Soil Investigation for Polyfluoroalkyl Substances
Work Plan

For

1 Shore Road, Glenwood Landing, New York 11547

SHORE REALTY SITE

NPL SITE NO. 13006

Prepared for:

Law Offices of Theodore W. Firetog

111 Thomas Powell Boulevard

Farmingdale, New York 11735-2251

On behalf of its client: Woodstock Construction Group

Prepared by:

Laurel Environmental Geosciences, D.P.C.

53 West Hills Road, Suite #1, Huntington Station, New York 11746

Project Number 21-215

Jamie Burgher
Project Manager, Geologist III

March 2023
Date

Introduction

Laurel Environmental Geosciences, D.P.C. ("***Laurel"***) was retained by Woodstock Construction Group, Ltd. to conduct a soil investigation for polyfluoroalkyl substances ("PFAS") on the industrial property located at 1 Shore Road, Glenwood Landing, New York ("Site").

The Site is currently undergoing remediation by the New York State Department of Environmental Conservation ("NYSDEC") with oversight provided by the United States Environmental Protection Agency ("USEPA"). ***Laurel*** previously issued a Waste Characterization Sampling Investigation report dated October 18, 2021, that documented the presence of perfluorooctanesulfonic acid ("PFOS") in subsurface soils at a concentration of 1.33 micrograms per kilogram ("µg/kg"), which is above the NYSDEC Part 375 Remedial Guidance Value for Unrestricted Use Soils (November 2022) at 0.88 µg/kg. Perfluorooctanoic acid ("PFOA") was also detected at 0.39 µg/kg, which is below the NYSDEC Part 375 Remedial Guidance Value for Unrestricted Use Soils (November 2022) at 0.66 µg/kg.

NYSDEC subsequently conducted a site-wide groundwater sampling event for PFAS, including two (2) rounds of sampling in June 2022 and October 2022. The groundwater sampling event documented PFOS and PFOA in groundwater above the NYSDEC proposed guidance value for Class GA Groundwater at 10 nanograms per liter ("ng/L") in fourteen (14) of eighteen (18) sampled monitoring wells. Concentrations of PFOS and PFOA were generally highest in the western area of the Site, near the recently conducted remedial excavations. The NYSDEC has not made a conclusion to date regarding the potential for on-site source areas for PFAS contamination.

Based on the PFAS findings available to date, ***Laurel*** has developed this work plan to investigate the potential for PFAS contamination to exist in subsurface soils within the areas of concern at the Site.

Subsurface Soil Investigation

A geophysical survey will be conducted to pre-clear soil sampling locations. An 800/300 MHz dual frequency Ground Penetrating Radar ("GPR") unit along with line locating equipment will be utilized for the geophysical survey. The geophysical survey is being conducted to ensure the safety of the operators, field staff, and prevent damaging the on-site remediation system.

A Community Air Monitoring Program ("CAMP") is required during ground intrusive activities at the Site. CAMP monitoring will include, at minimum, the use of an upwind and a downwind monitoring station capable of measuring dust particulate matter and volatile organic compounds ("VOCs") in accordance with New York State Department of Health ("NYSDOH") requirements. Daily reports will be produced at the end of each day and submitted to the NYSDEC and USEPA project managers. Daily reports will include, at minimum, a summary of ground intrusive work conducted on that day, a site sketch showing sample locations, any CAMP exceedances, and representative site photographs. The full CAMP document is included as Appendix A of this work plan.

For the purposes of this investigation, the Site has been split into nine (9) grids, each measuring approximately 100 feet by 100 feet, covering an area of the Site totaling approximately 90,000 square feet. The grids are biased towards areas where PFAS may have been stored or utilized in the past, such as around the former major oil storage tanks, and in the areas where subsurface contamination has historically been identified. The grids are shown on Figure 1.0.

Laurel proposes to collect a total of forty-four (44) soil samples for PFAS analysis at twenty-two (22) locations. At each sampling location, one (1) sample will be collected from the shallow soils (i.e., 1 to 3 feet bgs), and one (1) sample from the deep soils (i.e., the 2-foot interval immediately above the groundwater interface); groundwater at the Site is expected to range from 15 feet bgs to 5 bgs in the area of this investigation based on historic sampling conducted at the Site. To construct a representative sample of the subsurface soils, the soils in the targeted sampling interval will be composited in a stainless steel bowl prior to being transferred into a laboratory-supplied sampling container. The stainless steel bowl will be decontaminated with a laboratory-grade detergent (i.e., Alconox®) and deionized water before each use. The proposed sampling locations are shown on Figure 2.0. Samples are summarized in Table I below.

Samples will be collected via track mounted GeoProbe® unit advancing continuous soil borings to the stated depths. Damage and/or removal of existing in-land trees will be required to access the proposed sampling areas. Full access to the Site is required to ensure that the subsurface soils are fully characterized with no data gaps. The continuous soil cores will be field screened with a photoionization device ("PID") and logged in accordance with the United Soil Classification System ("USCS"). Soil boring logs will be constructed for inclusion in the final report. Any grossly impacted soils will be temporarily stored on-Site in a 55-gallon drum, sampled for waste characterization purposes, and subsequently disposed of in accordance with all applicable local, state, and federal regulations.

Laboratory Analysis

The soil samples will be submitted to York Analytical Laboratories Inc., a New York State Department of Health ("NYSDOH") certified Environmental Laboratory. The samples will be analyzed utilizing USEPA Method 1633 for PFAS.

Quality Assurance/Quality Control Procedures

QA/QC procedures will be used to provide performance information regarding accuracy, precision, sensitivity, representation, completeness, and comparability associated with the sampling and analysis for this investigation. Field QA/QC procedures will be used (1) to document that samples are representative of actual conditions at the Site and (2) identify possible cross-contamination from field activities. Any field equipment utilized during sampling and not considered to be disposable will be decontaminated with a laboratory-grade detergent between sampling events. The full Quality Assurance Project Plan ("QAPP") is included as Appendix B of this work plan.

Blind duplicate and matrix spike/matrix spike duplicate ("MS/MSD") samples will be collected at a rate of one (1) per twenty (20) samples collected. Equipment blank samples will be collected at a rate of one (1) per twenty (20) samples collected, with a minimum of one (1) blank collected per field day.

Reporting

A "Soil Investigation for Polyfluoroalkyl Substances Report" will be prepared following completion of the field activities and receipt of the laboratory data. The report will provide detailed summaries of the investigative findings. Soil analytical results will be compared to the following proposed standards or guidance values: NYSDEC Part 375 Remedial Guidance Values for Unrestricted Use Soils, NYSDEC Part 375 Remedial Guidance Values for Protection of Groundwater, NYSDEC Part 375 Remedial Guidance Values for Restricted Commercial Use, USEPA Regional Screening Levels for Residential and Industrial Properties. The report will include analytical data tables for all reported constituent compounds (including non-detectable concentrations) and will note any deviations to this work plan.

Table I
Sampling Matrix

Sample Type	Sample Designation	Matrix	# of Samples	Analysis	Data Reporting	Sample Bottles/Preservation	Holding Time
Composite	SB101 through SB122 (1-3') and (TBD)	Soil	Forty-four (44)	1633 for PFAS	Summary	1x – 250mL HDPE	48 hours to freeze
Composite QA/QC	Duplicate	Soil	Two (2)	1633 for PFAS	Summary	1x – 250ml HDPE	48 hours to freeze
Composite QA/QC	MS/MSD	Soil	Three (3)	1633 for PFAS	Summary	3x – 250ml HDPE	48 hours to freeze
QA/QC	Equipment Blank	DI Water	Two (2)	1633 for PFAS	Summary	3x – 250ml HDPE	48 hours to freeze

APPENDIX A

Community Air Monitoring Plan

COMMUNITY AIR MONITORING PLAN

SHORE REALTY SITE
1 SHORE ROAD, GLENWOOD LANDING, NEW YORK
NPL SITE #13006

MARCH 20, 2023
***LAUREL* PROJECT # 21-215**

PREPARED FOR:

LAW OFFICES OF THEODORE W. FIRETOG
111 THOMAS POWELL BOULEVARD
FARMINGDALE, NEW YORK, 11735-2251
On behalf its client: Woodstock Construction Group

PREPARED BY:

LAUREL ENVIRONMENTAL GEOSCIENCES, DPC.
53 WEST HILLS ROAD, SUITE 1 HUNTINGTON STATION, NEW YORK

COMMUNITY AIR MONITORING PLAN
FOR USE DURING INVESTIGATIVE AND REMEDIAL ACTIONS

Due to the nature of the potential contaminants at the property referred to as 1 Shore Road, Glenwood Landing, New York ("Site"), real-time air monitoring for volatile organic compounds ("VOCs") and/or particulate levels at the perimeter of the work area is necessary. The scope of work for the Site will require VOC and particulate monitoring. Due to the proximity of the Site to residential properties, continuous air monitoring will be completed during all ground intrusive activities. For this program, ground intrusive activities include, but are not limited to, drilling.

Periodic monitoring for VOCs will be completed during all non-ground intrusive activities, such as purging and sample collection from groundwater monitoring wells. "Periodic" monitoring during sample collection might reasonably consist of taking a reading upon arrival at a sample location, monitoring while conducting ground intrusive activities, 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. All CAMP readings will be submitted for New York State Department of Environmental Conservation ("NYSDEC") personnel to review on a weekly basis or as soon as possible if/when an exceedance occurs. Work is anticipated to be completed in less than one (1) week. All data from the CAMP will be included in the Soil Investigation for Polyfluoroalkyl Substances Report to be prepared upon receipt of final laboratory data.

VOC Monitoring, Response Levels, and Actions

VOCs will be monitored at the at the downwind and upwind perimeter of the work zone on a continuous basis during ground intrusive activities. The monitoring work will be performed using equipment appropriate to measure the types of contaminants known or suspected to be present (e.g., photoionization devices ("PIDs") and particulate monitoring stations). As applicable, the equipment will be calibrated at least daily for the contaminant(s) of concern or for an appropriate surrogate. The equipment will be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below. Prior to work beginning for the day, the equipment will be setup and allow to run for a 15-minute period to allow for background readings to be recorded.

- If the ambient air concentration of total organic vapors at the downwind perimeter exceeds 5 parts per million ("ppm") above background for the 15-minute average, work activities will be temporarily halted and monitoring continued. If the total organic

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vapor level readily decreases (per instantaneous readings) below 5 ppm over background, work activities will resume with continued monitoring.

- If total organic vapor levels at the downwind perimeter persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities will cease, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After which, work activities will resume provided that the total organic vapor level 200 feet downwind of the work zone or half the distance to the nearest potential receptor or residential structure, whichever is less - but in no case less than 20 feet, is below 5 ppm over background for the 15-minute average.

- If the organic vapor level is above 25 ppm at the perimeter of the work area, activities must be shutdown.

All 15-minute readings will be recorded and be available for NYSDEC personnel to review. Instantaneous readings used for decision purposes, if any, will also be recorded.

Particulate Monitoring, Response Levels, and Actions

Particulate concentrations will be monitored continuously at the downwind and upwind perimeters of the work zone. The particulate monitoring will 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 will be fitted with an audible alarm or modem with notification to email/text to indicate exceedance of the action level. In addition, fugitive dust migration will be visually assessed during all work activities.

- If the PM-10 particulate level at the downwind perimeter is 150 micrograms per cubic meter ("mcg/m^3") greater than background for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques will be employed. Work will continue with dust suppression techniques provided that the downwind perimeter PM-10 particulate levels do not exceed 150 mcg/m^3 above the background level and provided that no visible dust is migrating from the work area.

- If, after implementation of dust suppression techniques, PM-10 particulate levels at the downwind perimeter of the work zone are greater than 150 mcg/m^3 above the background level, work will be stopped and a re-evaluation of activities initiated. Work

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will resume provided that dust suppression measures and other controls are successful in reducing the PM-10 particulate concentration to within 150 mcg/m3 of the background level and in preventing visible dust migration.

All 15-minute readings will be recorded and be available for NYSDEC personnel to review. Instantaneous readings used for decision purposes, if any, will also be recorded.

VOC and Particulate Monitoring Requirements for Work Within 20 Feet of Potentially Exposed Individuals or Structures

When work areas are within 20 feet of potentially exposed populations or occupied structures, the continuous monitoring locations for VOCs and particulates must reflect the nearest potentially exposed individuals and the location of ventilation system intakes for nearby structures. The use of engineering controls such as vapor/dust barriers, temporary negative-pressure enclosures, or special ventilation devices will be considered to prevent exposures related to the work activities and to control dust and odors.

- If total VOC concentrations opposite the walls of occupied structures or next to intake vents exceed 1 ppm, monitoring should occur within the occupied structure(s). Background readings in the occupied spaces must be taken prior to commencement of the planned work. Any unusual background readings should be discussed with NYSDEC prior to commencement of the work.

- If total particulate concentrations opposite the walls of occupied structures or next to intake vents exceed 150 mcg/m3, work activities should be suspended until controls are implemented and are successful in reducing the total particulate concentration to 150 mcg/m3 or less at the monitoring point.

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APPENDIX B

Quality Assurance Project Plan

QUALITY ASSURANCE PROJECT PLAN

SHORE REALTY SITE
1 SHORE ROAD, GLENWOOD LANDING, NEW YORK
NPL SITE #13006

MARCH 20, 2023
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PREPARED FOR:

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PREPARED BY:

LAUREL ENVIRONMENTAL GEOSCIENCES, DPC.
53 WEST HILLS ROAD, SUITE 1 HUNTINGTON STATION, NEW YORK

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APPENDICES

1.0 INTRODUCTION

This document presents the Quality Assurance Project Plan ("QAPP") for the investigation of polyfluoroalkyl substances ("PFAS") at the Shore Realty Site, located at 1 Shore Road, Glenwood Landing, New York (the "Site"). This QAPP has been prepared to be incorporated into the proposed Soil Investigation for PFAS Work Plan for the Site.

1.1 Site Background and Previous Investigation Summary

As described in the Soil Investigation for PFAS Work Plan, a previous investigation conducted at the Site by ***Laurel Environmental Geosciences, DPC.*** ("***Laurel***") documented the presence of perfluorooctanesulfonic acid ("PFOS") in subsurface soils at a concentration of 1.33 micrograms per kilogram ("µg/kg"), which is above the NYSDEC Part 375 Remedial Guidance Value for Unrestricted Use Soils (November 2022) at 0.88 µg/kg. Perfluorooctanoic acid ("PFOA") was also detected at 0.39 µg/kg, which is below the NYSDEC Part 375 Remedial Guidance Value for Unrestricted Use Soils (November 2022) at 0.66 µg/kg.

1.2 Project Scope and Objective

The proposed Soil Investigation for PFAS intends to characterize subsurface soils and determine the horizontal and lateral extent of any PFAS contamination at the Site. The proposed scope of work for the is described in detail in the work plan and will include collection and laboratory analysis of a forty-four (44) additional subsurface soil samples. Proposed sample locations are shown on Figure 2 of the work plan.

2.0 PROJECT ORGANIZATION

Laurel will be responsible for implementation of the work plan once it has been approved by the NYSDEC. Mr. Jamie Burgher, the Project Manager, will ensure that there are suitable and verifiable data results from sampling and analysis. To achieve this objective, the quality assurance procedures detailed in this section will be followed for all sampling and laboratory analysis activities.

Mr. Brian McCabe, the Project QAO, will review sampling procedures and certify that the data was collected and analyzed using the appropriate procedures. The QAO may not have any responsibilities specific to the collection and analysis of samples from the site for which they are the QAO. The QAO will review the Data Validation and Data Acceptance associated with this project in accordance with the DER-10 DUSR Technical Guidance for Site Investigation and Remediation, Guidance for Data Deliverables and the Development of Data Usability Summary Reports ("DUSR") that will be completed by an independent data validation service. The Data Validation and Data Usability process will encompass Completeness, Compliance and Report Submittal.

Mr. Marc Califano, the Project Health and Safety Officer, will be responsible for reviewing the work plan and assuring that project staff are sufficiently briefed on hazards reasonably expected to be encountered during the investigation.

Resumes for relevant project staff are included in Appendix A of the QAPP.

Laurel will utilize subcontractors for laboratory work, and independent data validation services, as described below.

3.0 SAMPLING, ANALYTICAL, AND QUALITY ASSURANCE PROCEDURES

Samples will be collected using disposable, dedicated sampling equipment where possible. Any non-disposable sampling equipment will be rinsed with a water and Alconox, a laboratory grade detergent, solution to eliminate any cross-contamination between sampling locations.

3.1 Sampling Scope and Analytical Methods

This program includes collection and laboratory analysis of forty-four (44) soil samples. All samples will be analyzed by York Analytical Laboratories, Inc., which is approved under the New York State Department of Health ("NYSDOH") Environmental Laboratory Approval Program ("ELAP") for the required analyses. The reporting limit for perfluorooctanoic acid ("PFOA") and perfluorooctanesulfonic acid ("PFOS") will be 0.200 µg/kg and 0.186 µg/kg in soil, respectively. The remaining PFAS compound reporting limits, along with method detection limits, and standard laboratory procedures, are included in Appendix B of the QAPP. The sampling nomenclature is included in Appendix C of the QAPP. The samples and analyses are detailed in Table 1 below.

Table 1. Sample Summary and Rationale

Medium	Number of Samples	Analysis	Analytical Method	Container	Holding Time	Rationale
Soil	44*	PFASs	1633	250ml HDPE	48 hours to freeze 14 days to extract	Delineation of PFAS in soils

* Excluding QA/QC samples, which will include a blind duplicate sample, MS/MSD set, and equipment blank (at a rate of one per day).

All samples will be analyzed using standard laboratory turnaround time of ten business days, and all data will be provided with an Analytical Services Protocol ("ASP") Category B data package.

3.2 Sampling Procedures

All soil samples will be collected using the direct push method and will be collected using a new, dedicated disposable acetate sleeve. Upon retrieval, the sleeve will be opened and the soil within scanned for total VOCs using a photoionization detector ("PID") and geologically described, including observations regarding potential contamination such as odors, staining, etc. All sample descriptions and field observations will be documented. Soil samples will to be submitted for laboratory analysis will be directly transferred into pre-labeled, laboratory-supplied containers, using new, dedicated, disposable, non-PFAS equipment (e.g., plastic scoops, plastic spoons, wooden tongue depressor, etc.). The sample container for PFAS will be filled first, prior to any additional samples being collected.

Immediately after collection, each PFAS soil sample will be placed into a dedicated PFAS-only cooler and stored on wet ice for sample preservation. All samples will be delivered via laboratory courier under chain of custody procedures and will arrive at the laboratory within 48 hours after collection.

No equipment, supplies, or field clothing containing low-density polyethylene, Teflo®, Gore-Tex®, or Tyvek® will be used during the sampling program, due to the presence of PFAS compounds in these materials. Field personnel will not wear clothes having been laundered using fabric softener, nor will they use cosmetics, moisturizers, hand cream, or other personal care products (sunscreen, insect repellent, etc.) that may contain PFAS compounds. No eating or drinking will be allowed at the Site, except that bottled water and hydration drinks can be consumed, but only in the support area (i.e., away from areas where sample collection is occurring). In addition, notes taken in the field will not be recorded in a notebook with a water-resistant coating, no plastic clipboards or notebooks will be utilized, no aluminum foil or adhesives (e.g., sticky notes) will be used during sample collection, and sample labels and the Chain of Custody form will be completed using ballpoint pens (i.e., not permanent markers).

3.3 Quality Assurance/Quality Control Samples

In accordance with ASP and NYSDEC requirements, QA/QC samples will include blind duplicate samples, matrix spike/matrix spike duplicate ("MS/MSD") sets, and equipment blanks (the equipment blank water will be provided by the laboratory, and will be certified as "PFAS-free") for each sampled medium (i.e., soil). See Table 2 for a summary of QA/QC samples.

Table 2. QA/QC Sample Summary

Matrix Type	Field Parameters	Lab. Parameters	Analytical Methods	Sample Preservation	Sample Containers	Holding Time	Field Duplicates	Equipment Blanks	MS/MSD Samples
Soil	Total VOCs via PID	PFAS	EPA 1633	Cool to 4° C	One (1) 250mL HDPE	48 hours to freeze, 14 days to extract	1 per 20 samples (minimum 1)	1 per 20 samples, minimum once per day	1 per 20 samples (minimum 1)

3.4 Data Validation and Data Usability Summary Report

In accordance with NYSDEC requirements, the ASP Category B data packages generated for this project will be submitted for independent data validation, by a NYSDEC-approved, third-party data validator. The data validation process will include, at a minimum, review of sample custody documentation, instrument calibration results, surrogate and spike recovery data, chromatograms, raw data files, duplicate results, blank results, and internal standards. The results and findings of the data validation process will be documented in a Data Usability Summary Report ("DUSR").

QAPP
APPENDIX A

Project Personnel Resumes

JAMIE BURGHER

EDUCATION: STONY BROOK UNIVERSITY
B.S., Geology, May 2016
Departmental Honors, Magna Cum Laude

RELATED COURSES:

Field Geology, Geophysical Survey Methods, Introduction to Geochronology and Thermochronology, Introduction to Geophysics, Igneous and Metamorphic Petrology, Sedimentation and Stratigraphy, Structural Geology, Mineralogy, GIS Design and Application, GIS Database Design, GIS Project Management, Linear Algebra, Differential Equations.

EXPERIENCE:

Geologist II, Laurel Environmental Geosciences, DPC. Huntington, NY.
June 2016 - Present

- Perform Phase I & Phase II Environmental Site Assessments (ESAs)
- Calibrate and maintain instruments used in Phase II ESAs
- Conduct asbestos surveys
- Provide construction and remedial oversight to ensure compliance with environmental regulations
- Draft reports documenting fieldwork and clearly outlining any necessary corrective actions
- Interface with public agencies to achieve regulatory closure of oil spills, tank removals, and underground structure abandonments
- Construct Electronic Database Deliverables for submission to the NYSDEC
- Collaborate with project managers to develop proposals meeting site-specific needs
- Mentor junior staff in proper field investigative techniques while providing professional development assessments for review by senior management

Research Assistant, Stony Brook University. Stony Brook, NY.
September 2015 – December 2016

- Carry out geophysical investigations of suspected glaciotectonic structures
- Maintain and repair equipment used in fieldwork
- Give presentations to classes prior to undertaking fieldwork which established project goals
- Lead small groups in successfully completing fieldwork
- Use java-based GIS systems to map extent of objects and establish relationships

FIELD AND TECHNICAL SKILLS:

- Extensive experience using ground penetrating radar (GPR) units (50 – 800 MHz)
- Processing of GPR data for use in presentations and publications
- Practical experience in the use of magnetometers and gravitometers
- Knowledge of surveying techniques to make accurate topographic measurements
- Use of EQuIS Data Processor to create geodatabases meeting NYSDEC standards
- Use of ArcGIS and jMARS software to create information products
- Proficient in the use of Microsoft Office suite of software

CERTIFICATIONS:

OSHA 40 Hr. HAZWOPER Course, NIOSH 582
OSHA 30 Hr. Construction Safety
NYSDOL Asbestos Inspector
XRF Safety Training

BRIAN C. MCCABE

EDUCATION: STONY BROOK UNIVERSITY
B.S., Geology, September 1991, Minor in Marine Science
M.S., Hydrogeology, Pending.

RELATED COURSES:

Geophysics, Geochemistry,
Hydrogeology, Organic contaminant Hydrology, and Bioremediation.

EXPERIENCE:

Senior Geologist/Senior Project Manager, Laurel Environmental Geosciences, DPC.
January 2012 - present

- Project Health and Safety Officer
- Project QA/QC Officer
- Phase I Environmental Site Assessments.
- Phase II Environmental Assessments.
- Remedial Investigations/Feasibility Studies
- NYC Office of Environmental Restoration (OER) Investigations
- Underground Storage Tanks (UST's): testing, removal, closure.
- Underground Injection Well Closure (UIC)
- Hazardous Waste Site Remediation.
- Design and Management of *In-situ* Bioremediation Systems
- Installation of Sub-Slab Depressurization Systems
- Operation and Maintenance of Groundwater Extraction and Treatment Systems
- Installation of Vapor Barrier Systems
- Installation of Air Sparging/Soil Vapor Extraction Systems

Department Manager: Professional Services, Fenley & Nicol
September 1997 - January 2012

- Phase I Environmental Assessments.
- Phase II Environmental Assessments.
- Groundwater Contamination Studies.
- Underground Storage Tanks (UST'S): testing, removal, closure.
- Underground Injection Well Closure (UIC)
- Hazardous Site Remediation.
- Construction and operation of remediation system.

CERTIFICATIONS:

OSHA 40-Hr. HAZWOPER Course
Preston Groundwater Remediation Course
ASTM 1527-13 Environmental Site Assessment training course
Waterloo University, Groundwater Modeling Training Course
ExxonMobil, LPS training
LIRR Track Safety training

AFFILIATIONS:

Long Island Geologists
United States Coast Guard Auxiliary

MARC T. CALIFANO, P.G.

Project Director/Senior Geologist, *Laurel Environmental Geosciences, D.P.C.*
New York State Licensed Professional Geologist: #000669

EDUCATION:

B.S., ENVIRONMENTAL SCIENCE, 1999 - STATE UNIVERSITY OF NEW YORK AT ONEONTA

RELATED COURSES:

Environmental Geology, Earth Materials, Mineralogy, Environmental Geophysics, Field Geology, Structural Geology, Glacial Geology, Hydrogeology, Mapping Techniques in Geosciences, Geomorphology, Paleontology, Oceanography, Biology, Chemistry, Physics, and Calculus.

WORK EXPERIENCE:

Mr. Marc Califano, P.G. has over 25 years of experience in the environmental consulting field and has performed Phase I Environmental Site Assessments (ESA's), Phase II Soil, Groundwater and Vapor Intrusion Sampling Investigations, and Phase III Site Remediation projects for both municipal and private clients. Mr. Califano has extensive field experience, technical report writing skills and comprehensive knowledge of New York State, New York City and County (Nassau and Suffolk) regulatory compliance standards/requirements. Through this experience, Mr. Califano has closed many New York State Department of Environmental Conservation (NYSDEC) spill cases, remediated and closed several Suffolk County Department of Health Services (SCDHS) Underground Injection Control (UIC) cases and prepared several permit applications, technical reports, health and safety plans, remedial work plans and closure reports. Mr. Califano has been involved in many commercial redevelopment projects through the preparation of remedial work plans, sampling plans, engineering reports, site management plans and has extensive experience interrupting analytical data for the disposal of contaminated and hazardous materials.

LICENSES/CERTIFICATIONS

Licensed Professional Geologist, New York State, No. 000669
OSHA 40 Hour HAZWOPER Course
OSHA 8 Hour HAZWOPER Refresher Courses
Amtrak On-Track Safety Certification
MTA New York City Transit – Tack Safety Training
EPA Radon Training and Certification
Environmental Data Resources – ASTM E1527-21 Due Diligence update

AFFILIATIONS

Long Island Association of Professional Geologists

QAPP
APPENDIX B

Laboratory MDLs, RLs, and SOPs for PFAS Analaysis

Analytical Method Information

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PFAS, EPA 1633 Target List in Soil (EPA 1633 Draft 2)

Preservation: Cool 4°C

Container: 10_250mL Plastic Cool to 4° C **Amount Required:** 250 mL **Hold Time:** 28 days

Analyte	MDL	Reporting Limit	Surrogate %Rec	Duplicate RPD	----Matrix Spike---- %Rec	RPD	--Blank Spike / LCS-- %Rec	RPD
Perfluorobutanesulfonic acid (PFBS)	0.111	0.177 ug/kg		30	25-150	35	50-150	30
Perfluorohexanoic acid (PFHxA)	0.0530	0.200 ug/kg		30	25-150	35	50-150	30
Perfluoroheptanoic acid (PFHpA)	0.105	0.200 ug/kg		30	25-150	35	50-150	30
Perfluorohexanesulfonic acid (PFHxS)	0.179	0.183 ug/kg		30	25-150	35	50-150	30
Perfluorooctanoic acid (PFOA)	0.172	0.200 ug/kg		30	25-150	35	50-150	30
Perfluorooctanesulfonic acid (PFOS)	0.167	0.186 ug/kg		30	25-150	35	50-150	30
Perfluorononanoic acid (PFNA)	0.189	0.200 ug/kg		30	25-150	35	50-150	30
Perfluorodecanoic acid (PFDA)	0.191	0.200 ug/kg		30	25-150	35	50-150	30
Perfluoroundecanoic acid (PFUnA)	0.198	0.200 ug/kg		30	25-150	35	50-150	30
Perfluorododecanoic acid (PFDoA)	0.163	0.200 ug/kg		30	25-150	35	50-150	30
Perfluorotridecanoic acid (PFTrDA)	0.125	0.200 ug/kg		30	25-150	35	50-150	30
Perfluorotetradecanoic acid (PFTA)	0.103	0.200 ug/kg		30	25-150	35	50-150	30
N-MeFOSAA	0.148	0.200 ug/kg		30	25-150	35	50-150	30
N-EtFOSAA	0.194	0.200 ug/kg		30	25-150	35	50-150	30
Perfluoropentanoic acid (PFPeA)	0.109	0.400 ug/kg		30	25-150	35	50-150	30
Perfluoro-1-octanesulfonamide (FOSA)	0.146	0.200 ug/kg		30	25-150	35	50-150	30
Perfluoro-1-heptanesulfonic acid (PFHpS)	0.155	0.200 ug/kg		30	25-150	35	50-150	30
Perfluoro-1-decanesulfonic acid (PFDS)	0.191	0.193 ug/kg		30	25-150	35	50-150	30
1H,1H,2H,2H-Perfluorooctanesulfonic acid (6:2 FTS)	0.595	0.760 ug/kg		30	25-150	35	50-150	30
1H,1H,2H,2H-Perfluorodecanesulfonic acid (8:2 FTS)	0.755	0.768 ug/kg		30	25-150	35	50-150	30
Perfluoro-n-butanoic acid (PFBA)	0.109	0.800 ug/kg		30	25-150	35	50-150	30
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	0.139	0.356 ug/kg		30	25-150	30	50-150	30
Perfluoro-3,6-dioxaheptanoic acid (NFDHA)	0.193	0.400 ug/kg		30	25-150	30	50-150	30
Perfluoro-4-oxapentanoic acid (PFMPA)	0.0620	0.400 ug/kg		30	25-150	30	50-150	30
Perfluoro-5-oxahexanoic acid (PFMBA)	0.0960	0.400 ug/kg		30	25-150	30	50-150	30
Perfluoro-1-pentanesulfonate (PFPeS)	0.157	0.188 ug/kg		30	25-150	30	50-150	30
1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS)	0.595	0.750 ug/kg		30	25-150	30	50-150	30
HFPO-DA (Gen-X)	0.608	0.800 ug/kg		30	25-150	30	50-150	30
11CL-PF3OUdS	0.311	0.756 ug/kg		30	25-150	30	50-150	30
9CL-PF3ONS	0.246	0.748 ug/kg		30	25-150	30	50-150	30
ADONA	0.174	0.756 ug/kg		30	25-150	30	50-150	30
Perfluorododecanesulfonic acid (PFDoS)	0.169	0.194 ug/kg		30	25-150	30	50-150	30
Perfluoro-1-nonanesulfonic acid (PFNS)	0.124	0.192 ug/kg		30	25-150	30	50-150	30
3-Perfluoropropyl propanoic acid (FPrPA)	0.634	1.00 ug/kg		30	25-150	30	50-150	30
3-Perfluoropentyl propanoic acid (FPePA)	2.10	5.00 ug/kg		30	25-150	30	50-150	30
3-Perfluoroheptyl propanoic acid (FHpPA)	1.50	5.00 ug/kg		30	25-150	30	50-150	30
N-MeFOSE	0.611	2.00 ug/kg		30	25-150	30	50-150	30
N-MeFOSA	0.180	0.200 ug/kg		30	25-150	30	50-150	30
N-EtFOSE	0.697	2.00 ug/kg		30	25-150	30	50-150	30
N-EtFOSA	0.198	0.200 ug/kg		30	25-150	30	50-150	30
Surr: M3PFBS			25-150					

Analytical Method Information

(Continued)

PFAS, EPA 1633 Target List in Soil (EPA 1633 Draft 2) (Continued)

Analyte	MDL	Reporting Limit	Surrogate %Rec	Duplicate RPD	----Matrix Spike---- %Rec	RPD	--Blank Spike / LCS-- %Rec	RPD
Surr: M5PFHxA			25-150					
Surr: M4PFHpA			25-150					
Surr: M3PFHxS			25-150					
Surr: Perfluoro-n-[13C8]octanoic acid (M8PFOA)			25-150					
Surr: M6PFDA			25-150					
Surr: M7PFUdA			25-150					
Surr: Perfluoro-n-[1,2-13C2]dodecanoic acid (MPFDoA)			25-150					
Surr: M2PFTeDA			10-150					
Surr: Perfluoro-n-[13C4]butanoic acid (MPFBA)			25-150					
Surr: Perfluoro-1-[13C8]octanesulfonic acid (M8PFOS)			25-150					
Surr: Perfluoro-n-[13C5]pentanoic acid (M5PFPeA)			25-150					
Surr: Perfluoro-1-[13C8]octanesulfonamide (M8FOSA)			10-150					
Surr: d3-N-MeFOSAA			25-150					
Surr: d5-N-EtFOSAA			25-150					
Surr: M2-6:2 FTS			25-200					
Surr: M2-8:2 FTS			25-200					
Surr: M9PFNA			25-150					
Surr: M2-4:2 FTS			25-150					
Surr: d-N-MeFOSA			25-150					
Surr: d-N-EtFOSA			25-150					
Surr: M3HFPO-DA			25-150					
Surr: d9-N-EtFOSE			25-150					
Surr: d7-N-MeFOSE			25-150					
M3PFBA								
MPFDA								
MPFHxA								
MPFHxS								
MPFNA								
MPFOA								
MPFOS								
Perfluoro-n-[13C9]nonanoic acid (M9PFNA)-EIS								
Perfluoro-n-[13C8]octanoic acid (M8PFOA)-EIS								
Perfluoro-n-[13C54]pentanoic acid (M5PFPeA)-EIS								
Perfluoro-n-[13C4]butanoic acid (MPFBA)-EIS								
Perfluoro-n-[1,2-13C2]dodecanoic acid (MPFDoA)-EIS								
Perfluoro-1-[13C8]octanesulfonic acid (M8PFOS)-EIS								
Perfluoro-1-[13C8]octanesulfonamide (M8FOSA)-EIS								
M7PFUdA-EIS								
M6PFDA-EIS								
M5PFHxA-EIS								
M4PFHpA-EIS								
M3PFHxS-EIS								
M3PFBS-EIS								
M3-HFPO-DA-EIS								
M2PFTeDA-EIS								

Analytical Method Information

(Continued)

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PFAS, EPA 1633 Target List in Soil (EPA 1633 Draft 2) (Continued)

Analyte	MDL	Reporting Limit	Surrogate %Rec	Duplicate RPD	----Matrix Spike---- %Rec	RPD	--Blank Spike / LCS-- %Rec	RPD
M2-8-2FTS-EIS								
M2-6-2FTS-EIS								
M2-4-2FTS-EIS								
d9-NEtFOSE-EIS								
d7-NMeFOSE-EIS								
d5-NEtFOSA-EIS								
d5-N-EtFOSAA-EIS								
d3-NMeFOSA-EIS								
d3-N-MeFOSAA-EIS								

Analytical Method Information

(Continued)

PFAS, EPA 1633 Target List in Water (EPA 1633 Draft 2)

Preservation: Cool 4°C

Container: 10_250mL Plastic Cool to 4° C **Amount Required:** 250 mL **Hold Time:** 28 days

Analyte	MDL	Reporting Limit	Surrogate %Rec	Duplicate RPD	----Matrix Spike---- %Rec	RPD	--Blank Spike / LCS-- %Rec	RPD
Perfluorobutanesulfonic acid (PFBS)	0.470	1.77 ng/L		30	25-150	35	50-150	30
Perfluorohexanoic acid (PFHxA)	0.350	2.00 ng/L		30	25-150	35	50-150	30
Perfluoroheptanoic acid (PFHpA)	0.710	2.00 ng/L		30	25-150	35	50-150	30
Perfluorohexanesulfonic acid (PFHxS)	0.680	1.83 ng/L		30	25-150	35	50-150	30
Perfluorooctanoic acid (PFOA)	0.420	2.00 ng/L		30	25-150	35	50-150	30
Perfluorooctanesulfonic acid (PFOS)	0.820	1.86 ng/L		30	25-150	35	50-150	30
Perfluorononanoic acid (PFNA)	0.520	2.00 ng/L		30	25-150	35	50-150	30
Perfluorodecanoic acid (PFDA)	0.750	2.00 ng/L		30	25-150	35	50-150	30
Perfluoroundecanoic acid (PFUnA)	1.13	2.00 ng/L		30	25-150	35	50-150	30
Perfluorododecanoic acid (PFDoA)	0.880	2.00 ng/L		30	25-150	35	50-150	30
Perfluorotridecanoic acid (PFTrDA)	0.740	2.00 ng/L		30	25-150	35	50-150	30
Perfluorotetradecanoic acid (PFTA)	0.690	2.00 ng/L		30	25-150	35	50-150	30
N-MeFOSAA	0.790	2.00 ng/L		30	25-150	35	50-150	30
N-EtFOSAA	1.03	2.00 ng/L		30	25-150	35	50-150	30
Perfluoropentanoic acid (PFPeA)	0.230	4.00 ng/L		30	25-150	35	50-150	30
Perfluoro-1-octanesulfonamide (FOSA)	0.880	2.00 ng/L		30	25-150	35	50-150	30
Perfluoro-1-heptanesulfonic acid (PFHpS)	0.910	1.91 ng/L		30	25-150	35	50-150	30
Perfluoro-1-decanesulfonic acid (PFDS)	1.32	1.93 ng/L		30	25-150	35	50-150	30
1H,1H,2H,2H-Perfluorooctanesulfonic acid (6:2 FTS)	1.06	7.60 ng/L		30	25-150	35	50-150	30
1H,1H,2H,2H-Perfluorodecanesulfonic acid (8:2 FTS)	2.05	7.68 ng/L		30	25-150	35	50-150	30
Perfluoro-n-butanoic acid (PFBA)	0.330	8.00 ng/L		30	25-150	35	50-150	30
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	0.500	3.56 ng/L		30	25-150	30	50-150	30
Perfluoro-3,6-dioxaheptanoic acid (NFDHA)	2.14	4.00 ng/L		30	25-150	30	50-150	30
Perfluoro-4-oxapentanoic acid (PFMPA)	0.250	4.00 ng/L		30	25-150	30	50-150	30
Perfluoro-5-oxahexanoic acid (PFMBA)	0.370	4.00 ng/L		30	25-150	30	50-150	30
Perfluoro-1-pentanesulfonate (PFPeS)	0.760	1.88 ng/L		30	25-150	30	50-150	30
1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS)	1.79	7.50 ng/L		30	25-150	30	50-150	30
HFPO-DA (Gen-X)	3.23	8.00 ng/L		30	25-150	30	50-150	30
11CL-PF3OUdS	1.38	7.56 ng/L		30	25-150	30	50-150	30
9CL-PF3ONS	0.700	7.48 ng/L		30	25-150	30	50-150	30
ADONA	0.530	7.56 ng/L		30	25-150	30	50-150	30
Perfluorododecanesulfonic acid (PFDoS)	0.930	1.94 ng/L		30	25-150	30	50-150	30
Perfluoro-1-nonanesulfonic acid (PFNS)	0.860	1.92 ng/L		30	25-150	30	50-150	30
3-Perfluoropropyl propanoic acid (FPrPA)	2.03	5.00 ng/L		30	25-150	30	50-150	30
3-Perfluoropentyl propanoic acid (FPePA)	7.33	25.0 ng/L		30	25-150	30	50-150	30
3-Perfluoroheptyl propanoic acid (FHpPA)	9.47	25.0 ng/L		30	25-150	30	50-150	30
N-MeFOSE	3.99	20.0 ng/L		30	25-150	30	50-150	30
N-MeFOSA	1.58	2.00 ng/L		30	25-150	30	50-150	30
N-EtFOSE	3.99	20.0 ng/L		30	25-150	30	50-150	30
N-EtFOSA	1.80	2.00 ng/L		30	25-150	30	50-150	30
Surr: M3PFBS			25-150					

Analytical Method Information

(Continued)

PFAS, EPA 1633 Target List in Water (EPA 1633 Draft 2) (Continued)

Analyte	MDL	Reporting Limit	Surrogate %Rec	Duplicate RPD	----Matrix Spike---- %Rec	RPD	--Blank Spike / LCS-- %Rec	RPD
Surr: M5PFHxA			25-150					
Surr: M4PFHpA			25-150					
Surr: M3PFHxS			25-150					
Surr: Perfluoro-n-[13C8]octanoic acid (M8PFOA)			25-150					
Surr: M6PFDA			25-150					
Surr: M7PFUdA			25-150					
Surr: Perfluoro-n-[1,2-13C2]dodecanoic acid (MPFDoA)			25-150					
Surr: M2PFTeDA			10-150					
Surr: Perfluoro-n-[13C4]butanoic acid (MPFBA)			25-150					
Surr: Perfluoro-1-[13C8]octanesulfonic acid (M8PFOS)			25-150					
Surr: Perfluoro-n-[13C5]pentanoic acid (M5PFPeA)			25-150					
Surr: Perfluoro-1-[13C8]octanesulfonamide (M8FOSA)			10-150					
Surr: d3-N-MeFOSAA			25-150					
Surr: d5-N-EtFOSAA			25-150					
Surr: M2-6:2 FTS			25-200					
Surr: M2-8:2 FTS			25-200					
Surr: M9PFNA			25-150					
Surr: M2-4:2 FTS			25-150					
Surr: d-N-MeFOSA			25-150					
Surr: d-N-EtFOSA			25-150					
Surr: M3HFPO-DA			25-150					
Surr: d9-N-EtFOSE			25-150					
Surr: d7-N-MeFOSE			25-150					
M3PFBA								
MPFDA								
MPFHxA								
MPFHxS								
MPFNA								
MPFOA								
MPFOS								
Perfluoro-n-[13C9]nonanoic acid (M9PFNA)-EIS								
Perfluoro-n-[13C8]octanoic acid (M8PFOA)-EIS								
Perfluoro-n-[13C54]pentanoic acid (M5PFPeA)-EIS								
Perfluoro-n-[13C4]butanoic acid (MPFBA)-EIS								
Perfluoro-n-[1,2-13C2]dodecanoic acid (MPFDoA)-EIS								
Perfluoro-1-[13C8]octanesulfonic acid (M8PFOS)-EIS								
Perfluoro-1-[13C8]octanesulfonamide (M8FOSA)-EIS								
M7PFUdA-EIS								
M6PFDA-EIS								
M5PFHxA-EIS								
M4PFHpA-EIS								
M3PFHxS-EIS								
M3PFBS-EIS								
M3-HFPO-DA-EIS								
M2PFTeDA-EIS								

Analytical Method Information

(Continued)

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PFAS, EPA 1633 Target List in Water (EPA 1633 Draft 2) (Continued)

Analyte	MDL	Reporting Limit	Surrogate %Rec	Duplicate RPD	----Matrix Spike---- %Rec	RPD	--Blank Spike / LCS-- %Rec	RPD
M2-8-2FTS-EIS								
M2-6-2FTS-EIS								
M2-4-2FTS-EIS								
d9-NEtFOSE-EIS								
d7-NMeFOSE-EIS								
d5-NEtFOSA-EIS								
d5-N-EtFOSAA-EIS								
d3-NMeFOSA-EIS								
d3-N-MeFOSAA-EIS								

Standard Operating Procedure

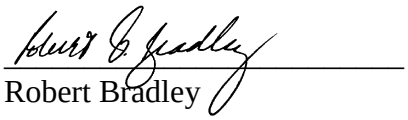
Determination of Target Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous and Solid matrices by Isotope Dilution Analysis by HPLC/MS-MS According to EPA Method 1633 Draft 2

Approvals

Laboratory Director/QA Officer

Krys Trafalski

Vice President/Chief Scientific Officer



Robert Bradley

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Issued to: NA

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 the Table 1.0 below.

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		
1*H*,1*H*, 2*H*, 2*H*-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4
1*H*,1*H*, 2*H*, 2*H*-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2
1*H*,1*H*, 2*H*, 2*H*-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*H*-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	9Cl-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
Fluorotelomer carboxylic acids		
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5
2*H*,2*H*,3*H*,3*H*-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 2.0-20.0 ng/L (ppt) for aqueous samples and 0.5-5.0 ug/kG for solids . The linear range for these PFAS can be extended by dilution. These MRLs are based upon a volume of 0.250L-0.500L extracted for aqueous samples and 2-5 g. for solids.

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 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 LC-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.

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 then analyzed by HPLC-MS/MS in the MRM mode. Extracts contain Non-extracted Internal Standards (NIS) to monitor instrument performance and 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 analytes for surrogate analog recoveries and these surrogates are essentially 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 ANALYSIS BATCH – A set of samples that is analyzed on the same instrument during a 24-hour period, including no more than 20 Field Samples, that begins and ends with the analysis of the appropriate Continuing Calibration Check (CCC) standards. Additional CCCs may be required depending on the length of the analysis batch and/or the number of Field Samples.

3.2 CALIBRATION STANDARD (CAL) – A solution of the method analytes, isotope dilution analogues, and isotope performance standards (Internal standards) prepared from the Primary Dilution Standards 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 the method analytes, internal standard(s) and surrogate(s). The CCV is analyzed periodically to verify the accuracy of the existing calibration for those analytes.

3.4 EXTRACTION BATCH – A set of up to 20 Field Samples (not including QC Samples) extracted together by the same person(s) during a work day using the same lot of SPE devices, solvents, surrogate, internal standard and fortifying solutions. Required QC samples include Method blank, and Matrix spike/duplicate pair.

3.5 FIELD DUPLICATES – Separate samples collected at the same time and sampling location, shipped and stored under identical conditions. Method precision, including the contribution from sample collection procedures, is estimated from the analysis of Field Duplicates. Field Duplicates are used to prepare matrix spike/matrix spike duplicate QC samples.

3.6 FIELD BLANK (FBLK) – An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FBLK is to determine if method analytes or other interferences are introduced into 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 our SOP and in the reference method.

3.8 ISOTOPE DILUTION TECHNIQUE - An analytical technique for measuring analyte concentration using the ratio of the peak area of the native analyte to that of an isotopically labeled analogue, added to the original sample in a known amount and carried through the entire analytical procedure.

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.

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 unpooled, 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 (with the exception of 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 commercially available. The purpose of the ICV is to verify the integrity of the primary 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 assure that the results produced by the 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 the 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 co-extracted from the 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 LC 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 multi-sampler system.

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 the 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 ºC 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 ºC during transport. Sample are to be received by the laboratory within 48 hours of collection. The laboratory must confirm that the sample temperature is 0 - 6 ºC upon receipt. Once received by the laboratory, the samples may be stored at ≤ -20 ºC, or at 0 - 6 ºC, 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 ºC and protected from the light. When stored at 0 - 6 ºC 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 ºC or ≤ -20 ºC, with the caveat that samples may need to be extracted as soon as possible if NFDHA is an important analyte.

5.4 **SAMPLE EXTRACT HOLDING TIMES** – Store sample extracts in the dark at less than 0 - 4 ºC until analyzed. If stored in the dark at less than 0 - 4 ºC, 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 uL, 10 uL 25 uL, 100 uL, 250 uL, 500 uL, 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.

6.13 LC/MS-MS system- Agilent 1260 or 1290 HPLC system interfaced to an Agilent 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 uL; 100-1000 uL; 1000-5000 uL-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 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 pipet to add the 2.5 mL (not strictly quantitative FYI)-**Make 4 bottles of this**. *Used for soil extractions*.- 1 month 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 to 242 mL PFAS free water. 3 month life- *used for pH adjustment*

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 stds and is used to dilute the extracts of samples that exceed the calibration range.**

7.2.14 **Formic Acid 0.1M-aqueous** – add 873 uL formic acid into 250 mL PFAS free water- **Make 2 bottles of this-***used to prepare 7 below.* 2 year life

7.2.15 **Formic Acid, 0.3M**-aqueous- add 2.62 mL (2619 uL) 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 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 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 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. The part no. is **MPFAC-HIF-ES**.

7.3.3 Stock Standard mixtures of both linear and branched isomers of the EPA 1633 - 40 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 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 five year life at stock concentrations.

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

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

Transfer 300 uL to an autosampler vials, add 3 uL of ISTD working mix, cap, vortex and store until needed. Life is 1 year.

The summary below details the procurement requirements for this method - All from Wellington Laboratories, Inc.:

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 Stds	MPFAC-HIF-IS	4 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 ug/mL levels. This insures their stability with respect to potential loss due to esterification. The basic solution insures 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.

In order 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 uL 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 uL of the 5.0 mM NaOH/MeOH.

For working calibration standards/CCV/SCV made to 500 uL 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.

7.4.2 Storage and Handling of Standards

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

Stock Standards may be stored at 4-10 deg. 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 Detailed Standards Preparation Procedure-EPA 1633

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 uL of the Stock and adding 500 uL MeOH.

Use as is by adding 3 uL to 300 uL 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 uL 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 uL of the Surrogate Stock, plus 25 uL of 5 mM NaOH/MeOH and 975 uL MeOH to give 2.0 mL final volume**. 50 uL are added to ALL preparation blanks, samples and QC**. This is sufficient for approx. 40 x 50 uL 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.

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 under the 5 part nos. described in Section 7.3.3 which contains 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 10 x 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 uL of each STOCK into a snap cap vial giving 1.0 mL final volume.

1. Element ID Y22B199- PFAC-MXF mix 200 uL
2. Element ID Y22B200- PFAC-MXG mix 200 uL
3. Element ID Y22B201- PFAC-MXH mix 200 uL
4. Element ID Y22B204- PFAC-MXI mix 200 uL
5. Element ID Y22B205- PFAC-MXJ mix 200 uL

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

7.4.7 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 uL 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 uL of each level into a 500 uL PP vial and add 3 uL 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.

EPA 1633 Calibration Standard Preparation
Rev 1.,0 10/03/22

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.

Level	*Stock: Y22B201* **1633 MXH Targets Intermediate @10x *** uL of MXH 10x Interm.	*Stock: Y22B200* **1633 MXG Targets Intermediate at 10x*** ul of MXG interm.	*Stock: Y22B199* **1633 MXF Targets Intermediate at 10x*** ul of MXF interm.	*Stock: Y22B204* **1633 MXI Targets Intermediate at 10x*** ul of MXI interm.	*Stock: Y22B205* **1633 MXJ Targets Intermediate at 10x*** ul of MXJ interm.	*Stock: Y22B198* **1633 EIS isotope Mix Intermediate at 10x** 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

* 100 uL up to 1 mL in MeOH

*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.

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

CAL LEVEL	uL 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 uL of STOCK ISTD at 250-1000 ng/mL with 500 uL of Methanol. This results in 125-500 ng/mL Intermediate ISTD. See 7.4.4.1.

Add 3.0 uL to 300 uL of each level 1-8 in a 500 uL PP autosampler vials and cap with polyolefin cap, vortex to mix and run. Add 3 uL to 300 uL 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.

7.4.8 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 uL of the surrogate (EIS) mix and 3 uL of the Spike mix into 294 uL MeOH/Water/Acetic Acid/Ammonium hydroxide from 7.1.13, then add 3 uL 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.

7.4.9 Second Source - Initial Calibration Verification (ICV)

Currently, the EPA method 1633 does not require a second source ICV. Rather, the initial calibration is verified by preparing a Level 5 -5.0 ng/mL (nominal) calibration standard independently from calibration standard preparation. This serves as the ICV.

8. PROCEDURE

8.1 Preventative and Routine Maintenance

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

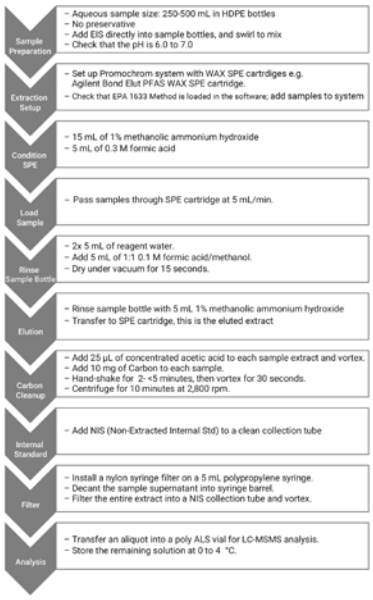
8.2 Sample Preparation (Extraction, Clean-up and Concentration)- Aqueous Matrices

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 mLs $\pm$ 0.02 mL through a tared 0.2 um PP filter. Dry filter $\geq$ 12 hours @ 105C, cool in dessicator. Calc % TSS
2. Check pH with short range pH paper to insure pH = 6.5 $\pm$ 0.5. Adjust if 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 uL of Spike mix for LLOPR and 100 uL of spike mix for mid-OPR.
6. Spike all with 25 uL 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 1g. = 1.0 mL. Enter this value into the element bench sheet and the initial volume.
11. Add 25 uL 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 <55ºC.. For example if final volume is 5.0 mL, concentrate to 2.0 mL final volume (2.5 x concentration). If 500 ml 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 uL portion of the extract into a 500 uL PP autosampler vial, add 3 uL of NIS (non-extracted internal std.). Cap, vortex, store at <6ºC.
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 uL 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 uL of EIS (isotopic surrogates) (250/5000 ng/mL) to each sample and QC sample, recap and invert to mix well.

8.2.5 Add, 5ul (low level spike), 50 uL (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.

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 mls 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 uL into a 500 uL autosampler vial (PP) and add 3.0 uL of ISTD (NIS) mix. .

8.2.16 Cap with polyolefin flexible caps and vortex to mix.

8.2.17 Store Extracts at <6^{o}C until analysis.

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

1. Determine % solids: use 5 grams; dry at 110C $\geq$ 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 $\pm$ 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 uL aliquot of EIS onto the soil. The current Element standard ID is Y22J305. For the OPRs add appropriate amount of spike solution (10 uL for LLOPR and 100 uL 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 rto 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.
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 benchsheet as final volume. Should be 5.0 mL. If less make up to 5.0 mL with MeOH.
28. Add 25 uL 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 uL of extract and transfer to a 500 uL autosampler vial. Add 3 uL of NIS (internal standard), vortex to mix. Cap with polyolefin flexible caps and vortex to mix.
33. Store Extracts at <6^{o}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 a 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 $[M-H]^-$ mode or M-COOH for HFPO-DA.

8.3.2 The QQQ 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 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)*

* Contains solvent system for calibration, NIS and EIS

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 ul aliquot of sample extract containing 3 uL 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 (injection sequence) 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 acceptable ion ratios for the abundance of both precursor and product ions along with retention time information. All positive detections of target PFAS must be less that the high point conc. of the Cal. 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. In order 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 RSD should be $\leq$ 20% where used.

9.1.1 The calibration levels as shown in Section 7.6.3 use 8 levels. All points are included in the calibration with exception of some species that saturate at levels 7 and 8.

9.2 ICV/SCV

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 $\pm$ 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 run 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 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 uL of the native spike solution OPR Mix Y22J304, 50 µL 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 are 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

MDL Determination –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 (we use only QQQ2 for EPA 1633 so all runs are on that system). 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 x SD (for seven replicates; SD = Standard Deviation)

Compute the MDLb (MDL based on method blanks-LRBs) as follows:

- If none of the blanks give numerical results then the MDLb 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 detections then use the following calculation to determine MDLb:

MDLb = Average of Blank Detections + (3.143 x Std. Dev.)

Calculate the final MDL by selecting the greater of MDLs or MDLb.

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 sample (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 METHOD BLANK - 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 LABORATORY CONTROL SAMPLES (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 is 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 Initial calibration verification (ICV) –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.
- Evaluate the initial calibration standards.
- Rerun the initial calibration.

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

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 model 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 or linear regression or quadratic regression FORCED through the origin. Weighted ($1/x$ 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 taking into account 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 are then evaluated using the York Qualinator ^{TM} 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.

- 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 the 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 be worn at all times 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 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 occurred.
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).
Formic Acid, conc.	-Flammable liquid and vapor -Harmful if swallowed -Causes severe skin burns and eye damage -Toxic if inhaled -May cause respiratory irritation	OSHA TWA: 5 ppm or 9 mg/m3 OSHA PEL: 10 ppm	Formic acid 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).

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.

Solvents

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

1. EPA METHOD 1633 Draft 2 June, 2022- Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS; EPA 821-D-22-001

15. REVISION HISTORY

Revision 1.0	10/24/2022	First issue.
Revision 1.1	02/10/2023	Modified LLOPR in Section 7.4.6.1 to reflect 2x the MRL

Attachment 1 – Non-Extracted Internal Standards (NIS)

YORK
ANALYTICAL LABORATORIES, INC.

Analytical Standard Record

Standard ID: **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:	PFAS
Vials:	1	Lot No.:	MPFACHIFIS0921
Vendor:	Wellington Laboratories		

Comments: Stock ISTD for EPA method 1633

Analyte	CAS Number	Concentration	Units
M3PFBA		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

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

MPFAC-HIF-IS

Mass-Labelled Perfluoroalkyl Substance Injection Standard Solution/Mixture

PRODUCT CODE:	MPFAC-HIF-IS
LOT NUMBER:	MPFACHIFIS0921
SOLVENT(S):	Methanol/Water (<1%)
DATE PREPARED: (mm/dd/yyyy)	09/07/2021
LAST TESTED: (mm/dd/yyyy)	09/07/2021
EXPIRY DATE: (mm/dd/yyyy)	09/07/2026
RECOMMENDED STORAGE:	Store ampoule in a cool, dark place

DESCRIPTION:

MPFAC-HIF-IS is a solution/mixture of five mass-labelled (^{13}C) perfluoroalkylcarboxylic acids (C_4, C_8, C_9-C_{10}) and two mass-labelled (^{18}O and ^{13}C) perfluoroalkanesulfonates (C_6 and C_8). 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 ^{13}C or >94% per ^{18}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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Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

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

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 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 $x_1, x_2, \ldots x_n$ on which it depends is:

$$u_c(y(x_1, x_2, \ldots x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

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).

CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED AR-1523 ISO 17034 REFERENCE MATERIAL PRODUCER

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

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

Compound	Acronym	Concentration (ng/mL)		Peak Assignment in Figure 1
Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	M3PFBA	1000		1
Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	MPFHxA	500		2
Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	MPFOA	500		4
Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	MPFNA	250		5
Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	MPFDA	250		7
Compound	**Acronym**	**Concentration* (ng/mL)**		**Peak Assignment in Figure 1**
		as the salt	**as the acid**	
Sodium perfluoro-1-hexane($^{18}O_2$)sulfonate	MPFHxS	500	474	3
Sodium perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonate	MPFOS	500	479	6

* Concentrations have been rounded to three significant figures.

Certified By: _____ **Date:** 10/13/2021 (mm/dd/yyyy)
B.G. Chittim, General Manager

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Revision#:9, Revised 2020-12-23

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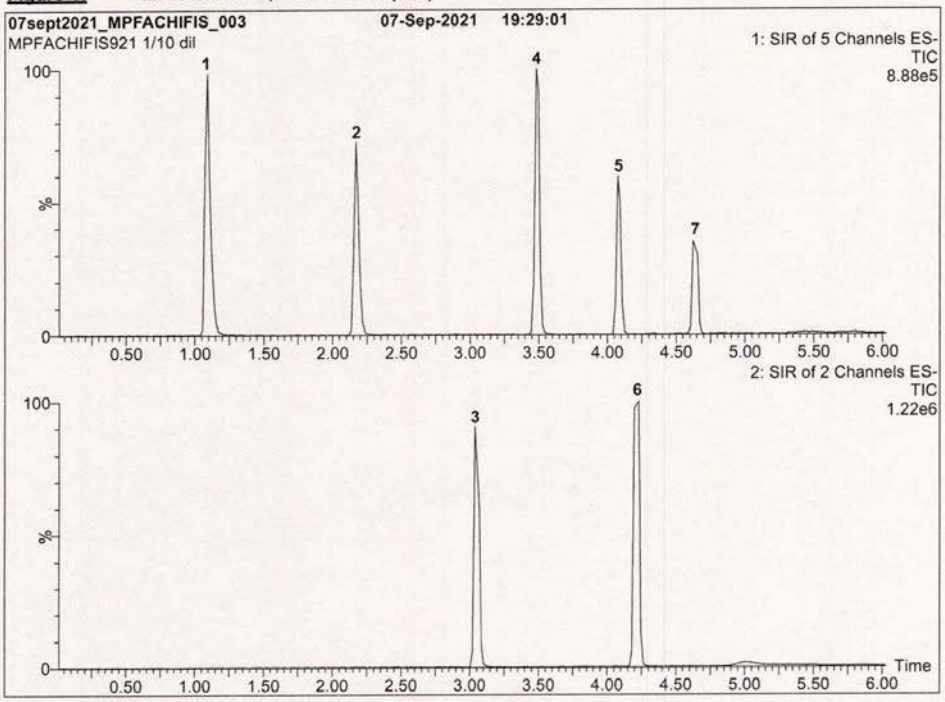
rev1

Figure 1: **MPFAC-HIF-IS; LC/MS Data (SIR)**



Conditions for Figure 1:

Waters Acquity Ultra Performance LC
Waters Xevo TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H_2O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 9 min and hold for
2 min before returning to initial conditions in 1 min.
Time: 15 min

Flow: 300 µL/min

MS Parameters:

Experiment: SIR

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = variable (2-6)
Desolvation Temperature (°C) = 350
Desolvation Gas Flow (L/hr) = 1000

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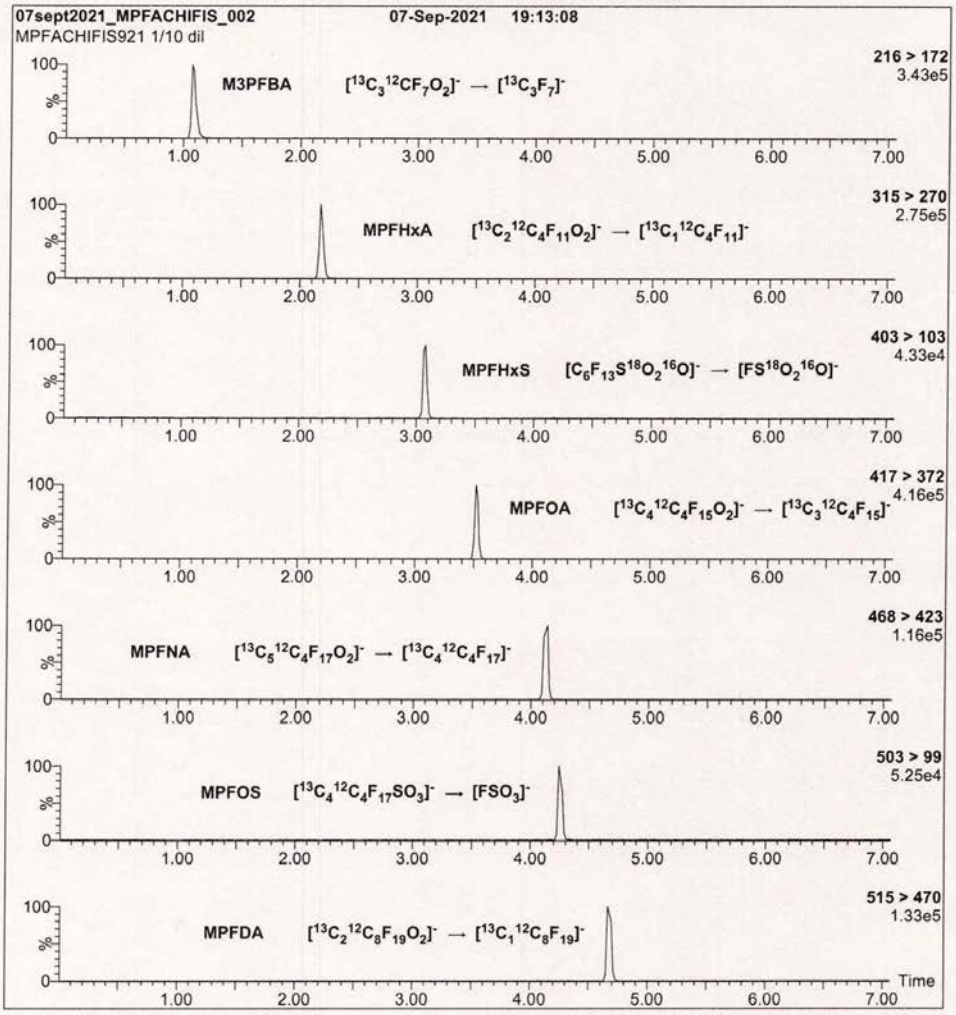
Figure 2: **MPFAC-HIF-IS; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (MPFAC-HIF-IS)

Mobile phase: Same as Figure 1

Flow: 300 µL/min

MS Parameters:

Collision Gas (mbar) = 3.18e-3
Collision Energy (eV) = 4-64 (variable)

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rev1

Attachment 2 – Extracted Internal Standards (EIS)

YORK
ANALYTICAL LABORATORIES, INC.

Analytical Standard Record

Standard ID: **Y22B198**

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

WELLINGTON LABORATORIES

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)	08/05/2021
LAST TESTED: (mm/dd/yyyy)	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 (^{13}C) perfluoroalkylcarboxylic acids (C_4-C_{12}, C_{14}), three mass-labelled (^{13}C) perfluoroalkanesulfonates (C_4, C_8, and C_8), three mass-labelled (one ^{13}C and two ^{2}H) perfluoro-1-octanesulfonamides, three mass-labelled (^{13}C) fluorotelomer sulfonates (4:2, 6:2, and 8:2), two mass-labelled (^{2}H) perfluorooctanesulfonamidoacetic acids, two mass-labelled (^{2}H) perfluorooctanesulfonamidoethanols, and mass-labelled (^{13}C) hexafluoropropylene oxide dimer acid. The components and their concentrations are given in Table A.

The individual mass-labelled perfluoroalkylcarboxylic acids, mass-labelled perfluoroalkanesulfonates, mass-labelled fluorotelomer sulfonates, perfluoro-1-($^{13}C_8$)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 (^{2}H) 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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Form#:13, Issued 2004-11-10
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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/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products, 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 $x_1, x_2, \ldots x_n$ on which it depends is:

$$u_c(y(x_1, x_2, \ldots x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

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).

CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

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

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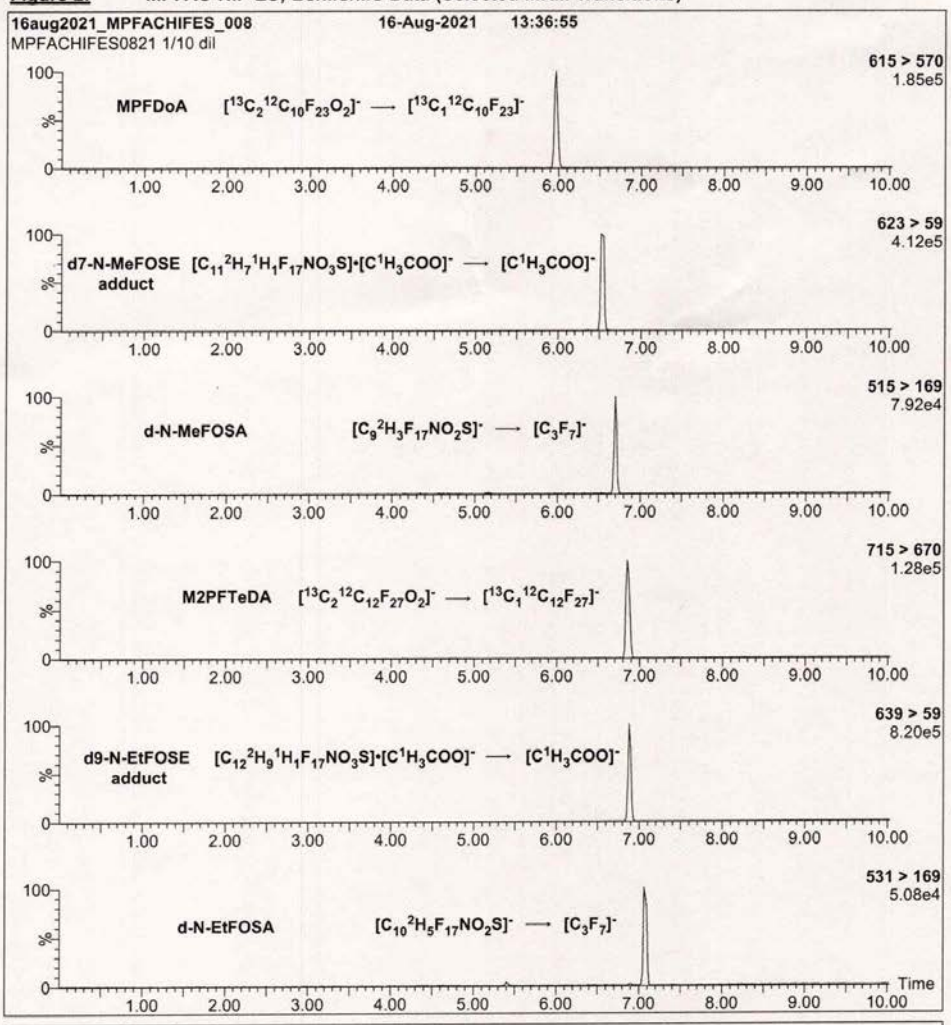
rev1

Figure 2: **MPFAC-HIF-ES; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (MPFAC-HIF-ES)

Mobile phase: Same as Figure 1

Flow: 300 µL/min

MS Parameters:

Collision Gas (mbar) = 3.41e-3
Collision Energy (eV) = 4-64 (variable)

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

MPFACHIFES0821 (7 of 7)
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Table A: **MPFAC-HIF-ES; Components and Concentrations (ng/mL, ± 5% in Methanol/Isopropanol (1%)/Water (<1%))**

Compound	Acronym	Concentration (ng/mL)	Peak Assignment in Figure 1
Perfluoro-n-($^{13}C_4$)butanoic acid	MPFBA	2000	1
Perfluoro-n-($^{13}C_5$)pentanoic acid	M5PFPeA	1000	2
Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	M5PFHxA	500	5
Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	M4PFHpA	500	7
Perfluoro-n-($^{13}C_8$)octanoic acid	M8PFOA	500	10
Perfluoro-n-($^{13}C_9$)nonanoic acid	M9PFNA	250	11
Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	M6PFDA	250	14
Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	M7PFUdA	250	17
Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	MPFDoA	250	19
Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	M2PFTeDA	250	22
Perfluoro-1-($^{13}C_8$)octanesulfonamide	M8FOSA	500	18
N-methyl-d_3-perfluoro-1-octanesulfonamide	d-N-MeFOSA	500	21
N-ethyl-d_5-perfluoro-1-octanesulfonamide	d-N-EtFOSA	500	24
N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	d3-N-MeFOSAA	1000	15
N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	d5-N-EtFOSAA	1000	16
2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	d7-N-MeFOSE	5000	20
2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	d9-N-EtFOSE	5000	23
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	M3HFPO-DA	2000	6

Compound	Acronym	Concentration* (ng/mL)		Peak Assignment in Figure 1
		as the salt	as the acid	
Sodium perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonate	M3PFBS	500	466	3
Sodium perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonate	M3PFHxS	500	474	8
Sodium perfluoro-1-($^{13}C_8$)octanesulfonate	M8PFOS	500	479	12
Sodium 1H,1H,2H,2H-perfluoro-(1,2-$^{13}C_2$)hexanesulfonate	M2-4:2FTS	1000	938	4
Sodium 1H,1H,2H,2H-perfluoro-(1,2-$^{13}C_2$)octanesulfonate	M2-6:2FTS	1000	951	9
Sodium 1H,1H,2H,2H-perfluoro-(1,2-$^{13}C_2$)decanesulfonate	M2-8:2FTS	1000	960	13

* Concentrations have been rounded to three significant figures.

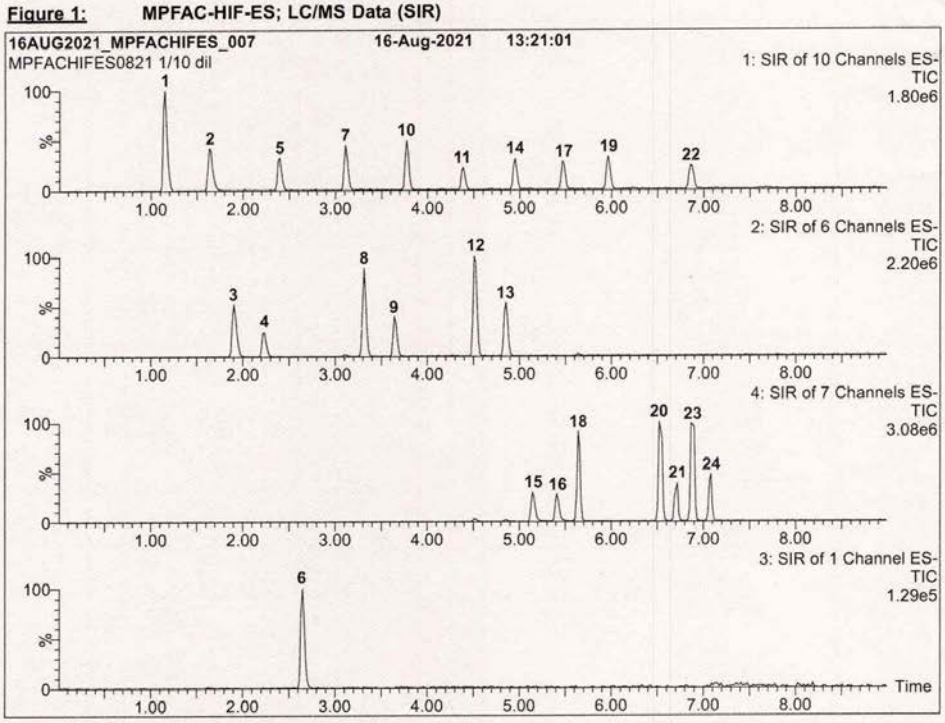
Certified By: _____ **Date:** 10/13/2021 (mm/dd/yyyy)

B.G. Chittim, General Manager

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

MPFACHIFES0821 (3 of 7)
rev1

Figure 1: **MPFAC-HIF-ES; LC/MS Data (SIR)**



Conditions for Figure 1:

Waters Acquity Ultra Performance LC
Waters Xevo TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H_2O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 9 min and hold for
2 min before returning to initial conditions in 1 min.
Time: 15 min

Flow: 300 µL/min

MS Parameters:

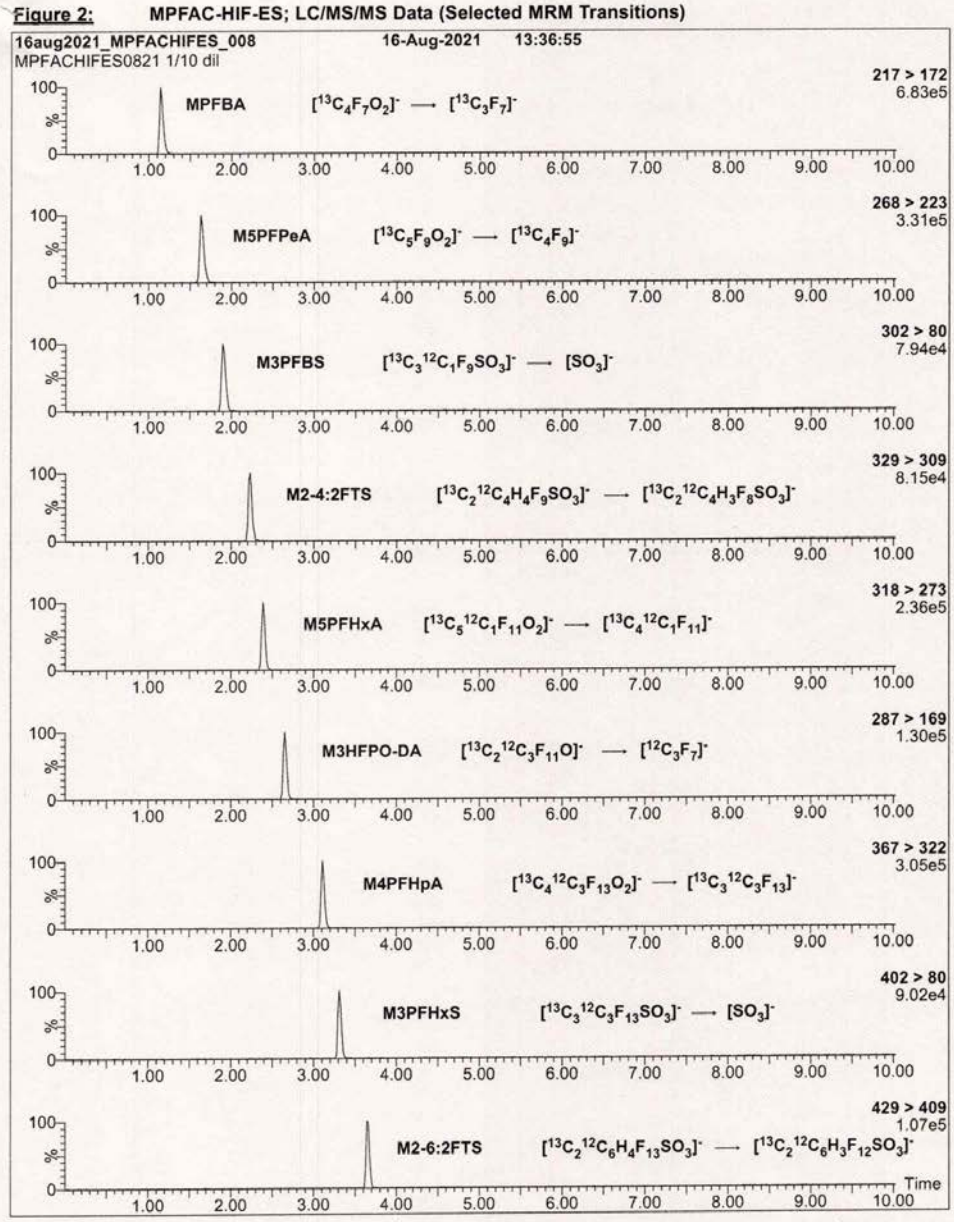
Experiment: SIR

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = variable (2-44)
Desolvation Temperature (°C) = 350
Desolvation Gas Flow (L/hr) = 1000

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

MPFACHIFES0821 (4 of 7)
rev1

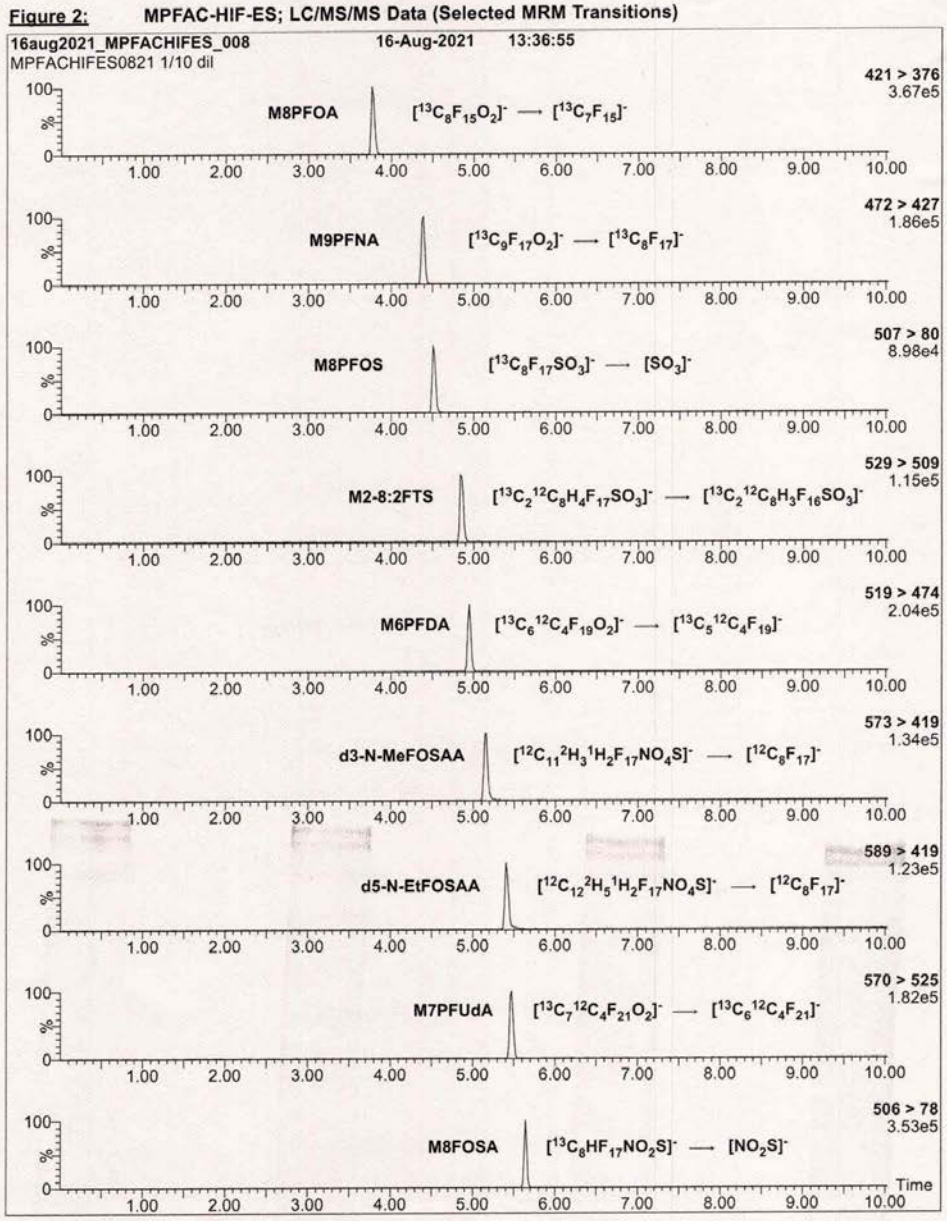
Figure 2: **MPFAC-HIF-ES; LC/MS/MS Data (Selected MRM Transitions)**



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

MPFACHIFES0821 (5 of 7)
rev1

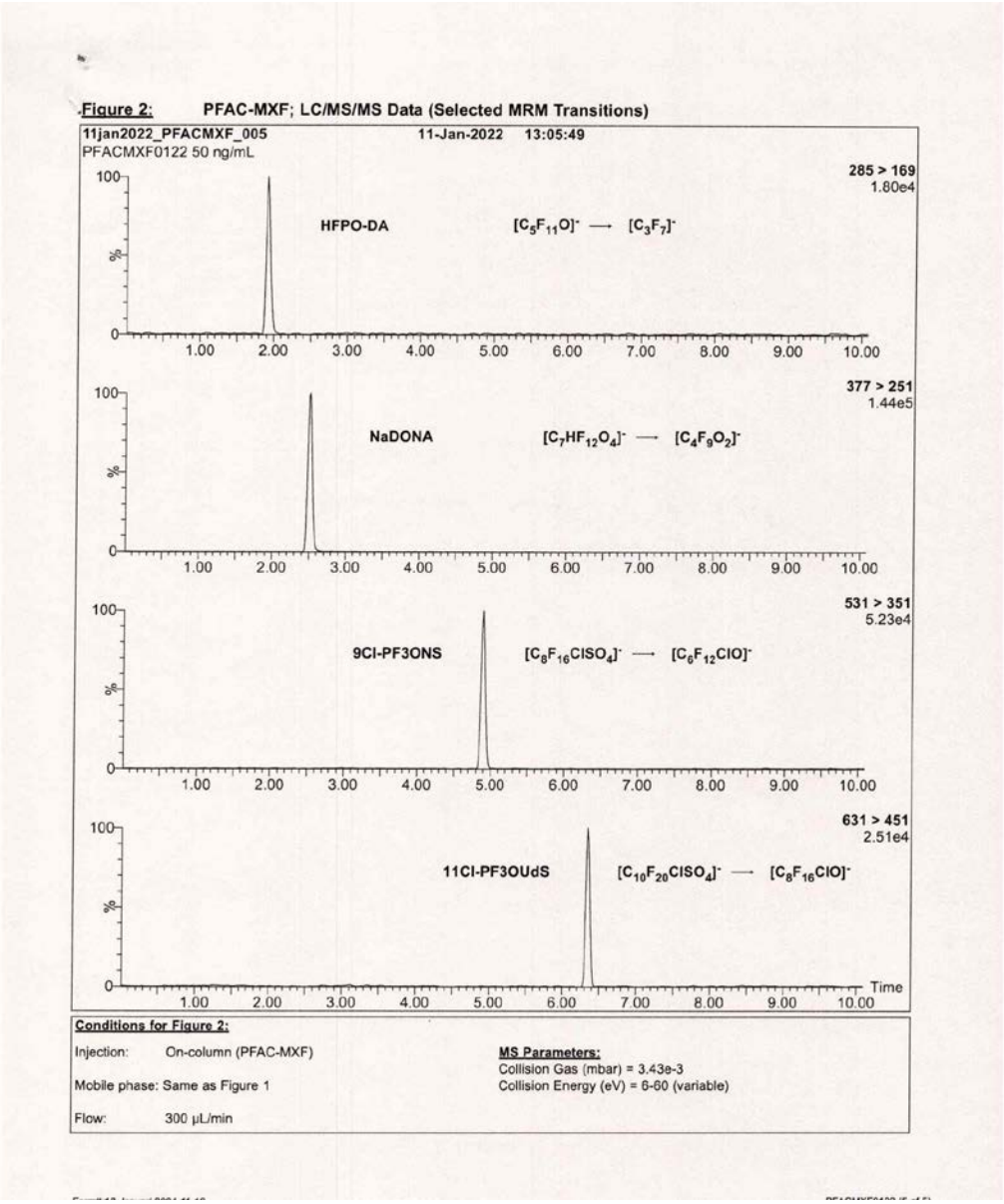
Figure 2: **MPFAC-HIF-ES; LC/MS/MS Data (Selected MRM Transitions)**



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

MPFACHIFES0821 (6 of 7)
rev1

Attachment 3 – Target Analyte Mixtures



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 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 $x_1, x_2, \ldots x_n$ on which it depends is:

$$u_c(y(x_1, x_2, \ldots x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

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).

CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

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	2000		A
Compound	**Acronym**	**Concentration* (ng/mL)**		**Peak Assignment in Figure 1**
		as the salt	**as the acid**	
Sodium dodecafluoro-3H-4,8-dioxanonanoate	NaDONA	2000	1890	B
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	9Cl-PF3ONS	2000	1870	C
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	11Cl-PF3OUdS	2000	1890	D

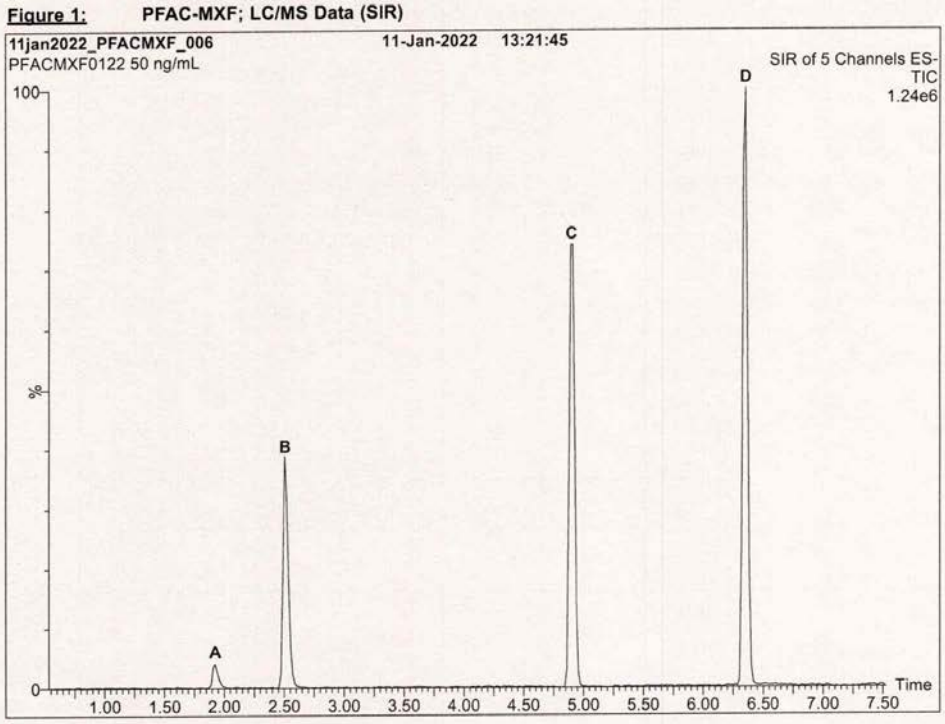
* Concentrations have been rounded to three significant figures.

Certified By: _____ **Date:** 01/12/2022 (mm/dd/yyyy)
B.G. Chittim, General Manager

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

PFACMXF0122 (3 of 5)
rev0

Figure 1: **PFAC-MXF; LC/MS Data (SIR)**



Conditions for Figure 1:

Waters Acquity Ultra Performance LC
Waters Xevo TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 45% H_2O / 55% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions in 0.75 min.
Time: 12 min

Flow: 300 µL/min

MS Parameters:

Experiment: SIR

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = variable (15-74)
Desolvation Temperature (°C) = 325
Desolvation Gas Flow (L/hr) = 1000

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

PFACMXF0122 (4 of 5)
rev0

YORK
ANALYTICAL LABORATORIES, INC.

Analytical Standard Record

Standard ID: **Y22B199**

Description:	PFAC-MXF-Native Repl.STOCK EPA 1633 PFAS	Prepared:	02/17/2022
Standard Type:	Other	**Expires:**	**01/11/2025**
Solvent:	MeOH/H20	Prepared By:	Robert Q. Bradley
Final Volume (mls):	1	Department:	PFAS
Vials:	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

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

PFAC-MXF

Native Replacement PFAS Solution/Mixture

PRODUCT CODE:	PFAC-MXF
LOT NUMBER:	PFACMXF0122
SOLVENT(S):	Methanol / Water (<1%)
DATE PREPARED: (mm/dd/yyyy)	01/10/2022
LAST TESTED: (mm/dd/yyyy)	01/11/2022
EXPIRY DATE: (mm/dd/yyyy)	01/11/2025
RECOMMENDED STORAGE:	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 (9Cl-PF3ONS and 11Cl-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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Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

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

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

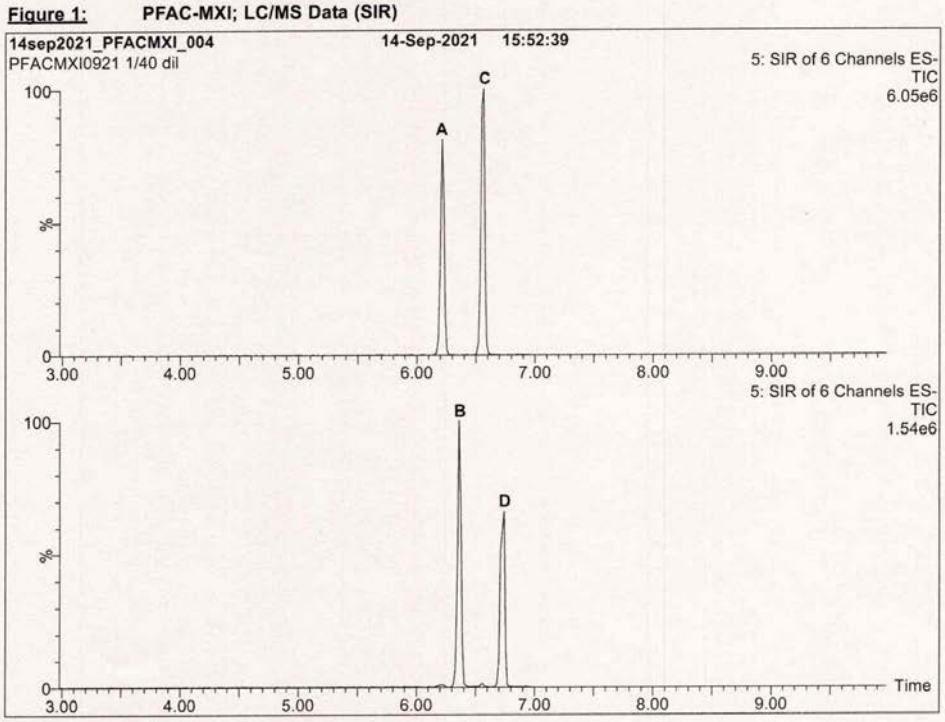
Compound	Acronym	Concentration (µg/mL)	Peak Assignment in Figure 1
N-methylperfluoro-1-octanesulfonamide	N-MeFOSA	1.00	B
N-ethylperfluoro-1-octanesulfonamide	N-EtFOSA	1.00	D
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	N-MeFOSE	10.0	A
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	N-EtFOSE	10.0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

Certified By: _____ **Date:** 09/23/2021
(mm/dd/yyyy)
B.G. Chittim, General Manager

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

PFACMXI0921 (3 of 5)
rev0

Figure 1: **PFAC-MXI; LC/MS Data (SIR)**



Conditions for Figure 1:

Waters Acquity Ultra Performance LC
Waters Xevo TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H_2O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 9 min and hold for
2 min before returning to initial conditions in 1 min.
Time: 15 min

Flow: 300 µL/min

MS Parameters:

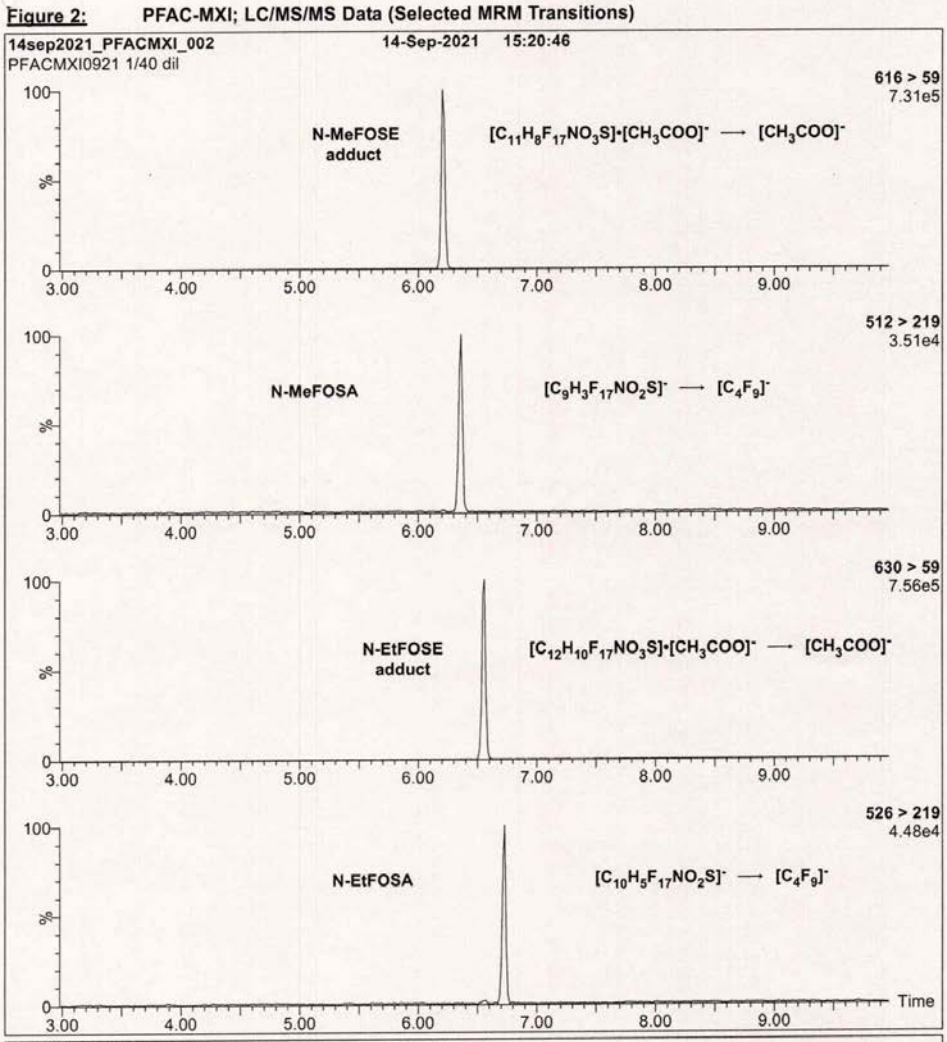
Experiment: SIR

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.50
Cone Voltage (V) = variable (2-74)
Desolvation Temperature (°C) = 350
Desolvation Gas Flow (L/hr) = 1000

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

PFACMXI0921 (4 of 5)
rev0

Figure 2: **PFAC-MXI; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (PFAC-MXI)

Mobile phase: Same as Figure 1

Flow: 300 µL/min

MS Parameters:
Collision Gas (mbar) = 3.31e-3
Collision Energy (eV) = 6-60 (variable)

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

PFACMXI0921 (5 of 5)
rev0

YORK
ANALYTICAL LABORATORIES, INC.

Analytical Standard Record

Standard ID: **Y22B204**

Description:	PFAC-MXI-EPA 1633 Stock	Prepared:	02/17/2022
Standard Type:	Other	**Expires:**	**02/17/2023**
Solvent:	Methanol	Prepared By:	Robert Q. Bradley
Final Volume (mls):	1	Department:	PFAS
Vials:	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

WELLINGTON LABORATORIES

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/yyyy)	09/08/2021
LAST TESTED: (mm/dd/yyyy)	09/14/2021
EXPIRY DATE: (mm/dd/yyyy)	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

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

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

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 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 $x_1, x_2, \ldots x_n$ on which it depends is:

$$u_c(y(x_1, x_2, \ldots x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

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).

CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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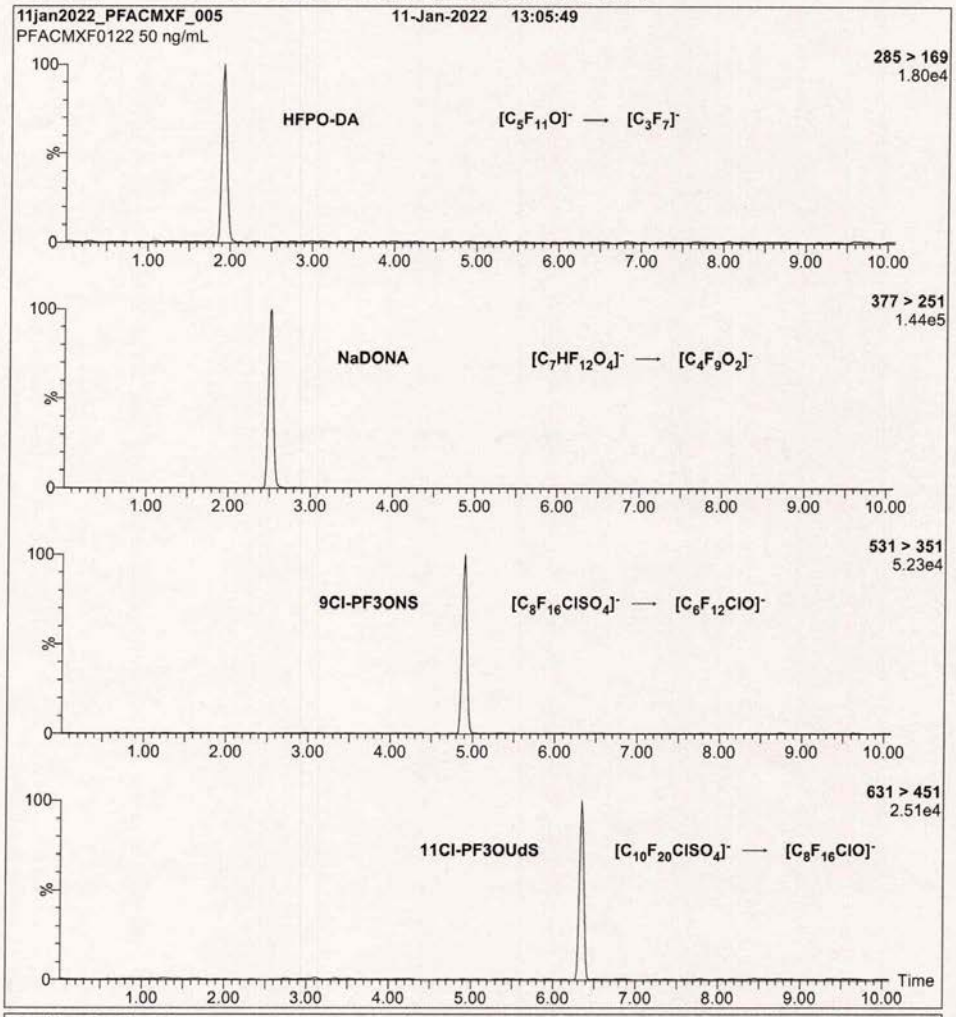
Form#:13, Issued 2004-11-10
Revision#:9, Revised 2020-12-23

PFACMXI0921 (2 of 5)
rev0

Figure 2: **PFAC-MXF; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (PFAC-MXF)

Mobile phase: Same as Figure 1

Flow: 300 µL/min

MS Parameters:
Collision Gas (mbar) = 3.43e-3
Collision Energy (eV) = 6-60 (variable)

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

PFACMXF0122 (5 of 5)
rev0

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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$$u_c(y(x_1, x_2, \ldots x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

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).

CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

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	2000		A
Compound	**Acronym**	**Concentration* (ng/mL)**		**Peak Assignment in Figure 1**
		as the salt	**as the acid**	
Sodium dodecafluoro-3H-4,8-dioxanonanoate	NaDONA	2000	1890	B
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	9Cl-PF3ONS	2000	1870	C
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	11Cl-PF3OUdS	2000	1890	D

* Concentrations have been rounded to three significant figures.

Certified By: _____ **Date:** 01/12/2022 (mm/dd/yyyy)
B.G. Chittim, General Manager

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

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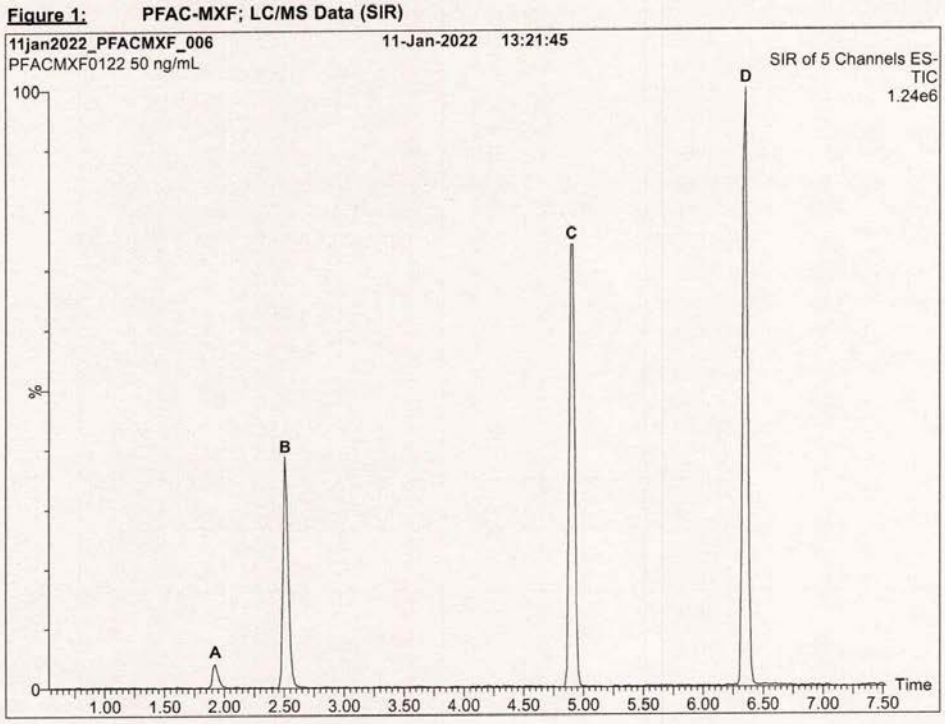
Figure 1: **PFAC-MXF; LC/MS Data (SIR)**



Conditions for Figure 1:

Waters Acquity Ultra Performance LC
Waters Xevo TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 45% H_2O / 55% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions in 0.75 min.
Time: 12 min

Flow: 300 µL/min

MS Parameters:

Experiment: SIR

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = variable (15-74)
Desolvation Temperature (°C) = 325
Desolvation Gas Flow (L/hr) = 1000

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Revision#:9, Revised 2020-12-23

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YORK
ANALYTICAL LABORATORIES, INC.

Analytical Standard Record

Standard ID: **Y22B199**

Description:	PFAC-MXF-Native Repl.STOCK EPA 1633 PFAS	Prepared:	02/17/2022
Standard Type:	Other	**Expires:**	**01/11/2025**
Solvent:	MeOH/H20	Prepared By:	Robert Q. Bradley
Final Volume (mls):	1	Department:	PFAS
Vials:	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

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WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

PFAC-MXF

Native Replacement PFAS Solution/Mixture

PRODUCT CODE:	PFAC-MXF
LOT NUMBER:	PFACMXF0122
SOLVENT(S):	Methanol / Water (<1%)
DATE PREPARED: (mm/dd/yyyy)	01/10/2022
LAST TESTED: (mm/dd/yyyy)	01/11/2022
EXPIRY DATE: (mm/dd/yyyy)	01/11/2025
RECOMMENDED STORAGE:	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 (9Cl-PF3ONS and 11Cl-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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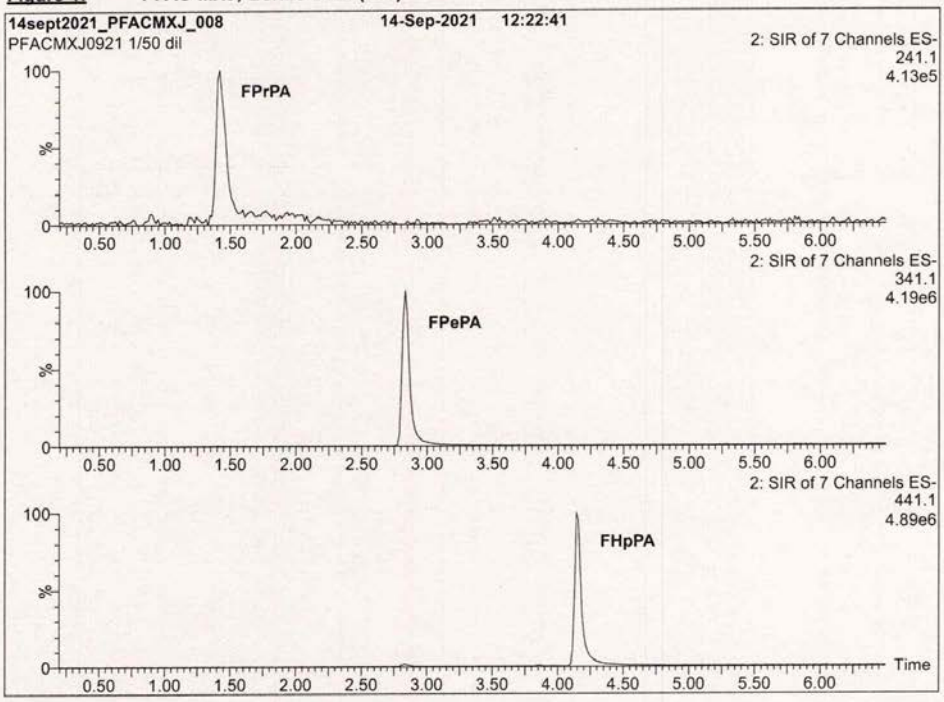
Form#:13, Issued 2004-11-10
Revision#:9, Revised 2020-12-23

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: _____ **Date:** 10/02/2021 (mm/dd/yyyy)
B.G. Chittim, General Manager

Figure 1: **PFAC-MXJ; LC/MS Data (SIR)**



Conditions for Figure 1:

Waters Acquity Ultra Performance LC
Waters Xevo TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H_2O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 9 min and hold for
2 min before returning to initial conditions in 1 min.
Time: 15 min

Flow: 300 µL/min

MS Parameters:

Experiment: SIR

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.50
Cone Voltage (V) = variable (2-74)
Desolvation Temperature (°C) = 350
Desolvation Gas Flow (L/hr) = 1000

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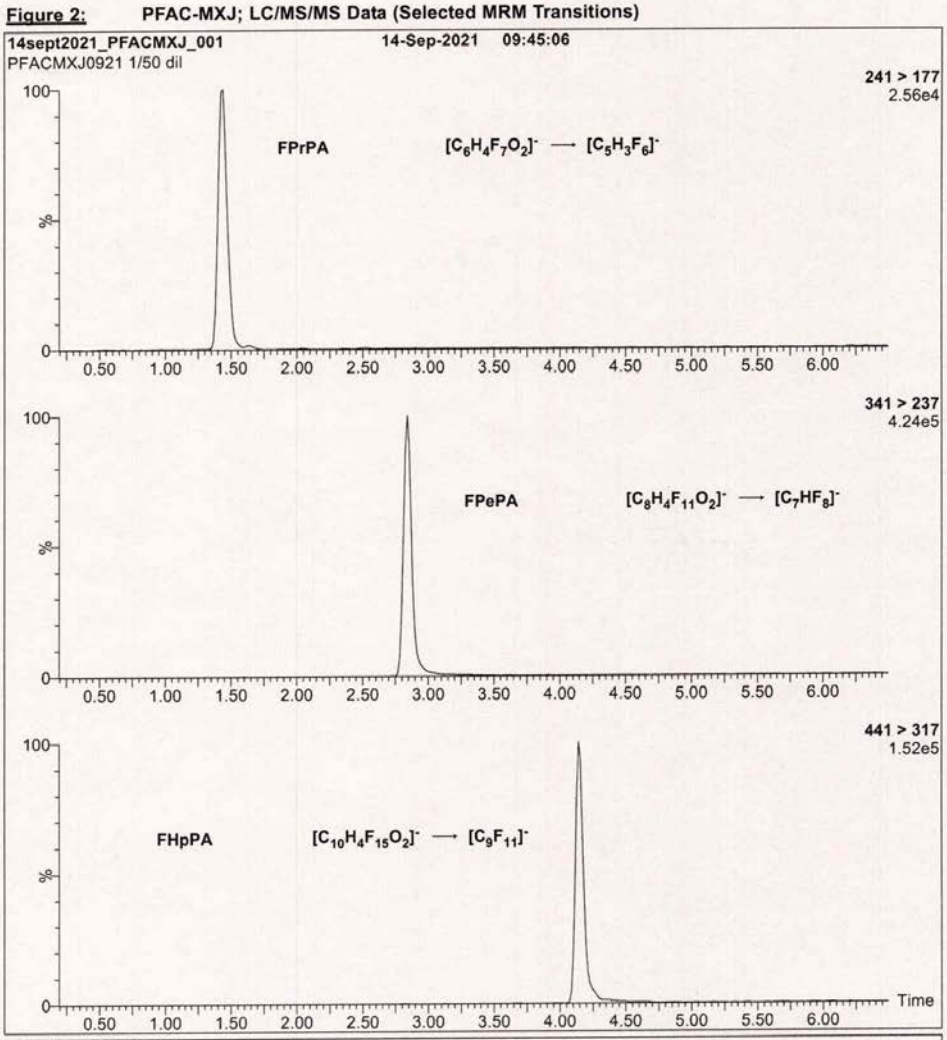
Figure 2: **PFAC-MXJ; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (PFAC-MXJ)

Mobile phase: Same as Figure 1

Flow: 300 µL/min

MS Parameters:
Collision Gas (mbar) = 3.31e-3
Collision Energy (eV) = 6-60 (variable)

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

PFACMXJ0921 (5 of 5)
rev1

YORK
ANALYTICAL LABORATORIES, INC.

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):	1	Department:	PFAS
Vials:	1	Lot No.:	PFACMXJ0921
Vendor:	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 Date

Page 1 of 1

WELLINGTON LABORATORIES

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/yyyy)	09/08/2021
LAST TESTED: (mm/dd/yyyy)	09/14/2021
EXPIRY DATE: (mm/dd/yyyy)	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.

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

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 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 $x_1, x_2, \ldots x_n$ on which it depends is:

$$u_c(y(x_1, x_2, \ldots x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

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).

CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

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PFACMXJ0921 (2 of 5)
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Figure 2: **PFAC-MXH; LC/MS/MS Data (Selected MRM Transitions)**

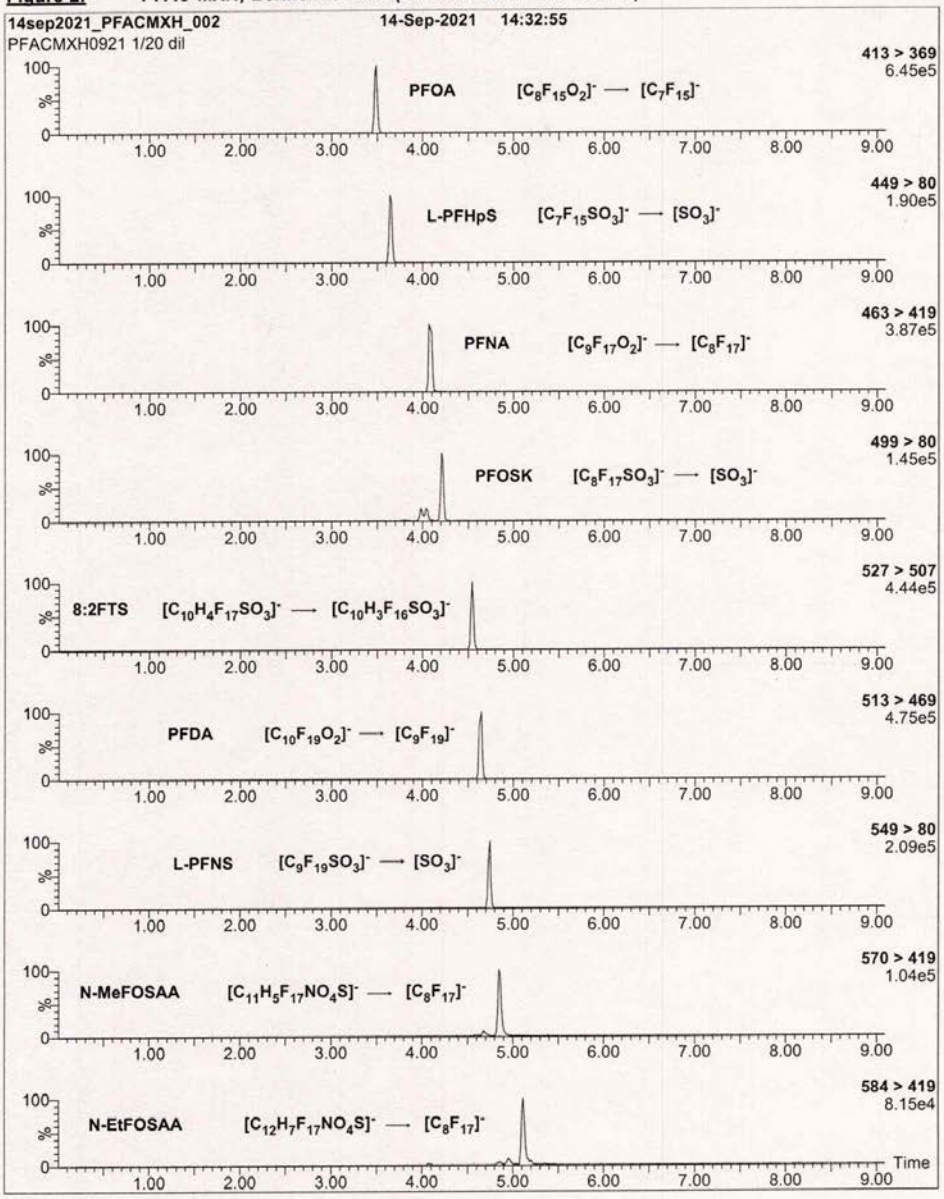

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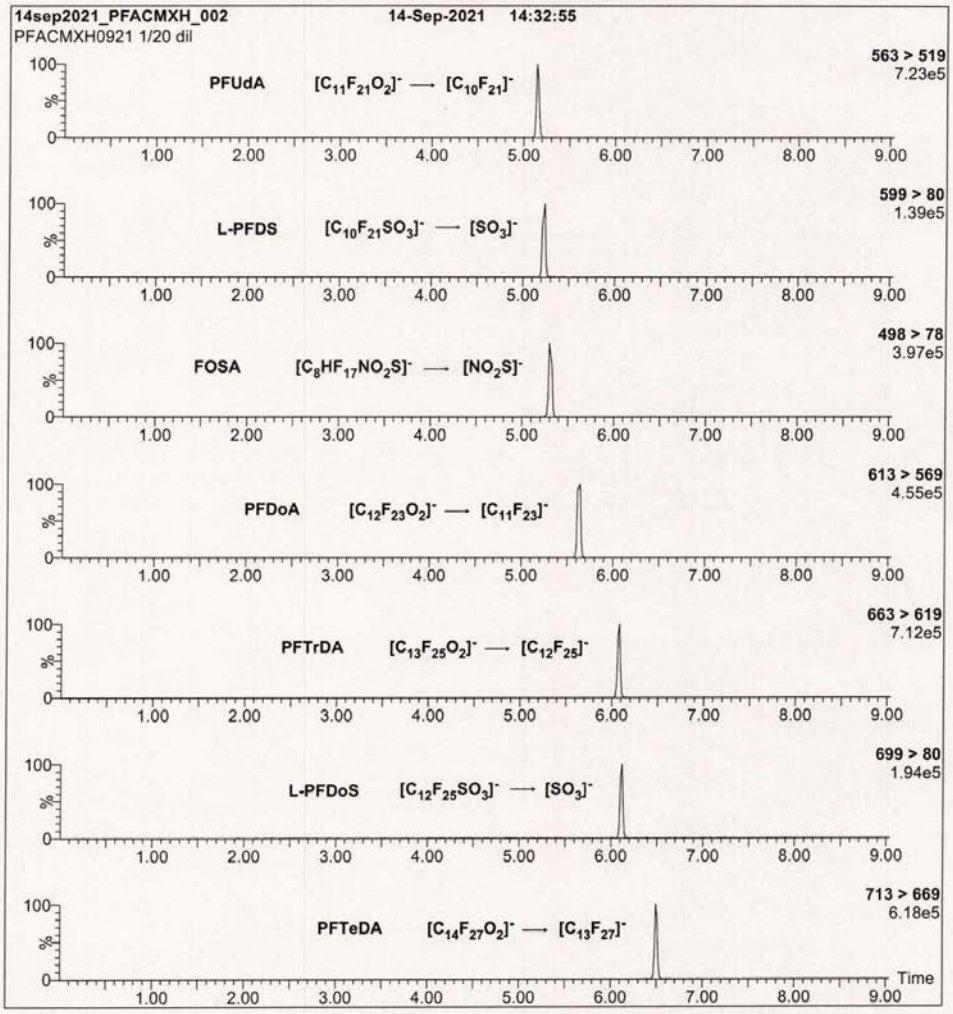
Figure 2: **PFAC-MXH; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (PFAC-MXH)

Mobile phase: Same as Figure 1

Flow: 300 µL/min

MS Parameters:
Collision Gas (mbar) = 3.31e-3
Collision Energy (eV) = 6-60 (variable)

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

PFACMXH0921 (11 of 11)
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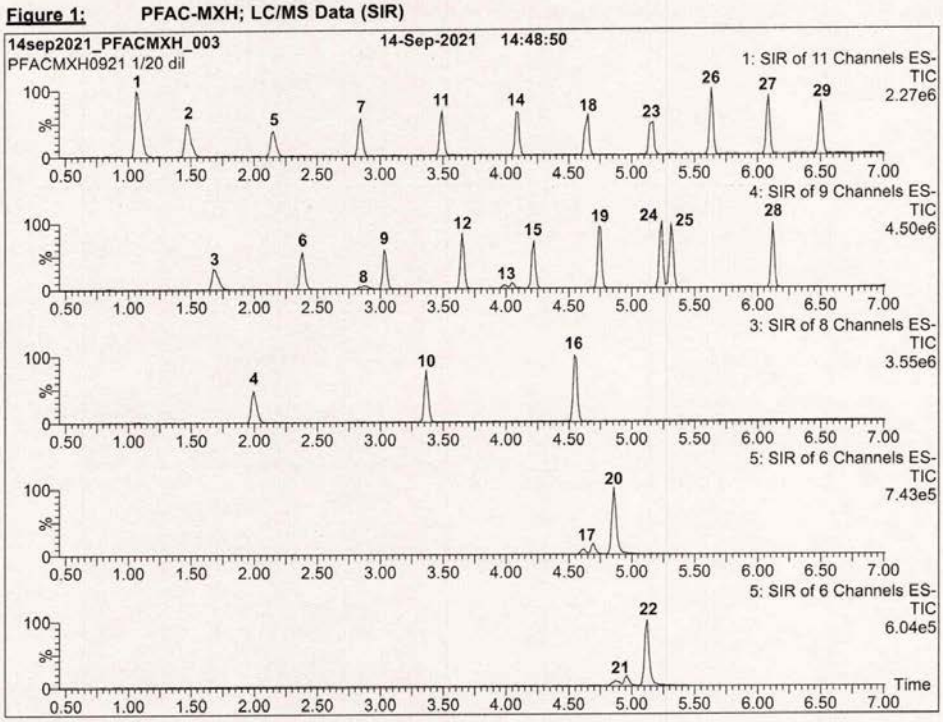
Table E: **PFOSK; Isomeric Components and Percent Composition (by ^{19}F-NMR)***

Isomer	Compound	Structure	Percent Composition by ^{19}F-NMR	
1	Potassium perfluoro-1-octanesulfonate	$CF_3CF_2CF_2CF_2CF_2CF_2CF_2CF_2SO_3^-K^+$	78.8	78.8
2	Potassium 1-trifluoromethylperfluoroheptanesulfonate**	$CF_3CF_2CF_2CF_2CF_2CF_2CF(CF_3)SO_3^-K^+$	1.2	21.1
3	Potassium 2-trifluoromethylperfluoroheptanesulfonate	$CF_3CF_2CF_2CF_2CF_2CF(CF_3)CF_2SO_3^-K^+$	0.6	
4	Potassium 3-trifluoromethylperfluoroheptanesulfonate	$CF_3CF_2CF_2CF_2CF(CF_3)CF_2CF_2SO_3^-K^+$	1.9	
5	Potassium 4-trifluoromethylperfluoroheptanesulfonate	$CF_3CF_2CF_2CF(CF_3)CF_2CF_2CF_2SO_3^-K^+$	2.2	
6	Potassium 5-trifluoromethylperfluoroheptanesulfonate	$CF_3CF_2CF(CF_3)CF_2CF_2CF_2CF_2SO_3^-K^+$	4.5	
7	Potassium 6-trifluoromethylperfluoroheptanesulfonate	$CF_3CF(CF_3)CF_2CF_2CF_2CF_2CF_2SO_3^-K^+$	10.0	
8	Potassium 5,5-di(trifluoromethyl)perfluorohexanesulfonate	$CF_3C(CF_3)CF_2CF_2CF_2CF_2SO_3^-K^+$	0.2	
9	Potassium 4,4-di(trifluoromethyl)perfluorohexanesulfonate	$CF_3CF_2C(CF_3)CF_2CF_2CF_2SO_3^-K^+$	0.03	
10	Potassium 4,5-di(trifluoromethyl)perfluorohexanesulfonate	$CF_3CF(CF_3)CF_2CF_2CF_2SO_3^-K^+$	0.4	
11	Potassium 3,5-di(trifluoromethyl)perfluorohexanesulfonate	$CF_3CF(CF_3)CF_2CF_2CF_2SO_3^-K^+$	0.07	

* Percent of total perfluorooctanesulfonate isomers only.
** Systematic Name: Potassium perfluorooctane-2-sulfonate.

Figure 1: **PFAC-MXH; LC/MS Data (SIR)**



Conditions for Figure 1:

Waters Acquity Ultra Performance LC
Waters Xevo TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP_{18}
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H_2O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 9 min and hold for 2 min
before returning to initial conditions in 1 min.
Time: 15 min

Flow: 300 µL/min

MS Parameters:

Experiment: SIR

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.50
Cone Voltage (V) = variable (2-74)
Desolvation Temperature (°C) = 350
Desolvation Gas Flow (L/hr) = 1000

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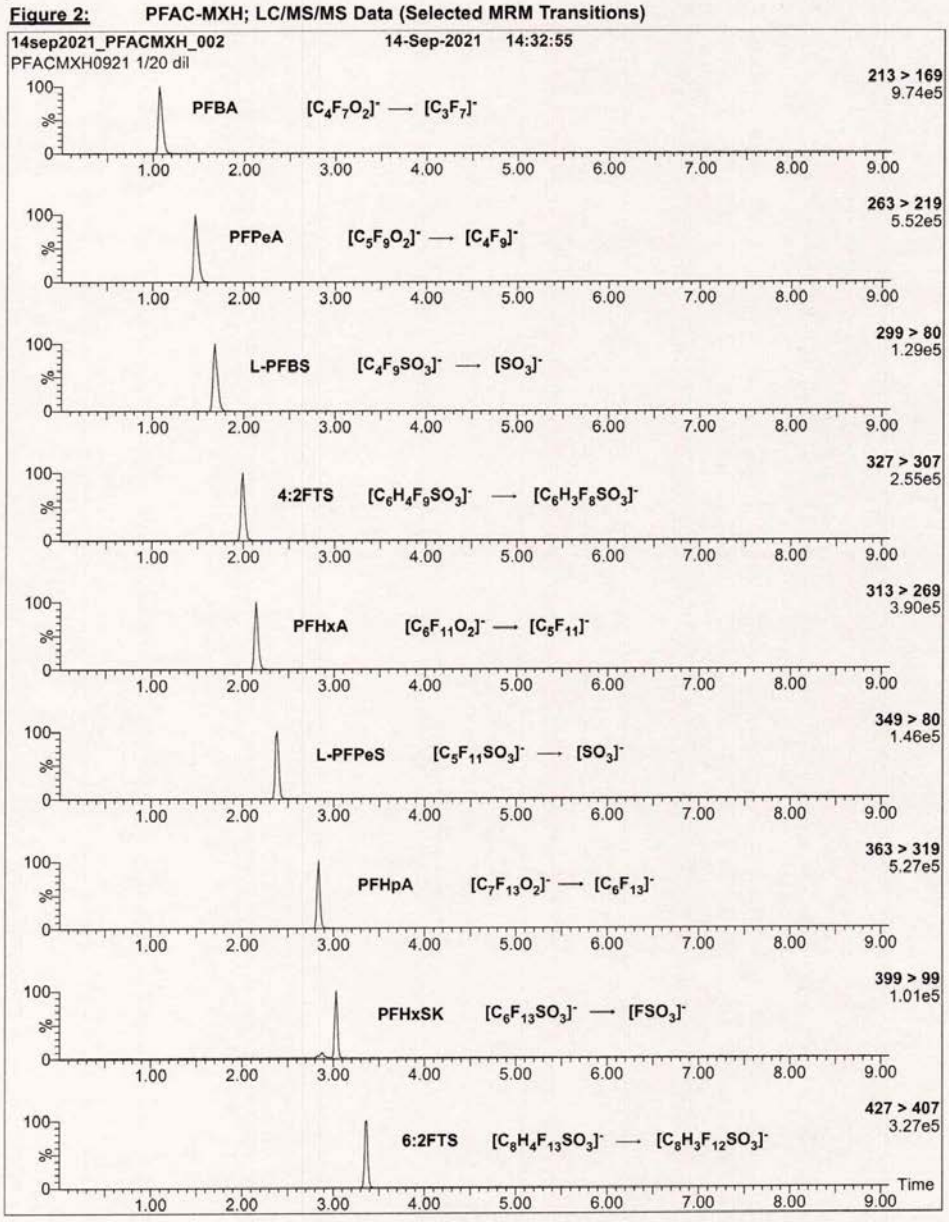
Figure 2: **PFAC-MXH; LC/MS/MS Data (Selected MRM Transitions)**



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Revision#:9, Revised 2020-12-23

PFACMXH0921 (9 of 11)
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Table B: **br-NMeFOSAA; Isomeric Components and Percent Composition (by ^{19}F-NMR)***

Isomer	Compound	Structure	Percent Composition by ^{19}F-NMR	
1	N-methylperfluoro-1-octanesulfonamidoacetic acid	$CF_3(CF_2)_7SO_2N(CH_3)CH_2CO_2H$	76.0	76.0
2	N-methylperfluoro-3-methylheptanesulfonamidoacetic acid	$CF_3(CF_2)_3CF(CF_3)(CF_2)_2SO_2N(CH_3)CH_2CO_2H$	0.7	24.0
3	N-methylperfluoro-4-methylheptanesulfonamidoacetic acid	$CF_3(CF_2)_2CF(CF_3)(CF_2)_3SO_2N(CH_3)CH_2CO_2H$	2.0	
4	N-methylperfluoro-5-methylheptanesulfonamidoacetic acid	$CF_3CF_2CF(CF_3)(CF_2)_4SO_2N(CH_3)CH_2CO_2H$	6.0	
5	N-methylperfluoro-6-methylheptanesulfonamidoacetic acid	$CF_3CF(CF_3)(CF_2)_5SO_2N(CH_3)CH_2CO_2H$	14.0	
6	N-methylperfluoro-5,5-dimethylhexanesulfonamidoacetic acid	$CF_3C(CF_3)_2(CF_2)_4SO_2N(CH_3)CH_2CO_2H$	0.2	
7	Other Unidentified Isomers		1.1	

* Percent of total N-methylperfluorooctanesulfonamidoacetic acid isomers only.

Table C: **br-NEtFOSAA; Isomeric Components and Percent Composition (by ^{19}F-NMR)***

Isomer	Compound	Structure	Percent Composition by ^{19}F-NMR	
1	N-ethylperfluoro-1-octanesulfonamidoacetic acid	$CF_3(CF_2)_7SO_2NCH_2CO_2H$ C_2H_5	77.5	77.5
2	N-ethylperfluoro-3-methylheptanesulfonamidoacetic acid	$CF_3(CF_2)_3CF(CF_2)_2SO_2NCH_2CO_2H$ CF_3 C_2H_5	2.3	22.5
3	N-ethylperfluoro-4-methylheptanesulfonamidoacetic acid	$CF_3(CF_2)_2CF(CF_2)_3SO_2NCH_2CO_2H$ CF_3 C_2H_5	2.2	
4	N-ethylperfluoro-5-methylheptanesulfonamidoacetic acid	$CF_3CF_2CF(CF_2)_4SO_2NCH_2CO_2H$ CF_3 C_2H_5	5.4	
5	N-ethylperfluoro-6-methylheptanesulfonamidoacetic acid	$CF_3CF(CF_2)_5SO_2NCH_2CO_2H$ CF_3 C_2H_5	10.4	
6	N-ethylperfluoro-5,5-dimethylhexanesulfonamidoacetic acid	$CF_3C(CF_2)_4SO_2NCH_2CO_2H$ CF_3 C_2H_5	0.3	
7	N-ethylperfluoro-4,5-dimethylhexanesulfonamidoacetic acid	$CF_3CFCF(CF_2)_3SO_2NCH_2CO_2H$ CF_3 C_2H_5	0.3	
8	N-ethylperfluoro-3,5-dimethylhexanesulfonamidoacetic acid	$CF_3CFCF_2CF(CF_2)_2SO_2NCH_2CO_2H$ CF_3 C_2H_5	0.3	
9	Other Unidentified Isomers		1.3	

* Percent of total N-ethylperfluorooctanesulfonamidoacetic acid isomers only.

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

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rev0

Table D: **PFHxSK; Isomeric Components and Percent Composition (by ^{19}F-NMR)***

Isomer	Compound	Structure	Percent Composition by ^{19}F-NMR	
1	Potassium perfluoro-1-hexanesulfonate	$CF_3CF_2CF_2CF_2CF_2CF_2SO_3^-K^+$	81.1	81.1
2	Potassium 1-trifluoromethylperfluoropentanesulfonate**	$CF_3CF_2CF_2CF_2CF(CF_3)SO_3^-K^+$	2.9	18.9
3	Potassium 2-trifluoromethylperfluoropentanesulfonate	$CF_3CF_2CF_2CF(CF_3)CF_2SO_3^-K^+$	1.4	
4	Potassium 3-trifluoromethylperfluoropentanesulfonate	$CF_3CF_2CF(CF_3)CF_2CF_2SO_3^-K^+$	5.0	
5	Potassium 4-trifluoromethylperfluoropentanesulfonate	$CF_3CF(CF_3)CF_2CF_2CF_2SO_3^-K^+$	8.9	
6	Potassium 3,3-di(trifluoromethyl)perfluorobutanesulfonate	$CF_3C(CF_3)CF_2CF_2SO_3^-K^+$	0.2	
7	Other Unidentified Isomers		0.5	

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

WELLINGTON LABORATORIES

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_8, 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 perfluorooctanesulfonamidoacetic acids, and perfluoro-1-octanesulfonamide (FOSA). The components and their concentrations are given in Table A.

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.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

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

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

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 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 $x_1, x_2,...x_n$ on which it depends is:

$$u_c(y(x_1, x_2,...x_n)) = \sqrt{\sum_{i=1}^{n} u(y, x_i)^2}$$

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

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of ±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).

CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

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

PFACMXH0921 (2 of 11)
rev0

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

Compound	Acronym	Concentration* (µg/mL)	Peak Assignment in Figure 1
Perfluoro-n-butanoic acid	PFBA	4.00	1
Perfluoro-n-pentanoic acid	PFPeA	2.00	2
Perfluoro-n-hexanoic acid	PFHxA	1.00	5
Perfluoro-n-heptanoic acid	PFHpA	1.00	7
Perfluoro-n-octanoic acid	PFOA	1.00	11
Perfluoro-n-nonanoic acid	PFNA	1.00	14
Perfluoro-n-decanoic acid	PFDA	1.00	18
Perfluoro-n-undecanoic acid	PFUdA	1.00	23
Perfluoro-n-dodecanoic acid	PFDoA	1.00	26
Perfluoro-n-tridecanoic acid	PFTrDA	1.00	27
Perfluoro-n-tetradecanoic acid	PFTeDA	1.00	29
Perfluoro-1-octanesulfonamide	FOSA	1.00	25
N-methylperfluorooctanesulfonamidoacetic acid [a]	N-MeFOSAA: linear isomer	0.760	20
	N-MeFOSAA: ∑ branched isomers	0.240	17
N-ethylperfluorooctanesulfonamidoacetic acid [b]	N-EtFOSAA: linear isomer	0.775	22
	N-EtFOSAA: ∑ branched isomers	0.225	21

Compound	Acronym	Concentration* (µg/mL)		Peak Assignment in Figure 1
		as the salt	as the acid	
Potassium perfluoro-1-butanesulfonate	L-PFBS	1.00	0.887	3
Sodium perfluoro-1-pentanesulfonate	L-PFPeS	1.00	0.941	6
Potassium perfluorohexanesulfonate [c]	PFHxSK: linear isomer	0.811	0.741	9
	PFHxSK: ∑ branched isomers	0.189	0.173	8
Sodium perfluoro-1-heptanesulfonate	L-PFHpS	1.00	0.953	12
Potassium perfluorooctanesulfonate [d]	PFOSK: linear isomer	0.788	0.732	15
	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

[a] See Table B for percent composition of linear and branched N-MeFOSAA isomers.
[b] See Table C for percent composition of linear and branched N-EtFOSAA isomers.
[c] See Table D for percent composition of linear and branched PFHxSK isomers.
[d] See Table E for percent composition of linear and branched PFOSK isomers.

* Concentrations have been rounded to three significant figures.

Certified By: _____ **Date:** 09/23/2021 (mm/dd/yyyy)
B.G. Chittim, General Manager

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

PFACMXH0921 (3 of 11)
rev0

YORK
ANALYTICAL LABORATORIES, INC.

Analytical Standard Record

Standard ID: **Y22B201**

Description:	PFAC-MXH STOCK PFAS EPA 1633	Prepared:	02/17/2022
Standard Type:	Other	**Expires:**	**09/14/2026**
Solvent:	MeOH/IPA/H2O	Prepared By:	Robert Q. Bradley
Final Volume (mls):	1	Department:	PFAS
Vials:	1	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

Attachment 4 – Calibration Concentrations, nominal

Calibration Solutions (ng/mL) Compound CS1 (LOQ) CS2 Perfluoroalkyl carboxylic acids			CS3	CS4 (CV[1])	CS5	CS6	CS7[2]
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
Perfluoroalkyl 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
Fluorotelomer 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
Perfluorooctane sulfonamides							
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
Perfluorooctane sulfonamidoacetic 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
Perfluorooctane sulfonamide ethanols							
NMeFOSE	2	5	12.5	25	50	125	625
NEtFOSE	2	5	12.5	25	50	125	625
Per- and polyfluoroether carboxylic 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-PF3OUdS	0.8	2	5	10	20	50	250
PFEESA	0.4	1	2.5	5	10	25	125

Calibration Solutions (ng/mL) Compound CS1 (LOQ) CS2 Fluorotelomer carboxylic			CS3	CS4 (CV[1])	CS5	CS6	CS7[2]
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 Standard (EIS) Analytes							
$^{13}C_4$-PFBA	10	10	10	10	10	10	10
$^{13}C_5$-PFPeA	5	5	5	5	5	5	5
$^{13}C_5$-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_4$-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_8$-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_9$-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_6$-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_7$-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_2$-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_2$-PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_3$-PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_3$-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_8$-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_2$-4:2FTS	5	5	5	5	5	5	5
$^{13}C_2$-6:2FTS	5	5	5	5	5	5	5
$^{13}C_2$-8:2FTS	5	5	5	5	5	5	5
$^{13}C_8$-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_3-NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D_3-NMeFOSAA	5	5	5	5	5	5	5
D_5-NEtFOSAA	5	5	5	5	5	5	5
D_7-NMeFOSE	25	25	25	25	25	25	25
D_9-NEtFOSE	25	25	25	25	25	25	25
$^{13}C_3$-HFPO-DA	10	10	10	10	10	10	10
Non-extracted Internal Standard (NIS) Analytes							
$^{13}C_3$-PFBA	5	5	5	5	5	5	5
$^{13}C_2$-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_4$-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_5$-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{13}C_2$-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
$^{18}O_2$-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
$^{13}C_4$-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5

[1] This calibration point is used as the calibration verification (CV)

[2] 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.

Attachment 5 -HPLC Method Parameters

HPLC Acquisition Method Report

Stroke A
Automatic Stroke Calculation A Yes

Compress A
Compressibility Mode A Compressibility Value Set
Compressibility A 70 10e-6/bar

Compress B
Compressibility Mode B Compressibility Value Set
Compressibility B 90 10e-6/bar

Stop Time
Stoptime Mode Time set
Stoptime 10.00 min

Post Time
Posttime Mode Time set
Posttime 1.50 min

Injection
Injection with needle wash
3.00 µL

Solvent Composition

	Channel	Name 1	Name 2	Selected	Used	Percent
1	A	Water 5mM ammonium acetate		Ch. 1	Yes	90.00 %
2	B	Methanol		Ch. 1	Yes	10.00 %

Timetable

	Time	A	B	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
Enable Analysis Left Temperature Value 0.8 °C

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

Attachment 6 - Triple Quadrupole Acquisition Method

Acquisition Method Report

Agilent Technologies

Acquisition Method Info

Method Name PFAS1633_ACQ_092922.m

Method Path D:\MassHunter\methods\PFAS1633_ACQ_092922.m

Method Description EPA 1633_Target PFAS Isotope Dilution_Acquisition

Device List

HiP Sampler
Binary Pump
Column Comp.
QQQ

MS QQQ Mass Spectrometer

Ion Source	AJS ESI	**Tune File**	D:\MassHunter\Tune\QQQ\G6460C\atunes.TUNE.XML
Stop Mode	No Limit/As Pump	**Stop Time (min)**	1
Time Filter	On	**Time Filter Width (min)**	0.07
LC->Waste Pre Row	N/A	**LC->Waste Post Row**	N/A

Time Segments

Index	Start Time (min)	Scan Type	Ion Mode	Div Valve	Delta EMV	Store	Cycle Time (ms)	Triggered?	MRM Repeats
1	0	DynamicMRM	ESI+Agilent Jet Stream	To MS	350	Yes	550	Yes	3

Time Segment 1

Scan Segments

Cpd Name	ISTD?	Prec Ion	MS1 Res	Prod Ion	MS2 Res	Primary	Trigger	Frag (V)	CE (V)	Cell Acc (V)	Ret Time (min)	Ret Window	Polarity
11-Cl-PF3OUdS	No	631	Unit/Enh (6490)	451	Unit/Enh (6490)	Yes	No	170	33	4	7.62	3	Negative
1H,1H,2H,2H-perfluoro-1-decanesulfonate (8 2FTS)	No	527	Unit/Enh (6490)	507	Unit/Enh (6490)	Yes	No	170	28	4	7.14	3	Negative
1H,1H,2H,2H-perfluoro-1-decanesulfonate (8 2FTS)	No	527	Unit/Enh (6490)	80.9	Unit/Enh (6490)	Yes	No	170	40	4	7.14	3	Negative
1H,1H,2H,2H-perfluoro-1-hexanesulfonate (4 2FTS)	No	327	Unit/Enh (6490)	307	Unit/Enh (6490)	Yes	No	162	20	4	4.788	3	Negative
1H,1H,2H,2H-perfluoro-1-hexanesulfonate (4 2FTS)	No	327	Unit/Enh (6490)	80.9	Unit/Enh (6490)	Yes	No	162	36	4	4.788	3	Negative
1H,1H,2H,2H-perfluoro-1-octanesulfonate (6 2FTS)	No	427	Unit/Enh (6490)	407	Unit/Enh (6490)	Yes	No	162	24	4	6.168	3	Negative
1H,1H,2H,2H-perfluoro-1-octanesulfonate (6 2FTS)	No	427	Unit/Enh (6490)	79.7	Unit/Enh (6490)	Yes	No	162	48	4	6.168	3	Negative
3:3FTCA	No	241	Unit/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

Acquisition Method Report

Agilent Technologies

Cpd Name	ISTD?	Prec Ion	MS1 Res	Prod Ion	MS2 Res	Primary	Trigger	Frag (V)	CE (V)	Cell Acc (V)	Ret Time (min)	Ret Window	Polarity
5-3FTCA	No	341	Unit/Enh (6490)	237	Unit/Enh (6490)	Yes	No	84	12	4	5.73	3	Negative
5-3FTCA	No	341	Unit/Enh (6490)	217	Unit/Enh (6490)	Yes	No	84	24	4	5.73	3	Negative
7-3FTCA	No	441	Unit/Enh (6490)	337	Unit/Enh (6490)	Yes	No	76	12	4	6.7	3	Negative
7-3FTCA	No	441	Unit/Enh (6490)	317	Unit/Enh (6490)	Yes	No	76	24	4	6.7	3	Negative
9-Cl-PF3ONS	No	531	Unit/Enh (6490)	351	Unit/Enh (6490)	Yes	No	175	29	4	6.89	3	Negative
ADONA	No	377	Unit/Enh (6490)	251	Unit/Enh (6490)	Yes	No	103	9	4	5.62	3	Negative
ADONA	No	377	Unit/Enh (6490)	85	Unit/Enh (6490)	Yes	No	103	37	4	5.62	3	Negative
d3-NMeFOSA	No	515	Unit/Enh (6490)	219	Unit/Enh (6490)	Yes	No	134	20	4	7.17	3	Negative
d3-N-MeFOSAA	No	572.99	Unit/Enh (6490)	418.8	Unit/Enh (6490)	Yes	No	130	20	4	7.17	3	Negative
d5-NEtFOSA	No	531	Unit/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-EtFOSAA	No	589.02	Unit/Enh (6490)	530.9	Unit/Enh (6490)	Yes	No	130	20	4	7.36	3	Negative
d5-N-EtFOSAA	No	589.02	Unit/Enh (6490)	418.8	Unit/Enh (6490)	Yes	No	130	20	4	7.36	3	Negative
d7-NMeFOSE	No	623	Unit/Enh (6490)	310	Unit/Enh (6490)	Yes	No	150	15	4	8.28	3	Negative
d7-NMeFOSE	No	623	Unit/Enh (6490)	59	Unit/Enh (6490)	Yes	No	88	15	4	8.28	3	Negative
d9-NEtFOSE	No	639	Unit/Enh (6490)	59	Unit/Enh (6490)	Yes	No	150	15	4	8.6	3	Negative
HFPO-DA	No	285	Unit/Enh (6490)	169.1	Unit/Enh (6490)	Yes	No	100	20	4	4.95	3	Negative
M2-4-2FTS	No	329	Unit/Enh (6490)	309	Unit/Enh (6490)	Yes	No	156	20	4	4.787	3	Negative
M2-4-2FTS	No	329	Unit/Enh (6490)	81	Unit/Enh (6490)	Yes	No	156	28	4	4.787	3	Negative
M2-6-2FTS	No	429	Unit/Enh (6490)	409	Unit/Enh (6490)	Yes	No	162	24	4	6.01	3	Negative
M2-6-2FTS	No	429	Unit/Enh (6490)	81	Unit/Enh (6490)	Yes	No	162	40	4	6.01	3	Negative
M2-8-2FTS	No	529	Unit/Enh (6490)	509	Unit/Enh (6490)	Yes	No	165	28	4	6.98	3	Negative
M2-8-2FTS	No	529	Unit/Enh (6490)	81	Unit/Enh (6490)	Yes	No	165	40	4	6.98	3	Negative
M2PFTeDA	No	715	Unit/Enh (6490)	670	Unit/Enh (6490)	Yes	No	62	12	4	8.25	3	Negative
M3-HFPO-DA	No	287	Unit/Enh (6490)	169	Unit/Enh (6490)	Yes	No	90	5	4	4.99	3	Negative
M3PFBA	Yes	216	Unit/Enh (6490)	172	Unit/Enh (6490)	Yes	No	90	5	4	1.2	2	Negative
M3PFBS	No	302	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	114	32	4	3.94	3	Negative
M3PFBS	No	302	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	114	40	4	3.94	3	Negative
M3PFHxS	No	402	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	165	40	4	5.55	3	Negative
M3PFHxS	No	402	Unit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	165	48	4	5.55	3	Negative
M4PFHpA	No	367	Unit/Enh (6490)	322	Unit/Enh (6490)	Yes	No	124	8	4	5.601	3	Negative
M5PFHxA	No	318	Unit/Enh (6490)	273	Unit/Enh (6490)	Yes	No	70	4	4	5.47	3	Negative
M5PFHxA	No	318	Unit/Enh (6490)	120	Unit/Enh (6490)	Yes	No	70	4	4	5.47	3	Negative
M6PFDA	No	519	Unit/Enh (6490)	473.9	Unit/Enh (6490)	Yes	No	59	8	4	6.99	3	Negative
M7PFUdA	No	570	Unit/Enh (6490)	525	Unit/Enh (6490)	Yes	No	64	8	4	7.38	3	Negative
MPFDA	Yes	514.98	Unit/Enh (6490)	469.8	Unit/Enh (6490)	Yes	No	94	5	4	6.972	2	Negative
MPFHxA	Yes	314.99	Unit/Enh (6490)	269.8	Unit/Enh (6490)	Yes	No	86	4	4	4.705	2	Negative
MPFHxA	Yes	314.99	Unit/Enh (6490)	120	Unit/Enh (6490)	Yes	No	86	4	4	4.705	2	Negative
MPFHxS	Yes	403	Unit/Enh (6490)	103	Unit/Enh (6490)	Yes	No	110	37	4	5.63	2	Negative
MPFHxS	Yes	403	Unit/Enh (6490)	84	Unit/Enh (6490)	Yes	No	110	40	4	5.63	2	Negative
MPFNA	Yes	468	Unit/Enh (6490)	423	Unit/Enh (6490)	Yes	No	66	4	4	6.541	2	Negative
MPFOA	Yes	417	Unit/Enh (6490)	372	Unit/Enh (6490)	Yes	No	84	4	4	6.03	2	Negative
MPFOS	Yes	502.96	Unit/Enh (6490)	99	Unit/Enh (6490)	Yes	No	148	48	4	6.57	3	Negative

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Acquisition Method Report

Agilent Technologies

Cpd Name	ISTD?	Prec Ion	MS1 Res	Prod Ion	MS2 Res	Primary	Trigger	Frag (V)	CE (V)	Cell Acc (V)	Ret Time (min)	Ret Window	Polarity
MPFOS	Yes	502.96	Unit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	148	54	4	6.57	3	Negative
NEtFOSA	No	526	Unit/Enh (6490)	219	Unit/Enh (6490)	Yes	No	120	20	4	8.528	3	Negative
NEtFOSA	No	526	Unit/Enh (6490)	169	Unit/Enh (6490)	Yes	No	120	20	4	8.528	3	Negative
N-EtFOSAA	No	584	Unit/Enh (6490)	525.9	Unit/Enh (6490)	Yes	No	130	20	4	7.521	3	Negative
N-EtFOSAA	No	584	Unit/Enh (6490)	418.8	Unit/Enh (6490)	Yes	No	130	20	4	7.521	3	Negative
NEtFOSE	No	630	Unit/Enh (6490)	59	Unit/Enh (6490)	Yes	No	120	20	4	8.301	3	Negative
NFDHA	No	295	Unit/Enh (6490)	201.1	Unit/Enh (6490)	Yes	No	92	2	4	4.641	3	Negative
NFDHA	No	295	Unit/Enh (6490)	84.9	Unit/Enh (6490)	Yes	No	92	34	4	4.641	3	Negative
NMeFOSA	No	512	Unit/Enh (6490)	219	Unit/Enh (6490)	Yes	No	120	20	4	8.298	3	Negative
NMeFOSA	No	512	Unit/Enh (6490)	169	Unit/Enh (6490)	Yes	No	120	20	4	8.298	3	Negative
N-MeFOSAA	No	570	Unit/Enh (6490)	511.9	Unit/Enh (6490)	Yes	No	150	20	4	7.335	3	Negative
N-MeFOSAA	No	570	Unit/Enh (6490)	418.9	Unit/Enh (6490)	Yes	No	150	20	4	7.335	3	Negative
NMeFOSE	No	616	Unit/Enh (6490)	59	Unit/Enh (6490)	Yes	No	120	20	4	8.301	3	Negative
Perfluoro-1-[13C8]octanesulfonamide (M8FOSA)	No	506	Unit/Enh (6490)	78	Unit/Enh (6490)	Yes	No	162	48	4	7.59	3	Negative
Perfluoro-1-[13C8]octanesulfonic acid (M8PFOS)	No	507	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	174	48	4	6.59	3	Negative
Perfluoro-1-[13C8]octanesulfonic acid (M8PFOS)	No	507	Unit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	174	54	4	6.59	3	Negative
Perfluoro-1-decanesulfonate (L-PFDS)	No	598.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	156	50	4	7.546	3	Negative
Perfluoro-1-decanesulfonate (L-PFDS)	No	598.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	100	60	4	7.546	3	Negative
Perfluoro-1-heptanesulfonate (L-PFHpS)	No	448.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	162	48	4	6.252	3	Negative
Perfluoro-1-heptanesulfonate (L-PFHpS)	No	448.9	Unit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	162	48	4	6.252	3	Negative
Perfluoro-1-octanesulfonamide (FOSA)	No	497.9	Unit/Enh (6490)	478	Unit/Enh (6490)	Yes	No	156	100	4	7.651	3	Negative
Perfluoro-1-octanesulfonamide (FOSA)	No	497.9	Unit/Enh (6490)	78	Unit/Enh (6490)	Yes	No	156	40	4	7.651	3	Negative
Perfluoro-1-pentanesulfonate (L-PFPeS)	No	348.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	36	4	5.042	3	Negative
Perfluoro-1-pentanesulfonate (L-PFPeS)	No	348.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	40	4	5.042	3	Negative

Acquisition Method Report

Agilent Technologies

Cpd Name	ISTD?	Prec Ion	MS1 Res	Prod Ion	MS2 Res	Primary	Trigger	Frag (V)	CE (V)	Cell Acc (V)	Ret Time (min)	Ret Window	Polarity
Perfluorobutanesulfonic acid (PFBS)	No	298.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	32	4	4.042	3	Negative
Perfluorobutanesulfonic acid (PFBS)	No	298.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	36	4	4.042	3	Negative
Perfluorodecanoic acid (PFDA)	No	513	Unit/Enh (6490)	468.8	Unit/Enh (6490)	Yes	No	90	8	4	7.158	3	Negative
Perfluorodecanoic acid (PFDA)	No	513	Unit/Enh (6490)	268.8	Unit/Enh (6490)	Yes	No	90	16	4	7.158	3	Negative
Perfluorododecanesulfonic acid (PFDoS)	No	699	Unit/Enh (6490)	99	Unit/Enh (6490)	Yes	No	100	60	4	7.984	3	Negative
Perfluorododecanesulfonic acid (PFDoS)	No	699	Unit/Enh (6490)	80	Unit/Enh (6490)	Yes	No	156	50	4	7.984	3	Negative
Perfluorododecanoic acid (PFDoA)	No	613	Unit/Enh (6490)	568.8	Unit/Enh (6490)	Yes	No	90	12	4	7.876	3	Negative
Perfluorododecanoic acid (PFDoA)	No	613	Unit/Enh (6490)	168.7	Unit/Enh (6490)	Yes	No	90	28	4	7.876	3	Negative
Perfluoroheptanoic acid (PFHpA)	No	363	Unit/Enh (6490)	318.8	Unit/Enh (6490)	Yes	No	90	8	4	5.601	3	Negative
Perfluoroheptanoic acid (PFHpA)	No	363	Unit/Enh (6490)	168.9	Unit/Enh (6490)	Yes	No	90	16	4	5.601	3	Negative
Perfluorohexanesulfonic acid (PFHxS)	No	398.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	40	4	5.685	3	Negative
Perfluorohexanesulfonic acid (PFHxS)	No	398.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	44	4	5.685	3	Negative
Perfluorohexanoic acid (PFHxA)	No	313	Unit/Enh (6490)	268.9	Unit/Enh (6490)	Yes	No	70	4	4	4.856	3	Negative
Perfluorohexanoic acid (PFHxA)	No	313	Unit/Enh (6490)	119	Unit/Enh (6490)	Yes	No	70	20	4	4.856	3	Negative
Perfluoro-n-[1,2-13C2]dodecanoic acid (MPFDoA)	No	615	Unit/Enh (6490)	570	Unit/Enh (6490)	Yes	No	53	8	4	7.71	3	Negative
Perfluoro-n-[13C4]butanoic acid (MPFBA)	No	217	Unit/Enh (6490)	172	Unit/Enh (6490)	Yes	No	59	4	4	1.22	3	Negative
Perfluoro-n-[13C5]pentanoic acid (M5PFPeA)	No	268	Unit/Enh (6490)	223	Unit/Enh (6490)	Yes	No	62	4	4	3.44	3	Negative
Perfluoro-n-[13C8]octanoic acid (M8PFOA)	No	421	Unit/Enh (6490)	376	Unit/Enh (6490)	Yes	No	59	4	4	6.05	3	Negative
Perfluoro-n-[13C8]octanoic acid (M8PFOA)	No	421	Unit/Enh (6490)	172	Unit/Enh (6490)	Yes	No	59	16	4	6.05	3	Negative

Acquisition Method Report

Agilent Technologies

Cpd Name	ISTD?	Prec Ion	MS1 Res	Prod Ion	MS2 Res	Primary	Trigger	Frag (V)	CE (V)	Cell Acc (V)	Ret Time (min)	Ret Window	Polarity
Perfluoro-n-[13C9]nonanoic acid (M9PFNA)	No	472	Unit/Enh (6490)	427	Unit/Enh (6490)	Yes	No	59	8	4	6.56	3	Negative
Perfluoro-n-[13C9]nonanoic acid (M9PFNA)	No	472	Unit/Enh (6490)	223	Unit/Enh (6490)	Yes	No	59	16	4	6.56	3	Negative
Perfluoro-n-butanoic acid (PFBA)	No	213	Unit/Enh (6490)	169.9	Unit/Enh (6490)	Yes	No	70	4	4	1.246	3	Negative
Perfluorononanesulfonate (L-PFNS)	No	548.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	159	48	4	7.174	3	Negative
Perfluorononanesulfonate (L-PFNS)	No	548.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	159	48	4	7.174	3	Negative
Perfluorononanoic acid (PFNA)	No	463	Unit/Enh (6490)	418.8	Unit/Enh (6490)	Yes	No	90	8	4	6.718	3	Negative
Perfluorononanoic acid (PFNA)	No	463	Unit/Enh (6490)	218.8	Unit/Enh (6490)	Yes	No	90	16	4	6.718	3	Negative
Perfluoro-n-pentanoic acid (PFPeA)	No	263	Unit/Enh (6490)	219	Unit/Enh (6490)	Yes	No	62	4	4	3.526	3	Negative
Perfluorooctanesulfonic acid (PFOS)	No	498.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	44	4	6.743	3	Negative
Perfluorooctanesulfonic acid (PFOS)	No	498.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	84	4	6.743	3	Negative
Perfluorooctanoic acid (PFOA)	No	413	Unit/Enh (6490)	368.8	Unit/Enh (6490)	Yes	No	90	8	4	6.202	3	Negative
Perfluorooctanoic acid (PFOA)	No	413	Unit/Enh (6490)	168.9	Unit/Enh (6490)	Yes	No	90	16	4	6.202	3	Negative
Perfluorotetradecanoic acid (PFTA)	No	713	Unit/Enh (6490)	669	Unit/Enh (6490)	Yes	No	110	12	4	8.414	3	Negative
Perfluorotetradecanoic acid (PFTA)	No	713	Unit/Enh (6490)	168.8	Unit/Enh (6490)	Yes	No	110	28	4	8.414	3	Negative
Perfluorotridecanoic acid (PFTrDA)	No	663	Unit/Enh (6490)	618.8	Unit/Enh (6490)	Yes	No	90	12	4	8.164	3	Negative
Perfluoroundecanoic acid (PFUnA)	No	563	Unit/Enh (6490)	519	Unit/Enh (6490)	Yes	No	90	8	4	7.538	3	Negative
Perfluoroundecanoic acid (PFUnA)	No	563	Unit/Enh (6490)	169	Unit/Enh (6490)	Yes	No	90	24	4	7.538	3	Negative
PFEESA	No	315	Unit/Enh (6490)	135	Unit/Enh (6490)	Yes	No	112	26	4	4.464	3	Negative
PFEESA	No	315	Unit/Enh (6490)	83	Unit/Enh (6490)	Yes	No	112	14	4	4.464	3	Negative
PFMBA	No	279	Unit/Enh (6490)	85	Unit/Enh (6490)	Yes	No	75	18	4	4.011	3	Negative
PFMPA	No	229	Unit/Enh (6490)	85	Unit/Enh (6490)	Yes	No	59	6	4	2.15	3	Negative

Scan Parameters

Data Stg	Threshold
Centroid	0

Acquisition Method Report

Agilent Technologies

Source Parameters

Parameter	Value (+)	Value (-)
Gas Temp (°C)	230	230
Gas Flow (l/min)	5	5
Nebulizer (psi)	15	15
SheathGasHeater	350	350
SheathGasFlow	12	12
Capillary (V)	3500	2500
VCharging	500	0

Chromatograms

Chrom Type	Label	Offset	Y-Range
TIC	TIC	0	10000000

Instrument Curves

Actual

Name: HiP Sampler **Module: G4226A**

Auxiliary	
Draw Speed	100.0 µL/min
Eject Speed	400.0 µL/min
Draw Position Offset	1.5 mm
Wait Time After Drawing	1.2 s
Sample Flush Out Factor	5.0
Vial/Well bottom sensing	Yes
Injection	
Injection Mode	Injection with needle wash
Injection Volume	3.00 µL
Needle Wash	
Needle Wash Location	Flush Port
Wash Time	10.0 s
High throughput	
Automatic Delay Volume Reduction	No
Overlapped Injection	
Enable Overlapped Injection	No
Valve Switching	
Valve Movements	0
Valve Switch Time 1	
Switch Time 1 Enabled	No
Valve Switch Time 2	
Switch Time 2 Enabled	No
Valve Switch Time 3	
Switch Time 3 Enabled	No
Valve Switch Time 4	
Switch Time 4 Enabled	No
Stop Time	
Stoptime Mode	As pump/No limit
Post Time	
Posttime Mode	Off

Name: Binary Pump **Module: G4220A**

Flow	0.400 mL/min
Use Solvent Types	No
Stroke Mode	Synchronized
Low Pressure Limit	0.00 bar
High Pressure Limit	600.00 bar
Max. Flow Ramp Up	100.000 mL/min²
Max. Flow Ramp Down	100.000 mL/min²
Expected Mixer	No check

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QAPP
APPENDIX C

Sample Nomenclature

APPENDIX C
SAMPLE NOMENCLATURE

The sample nomenclature outlined below provides consistency between sample events and projects but, most importantly, establish unique sample IDs that will avoid confusion months or years after the sample has been collected.

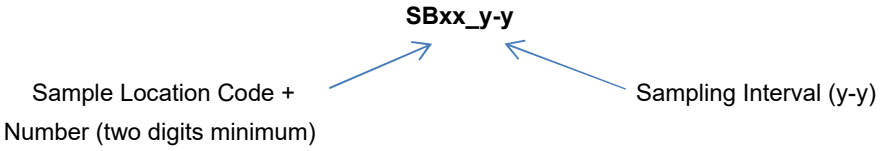
1.0 INVESTIGATION LOCATION CODES

SB	Soil Boring	SV	Soil Vapor Point
WC	Waste Characterization Boring	IA	Indoor Air
TP	Test Pit	AA	Ambient Air
EPSW	Endpoint Location (Sidewall)	SVE	Vapor Extraction Well
EPB	Endpoint Location (Bottom)	DS	Drum
MW	Monitoring Well	IDW	Investigation Derived Waste
TMW	Temporary Monitoring Well	SL	Sludge
SW	Surface Water	FP	Free Product

2.0 SAMPLE NOMENCLATURE

Each sample at a site must have a unique value.

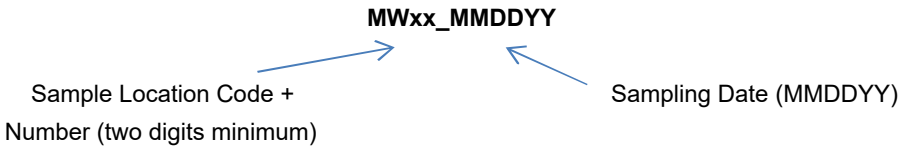
- **Soil/Sediment Samples:**



Sample Type	Sample Location Code	Sampling Depth or Interval (feet bgs or approx. elevation)	Sample Name
Phase II/Remedial Investigation			
Grab Soil Sample	SB01	2 to 4	SB01_2-4
	SB02	4	SB02_4
Waste Characterization			
Grab Soil Sample	WC01	2 to 4	WC01_2-4
	WC02	4	WC02_4
Composite Soil Sample from one or more locations	COMP01 or COMP02 + COMP03	0 to 10 (Fill)	COMP01_0-10

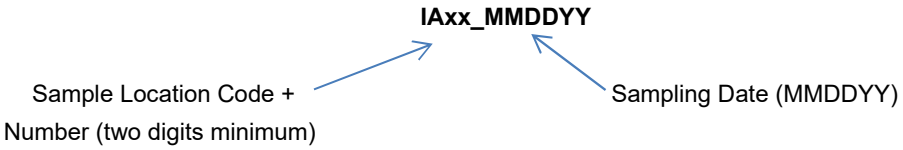
Sample Type	Sample Location Code	Sampling Depth or Interval (feet bgs or approx. elevation)	Sample Name
Endpoint Sampling			
Grab Soil Sample	EPSW01_N	5	EPSW01_N_5
	EPSW01_S	5	EPSW01_S_5
	EPSW01_E	5	EPSW01_E_5
	EPSW01_W	5	EPSW01_W_5
	EPB01	6	EPB01_6

- **Groundwater/Surface Water Samples:**



Sample Type	Sample Location Code	Sampling Date	Sample Name
Groundwater Sample	MW 01	02/21/2013	MW01_022113

- **Air/Soil Vapor Samples:**



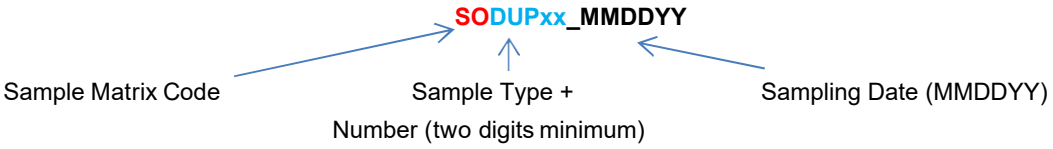
Sample Type	Sample Location Code	Date	Sample Name
Air Sample	IA01	02/21/2013	IA01_022113
Soil Vapor Sample	SV01	02/21/2013	SV01_022113
Vapor Extraction Well Sample	SVE01 (INLET/MIDPOINT/OUTLET)	02/21/2013	SVE01_IN_022113 SVE01_ MID_022113 SVE01_ OUT_022113

- **QA/QC Samples:**

Sample Matrix Codes

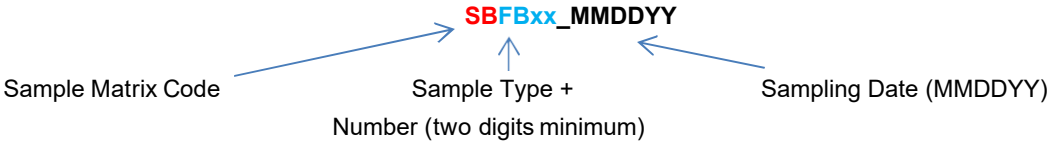
SO	Soil	AS	Air
SE	Sediment	SV	Soil Vapor
GW	Groundwater	SL	Sludge
SW	Surface Water	FP	Free Product

- Duplicates Samples



Sample Type	Parent Sample Code	Date	Sample Name
Groundwater Duplicate Sample (DUP)	MW01_022113	02/21/2013	GWDUP01_022113
Soil boring Duplicate Sample (DUP)	SBP01_022113	02/21/2013	SODUP01_022113
Grab Waste Characterization	WC01	02/21/2013	WCDUP01_022113
Composite Waste Characterization	COMP01	02/21/2013	COMPDUP01_022113

- Field Blanks and Trip Blanks



Sample Type	Date	Sample Name
Groundwater Field Blank (FB)	02/21/2013	GWFB01_022113
Groundwater Trip Blank (TB)	02/21/2013	GWTB01_022113
Soil Field Blank	02/21/2013	SOFB01_022113
Soil Trip Blank	02/21/2013	SOTB01_022113

- Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Parent Sample Name_MS or MSD

Sample Type	Sample Location	Parent Sample Name	Sample Name
Matrix Spike Soil (MS)	SB01	SB01_2-4	SB01_2-4_MS
Matrix Spike Soil Duplicate (MSD)	SB01	SB01_2-4	SB01_2-4_MSD
Matrix Spike GW (MS)	MW01	MW01	MW01_MS
Matrix Spike GW Duplicate (MSD)	MW01	MW01	MW01_MSD

3.0 NOTES

1 The sample location code should not exceed 20 characters and the sample name should not exceed 40 characters.

2 Sample location code (**SB01, MW01, etc.)** is a sequential number (starting with 01) and should be a minimum of two digits.

3 Sample Interval (SB01_**0-5**) is separated from the sample location code with an underscore, and the top and bottom interval with a dash. Soil and sediment sample intervals should always be in feet. Soil and sediment sample intervals should contain no " /" or "()" or unit.

4 Sample date (MW01_ **022113**) is separated from the sample location code with an underscore and should be provided in MMDDYY format [the date should contain no " /" or " -"].

5 If groundwater samples are collected from multiple intervals within one well, you may assign a letter designation (in lower case) to the well ID to differentiate between intervals (i.e., MW01a_022113, M W01b_022113, and MW01c_022113). The letter " a" would indicate the shallowest interval and " c" the deepest. The actual depth intervals should be documented in the project field book or field sheets and the letter designations should be used consistently between sampling events.

6 According to USEPA's Contract Laboratory Program (CLP) Guidance for Field Samplers (January 2011), field duplicate samples should remain "blind" to the laboratory (i.e., they should have separate CLP Sample numbers). Assign two separate (unique) CLP sample numbers (i.e., one number to the field sample and one to the duplicate). Submit blind to the laboratory. (http://www.epa.gov/superfund/programs/clp/download/sampler/CLPSamp -01-2011.pdf)