N- MeFOSAA	No	572.99	Unit/Enh (6490)	418.8	Unit/Enh (6490)	Yes	No 	130	20		7.17	3	Negative
d5- NE1FOSA	No	531	U nit/Enh (6490)	219	Unit/Enh (6490)	Yes	No	150	20	4	8.52	3	Negative
d5- NetFosa	No	531	Unit/Enh (6490)	169	Unit/Enh (6490)	Yes	No	150	20	4	8.52	3	Negative
d5-N- EtF OSAA	No	589.02	Unit/Enh (6490)	530.9	Unit/Enh (6490)	Yes	No	130	20	4	7.36	3	Negative
d5-N- EtF OSAA	Νo	589.02	U nit/Enh (6490)	418.8		Yes	Νo	130	20	4	7.36	3	Negative
d7-	No	623	Unit/Enh	310	Ùnit/Énh	Yes	No	150	15	4	8.28	3	Negative
NMeFOSE d7-	No	623	(6490) U nit/Enh	59	(6490) Unit/Enh	Yes	No	88	15	4	8.28	3	Negative
NMeFOSE d9-	No	639	(6490) U nit/Enh	59	(6490) Unit/Enh	Yes	No	150	15	4	8.6	3	Negative
NETFOSE HEPO-DA	No	285	(6490) Hnit/Enh	169.1	(6490) Unit/Enh	Yes	Nο	100	20	4	4.95	3	Negative
M2-4-2FTS	No	329	(6490) U nit/Enh	309	(6490) Unit/Enh	Yes	No	156	20	4	4.787	3	Negative
			(6490)		(6490)								
M2-4-2FTS	No	329	U nit/Enh (6490)	81	Unit/Enh (6490)	Yes	No	156	28	4	4.787	3	Negative
M2-6-2FTS	No	429	U nit/Enh (6490)	409	Unit/Enh (6490)	Yes	No	162	24	4	6.01	3	Negative
M2-6-2FTS	No	429	U nit/Enh (6490)	81	Unit/Enh (6490)	Yes	No	162	40	4	6.01	3	Negative
M2-8-2FTS	No	529	Ù nit/Enh (6490)	509	Unit/Enh (6490)	Yes	No	165	28	4	6.98	3	Negative
M2-8-2FTS	Νo	529	Unit/Enh (6490)	81	Unit/Enh (6490)	Yes	Νo	165	40	4	6.98	3	Negative
M2PFTeD	No	7 15	Unit/Enh	670	Unit/Enh	Yes	No	62	12	4	8.25	3	Negative
M3-HFPO-	No	287	(6490) U nit/Enh	169	(6490) Unit/Enh	Yes	No	90	5	4	4.99	3	Negative
DA M3PFBA	Yes	216	(6490) U nit/Enh	172	(6490) Unit/Enh	Yes	No	90	5	4	1.2	2	Negative
M3PFBS	No	302	(6490) U nit/Enh	98.9	(6490) Unit/Enh	Yes	No	114	32	4	3.94	3	Negative
M3PFBS	No	302	(6490) U nit/Enh	79.9	(6490) Unit/Enh	Yes	No	114	40	4	3.94	3	Negative
M3PFHxS	No	402	(6490) U nit/Enh	98.9	(6490) Unit/Enh	Yes	No	165	40	4	5.55	3	Negative
			(6490)		(6490)		***					-	
M3PFHxS	No	402	U nit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	165	48	4	5.55	3	Negative
M4PF HpA	No	367	U nit/Enh (6490)	322	Unit/Enh (6490)	Yes	No	124	8	4	5.601	3	Negative
M5PFHxA	Νo	318	Unit/Enh (6490)	273	Unit/Enh (6490)	Yes	Νo	70	4	4	5.47	3	Negative
M5PFHxA	No	318	Ù nit/Enh (6490)	120	Unit/Enh (6490)	Yes	No	70	4	4	5.47	3	Negative
M6PFDA	Νo	519	Unit/Enh (6490)	473.9	Unit/Enh (6490)	Yes	No	59	8	4	6.99	3	Negative
M7PF UdA	No	570	Ùnit/Énh	525	Ùnit/Énh	Yes	No	64	8	4	7.38	3	Negative
MPFDA	Yes	514.98	(6490) Unit/Enh	469.8	(6490) Unit/Enh	Yes	No	94	5	4	6.972	2	Negative
MPFHxA	Yes	314.99	(6490) U nit/Enh	269.8	(6490) Unit/Enh	Yes	No	86	4	4	4.705	2	Negative
MPFHxA	Yes	314.99	(6490) U nit/Enh	120	(6490)	Yes	No	86	4	4	4.705	2	Negative
MPFHxS	Yes	403	(6490) U nit/Enh	103	(6490) Unit/Enh	Yes	No	110	37	4	5.63	2	Negative
			(6490)		(6490)		***					_	
MPFHxS	Yes	403	Unit/Enh (6490)	84	Unit/Enh (6490)	Yes	No 	110	40	4	5.63	2	Negative
MPFNA	Yes	468	U nit/Enh (6490)	423	Unit/Enh (6490)	Yes	No	66	4	4	6.541	2	Negative
MPFOA	Yes	417	Ú nit/Enh (6490)	372	Unit/Enh (6490)	Yes	Νo	84	4	4	6.03	2	Negative
MPFOS	Yes	502.96	U nit/Enh (6490)	99	Unit/Enh (6490)	Yes	No	148	48	4	6.57	3	Negative

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										•			
Cpd Name	ISTD?	Prec lon	MS1 Res	Prod lon	MS2 Res	Primary	Trigger	Frag (V)	CE(V)	Cell Acc (V)	Ret Time (min)	Ret Window	Polarit
MPFOS	Yes	502.96	Unit/Enh	80	Unit/Enh	Yes	No	148	54	4	6.57	3	Negativ
NETFOSA	No	526		219	(6490) Unit/Enh	Yes	No	120	20	4	8.528	3	Negativ
NEFOSA	No	526		169	(6490) Unit/Enh	Yes	No	120	20	4	8.528	3	Negativ
N-	No	584		525.9		Yes	No	130	20	4	7.521	3	Negativ
EtFOSAA N-	No	584		418.8		Yes	No	130	20	4	7.521	3	Negativ
EtFOSAA NE#FOSE	No	630	(6490) U nit/Enh	59		Yes	No	120	20	4	8.301	3	Negativ
NFDHA	No	295		201.1	(6490) Unit/Enh	Yes	No	92	2	4	4.641	3	Negativ
NFDHA	No	295		84.9		Yes	No	92	34	4	4.641	3	Negativ
NMeFOSA	No	512		219		Yes	No	120	20	4	8.298	3	Negati
NMeFOSA	No	512		169		Yes	No	120	20	4	8.298	3	Negati
N-	No	570	(6490) U nit/Enh	511.9	(6490) Unit/Enh	Yes	No	150	20	4	7.335	3	Negati
MeFOSAA N-	No	570	(6490) U nit/Enh	418.9	(6490) Unit/Enh	Yes	No	150	20	4	7.335	3	Negati
MeFOSAA NMeFOSE	No	616	(6490) U nit/Enh	59	(6490) Unit/Enh	Yes	No	120	20	4	8.301	3	Negativ
Perfluoro-1	No	506	(6490)	78	(6490)	Yes	No	162	48	4	7.59	3	Negativ
[1308]octa nesulfona			(6490)		(6490)								
mide (MBFOSA) Perfluoro 1	No	507	U nit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	174	48	4	6.59	3	Negati
1308]octa resulfonic acid													
MBPFOS) Perfluoro-1	No	507	U nit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	174	54	4	6.59	3	Negati
1308]octa nesulfonic acid (MBPFOS) Perfluoro-1	No	598.9	U nit/Enh	98.9	Unit/Enh	Yes	No	156	50	4	7.546	3	Negati
decanesulf onate (L- PFDS)			(6490)		(6490)								-
erfluoro-1 lecanesulf	No	598.9	U nit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	100	60	4	7.546	3	Negat
опаte (L: PFDS) Perfluoro-1	No	448.9	U nit/Enh	98.9	Unit/Enh	Yes	No	162	48	4	6.252	3	Negat
eptanesul fonate (L-		770.0	(6490)	30.3	(6490)			102		·	0.202	· ·	Nogal
PFHpS) Perfluoro-1	No	448.9	U nit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	162	48	4	6.252	3	Negati
ieptanesul fonate (L- PF HpS)													
erfluoro 1 octanes ulf on amide	No	497.9	U nit/Enh (6490)	478	Unit/Enh (6490)	Yes	No	156	100	4	7.651	3	Negat
(FOSA) erfluoro 1	No	497.9	U nit/Enh (6490)	78	Unit/Enh (6490)	Yes	No	156	40	4	7.651	3	Negat
octanesulf onamide (FOSA)	<b>N</b> -	0.40-				36	N -	450			F0#		No.
entanesul	No	348.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	36	4	5.042	3	Negat
fonate (L- PFPeS) rerfluoro-1	No	348.9	U nit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	40	4	5.042	3	Negat
entanesul fonate (L- PFPeS)													

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Cpd Name	ISTD?	Prec lon	MS1 Res	Prod lon	MS2 Res	Primary	Trigger	Frag (V)	CE(V)	Cell Acc	Ret Time (min)	Ret Window	Polarity
Perfluorob utanesulfo nic acid	No	298.9	U nit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	32	(V) 4	4.042	3	Negative
(PFBS) Perfluorob utanesulfo nic acid	No	298.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	36	4	4.042	3	Negative
(PFBS) Perfluorod ecanoic acid	No	513	Unit/Enh (6490)	468.8	Unit/Enh (6490)	Yes	No	90	8	4	7.158	3	Negative
(PFDA) Perfluorod ecanoic acid	No	513	U nit/Enh (6490)	268.8	Unit/Enh (6490)	Yes	No	90	16	4	7.158	3	Negative
(PFDA) Perfluorod odecanes u Ifonic acid	No	699	Unit/Enh (6490)	99	Unit/Enh (6490)	Yes	No	100	60	4	7.984	3	Negative
(PFD oS) Perfluorod odecanes u Ifonic acid	No	699	Unit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	156	50	4	7.984	3	Negative
(PFD oS) Perfluorod odecanoic acid	No	613	U nit/Enh (6490)	568.8	Unit/Enh (6490)	Yes	No	90	12	4	7.876	3	Negative
(PFD oA) Perfluorod odecanoic acid	No	613	U nit/Enh (6490)	168.7	Unit/Enh (6490)	Yes	No	90	28	4	7.876	3	Negative
(PFD oA) Perfluoroh eptanoic acid (PFHpA)	No	363	U nit/Enh (6490)	318.8	Unit/Enh (6490)	Yes	No	90	8	4	5.601	3	Negative
Perfluoroh eptanoic acid (PFHpA)	No	363	Unit/Enh (6490)	168.9	Unit/Enh (6490)	Yes	No	90	16	4	5.601	3	Negative
Perfluoroh exanesulfo nic acid (PF HxS)	No	398.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	40	4	5.685	3	Negative
Perfluoroh exanesulfo nic acid (PF HxS)	No	398.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	44	4	5.685	3	Negative
Perfluoroh exanoic acid (PF HxA)	No	313	Unit/Enh (6490)	268.9	Unit/Enh (6490)	Yes	No	70	4	4	4.856	3	Negative
Perfluoroh exanoic acid (PF HxA)	No	313	Unit/Enh (6490)	119	Unit/Enh (6490)	Yes	No	70	20	4	4.856	3	Negative
Perfluoro- n-[1,2- 1302]dode canoic acid	No	615	U nit/Enh (6490)	570	Unit/Enh (6490)	Yes	No	53	8	4	7.71	3	Negative
(MPF DoA) Perfluoro- n- [13C4]buta noic acid	No	217	U nit/Enh (6490)	172	Unit/Enh (6490)	Yes	No	59	4	4	1.22	3	Negative
(MPFBA) Perfluoro- n- [13054]pe	No	268	Unit/Enh (6490)	223	Unit/Enh (6490)	Yes	No	62	4	4	3.44	3	Negative
ntanoic acid (M5PF PeA )													
Perfluoro- n- [1308]octa noic acid	No	421	Unit/Enh (6490)	376	Unit/Enh (6490)	Yes	No	59	4	4	6.05	3	Negative
(M8PFOA) Perfluoro- n- [13C8]octa noic acid (M8PFOA)	No	421	U nit/Enh (6490)	172	Unit/Enh (6490)	Yes	No	59	16	4	6.05	3	Negative

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Cpd Name	ISTD?	Prec lon	MS1 Res	Prod lon	MS2 Res	Primary	Trigger	Frag (V)	CE(V)	Cell Acc	Ret Time	Ret	Polarity
Perfluoro-	No	472	Unit/Enh	427	Unit/Enh	Yes	No	59	8	(V) 4	(min.) 6.56	Window 3	Negative
n- [1309]non anoic acid			(6490)		(6490)								
(M9PFNA) Perfluoro- n-	No	472	U nit/Enh (6490)	223	Unit/Enh (6490)	Yes	No	59	16	4	6.56	3	Negativ
[1309]non anoic acid (M9PFNA)			(=)		(- 1)								
Perfluoro- n-butanoic acid	No	213	U nit/Enh (6490)	168.9	Unit/Enh (6490)	Yes	No	70	4	4	1.246	3	Negativ
(PFBA) Perfluoron onanes ulfo nate (L-	No	548.9	U nit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	159	48	4	7.174	3	Negativ
PFNS) Perfluoron onanes ulfo nate (L-	No	548.9	U nit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	159	48	4	7.174	3	Negativ
PFNS) Perfluoron onanoic acid	No	463	U nit/Enh (6490)	418.8	Unit/Enh (6490)	Yes	No	90	8	4	6.718	3	Negativ
(PFNA) Perfluoron onanoic acid	No	463	U nit/Enh (6490)	218.8	Unit/Enh (6490)	Yes	No	90	16	4	6.718	3	Negative
(PFNA) Perfluoro- n-	No	263	U nit/Enh (6490)	219	Unit/Enh (6490)	Yes	No	62	4	4	3.526	3	Negativ
pentanoic acid (PF PeA) Perfluoroo	No	498 9	Unit/Enh	98.9	Unit/Enh	Yes	No	150	44	4	6.743	3	Negativ
ctanesulfo nic acid (PFOS)			(6490)		(6490)								
Perfluoroo ctanesulfo nic acid (PFOS)	No	498.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	84	4	6.743	3	Negativ
Perfluoroo ctanoic acid (PFOA)	No	413	U nit/Enh (6490)	368.8	Unit/Enh (6490)	Yes	No	90	8	4	6.202	3	Negativ
Perfluoróo ctanoic acid	No	413	U nit/Enh (6490)	168.9	Unit/Enh (6490)	Yes	No	90	16	4	6.202	3	Negativ
(PFOA) Perfluorote tradecanoi c acid	No	713	U nit/Enh (6490)	669	Unit/Enh (6490)	Yes	No	110	12	4	8.414	3	Negativ
(PFTA) Perfluorote tradecanoi c acid	No	713	U nit/Enh (6490)	168.8	Unit/Enh (6490)	Yes	No	110	28	4	8.414	3	Negativ
(PFTA) Perfluorotri decanoic acid	No	663	U nit/Enh (6490)	618.8	Unit/Enh (6490)	Yes	No	90	12	4	8.164	3	Negativ
(PFTrDA) Perfluorou ndecanoic acid	No	563	U nit/Enh (6490)	519	Unit/Enh (6490)	Yes	No	90	8	4	7.538	3	Negativ
(PFUnA) Perfluorou ndecanoic acid	No	563	U nit/Enh (6490)	169	Unit/Enh (6490)	Yes	No	90	24	4	7.538	3	Negativ
(PFUπA) PFEESA	No	315	U nit/Enh (6490)	135	Unit/Enh (6490)	Yes	No	112	26	4	4.464	3	Negativ
PFEESA	No	315		83		Yes	No	112	14	4	4.464	3	Negativ
PFMBA	No	279	Unit/Enh (6490)	85	Unit/Enh (6490)	Yes	No	75	18	4	4.011	3	Negative
PF MPA	Νo	229	Unit/Enh (6490)	85	Unit/Enh (6490)	Yes	No	59	6	4	2.15	3	Negative

Data Stg Centroid Threshold

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Source Parameters			
Parameter	Value (+)	Value (-)	
Gas Temp (*C)	230	230	
Gas Flow (I/min)	5	5	
Nebulizer (psi)	15	15	
SheathGasHeater	350	350	
SheathGasFlow	12	12	
Capillary (V)	3500 500	2500 0	
VCharging	500	v	
Chromatograms			
Chrom Type	Label	Offset 0	Y-Range
TIC	TIC	U	10000000
Instrument Curves			
Actual			
Name: HiP Sample	r		Module: G4226A
Auxiliary			
Draw Speed			100.0 µL/min
Eject Speed			400.0 μL/min
Draw Position C			1.5 mm
Wait Time After			1.2 s
Sample Flush O			5.0
Vial/Well botton	1 sensing		Yes
Injection			
Injection Mode			Injection with needle wash
Injection Volum	e		3.00 µL
Needle Wash			
Needle Wash	Location		Flush Port
Wash Time			10.0 s
High throughput			
	/ Volume Reduction		No
Overlapped Inje			· ·-
	apped Injection		No
Valve Switching	appea aljection		140
Valve Movemen	te		0
Valve Switch Til			U
			hlo
Switch Time			No
Valve Switch Ti			
Switch Time			No
Valve Switch Ti			
Switch Time			No
Valve Switch Ti			
Switch Time	4 Enabled		No
Stop Time			
Stoptime Mode			As pump/No limit
Post Time			
Posttime Mode			ОП
Name: Binary Pun	р		Module: G4220A
-			0.400 mL/min
Flow	i		No
Use Solvent Types			Synchronized
Use Solvent Types Stroke Mode	t		Synchronized 0.00 bar
Flow Use Solvent Types Stroke Mode Low Pressure Lim High Pressure Lim			0.00 bar
Use Solvent Types Stroke Mode Low Pressure Lim High Pressure Lim	it		0.00 bar 600.00 bar
Use Solvent Types Stroke Mode Low Pressure Lim	it P		0.00 bar

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## Appendix C Health and Safety Plan

## Health and Safety Plan

## for 260 West 126<sup>th</sup> Street Remedial Investigation Work Plan

260 West 126<sup>th</sup> Street New York, New York 10027 Block 1931, Lot No. 56 BCP Site # TBD

#### Submitted to:

New York State Department of Environmental Conservation Division of Environmental Remediation Remedial Bureau B 625 Broadway, 12<sup>th</sup> Floor Albany, NY 12233-7016

Prepared for: S & F 126 ST. LLC 320 Roebling Street, Suite 749 Brooklyn, NY 11211

#### Prepared by:



121 West 27<sup>th</sup> Street, Suite 303 New York, NY 10001

#### Tenen Environmental, LLC Health and Safety Plan

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Appendix A – Acknowledgement of HASP

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Appendix C – Material Safety Data Sheets

#### 1.0 INTRODUCTION

This Health and Safety Plan (HASP) has been prepared in conformance with the Occupational Safety and Health Administration (OSHA) standards and guidance that govern site investigation activities, other applicable regulations, and Tenen Environmental, LLC (Tenen) health and safety policies and procedures. The purpose of this HASP is the protection of Tenen field personnel and others during the implementation of a Remedial Investigation.

The Site, located at 260 West 126<sup>th</sup> Street, New York, New York (Tax Block 1931, Lot 56), is a rectangular shaped parcel located on the southern side of West 126<sup>th</sup> Street, between Frederick Douglass Boulevard (formerly Eighth Avenue) and Adam C. Powell Boulevard, in the Harlem section of Manhattan. The Site is approximately 12,490 square feet (SF) and has approximately 125 feet of frontage along West 126<sup>th</sup> Street and is approximately 100 feet deep.

The Site is developed with a two-story church building with a full cellar level covering approximately 2,725 SF of the western portion of the property. The church building is currently vacant and recently experienced a fire, rendering it unsafe to occupy. The remainder of the property is paved and utilized as a vacant parking lot.

#### 1.1 Scope of HASP

This HASP includes safety procedures to be used by Tenen staff during the following activities:

- Advancement of soil borings, installation of permanent monitoring wells, and installation of temporary soil vapor points, and;
- Collection of soil, groundwater, soil vapor, and ambient air samples from soil borings, new permanent monitoring wells, and temporary soil vapor points.

Subcontractors will ensure that performance of the work is in compliance with this HASP and applicable laws and regulations.

#### 2.0 PROJECT SAFETY AUTHORITY

The following personnel are responsible for project health and safety under this HASP.

- Project Manager, Mohamed Ahmed
- Health and Safety Officer (HSO), Ashley Platt

In addition, each individual working at the Site will be responsible for compliance with this HASP and general safe working practices. All Site workers will have the authority to stop work if a potentially hazardous situation or event is observed.

#### 2.1 Designated Personnel

The Project Manager is responsible for the overall operation of the project, including compliance with the HASP and general safe work practices. The Project Manager may also act as the Health and Safety Officer (HSO) for this project.

Tenen will appoint one of its on-site personnel as the on-site HSO. This individual will be responsible for the implementation of the HASP. The HSO will have a 4-year college degree in occupational safety or a related science/engineering field, and at least two (2) years of experience in implementation of air monitoring and hazardous materials sampling programs. The HSO will have completed a 40-hour training course that meets OSHA requirements of 29 CFR Part 1910, Occupational Safety and Health Standards.

The HSO will be present on-site during all field operations involving drilling or other subsurface disturbance, and will be responsible for all health and safety activities and the delegation of duties to the field crew. The HSO has stop-work authorization, which he/she will execute on his/her determination of an imminent safety hazard, emergency situation, or other potentially dangerous situation. If the HSO must be absent from the field, a replacement who is familiar with the Construction Health and Safety Plan, air monitoring and personnel protective equipment (PPE) will be designated.

#### 3.0 HAZARD ASSESSMENT AND CONTROL MEASURES

The Site has been developed since at least 1902. In 1902, the Site lot was divided into six separate tax parcels with six tightly grouped four-story residential buildings present on the north side of the Site, along West 126<sup>th</sup> Street. By 1951, the residential buildings had been demolished and the Site was mostly undeveloped except for the western portion of the Site, where a four-story storefront building with lumber storage remained. By 1962, the existing two-story church building with a full cellar level was present on the western portion of the Site. The Site has generally remained unchanged through current day with a two-story church building on the western portion of the Site and licensed parking on the eastern portion of the Site. The following previous investigations summarize contaminants of concern detected on the site:

Phase I Environmental Site Assessment Report, 260 West 126<sup>th</sup> Street, Manhattan, NY, Environmental Business Consultants (EBC), October 2016.

EBC conducted a Phase I Environmental Site Assessment (ESA) at the Site in October 2016 and concluded that there are no recognized environmental conditions (RECs) in connection with the property.

Remedial Investigation Report, 260 West 126<sup>th</sup> Street, Manhattan, NY, Environmental Business Consultants (EBC), December 2016.

A Remedial Investigation Report was submitted by EBC in December 2016, reporting the Phase II Remedial Investigation in November 2016. The Investigation consisted of the advancement of eight soil borings, collection of sixteen soil samples, installation of four groundwater monitoring wells, collection of four groundwater samples, installation of five temporary sub-slab soil vapor points, and the collection of five sub-slab soil vapor samples. The results of the Phase II Remedial Investigation are as follows:

- No PCBs were detected in soil/fill and groundwater samples.
- Trace concentrations of VOCs were detected in soil/fill samples, but none exceeded Unrestricted Use SCOs. No VOCs were detected in groundwater samples.
- Several SVOCs, including benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene and indeno(1,2,3-cd)pyrene, were detected in soil samples above Restricted-Residential SCOs, benzo(k)fluoranthene and chrysene were detected above Unrestricted Use SCOs. The highest concentrations were detected in shallow soil (0-2 ft-bg). All mentioned SVOCs above were also detected above New York State 6NYCRR Part 703.5 Class GA groundwater quality standards (GQS) in groundwater samples.
- Two pesticides, 4,4'-DDE and 4,4'-DDT were detected above Unrestricted Use SCOs in soil samples. No pesticides were detected in groundwater samples.
- Several metals, including arsenic, barium, chromium, copper, lead, mercury, nickel, and zinc were detected above Unrestricted Use SCOs for the soil samples, with barium and lead, detected above Restricted-Residential SCOs in four shallow samples and one deep sample. For groundwater samples, aluminum, arsenic, barium, beryllium, cadmium, chromium, copper, iron, lead, magnesium, manganese, nickel, sodium, and thallium were detected above their respective GQS in the unfiltered sample. A filtered sample of groundwater was not collected.

 Soil vapor sample during Remedial Investigation showed moderate levels of petroleumrelated VOCs, of which toluene had the highest concentration, and chlorinated VOCs, of which tetrachloroethene (PCE) had the highest concentrations, with PCE notably elevated compared to other VOCs. Trichloroethene (TCE) was also detected in two samples and the duplicate.

#### 3.1 Human Exposure Pathways

The media of concern at the Site include potentially-impacted soil, groundwater and soil vapor. Potential exposure pathways include dermal contact, incidental ingestion and inhalation of vapors. The risk of dermal contact and incidental ingestion will be minimized through general safe work practices, a personal hygiene program and the use of PPE. The risk of inhalation will be minimized through the use of an air monitoring program for VOCs and particulates.

#### 3.2 Chemical Hazards

Based on historic uses, the following contaminants of concern may be present at the Site:

#### Chlorinated Solvents

• Tetrachloroethene (PCE)

#### **SVOCs**

• PAHs

#### Metals

- Barium
- Lead

Material Safety Data Sheets (MSDSs) for each contaminant of concern are included in Appendix C. All personnel are required to review the MSDSs included in this HASP.

#### 3.3 Physical Hazards

The physical hazards associated with the field activities likely present a greater risk of injury than the chemical constituents at the Site. Activities within the scope of this project shall comply with New York State and Federal OSHA construction safety standards.

#### Head Trauma

To minimize the potential for head injuries, field personnel will be required to wear National Institutes of Occupational Safety and Health (NIOSH)-approved hard hats during field activities. Hats must be worn properly and not altered in any way that would decrease the degree of protection provided.

#### Foot Trauma

To avoid foot injuries, field personnel will be required to wear steel-toed safety shoes while field activities are being performed. To afford maximum protection, all safety shoes must meet American National Standards Institute (ANSI) standards.

#### Eye Trauma

Field personnel will be required to wear eye protection (safety glasses with side shields) while field activities are being performed to prevent eye injuries caused by contact with chemical or physical agents.

#### Noise Exposure

Field personnel will be required to wear hearing protection (ear plugs or muffs) in high noise areas (noise from heavy equipment) while field activities are being performed.

#### Buried Utilities and Overhead Power Lines

Boring locations will be cleared by an underground utility locator service. In addition, prior to intrusive activities, the drilling subcontractor will contact the One Call Center to arrange for a utility mark-out, in accordance with New York State requirements. Protection from overhead power lines will be accomplished by maintaining safe distances of at least 15 feet at all times.

#### Thermal Stress

The effects of ambient temperature can cause physical discomfort, personal injury, and increase the probability of accidents. In addition, heat stress due to lack of body ventilation caused by protective clothing is an important consideration. Heat-related illnesses commonly consist of heat stroke and heat exhaustion.

The symptoms of heat stroke include: sudden onset; change in behavior; confusion; dry, hot and flushed skin; dilated pupils; fast pulse rate; body temperature reaching 105° or more; and/or, deep breathing later followed by shallow breathing.

The symptoms of heat exhaustion include: weak pulse; general weakness and fatigue; rapid shallow breathing; cold, pale and clammy skin; nausea or headache; profuse perspiration; unconsciousness; and/or, appearance of having fainted.

Heat-stress monitoring will be conducted if air temperatures exceed 70 degrees Fahrenheit. The initial work period will be set at 2 hours. Each worker will check his/her pulse at the wrist for 30 seconds early in each rest period. If the pulse rate exceeds 110 beats per minute, the next work period will be shortened by one-third.

One or more of the following precautions will reduce the risk of heat stress on the Site:

- Provide plenty of liquids to replace lost body fluids; water, electrolytic drinks, or both will be made available to minimize the risk of dehydration and heat stress
- Establish a work schedule that will provide appropriate rest periods
- Establish work regimens consistent with the American Conference of Governmental Industrial Hygienists (ACGIH) guidelines
- Provide adequate employee training on the causes of heat stress and preventive measures

In the highly unlikely event of extreme low temperatures, reasonable precautions will be made to avoid risks associated with low temperature exposure.

#### **Traffic**

# Tenen Environmental, LLC Health and Safety Plan

Field activities will occur near public roadways. As a result, vehicular traffic will be a potential hazard during these activities and control of these areas will be established using barricades or traffic cones. Additional staff will be assigned, as warranted, for the sole purpose of coordinating traffic. Personnel will also be required to wear high-visibility traffic vests while working in the vicinity of the public roadways and local requirements for lane closure will be observed as needed. All work in public rights-of-way will be coordinated with local authorities and will adhere to their requirements for working in traffic zones.

#### **Hazardous Weather Conditions**

All Site workers will be made aware of hazardous weather conditions, specifically including extreme heat, and will be requested to take the precautions described herein to avoid adverse health risks. All workers are encouraged to take reasonable, common sense precautions to avoid potential injury associated with possible rain or high wind, sleet, snow or freezing.

#### Slip, Trip and Fall

Areas at the Site may be slippery from mud or water. Care should be taken by all Site workers to avoid slip, trip, and fall hazards. Workers shall not enter areas that do not have adequate lighting. Additional portable lighting will be provided at the discretion of the HSO.

#### **Biological Hazards**

Drugs and alcohol are prohibited from the Site. Any on-site personnel violating this requirement will be immediately expelled from the site.

Any worker or oversight personnel with a medical condition that may require attention must inform the HSO of such condition. The HSO will describe appropriate measures to be taken if the individual should become symptomatic.

Due to the Site location in an urban area, it is highly unlikely that poisonous snakes, spiders, plants and insects will be encountered. However, other animals (dogs, cats, etc.) may be encountered and care should be taken to avoid contact.

#### 4.0 COVID-19 HEALTH AND SAFETY

The following requirements apply to all Tenen employees working on project sites for the duration of the COVID-19 pandemic. These guidelines are based on information provided by the Centers for Disease Control, the Occupational Safety and Health Administration and the New York State "New York Forward" Covid-19 management plans. Information regarding the health status of Tenen employees will be kept confidential, with the exception of required notifications to health authorities. The following are guidelines. As with any potential workplace hazard, employees should report any concerns related to potential Covid-19 exposure to the Project Manager.

#### **Communication/Reporting:**

Employees should not report to work and should notify the Project Manager immediately in the event of the following:

- You are exhibiting flu-like symptoms (fever, body aches, cough, difficulty breathing). Contact your health care provider and follow their instructions.
- You do not exhibit symptoms but have a sick (i.e., diagnosed with Covid-19 or exhibiting flu-like symptoms) family member at home. Remember that the virus can be spread by asymptomatic individuals.
- You have been exposed to someone who has been diagnosed with Covid-19.

In each of the above cases, inform your Project Manager regarding others who may have been exposed in order to facilitate any necessary notification or contact tracing efforts.

#### Hygiene

- Wash hands frequently with soap and water for at least 20 seconds or use hand sanitizer with at least 60% alcohol if soap and water are not available. Key times for employees to clean their hands include:
  - Before and after work shifts
  - Before and after work breaks
  - o After blowing the nose, coughing, or sneezing
  - o After using the restroom
  - o Before eating or preparing food
  - o After putting on, touching, or removing face coverings
- Avoid touching the eyes, nose, and mouth with unwashed hands.
- Practice good respiratory etiquette, including covering coughs and sneezes.
- To the extent possible, avoid sharing tools and sampling equipment. Shared tools and equipment should be regularly disinfected.

#### **Physical Distancing**

- Minimize contact with others, maintaining a distance of at least six feet to the extent possible
- Employees should wear masks over their nose and mouth to prevent spread of the virus; this is especially important when a minimum 6-foot distance cannot be maintained.
- Maintain the 6-foot distance to the extent possible during sampling efforts and pickup and delivery of sampling equipment and containers.
- Keep job site meetings to a minimum and of short duration; limit the number of people involved and maintain social distance.

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#### 5.0 AIR MONITORING

The NYSDOH Generic Community Air Monitoring Plan (CAMP), included as Appendix 1A of DER-10, will be implemented during all ground-intrusive sampling activities.

#### 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 should be performed using equipment appropriate for 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 shut down.
- 4. All 15-minute readings must be recorded and be available for State (NYSDEC 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/m3) 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 (NYSDEC and NYSDOH) personnel to review.

The NYSDOH Generic CAMP is included as Appendix D of the RIWP.

#### 6.0 PERSONAL PROTECTIVE EQUIPMENT

The personal protection equipment required for various kinds of site investigation tasks is based on 29 CFR 1910.120, Hazardous Waste Operations and Emergency Response, "General Description and Discussion of the Levels of Protection and Protective Gear" and the Centers for CDC COVID-19 "Guidelines on How to Protect Yourselves and Others".

Tenen field personnel and other site personnel will wear Modified Level D-1 personal protective equipment. During activities such as drilling, well installation, or sampling, where there is a chance of contact with contaminated materials, Modified Level D-2 equipment will be worn. The protection will be upgraded to Level C if warranted by the results of the air monitoring. A six-foot minimum distance between individuals (both workers and non-workers) will be maintained at all times. A description of the personnel protective equipment for Levels D and C is provided below.

#### **Modified Level D-1**

Respiratory Protection: Cloth face covering

Protective Clothing: Hard hat, steel-toed shoes, long pants, nitrile gloves

**Modified Level D-2** 

Respiratory Protection: Cloth face covering

Protective Clothing: Hard hat, steel-toed shoes, coveralls/tyvek, nitrile gloves

Level C

Respiratory Protection: Air purifying respirator with organic vapor cartridges and filters.

Protective Clothing: Same as Modified Level D-2

#### 7.0 EXPOSURE MONITORING

#### 7.1 Hazardous Materials

Selective monitoring of workers in the exclusion area may be conducted, as determined by the HSO, if sources of hazardous materials are identified. Personal monitoring may be conducted in the breathing zone at the discretion of the Project Manager or HSO. All monitoring will comply with the CDCs Guidance on Social Distancing.

#### 7.2 COVID-19

For any employee that may have come into contact with a person who has COVID-19, a 14-day quarantine will be imposed for that individual and any employee that individual was in contact with.

#### 8.0 SITE ACCESS

Access to the Site during the investigation will be controlled by the Project Manager or HSO. Unauthorized personnel will not be allowed access to the sampling areas.

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#### 9.0 WORK AREAS

During any activities involving drilling or other subsurface disturbance, the work area must be divided into various zones to prevent the spread of contamination, clarify the type of protective equipment needed, and provide an area for decontamination.

The Exclusion Zone is defined as the area where potentially contaminated materials are generated as the result of drilling, sampling, or similar activities. The Contamination Reduction Zone (CRZ) is the area where decontamination procedures take place and is located adjacent to the Exclusion Zone. The Support Zone is the area where support facilities such as vehicles, a field phone, fire extinguisher and/or first aid supplies are located. The emergency staging area (part of the Support Zone) is the area where all Site workers will assemble in the event of an emergency. These zones shall be designated daily, depending on that day's activities. All field personnel will be informed of the location of these zones before work begins.

Control measures such as "Caution" tape and traffic cones will be placed around the perimeter of the work area when work is being done in the areas of concern (i.e., areas with exposed soil) to prevent unnecessary access.

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#### 10.0 DECONTAMINATION PROCEDURES

#### **Personnel Decontamination**

Personnel decontamination (decon), if deemed necessary by the HSO, will take place in the designated decontamination area delineated for each sampling location. Personnel decontamination will consist of the following steps:

- Soap and potable water wash and potable water rinse of gloves;
- Tyvek removal;
- Glove removal;
- Disposable clothing removal; and
- Field wash of hands and face.

#### **Equipment Decontamination**

Sampling equipment, such as split-spoons and bailers, will be decontaminated in accordance with U.S. Environmental Protection Agency methodologies, as described in the work plan.

#### **Disposal of Materials**

Purged well water, water used to decontaminate any equipment and well cuttings will be containerized and disposed off-site in accordance with federal, state and local regulations.

#### 11.0 GENERAL SAFE WORK PRACTICES

To protect the health and safety of the field personnel, all field personnel will adhere to the guidelines listed below during activities involving subsurface disturbance.

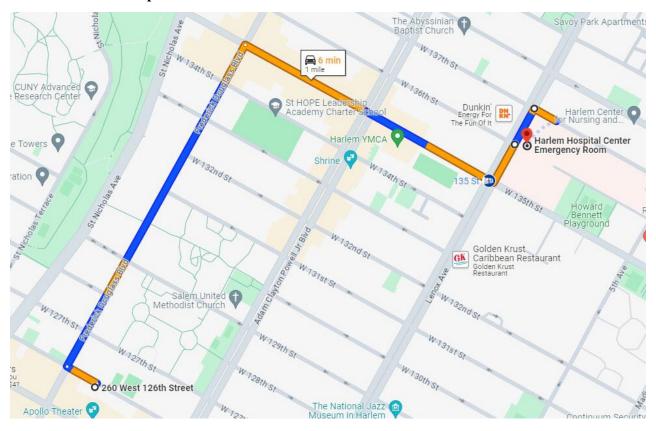
- Eating, drinking, chewing gum or tobacco, and smoking are prohibited, except in designated areas on the site. These areas will be designated by the HSO.
- Workers must wash their hands and face thoroughly on leaving the work area and before eating, drinking, or any other such activity. The workers should shower as soon as possible after leaving the site.
- Removal of potential contamination from PPE and equipment by blowing, shaking or any means that may disperse materials into the air is prohibited.
- Contact with contaminated or suspected surfaces should be avoided.
- The buddy system should always be used; each buddy should watch for signs of fatigue, exposure, and heat stress.
- Personnel will be cautioned to inform each other of symptoms of chemical exposure such as headache, dizziness, nausea, and irritation of the respiratory tract and heat stress.
- No excessive facial hair that interferes with a satisfactory fit of the face-piece of the respirator to the face will be allowed on personnel required to wear respiratory protective equipment.
- On-site personnel will be thoroughly briefed about the anticipated hazards, equipment requirements, safety practices, emergency procedures, and communications methods.

#### 12.0 EMERGENCY PROCEDURES

The field crew will be equipped with emergency equipment, such as a first aid kit and disposable eye washes. In the case of a medical emergency, the HSO will determine the nature of the emergency and will have someone call for an ambulance, if needed. If the nature of the injury is not serious—i.e., the person can be moved without expert emergency medical personnel—onsite personnel should drive injured person to a hospital. The nearest emergency room is located at the Harlem Hospital Center located at 506 Lenox Ave, New York, NY 10037. The phone number is (212) 939-1000. The route to the hospital is shown and detailed on the next page.

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#### 12.1 Route to Hospital



Driving directions to the Harlem Hospital Center Emergency Room from 260 West 126<sup>th</sup> Street, New York, New York.

#### **Driving Directions**

- 1. Head northwest on W 126th St toward Frederick Douglass Blvd (220 ft).
- 2. Turn right at the 1st cross street onto Frederick Douglass Blvd (0.4 mi).
- 3. Turn right onto W 135th St (0.3 mi).
- 4. Turn left onto Lenox Ave/Malcolm X Blvd (0.1 mi).
- 5. Turn right onto the Emergency Room entrance on W 137th St (164 ft).

#### 12.2 Emergency Contacts

There will be an on-site field phone. Emergency and contact telephone numbers are listed below:

<u>Table 1 – Emergency Contacts</u>	
Ambulance	911
Emergency Room	(212) 939-1000
NYSDEC Spill Hotline	(800) 457-7362
NYSDEC	(518) 402-8013
Project Manager, Mohamed Ahmed	(917) 612-6018
On-site Personnel, Da Lin	(917) 530-5761
Client representative, Ushi Goldman	(646) 256-2188

#### 13.0 TRAINING

All personnel performing the field activities described in this HASP will have received the initial safety training required by 29 CFR, 1910.120. Current refresher training status also will be required for all personnel engaged in field activities.

All those who enter the work area while intrusive activities are being performed must recognize and understand the potential hazards to health and safety. All field personnel must attend a training program covering the following areas:

- potential hazards that may be encountered;
- the knowledge and skills necessary for them to perform the work with minimal risk to health and safety;
- the purpose and limitations of safety equipment; and
- protocols to enable field personnel to safely avoid or escape from emergencies.

Each member of the field crew will be instructed in the above objectives before he/she goes onto the site. The HSO will be responsible for conducting the training program.

#### 14.0 MEDICAL SURVEILLANCE

All Tenen and subcontractor personnel performing field work involving drilling or other subsurface disturbance at the site are required to have passed a complete medical surveillance examination in accordance with 29 CFR 1910.120 (f). The medical examination for Tenen employees will, at a minimum, be provided annually and upon termination of hazardous waste site work.

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## Appendix A

Acknowledgement of HASP

#### ACKNOWLEDGMENT OF HASP

Below is an affidavit that must be signed by all Tenen Environmental employees who enter the site. A copy of the HASP must be on-site at all times and will be kept by the HSO.

#### **AFFIDAVIT**

I have read the Construction Health and Safety Plan (HASP) for the 260 West 126<sup>th</sup> Street site in New York, NY. I agree to conduct all on-site work in accordance with the requirements set forth in this HASP and understand that failure to comply with this HASP could lead to my removal from the site.

Signature:	Date:
Signature:	Date:

## Appendix B

Injury Reporting Form (OSHA Form 300)

# How to Fill Out the Log

The Log of Work-Related Injuries and Illnesses is used to classify work-related injuries and illnesses and to note the extent and severity of each case. When an incident occurs, use the Log to record specific details about what happened and how it happened.

If your company has more than one establishment or site, you must keep separate records for each physical location that is expected to remain in operation for one year or longer.

We have given you several copies of the *Log* in this package. If you need more than we provided, you may photocopy and use as many as you need.

The *Summary* — a separate form — shows the work-related injury and illness totals for the year in each category. At the end of the year, count the number of incidents in each category and transfer the totals from the *Log* to the *Summary*. Then post the *Summary* in a visible location so that your employees are aware of injuries and illnesses occurring in their workplace.

You don't post the Log. You post only the Summary at the end of the year.

OSHA's Form 300 (Rev. 01/2004)

### Log of Work-Related Injuries and Illnesses

You must record information about every work-related death and about every work-related injury or illness that involves loss of consciousness, restricted work activity or job transfer, days away from work, or medical treatment beyond first aid. You must also record significant work-related injuries and illnesses that are diagnosed by a physician or licensed health

care professional. You must also record work-related injuries and illnesses that meet any of the specific recording criteria listed in 29 CFR Part 1904.8 through 1904.12. Feel free to use two lines for a single case if you need to. You must complete an Injury and Illness Incident Report (OSHA Form 301) or equivalent form for each injury or illness recorded on this

Attention: This form contains information relating to employee health and must be used in a manner that protects the confidentiality of employees to the extent possible while the information is being used for occupational safety and health purposes.

Year 20\_\_\_\_\_
U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

Establishment name XYZ Company

City Anywhere State MA

form. If you're not sure whether a case is recordable, call your local OSHA office for help Identify the person (C) (D) (E) Employee's name Job title Where the event occurred Describe injury or illness, parts of body affected, Date of injury  $(e.g.\ Welder)$ (e.g. Loading dock north end) and object/substance that directly injured or onset or made person ill of illness ď fracture, left arm and left leg, fell from ladder pouring deck poisoning from lead fumes 2nd floor storeroom broken left foot, fell over box ▲ Back strain lifting boxes packaging dept production floor dust in eye /- - -\_\_\_\_ days \_\_\_\_ days \_\_\_ 

Be as specific as possible. You can use two lines if you need more room.

Revise the log if the injury or illness progresses and the outcome is more serious than you originally recorded for the case. Cross out, erase, or white-out the original entry. Choose ONLY ONE of these categories. Classify the case by recording the most serious outcome of the case, with column G (Death) being the most serious and column J (Other recordable cases) being the least serious.

Note whether the case involves an injury or an illness.



## OSHA's Form 300 (Rev. 01/2004)

# Log of Work-Related Injuries and Illnesses

**Attention:** This form contains information relating to employee health and must be used in a manner that protects the confidentiality of employees to the extent possible while the information is being used for occupational safety and health purposes.



U.S. Department of Labor
Occupational Safety and Health Administration

Establishment name

Form approved OMB no. 1218-0176

You must record information about every work-related death and about every work-related injury or illness that involves loss of consciousness, restricted work activity or job transfer,
days away from work, or medical treatment beyond first aid. You must also record significant work-related injuries and illnesses that are diagnosed by a physician or licensed health
care professional. You must also record work-related injuries and illnesses that meet any of the specific recording criteria listed in 29 CFR Part 1904.8 through 1904.12. Feel free to
use two lines for a single case if you need to. You must complete an Injury and Illness Incident Report (OSHA Form 301) or equivalent form for each injury or illness recorded on this
form. If you're not sure whether a case is recordable, call your local OSHA office for help.

m. If yo	ou're not sure whether a case	e is recordable, call your i	local OSHA office f	or help.						City			Sta	te		
dent	ify the person		Describe t	he case			sify the ca									
A) Case	(B) Employee's name	(C) Job title	(D)  Date of injury	(E) Where the event occurred	(F) Describe injury or illness, parts of body affected,		on the mos	E box for each		Enter to days the ill work	he number of ne injured or ker was:	Chec	ck the	"Injury e type	," colu of illn	mn d ess:
10.		(e.g., Welder)	or onset of illness	(e.g., Loading dock north end)	and object/substance that directly injured or made person ill (e.g., Second degree burns on			Remaine	ed at Work	Away	On job	(M)	order	ory n	g loss	
					right forearm from acetylene torch)	Death		Job transfer or restriction	Other record- able cases	from work	transfer or restriction	Injury	Skin dis	Respirat	Poisonir Hearing	All other
						(G)	(H)	(I)	(J)	(K)	(L)	(1)			(4) (5)	) (6
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Public reporting burden for this collection of information is estimated to average 14 minutes per response, including time to review the instructions, search and gather the data needed, and complete and review the collection of information. Persons are not required to respond to the collection of information unless it displays a currently valid OMB control number. If you have any comments about these estimates or any other aspects of this data collection, contact: US Department of Labor, OSHA Office of Statistical Analysis, Room N-3644, 200 Constitution Avenue, NW, Washington, DC 20210. Do not send the completed forms to this office.

Be sure to transfer these totals to the Summary page (Form 300A) before you post it.

Injury	Skin disorder	Respiratory condition	Poisoning	Hearing loss	All other lillnesses
(1)	(2)	(3)	(4)	(5)	(6)

## OSHA's Form 300A (Rev. 01/2004)

# Summary of Work-Related Injuries and Illnesses



U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

All establishments covered by Part 1904 must complete this Summary page, even if no work-related injuries or illnesses occurred during the year. Remember to review the Log to verify that the entries are complete and accurate before completing this summary.

Using the Log, count the individual entries you made for each category. Then write the totals below, making sure you've added the entries from every page of the Log. If you had no cases, write "0."

Employees, former employees, and their representatives have the right to review the OSHA Form 300 in its entirety. They also have limited access to the OSHA Form 301 or its equivalent. See 29 CFR Part 1904.35, in OSHA's recordkeeping rule, for further details on the access provisions for these forms.

Number of C	ases		
Total number of deaths	Total number of cases with days away from work	Total number of cases with job transfer or restriction	Total number of other recordable cases
(G)	(H)	(1)	(J)
Number of D	)ays		
Total number of da from work		otal number of days of job ansfer or restriction	
(K)	_	(L)	
Injury and II	Iness Types		
Total number of (M)			
) Injuries		(4) Poisonings	
		(5) Hearing loss	
) Skin disorders		(6) All other illness	es
Respiratory condit	ions		

#### Post this Summary page from February 1 to April 30 of the year following the year covered by the form.

Public reporting burden for this collection of information is estimated to average 58 minutes per response, including time to review the instructions, search and gather the data needed, and complete and review the collection of information. Persons are not required to respond to the collection of information unless it displays a currently valid OMB control number. If you have any comments about these estimates or any other aspects of this data collection, contact: US Department of Labor, OSHA Office of Statistical Analysis, Room N-3644, 200 Constitution Avenue, NW, Washington, DC 20210. Do not send the completed forms to this office.

Your establishment name _	
Street	
City	State ZIP
Industry description (e.g., M	lanufacture of motor truck trailers)
Standard Industrial Classific	cation (SIC), if known (e.g., 3715)
OR	
North American Industrial	Classification (NAICS), if known (e.g., 336212)
	nation (If you don't have these figures, see the
Employment inform	<b>mation</b> (If you don't have these figures, see the age to estimate.)
<b>Employment inforr</b> Worksheet on the back of this pa Annual average number of o	mation (If you don't have these figures, see the age to estimate.) employees
<b>Employment inform</b> Worksheet on the back of this per Annual average number of o	mation (If you don't have these figures, see the age to estimate.) employees
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# Worksheet to Help You Fill Out the Summary

At the end of the year, OSHA requires you to enter the average number of employees and the total hours worked by your employees on the summary. If you don't have these figures, you can use the information on this page to estimate the numbers you will need to enter on the Summary page at the end of the year.

# How to figure the average number of employees who worked for your establishment during the year:

**1 Add** the total number of employees your establishment paid in all pay periods during the year. Include all employees: full-time, part-time, temporary, seasonal, salaried, and hourly.

The number of employees paid in all pay periods =

**2 Count** the number of pay periods your establishment had during the year. Be sure to include any pay periods when you had no employees.

The number of pay periods during the year =

**3 Divide** the number of employees by the number of pay periods.

<u>0</u> — = <u>0</u>

**4 Round the answer** to the next highest whole number. Write the rounded number in the blank marked *Annual average number of employees*.

The number rounded = **4** 

For example, Acme Construction figured its average employment this way:

For pay period	Acme paid this number of employees		
1	10	Number of employees paid = 830	0
2	0	1 / 1	
3	15	Number of pay periods $= 26$	2
4	30	830 = 31.92	•
5	40	<del></del>	0
▼	▼	26	
24	20	31.92 rounds to 32	A
25	15	31.72 Totalids to 32	•
26	+10	32 is the annual average number of emple	oyees
	830		•

#### How to figure the total hours worked by all employees:

Include hours worked by salaried, hourly, part-time and seasonal workers, as well as hours worked by other workers subject to day to day supervision by your establishment (e.g., temporary help services workers).

Do not include vacation, sick leave, holidays, or any other non-work time, even if employees were paid for it. If your establishment keeps records of only the hours paid or if you have employees who are not paid by the hour, please estimate the hours that the employees actually worked.

If this number isn't available, you can use this optional worksheet to estimate it.

#### **Optional Worksheet**

	 <b>Find</b> the number of full-time employees in your establishment for the year.
X	 <b>Multiply</b> by the number of work hours for a full-time employee in a year.
	 This is the number of full-time hours worked.
+	 <b>Add</b> the number of any overtime hours as well as the hours worked by other employees (part-time, temporary, seasonal)

Write the rounded number in the blank marked *Total hours worked by all employees last year.* 



# OSHA's Form 301

# Injury and Illness Incident Report

**Attention:** This form contains information relating to employee health and must be used in a manner that protects the confidentiality of employees to the extent possible while the information is being used for occupational safety and health purposes.



Form approved OMB no. 1218-0176

This *Injury and Illness Incident Report* is one of the first forms you must fill out when a recordable work-related injury or illness has occurred. Together with the *Log of Work-Related Injuries and Illnesses* and the accompanying *Summary*, these forms help the employer and OSHA develop a picture of the extent and severity of work-related incidents.

Within 7 calendar days after you receive information that a recordable work-related injury or illness has occurred, you must fill out this form or an equivalent. Some state workers' compensation, insurance, or other reports may be acceptable substitutes. To be considered an equivalent form, any substitute must contain all the information asked for on this form.

According to Public Law 91-596 and 29 CFR 1904, OSHA's recordkeeping rule, you must keep this form on file for 5 years following the year to which it pertains.

If you need additional copies of this form, you may photocopy and use as many as you need.

Completed by		 				_
Title						
Phone (	_)	 	Date	/	/	_

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) <b>D</b> a	ate hired//
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5) N: 7) If	rofessional  ame of physician or other health care professional
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66) No	ame of physician or other health care professional

	Information about the case	
10)	Case number from the Log	_ (Transfer the case number from the Log after you record the case.)
11)	Date of injury or illness//	-
12)	Time employee began work	AM / PM
13)	Time of event	AM / PM Check if time cannot be determined
14)	tools, equipment, or material the employee v	the incident occurred? Describe the activity, as well as the was using. Be specific. Examples: "climbing a ladder while rine from hand sprayer"; "daily computer key-entry."
15)		nred. Examples: "When ladder slipped on wet floor, worker rine when gasket broke during replacement"; "Worker
16)		part of the body that was affected and how it was affected; be Examples: "strained back"; "chemical burn, hand"; "carpal
17)	What object or substance directly harmed "radial arm saw." If this question does not app	the employee? Examples: "concrete floor"; "chlorine"; oly to the incident, leave it blank.
18)	If the employee died, when did death occu	r? Date of death//

# U.S. Department of Labor

# If You Need Help...

If you need help deciding whether a case is recordable, or if you have questions about the information in this package, feel free to contact us. We'll gladly answer any questions you have.

- **▼** Visit us online at www.osha.gov
- ▼ Call your OSHA Regional office and ask for the recordkeeping coordinator

or

**▼ Call your State Plan office** 

#### **Federal Jurisdiction**

Region 1 - 617 / 565-9860 Connecticut; Massachusetts; Maine; New Hampshire; Rhode Island

Region 2 - 212 / 337-2378

New York; New Jersey

Region 3 - 215 / 861-4900

DC; Delaware; Pennsylvania; West Virginia

Region 4 - 404 / 562-2300 Alabama; Florida; Georgia; Mississippi

Region 5 - 312 / 353-2220 Illinois; Ohio; Wisconsin

Region 6 - 214 / 767-4731 Arkansas; Louisiana; Oklahoma; Texas

Region 7 - 816 / 426-5861 Kansas; Missouri; Nebraska

Region 8 - 303 / 844-1600 Colorado; Montana; North Dakota; South Dakota

**Region 9 - 415 / 975-4310** 

Region 10 - 206 / 553-5930 *Idaho* 

#### State Plan States

Alaska - 907 / 269-4957

Arizona - 602 / 542-5795

California - 415 / 703-5100

\*Connecticut - 860 / 566-4380

Hawaii - 808 / 586-9100

Indiana - 317 / 232-2688

Iowa - 515 / 281-3661

Kentucky - 502 / 564-3070

Maryland - 410 / 527-4465

Michigan - 517 / 322-1848

Minnesota - 651 / 284-5050

Nevada - 702 / 486-9020

\*New Jersey - 609 / 984-1389

New Mexico - 505 / 827-4230

\*New York - 518 / 457-2574

North Carolina - 919 / 807-2875

Oregon - 503 / 378-3272

Puerto Rico - 787 / 754-2172

South Carolina - 803 / 734-9669

Tennessee - 615 / 741-2793

Utah - 801 / 530-6901

Vermont - 802 / 828-2765

Virginia - 804 / 786-6613

Virgin Islands - 340 / 772-1315

Washington - 360 / 902-5554

Wyoming - 307 / 777-7786

\*Public Sector only



## Appendix C

Material Safety Data Sheets (MSDS)



### **BARIUM**

CAS # 7440-39-3

### Division of Toxicology and Human Health Sciences ToxFAQs<sup>TM</sup>

**June 2013** 

This fact sheet answers the most frequently asked health questions (FAQs) about barium and barium compounds. For more information, call the ATSDR Information Center at 1-800-232-4636. This fact sheet is one in a series of summaries about hazardous substances and their health effects. It is important you understand this information because these substances may harm you. The effects of exposure to any hazardous substance depend on the dose, the duration, how you are exposed, personal traits and habits, and whether other chemicals are present.

HIGHLIGHTS: Exposure to barium occurs mostly in the workplace or from drinking contaminated water. Ingesting drinking water containing levels of barium above the EPA drinking water guidelines for relatively short periods of time can cause gastrointestinal disturbances and muscle weakness. Ingesting high levels for a long time can damage the kidneys. Barium and barium compounds have been found in at least 798 of the 1,684 National Priority List sites identified by the Environmental Protection Agency (EPA).

#### What is barium?

Barium is a silvery-white metal which exists in nature only in ores containing mixtures of elements. It combines with other chemicals such as sulfur or carbon and oxygen to form barium compounds.

Barium compounds are used by the oil and gas industries to make drilling muds. Drilling muds make it easier to drill through rock by keeping the drill bit lubricated. They are also used to make paint, bricks, ceramics, glass, and rubber.

Barium sulfate is sometimes used by doctors to perform medical tests and to take x-rays of the gastrointestinal tract.

# What happens to barium when it enters the environment?

- □ Barium gets into the air during the mining, refining, and production of barium compounds, and from the burning of coal and oil.
- ☐ The length of time that barium will last in air, land, water, or sediments depends on the form of barium released.
- □ Barium compounds, such as barium sulfate and barium carbonate, which do not dissolve well in water, can last a long time in the environment.
- □ Barium compounds, such as barium chloride, barium nitrate, or barium hydroxide, that dissolve easily in water usually do not last in these forms for a long time in the environment. The barium in these compounds that is dissolved in water quickly combines with sulfate or carbonate that are naturally found in water and become

- the longer lasting forms (barium sulfate and barium carbonate).
- ☐ Fish and aquatic organisms can accumulate barium.

#### How might I be exposed to barium?

- □ Ingesting small amounts present in your food and water or breathing air containing very low levels of barium.
- □ Living in areas with unusually high natural levels of barium in the drinking water.
- □ Working in a job that involves barium production or use.
- ☐ Living or working near waste sites where barium has been disposed of.

### How can barium affect my health?

The health effects of the different barium compounds depend on how well the compound dissolves in water or in the stomach contents. Barium compounds that do not dissolve well, such as barium sulfate, are not generally harmful.

Barium has been found to potentially cause gastrointestinal disturbances and muscular weakness when people are exposed to it at levels above the EPA drinking water standards for relatively short periods of time. Some people who eat or drink amounts of barium above background levels found in food and water for a short period may experience vomiting, abdominal cramps, diarrhea, difficulties in breathing, increased or decreased blood pressure, numbness around the face, and muscle weakness. Eating or drinking very large amounts of barium compounds that easily dissolve can cause changes in heart

### ToxFAQs<sup>TM</sup> Internet address is http://www.atsdr.cdc.gov/toxfaqs/index.asp

rhythm or paralysis and possibly death. Animals that drank barium over long periods had damage to the kidneys, decreases in body weight, and some died.

### How likely is barium to cause cancer?

The Department of Health and Human Services (DHHS) and the International Agency for Research on Cancer (IARC) have not classified barium as to its carcinogenicity. The EPA has determined that barium is not likely to be carcinogenic to humans following ingestion and that there is insufficient information to determine whether it will be carcinogenic to humans following inhalation exposure.

#### How can barium affect children?

We do not know whether children will be more or less sensitive than adults to barium toxicity. A study in rats that swallowed barium found a decrease in newborn body weight; we do not know if a similar effect would be seen in humans.

### How can families reduce the risk of exposure to barium?

☐ The greatest potential source of barium exposure is through food and drinking water. However, the amount of barium in foods and drinking water are typically too low to be of concern.

# Is there a medical test to determine whether I've been exposed to barium?

There is no routine medical test to determine whether you have been exposed to barium. Doctors can measure barium in body tissues and fluids, such as bones, blood, urine, and feces, using very complex instruments. These tests cannot be used to predict the extent of the exposure or potential health effects.

The geometric mean barium level measured in the U.S. general population aged 6 and older is reported by the Centers for Disease Control and Prevention (CDC) as 1.56  $\mu$ g/g creatinine (measured in urine).

# Has the federal government made recommendations to protect human health?

The EPA has set a limit of 2.0 milligrams of barium per liter of drinking water (2.0 mg/L), which is the same as 2 ppm.

The Occupational Safety and Health Administration (OSHA) has set Permissible Exposure Limits (PELs) of 0.5 milligrams of soluble barium compounds per cubic meter of workplace air (0.5 mg/m³) for 8 hour shifts and 40 hour work weeks. The OSHA limits for barium sulfate dust are 15 mg/m³ of total dust and 5 mg/m³ for respirable fraction.

The National Institute for Occupational Safety and Health (NIOSH) has set Recommended Exposure Limits (RELs) of 0.5 mg/m³ for soluble barium compounds. The NIOSH has set RELs of 10 mg/m³ (total dust) for barium sulfate and 5 mg/m³ (respirable fraction).

#### References

Agency for Toxic Substances and Disease Registry (ATSDR). 2007. Toxicological Profile for Barium and Compounds (*Update*). Atlanta, GA: U.S. Department of Public Health and Human Services, Public Health Service.

http://www.cdc.gov/exposurereport/pdf/FourthReport\_Upd atedTables Sep2012.pdf

Where can I get more information? For more information, contact the Agency for Toxic Substances and Disease Registry, Division of Toxicology and Human Health Sciences, 1600 Clifton Road NE, Mailstop F-57, Atlanta, GA 30333. Phone: 1-800-232-4636, FAX: 770-488-4178. ToxFAQs Internet address via WWW is http://www.atsdr.cdc.gov/toxfaqs/index.asp. ATSDR can tell you where to find occupational and environmental health clinics. Their specialists can recognize, evaluate, and treat illnesses resulting from exposure to hazardous substances. You can also contact your community or state health or environmental quality department if you have any more questions or concerns.



# Lead - ToxFAQs™

### What is lead?

Lead is a metal found naturally in the earth's crust. It can be found in all parts of our environment, including air, water, and soil. Lead can combine with other chemicals to make different compounds.



Lead is used in the production of batteries, ammunition, and metal products (solder and pipes). Because of health concerns, the use of lead in paints, ceramic products, caulking, and pipe solder has been dramatically reduced. The use of lead as an additive to automobile gasoline was banned in 1996 in the United States.

### What happens to lead in the environment?

- Lead is an element, so it does not break down.
- When lead is released into the air, it may be transported long distances before it lands and stays on the ground.
- Once on the ground, lead can often stick to soil particles.
- Lead in soil can get into groundwater, but the amount of lead that moves into groundwater will depend on the lead compound and soil type.

### How can I be exposed to lead?

- Eating food or drinking water that contains lead.
- Drinking water from pipes that were soldered with lead can cause exposure.
- Spending time or living in homes with lead-based paints can result in exposure when the paint breaks down and forms dust, which can get on your hands, or into your mouth and nose and be swallowed.
- Lead can cause health problems in almost every organ and system in your body.
- Spending time in areas where the soil is contaminated with lead.
- Working in a job where lead is used or participating in certain hobbies where lead is used, such as making stained glass.
- Using healthcare products from other countries, alternative treatments, or folk remedies.

### How can lead affect my health?

The effects of lead are the same whether it enters the body by breathing it in or eating it. Lead can affect almost every organ and system in your body. The nervous system is the main target for lead poisoning in children and adults. Long-term exposure can result in decreased learning, memory, and attention, and weakness in fingers, wrists, or ankles. Lead exposure can cause anemia (low iron in the blood) and damage to the kidneys. It can also cause increases in blood pressure, particularly in middle-aged and older individuals. Exposure to high lead levels can severely damage the brain and kidneys and can cause death. In pregnant women, exposure to high levels of lead may cause a miscarriage. In men, it can cause damage to reproductive organs.



### Lead

### How can lead affect children?

Children are more vulnerable to lead poisoning than adults because their nervous system is still developing. Children can be exposed to lead in their environment and before birth from lead in their mother's body. At lower levels of exposure, lead can decrease mental development, especially learning, intelligence, and behavior. Physical growth may also be decreased. A child who swallows large amounts of lead may develop anemia, severe stomachache, muscle weakness, and brain damage. Exposure to lead during pregnancy can also result in premature births. Some effects of lead poisoning in a child may continue into adulthood.

### Can lead cause cancer?

Several agencies and organizations both in the United States and internationally have reviewed studies and made an assessment about whether lead can cause cancer.

- The Department of Health and Human Services (HHS) has determined that lead and lead compounds are reasonably anticipated to be human carcinogens (causing cancer in people).
- The U.S. Environmental Protection Agency (EPA) has classified lead as a probable human carcinogen.
- The International Agency for Research on Cancer (IARC) has determined that inorganic lead is probably carcinogenic to humans, and that there is insufficient information to determine whether organic lead compounds will cause cancer in humans.

### Can I get a medical test to check for lead?

A blood test is available to measure the amount of lead in your blood. Blood tests are commonly used to screen children for lead poisoning. Your doctor can draw blood samples and send them to appropriate laboratories for analysis. If you think you or anyone in your family has been exposed to lead, contact your doctor, nurse, or poison control center.

### How can I protect my family from lead exposure?

- Avoid exposure to sources of lead.
- Do not allow children to chew or mouth surfaces that may have been painted with lead-based paint.
- If your home contains lead-based paint (built before 1978), or if you live in an area contaminated with lead, wash children's hands and faces often to remove lead dusts and soil, and regularly clean the house to remove lead dust and lead tracked in soil.
- Certain water pipes may contain lead, so if you know that pipes have lead solder, you should avoid drinking from that source.
- Check for lead in some products such as toys and jewelry and avoid such products.
- Lead is sometimes in candies imported from other countries or traditional home remedies; find out if yours has any lead and avoid using these products or giving them to children.
- You can learn more about preventing lead poisoning here: <a href="https://www.cdc.gov/nceh/lead/faqs/lead-faqs.htm">https://www.cdc.gov/nceh/lead/faqs/lead-faqs.htm</a>

### Want more information?

Call **CDC-INFO** at 1-800-232-4636, or submit your question online at <a href="https://wwwn.cdc.gov/dcs/ContactUs/Form">https://wwwn.cdc.gov/dcs/ContactUs/Form</a> Go to ATSDR's Toxicological Profile for Lead

CDC Lead Poisoning Prevention Program <a href="https://www.cdc.gov/nceh/lead/default.htm">https://www.cdc.gov/nceh/lead/default.htm</a>

Environmental Protection Agency <a href="https://www.epa.gov/lead/protect-your-family-exposures-lead">https://www.epa.gov/lead/protect-your-family-exposures-lead</a>

Go to ATSDR's Toxic Substances Portal: <a href="https://wwwn.cdc.gov/TSP/index.aspx">https://wwwn.cdc.gov/TSP/index.aspx</a>

If you have any more questions or concerns, you can also find & contact your ATSDR Regional Representative at <a href="http://www.atsdr.cdc.gov/DRO/dro\_org.html">http://www.atsdr.cdc.gov/DRO/dro\_org.html</a>

August 2020 Page 2 of 2

# Polycyclic Aromatic Hydrocarbons (PAHs) - ToxFAQs™

This fact sheet answers the most frequently asked health questions (FAQs) about polycyclic aromatic hydrocarbons (PAHs). For more information, call the CDC Information Center at 1-800-232-4636. This fact sheet is one in a series of summaries about hazardous substances and their health effects. This information is important because this substance may harm you. The effects of exposure to any hazardous substance depend on the dose, the duration, how you are exposed, personal traits and habits, and whether other chemicals are present.

HIGHLIGHTS: Exposure to polycyclic aromatic hydrocarbons usually occurs by breathing air contaminated by wild fires or coal tar, or by eating foods that have been grilled. PAHs have been found in at least 600 of the 1,430 National Priorities List (NPL) sites identified by the Environmental Protection Agency (EPA).

# What are polycyclic aromatic hydrocarbons?

(Pronounced pŏl'ĭ-sī'klĭk ăr'ə-măt'ĭk hī'drə-kar'bənz)

Polycyclic aromatic hydrocarbons (PAHs) are a group of over 100 different chemicals that are formed during the incomplete burning of coal, oil and gas, garbage, or other organic substances like tobacco or charbroiled meat. PAHs are usually found as a mixture containing two or more of these compounds, such as soot.

Some PAHs are manufactured. These pure PAHs usually exist as colorless, white, or pale yellow-green solids. PAHs are found in coal tar, crude oil, creosote, and roofing tar, but a few are used in medicines or to make dyes, plastics, and pesticides.

# What happens to PAHs when they enter the environment?

- PAHs enter the air mostly as releases from volcanoes, forest fires, burning coal, and automobile exhaust.
- PAHs can occur in air attached to dust particles.
- Some PAH particles can readily evaporate into the air from soil or surface waters.
- PAHs can break down by reacting with sunlight and other chemicals in the air, over a period of days to weeks.
- PAHs enter water through discharges from industrial and wastewater treatment plants.

- Most PAHs do not dissolve easily in water. They stick to solid particles and settle to the bottoms of lakes or rivers.
- Microorganisms can break down PAHs in soil or water after a period of weeks to months.
- In soils, PAHs are most likely to stick tightly to particles; certain PAHs move through soil to contaminate underground water.
- PAH contents of plants and animals may be much higher than PAH contents of soil or water in which they live.

### How might I be exposed to PAHs?

- Breathing air containing PAHs in the workplace of coking, coal-tar, and asphalt production plants; smokehouses; and municipal trash incineration facilities.
- Breathing air containing PAHs from cigarette smoke, wood smoke, vehicle exhausts, asphalt roads, or agricultural burn smoke.
- Coming in contact with air, water, or soil near hazardous waste sites.
- Eating grilled or charred meats; contaminated cereals, flour, bread, vegetables, fruits, meats; and processed or pickled foods.
- Drinking contaminated water or cow's milk.
- Nursing infants of mothers living near hazardous waste sites may be exposed to PAHs through their mother's milk.



### **Polycyclic Aromatic Hydrocarbons**

### How can PAHs affect my health?

Mice that were fed high levels of one PAH during pregnancy had difficulty reproducing and so did their offspring. These offspring also had higher rates of birth defects and lower body weights. It is not known whether these effects occur in people.

Animal studies have also shown that PAHs can cause harmful effects on the skin, body fluids, and ability to fight disease after both short- and long-term exposure. But these effects have not been seen in people.

### How likely are PAHs to cause cancer?

The Department of Health and Human Services (DHHS) has determined that some PAHs may reasonably be expected to be carcinogens.

Some people who have breathed or touched mixtures of PAHs and other chemicals for long periods of time have developed cancer. Some PAHs have caused cancer in laboratory animals when they breathed air containing them (lung cancer), ingested them in food (stomach cancer), or had them applied to their skin (skin cancer).

# Is there a medical test to show whether I've been exposed to PAHs?

In the body, PAHs are changed into chemicals that can attach to substances within the body. There are special tests that can detect PAHs attached to these substances in body tissues or blood. However, these tests cannot tell whether any health effects will occur or find out the extent or source of your exposure to the PAHs. The tests aren't usually available in your doctor's office because special equipment is needed to conduct them.

# Has the federal government made recommendations to protect human health?

The Occupational Safety and Health Administration (OSHA) has set a limit of 0.2 milligrams of PAHs per cubic meter of air (0.2 mg/m³). The OSHA Permissible Exposure Limit (PEL) for mineral oil mist that contains PAHs is 5 mg/m³ averaged over an 8-hour exposure period.

The National Institute for Occupational Safety and Health (NIOSH) recommends that the average workplace air levels for coal tar products not exceed 0.1 mg/m³ for a 10-hour workday, within a 40-hour workweek. There are other limits for workplace exposure for things that contain PAHs, such as coal, coal tar, and mineral oil.

### **Glossary**

Carcinogen: A substance that can cause cancer.

Ingest: Take food or drink into your body.

### References

Agency for Toxic Substances and Disease Registry (ATSDR). 1995. Toxicological profile for polycyclic aromatic hydrocarbons. Atlanta, GA: U.S. Department of Health and Human Services, Public Health Service.

### Where can I get more information?

For more information, contact the Agency for Toxic Substances and Disease Registry, Division of Toxicology and Human Health Sciences, 1600 Clifton Road NE, Mailstop F-57, Atlanta, GA 30329-4027.

Phone: 1-800-232-4636.

ToxFAQs™ Internet address via WWW is http://www.atsdr.cdc.gov/toxfaqs/index.asp.

ATSDR can tell you where to find occupational and environmental health clinics. Their specialists can recognize, evaluate, and treat illnesses resulting from exposure to hazardous substances. You can also contact your community or state health or environmental quality department if you have any more questions or concerns.

September 1996 Page 2 of 2

# Tetrachloroethylene - ToxFAQs™

CAS # 127-18-4

This fact sheet answers the most frequently asked health questions (FAQs) about tetrachloroethylene. For more information, call the ATSDR Information Center at 1-800-232-4636. This fact sheet is one in a series of summaries about hazardous substances and their health effects. It is important you understand this information because this substance may harm you. The effects of exposure to any hazardous substance depend on the dose, the duration, how you are exposed, personal traits and habits, and whether other chemicals are present.

HIGHLIGHTS: Tetrachloroethylene is a manufactured chemical used for dry cleaning and metal degreasing and in the aerospace industry. Exposure to very high concentrations of tetrachloroethylene can cause dizziness headaches, sleepiness, incoordination confusion, nausea, unconsciousness, and even death. Tetrachloroethylene has been found in at least 949 of the 1,854 National Priorities List sites identified by U.S. Environmental Protection Agency (EPA).

### What is tetrachloroethylene?

Tetrachloroethylene is a nonflammable colorless liquid. Other names for tetrachloroethylene include perchloroethylene, PCE, perc, tetrachloroethene, and perchlor. Most people can smell tetrachloroethylene when it is present in the air at a level of 1 part in 1 million parts of air (1 ppm) or more.

Tetrachloroethylene is used as a dry cleaning agent and metal degreasing solvent. It is also used as a starting material (building block) for making other chemicals and is used in some consumer products.

# What happens to tetrachloroethylene when it enters the environment?

- Tetrachloroethylene can be released into air, water, and soil at places where it is produced or used.
- Tetrachloroethylene breaks down very slowly in the air and so it can be transported long distances in the air.
   Half of the amount in the air will degrade in approximately 100 days.
- Tetrachloroethylene evaporates quickly from water into air. It is generally slow to break down in water.
- Tetrachloroethylene may evaporate quickly from shallow soils or may filter through the soil and into the groundwater below. It is generally slow to break down in soil.

### How might I be exposed to tetrachloroethylene?

- When you bring clothes from the dry cleaners, they will release small amounts of tetrachloroethylene into the air.
- When you drink water containing tetrachloroethylene, you are exposed to it. You might also be exposed to tetrachloroethylene that is released into the air during showering and bathing.
- People residing near contaminated sites or dry cleaning locations may be exposed to higher levels than the general population.
- People working in the dry cleaning industries or using metal degreasing products may be exposed to elevated levels of tetrachloroethylene.

### How can tetrachloroethylene affect my health?

Breathing high levels of tetrachloroethylene for a brief period may cause dizziness or drowsiness, headache, and incoordination; higher levels may cause unconsciousness and even death.

Exposure for longer periods to low levels of tetrachloroethylene may cause changes in mood, memory, attention, reaction time, and vision.

Studies in animals exposed to tetrachloroethylene have shown liver and kidney effects, and changes in brain chemistry, but we do not know what these findings mean for humans.



### **Tetrachloroethylene**

### CAS # 127-18-4

# How likely is tetrachloroethylene to cause cancer?

Studies in humans suggest that exposure to tetrachloroethylene might lead to a higher risk of getting bladder cancer, multiple myeloma, or non-Hodgkin's lymphoma.

In animals, tetrachloroethylene has been shown to cause cancers of the liver, kidney, and blood system.

The Department of Health and Human Services (DHHS) considers tetrachloroethylene to be reasonably anticipated to be a human carcinogen. EPA considers tetrachloroethylene likely to be carcinogenic to humans by all routes of exposure. The International Agency for Research on Cancer (IARC) considers tetrachloroethylene probably carcinogenic to humans.

### How can tetrachloroethylene affect children?

It is not known whether children are more susceptible than adults to the effects of tetrachloroethylene.

A few studies in humans have suggested that exposure to tetrachloroethylene increased the numbers of babies with birth defects, but these studies were not large enough to clearly answer the question. Studies in animals exposed by inhalation or stomach tube have not shown clear evidence of specific birth defects.

# How can families reduce the risk of exposure to tetrachloroethylene?

- Tetrachloroethylene has been found in low levels in some food. You can minimize the risk of your family's exposure by peeling and thoroughly washing fruits and vegetables before cooking.
- Use bottled water if you have concerns about the presence of tetrachloroethylene in your tap water. You may also contact local drinking water authorities and follow their advice.

- Prevent children from playing in dirt or eating dirt if you live near a waste site that has tetrachloroethylene.
- Tetrachloroethylene is widely used as a scouring solvent that removes oils from fabrics, as a carrier solvent, as a fabric finish or water repellant, and as a metal degreaser/cleaner. Follow instructions on product labels to minimize exposure to tetrachloroethylene.

# Is there a medical test to determine whether I've been exposed to tetrachloroethylene?

Tetrachloroethylene and its breakdown products (metabolites) can be measured in blood and urine. However, the detection of tetrachloroethylene or its metabolites cannot predict the kind of health effects that might develop from that exposure. Because tetrachloroethylene and its metabolites leave the body fairly rapidly, the tests need to be conducted within days after exposure.

# Has the federal government made recommendations to protect human health?

The Occupational Safety and Health Administration (OSHA) has set an 8-hour time weighted average permissible exposure limit of 100 ppm, an acceptable ceiling exposure limit of 200 ppm, and a maximum peak of 300 ppm (not to be exceeded for more than 5 minutes of any 3-hour period).

The National Institute for Occupational Safety and Health (NIOSH) recommends that workplace exposure to tetrachloroethylene be minimized due to concerns about its carcinogenicity.

#### Reference

This ToxFAQs™ information is taken from the 2019
Toxicological Profile for Tetrachloroethylene produced by
the Agency for Toxic Substances and Disease Registry,
Public Health Service, U.S. Department of Health and
Human Services, Public Health Service in Atlanta, GA.

### Where can I get more information?

For more information, contact the Agency for Toxic Substances and Disease Registry, Division of Toxicology and Human Health Sciences, 1600 Clifton Road NE, Mailstop F-57, Atlanta, GA 30329-4027.

Phone: 1-800-232-4636

ToxFAQs<sup>™</sup> on the web: www.atsdr.cdc.gov/ToxFAQs

ATSDR can tell you where to find occupational and environmental health clinics. Their specialists can recognize, evaluate, and treat illnesses resulting from exposure to hazardous substances. You can also contact your community or state health or environmental quality department if you have any more questions or concerns.

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# Appendix D Community Air Monitoring Plan

### 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

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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.

- 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.
- 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.
- All 15-minute readings must be recorded and be available for State (DEC and NYSDOH) 4. 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.

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- If the downwind PM-10 particulate level is 100 micrograms per cubic meter (mcg/m<sup>3</sup>) 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/m<sup>3</sup> above the upwind level and provided that no visible dust is migrating from the work area.
- If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than 150 mcg/m<sup>3</sup> 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/m<sup>3</sup> of the upwind level and in preventing visible dust migration.
- All readings must be recorded and be available for State (DEC and NYSDOH) and County Health personnel to review.

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# 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.
- 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.

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.

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