

July 31, 2023

Ronnie E. Lee, P.E.
Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, NY 12233-7016

via email: ronnie.lee@dec.ny.gov

Re: 495 Eleventh, NY BCP Site Number: C231141
Pre-Design Investigation Work Plan
GBTS Project: 22003-0076

Dear Mr. Lee:

This Pre-Design Investigation Work Plan (PDIWP) describes additional soil and groundwater investigations that will be conducted at the above referenced BCP Site, in accordance with the requirements of the NYSDEC-approved Remedial Action Work Plan (RAWP; June 2022). The purpose of the proposed fieldwork is to provide additional data regarding the presence or absence of per- and polyfluoroalkyl substances (PFAS) in Site media (sampling for PFAS was not conducted during the Site remedial investigation).

The following Scope of Work will be implemented:

- Extension of five (5) soil borings and collection of five (5) soil samples;
- Collection of groundwater samples from the four (4) permanent on-site monitoring wells; and,
- Submission of soil and groundwater samples for laboratory analysis of PFAS (NYSDEC target list).

All Site investigative work will be in conformance with the RAWP, and that document is incorporated by reference into this PDIWP. Sampling locations are shown on the attached Proposed Fieldwork Map.

SITE PREPARATION SERVICES

Qualifications of On-Site Remedial Personnel

Prior to the initiation of work, the identities and qualifications of the project managers and associated staff will be supplied to the NYSDEC. The Volunteer will ensure that qualified contractors are used. All on-site staff will be appropriately trained in accordance with Occupational Safety and Health Administration (OSHA) practices (29 CFR, Part 1910). The NYSDEC will also be notified of any changes in the senior on-site personnel. Prior to the initiation of fieldwork, a Site Health and Safety Officer will be designated by the Volunteer, and a complete Health and Safety Plan will be provided.

Health and Safety Plan (HASP)

All activities will be performed per the HASP (attached), which will be reviewed with all Site personnel prior to the start of fieldwork. All proposed work will be performed in "Level D" personal protective equipment. Field personnel (including subcontractors) will be prepared to continue services wearing more protective levels of equipment should field conditions warrant.

Utility Markouts

A utility markout will be requested prior to initiating intrusive sampling activities.

Quality Assurance Project Plan (QAPP)

Quality Assurance/Quality Control will be implemented during sampling in accordance with the QAPP (attached) prepared for this PDIWP, which includes Standard Operating Procedures (SOPs) for sampling and other fieldwork activities, as well as laboratory-specific protocols.

Sample collection and laboratory analysis for PFAS will comply with NYSDEC guidance (*Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances Under NYSDEC's Part 375 Remedial Programs, April 2023*), which includes NYSDEC's current PFAS target list. The NYSDEC Guidance document, as well as additional fieldwork SOPs for PFAS sampling (detailed protocols, including lists of prohibited behaviors and materials) are provided in the QAPP.

Laboratory

All samples will be collected in accordance with applicable NYSDEC guidelines and will be submitted to a New York State Department of Health (NYSDOH) ELAP-certified laboratory using appropriate chain of custody procedures. USEPA Method 1633 will be used for PFAS analysis. Reporting limits will not exceed 0.5 µg/Kg for soil and 2 ng/L for groundwater. A complete list of PFAS compounds (with reporting limits) is included as an Attachment of the QAPP.

Dedicated high density polyethylene (HDPE) tubing and laboratory supplied glassware appropriate for PFAS will be used for sample collection. One field blank and one trip blank will be supplied for each day of fieldwork involving sample collection. Field personnel will maintain all samples at cold temperatures and complete all chain of custody forms.

Laboratory reports will include detailed Quality Assurance/Quality Control (QA/QC) analyses and Category B ASP deliverables. A Data Usability Summary Report (DUSR) will be prepared by a third, independent party, which maintains NYSDOH ELAP CLP Certification. Data validation will be conducted by an independent validator if required by the NYSDEC.

Notifications

The NYSDEC will be notified in writing at least one week prior to the initiation of any of the on-site work and during the course of the fieldwork if deemed necessary by on-site personnel. Changes to fieldwork scheduling will be provided via facsimile transmission and/or email. All applicable local agencies will also be notified prior to the initiation of Site work.

Prior to the implementation of any of the investigative tasks outlined below, a request for a complete utility markout of the subject property will be submitted as required by New York State Department of Labor regulations. Confirmation of underground utility locations will be secured, and a field check of the utility markout will be conducted prior to the initiation of work. Any utilities on the Site will be protected (as necessary) by the contractor or owner.

PROPOSED FIELDWORK

General Fieldwork Methodology

All sampling locations will be determined in the field, measured to the nearest 0.5-foot relative to a permanent fixed on-site marker, and will be recorded in logbooks for inclusion in all final maps. An assessment of media characteristics, including soil type, presence or absence of foreign materials, field indications of contamination (e.g., unusual coloration patterns or odors), and instrument readings, will be made during all Site investigative work.

A Qualified Environmental Professional (QEP) will be responsible for identifying any materials that require special handling (media that may contain elevated contaminant levels or is grossly contaminated, hazardous materials, etc.) and will ensure that they are properly securely stored on-Site (soil stockpiled on plastic and covered, or soil and water placed in approved containers) pending characterization and proper disposition.

The QEP will ensure that unforeseen environmental conditions are managed in accordance with applicable federal and state regulations.

Community Air Monitoring Program (CAMP)

A CAMP (attached) will be implemented during all ground-intrusive activities. Air quality will be monitored for the presence of volatile organic compounds (VOCs) and respirable dust in the work zone during intrusive activities.

Monitoring will include the use of a photoionization detector to measure the concentration of VOCs and dust monitors to measure particulate matter in the breathing zone. All equipment will be calibrated. Monitoring stations will be established at both upwind and downwind locations in order to provide a measure of protection for the downwind community (i.e., off-site receptors including residences and businesses, and on-Site workers) from potential airborne contaminant releases as a direct result of investigative and remedial work activities.

CAMP findings (narrative and data summary tables) will be provided in Daily Reports submitted to the NYSDEC and NYSDOH project managers. Exceedances of CAMP action levels and corrective measures will be reported to the NYSDEC and NYSDOH project managers within 24 hours of occurrence.

Investigation Derived Waste

Investigation derived waste, including well development water, purge water and soil cuttings exhibiting field evidence of contamination (odors, staining, indications of free product, or elevated PID readings) will be placed in appropriate containers and stored on Site pending characterization and proper disposal at an appropriately licensed facility.

Soil Sampling

Five (5) soil borings will be advanced using a mechanized Geoprobe (see Proposed Fieldwork Map).

Boring equipment will be capable of collecting soil cores at discreet intervals. Samples will be collected from the base of the boring interval representing the maximum depth of construction excavations at the

boring location (generally expected to terminate on bedrock). If the construction excavation depth is above bedrock, and underlying soils appear to be fill materials or exhibit overt field evidence of contamination, additional deeper samples will be collected.

After sampling, the boreholes will be abandoned by backfilling with uncontaminated soil spoils and/or clean sand/gravel, and then completing the upper 6 inches (minimum) with concrete or asphalt.

The CAMP will be implemented during all boring and soil sampling activities.

Groundwater Sampling

The four (4) existing permanent monitoring wells will be developed and then sampled using USEPA Low-Stress (“low flow”) methodology (SOP provided in the QAPP).

DOCUMENTATION

Fieldwork observations and laboratory results from the soil and groundwater investigation will be incorporated into a draft Pre-Design Investigation Report (PDIR) to be submitted to NYSDEC in accordance with RAWP requirements.

SCHEDULE


The activities described in this work plan will be conducted in accordance with the following schedule:

<u>Week(s)</u>	<u>Activity</u>
2	Approval of the PDIWP by NYSDEC
2 - 8	TA approvals/Site Access arrangements.
9	Utility mark out; Site preparation; groundwater gauging, contractor coordination
10	Installation of borings; soil and groundwater sampling
11-13	Laboratory Analysis
14-18	Preparation and submittal of PDIR and electronic data deliverable (EDD) to NYSDEC

Please review this PDIWP and contact Richard Hooker at (845) 452-1658 with any questions.

Respectfully submitted,

Gallagher Bassett Services Inc.



Richard Hooker
Manager, Environmental Consulting

I, Victoria Panico, CHMM, certify that I am currently a Qualified Environmental Professional as defined in 6 NYCRR Part 375 and that this Pre-design Investigation Work Plan was prepared in accordance with all applicable statutes and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10).

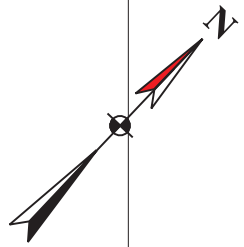
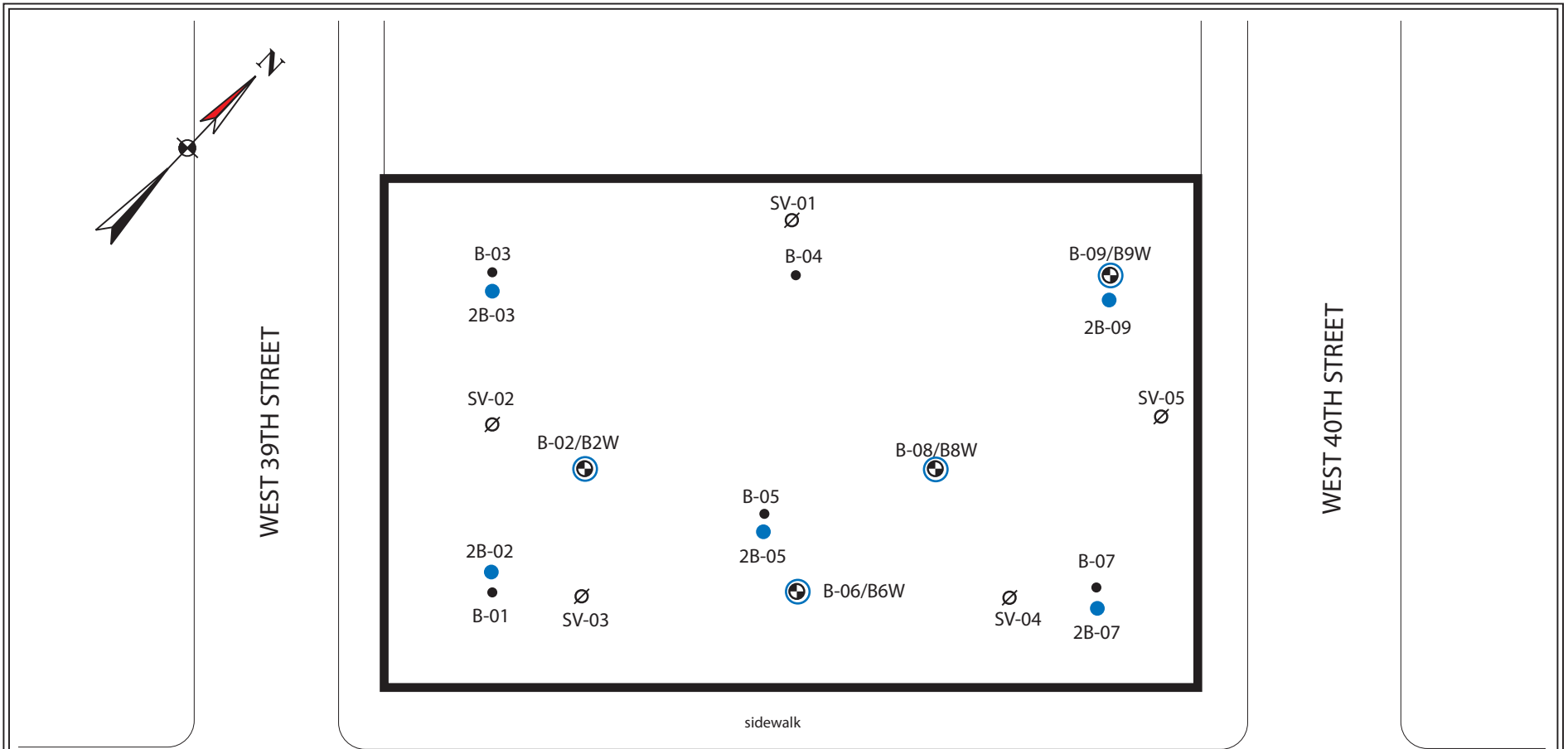
Victoria Panico

July 31, 2023

Victoria Panico, CHMM, Gallagher Bassett Technical Services
Project Manager

Attachments: Proposed Fieldwork Map
Health and Safety Plan
Quality Assurance Project Plan
Community Air Monitoring Plan

ATTACHMENT – Proposed Fieldwork Map



WEST 39TH STREET

WEST 40TH STREET

11th AVENUE



Base map provided by HAKS Engineers, Architects & Land Surveyors PC survey dated 11/5/14. All feature locations are approximate. This map is intended as a schematic to be used in conjunction with the associated report, and it should not be relied upon as a survey for planning or other activities.

Proposed Fieldwork Map
495 11th Avenue
Borough of Manhattan, New York

- Legend: subject property border
- previous boring location
 - previous soil vapor location
 - previous boring location and existing monitoring well location
 - proposed boring location
 - existing monitoring well to be sampled

File: 22003-0076
July 2023
Scale as shown
Figures

ATTACHMENT - Health and Safety Plan



TECHNICAL
SERVICES

**INVESTIGATION
HEALTH AND SAFETY PLAN**

495 ELEVENTH

**495 Eleventh Avenue
Manhattan, New York**

NYSDEC BCP Site: C231141

July 2023

GBTS Project: 22003-0076

Technical Services Division

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INVESTIGATION HEALTH AND SAFETY PLAN

July 2023

GBTS Project:

22003-0076

Prepared By:

**Gallagher Bassett Technical Services
22 IBM Road, Suite 101
Poughkeepsie, New York 12601**

Prepared For:

**495 11th Avenue Owner Realty LLC
PO Box 234550
Great Neck, New York, 11023**

The undersigned have reviewed this Construction Health And Safety Plan and certify to 495 11th Avenue Owner Realty LLC and to the New York State Department of Environmental Conservation that the information provided in this document is accurate as of the date of issuance by this office.



Scott Spitzer
Gallagher Bassett Technical Services
Technical Director – Environmental Consulting



Richard Hooker
Gallagher Bassett Technical Services
Manager – Environmental Consulting

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Attachments:

Figure: Proposed Fieldwork Map

1.0 INTRODUCTION

1.1 Purpose

This Investigation Health and Safety Plan (HASP) has been developed to provide the requirements and general procedures to be followed by Gallagher Bassett Technical Services (GBTS) and on-Site subcontractors while performing investigation services at the 495 Eleventh BCP Site (ID: C231141) located in Manhattan, New York.

This HASP incorporates policies, guidelines and procedures intended to protect the public health of the community during fieldwork activities, and therefore serves as a Community Health and Safety Plan. The objectives of the HASP are met by establishing guidelines to minimize potential exposures during fieldwork, and by planning for and responding to emergencies affecting the public adjacent to the site.

This HASP describes the responsibilities, training requirements, protective equipment and standard operating procedures to be utilized by all personnel while on the Site. All on-site personnel and visitors shall follow the guidelines, rules, and procedures contained in this HASP. The Project Manager or Site Health and Safety Officer (SHSO) may impose any other procedures or prohibitions necessary for safe operations. This HASP incorporates by reference applicable Occupational Safety and Health Administration (OSHA) requirements in 29 CFR 1910 and 1926.

The requirements and guidelines in this HASP are based on a review of available information and evaluation of potential on-site hazards. This HASP will be discussed with Site personnel and will be available on-site for review while work is underway. On-site personnel will report to the SHSO in matters of health and safety. The on-site project supervisor(s) are responsible for the enforcement and implementation of this HASP, which is applicable to all on-site field personnel, including contractors and subcontractors.

This HASP is specifically intended for the conduct of activities within the defined scope of work in specified areas of the Site. Changes in conditions or future actions that may be conducted at the Site may necessitate the modification of the requirements of the HASP. Although this HASP can be made available to interested persons for informational purposes, GBTS cannot be held accountable for the interpretations or activities of any other persons or entities other than the employees of GBTS or its subcontractors.

1.2 Site Location and Description

The Site is defined as the property located at 495 Eleventh Avenue, Manhattan, City of New York, New York. A figure illustrating the Site configuration and areas of proposed investigation activities is included as an Attachment to this HASP.

1.3 Work Activities

Investigation activities are detailed in the NYSDEC-approved Pre-Design Investigation Work Plan (PDIWP) dated July 2023. The specific tasks detailed in the PDIWP are wholly incorporated by reference into this HASP. The PDIWP describes the tasks required to investigate environmental contamination at the Site.

The Remedial Investigation Report (RIR) prepared for the Site documented the presence of fill materials and groundwater with elevated levels of metals and polycyclic aromatic hydrocarbons (PAHs). Low-level soil vapor contamination, typical of urban settings, is present at the Site.

2.0 HEALTH AND SAFETY HAZARDS

2.1 Hazard Overview for On-Site Personnel

Elevated concentrations of organic compounds (PAHs) and metals are present in Site soil and groundwater, and low-grade volatile compounds are present in soil vapor. The possibility exists for on-site personnel to have contact with contaminated soils, groundwater and/or vapor during investigative activities. Contact with contaminated substances may present a skin contact, inhalation and/or ingestion hazard. These potential hazards are addressed in Sections 3.0 through 11.0, below.

2.2 Potential Hazards to the Public from Fieldwork Activities

The potential exists for the public to be exposed to contaminated soils, groundwater and/or vapor, which may present a skin contact, inhalation and/or ingestion hazard. Additional potential hazards to the public that are associated with fieldwork activities include mechanical/physical hazards, traffic hazards from fieldwork vehicles, and noise impacts associated with operation of mechanical equipment.

Impacts to public health and safety are expected to be limited to hazards that could directly affect on-Site visitors and/or trespassers. These effects will be mitigated through site access and control measures (see Section 6.0, below). Specific actions taken to protect the public health (presented in Sections 3.0 through 11, below) are anticipated to minimize any potential off-site impacts from contaminant migration, noise and traffic hazards.

3.0 PERSONAL PROTECTIVE EQUIPMENT

The levels of protection identified for the services specified in the RAWP represent a best estimate of exposure potential and protective equipment needed for that exposure. Determination of levels was based on data provided by previous studies of the Site and information reviewed on current and past Site usage.

The SHSO may recommend revisions to these levels based on an assessment of actual exposures and may at any time require Site workers, supervisors and/or visitors to use specific safety equipment.

The level of protective clothing and equipment selected for this project is Level D. Level D PPE provides minimal skin protection and no respiratory protection, and is used when the atmosphere contains no known hazard, oxygen concentrations are not less than 19.5%, and work activities exclude splashes, immersion or the potential for unexpected inhalation or contact with hazardous levels of chemicals. Workers will wear Level D protective clothing including, but not limited to, a hard hat, steel-toed boots, nitrile gloves (when handling soils and/or groundwater), hearing protection (foam ear plugs or ear muffs, as required), and safety goggles (in areas of exposed groundwater and when decontaminating equipment). Personal protective equipment (PPE) will be worn at all times, as designated by this HASP.

Disposable gloves will be changed immediately following the handling of contaminated soils, water, or equipment. Tyvek suits will be worn during activities likely to excessively expose work clothing to contaminated dust or soil (chemically-resistant over garments will be required in situations where exposures could lead to penetration of clothing and direct dermal contact by contaminants).

The requirement for the use of PPE by official on-site visitors shall be determined by the SHSO, based on the most restrictive PPE requirement for a particular Work Zones (see Section 6 for Work Zone definitions). All on-site visitors shall, at a minimum, be required to wear an approved hardhat and be provided with appropriate hearing protection as necessary.

The need for an upgrade in PPE will be determined based upon encountered Site conditions, including measurements taken in the breathing zone of the work area using a photo-ionization detector (PID). An upgrade to a higher level of protection (Level C) will begin when specific action levels are reached (see Section 5.0, below), or as otherwise required by the SHSO. Level C PPE includes a full-face or half-mask air-purifying respirator (NIOSH approved for compound[s] of concern), hooded chemical-resistant clothing, outer and inner chemical-resistant gloves, and (as needed) coveralls, outer boots/boot covers, escape mask, and face shield. Level C PPE may be used only when: oxygen concentrations are not less than 19.5%; contaminant contact will not adversely affect exposed skin; types of air contaminants have been identified, concentrations measured, and a cartridge/canister is available that can remove the contaminant; atmospheric contaminant concentrations do not exceed immediately dangerous to life or health (IDLH) levels; and job functions do not require self-contained breathing apparatus (SCBAs). The need for Level B or Level A PPE is not anticipated for the planned remedial activities at this Site.

If any equipment fails and/or any employee experiences a failure or other alteration of their protective equipment that may affect its protective ability, that person will immediately leave the work area. The Project Manager and the SHSO will be notified and, after reviewing the situation,

determine the effect of the failure on the continuation of on-going operations. If the failure affects the safety of personnel, the work site, or the surrounding environment, personnel will be evacuated until appropriate corrective actions have been taken.

4.0 CONTAMINANT CONTROL

Precautions will be taken during dry weather (e.g., wetting or covering exposed soils) to avoid generating and breathing dust-generated from soils. A PID (or equivalent equipment) will be used to monitor potential contaminant levels. Response to the monitoring will be in accordance with the action levels provided in Section 5.0.

5.0 MONITORING AND ACTION LEVELS

Concentrations of petroleum compounds in the air are expected to be below the OSHA Permissible Exposure Limits (PELs). Air monitoring will be conducted during Site remediation in accordance with a Community Air Monitoring Plan (CAMP; RAWP Appendix F). Monitoring will be conducted at all times that fieldwork includes ground intrusive activities or other work likely to generate emissions. PID and dust readings consistently in excess of CAMP limits will be used as an indication of the need to initiate personnel monitoring, increase worker protective measures, and/or modify or cease on-site operations in order to mitigate off-site community exposure. Note that the CAMP includes special requirements for work within 20 feet of potential receptors, and for any indoor work.

PID readings that consistently exceed background in the breathing zone (during any proposed tasks) will necessitate moving away from the source or implementing a higher PPE level.

6.0 SITE CONTROL/WORK ZONES

Site control procedures will be established to reduce the possibility of worker/visitor contact with environmental contaminants, to protect the public in the area surrounding the Site and to limit access to the Site to only those persons required to be in the work zone. Notices placed near the Site will warn the public not to enter fieldwork areas and direct visitors to report to the Project Manager or SHSO. Measures will be taken to limit the entry of unauthorized personnel into the specific areas of field activity and to safely direct and control all vehicular traffic in and near the Site (e.g., placement of traffic cones and warning tape).

Work Areas are defined as follows:

Exclusion Zone - The exclusion zone will be that area immediately surrounding the work being performed to accomplish fieldwork activities involving the handling or potential exposures to contaminated media. Only individuals with appropriate PPE and training are allowed into this zone. It is the responsibility of the SHSO to prevent unauthorized personnel from entering the exclusion zone. When necessary (e.g., high traffic areas) the exclusion zone will be delineated with barricade tape, cones and/or barricades.

Dedicated Decontamination Area - A dedicated decontamination area for personnel and equipment (including contamination reduction and support zones) is not anticipated to be required during completion of fieldwork activities, but will be established and utilized, as warranted, based on changes in Site conditions. Care will be taken at all times to remove gloves, excess soil from boots, and soiled clothing (if necessary) before entering the Intermediate Zone.

Intermediate Zone - The intermediate zone, also known as the decontamination zone, is where patient decontamination should take place, if necessary. A degree of contamination still is found in this zone and some PPE is required, although it is usually of a lesser degree than that required for the exclusion zone.

Command Zone - The command zone is located outside the decontamination zone. All exposed individuals and equipment from the exclusion zone and the decontamination zone should be decontaminated before entering the command zone. Access to all zones must be controlled. Keeping onlookers, media, etc. well away from the Site is critical and will be the responsibility of both the SHSO and the Project Manager, and other Site personnel as appropriate.

7.0 NOISE CONTROL

All fieldwork activities will be conducted in a manner designed to reduce unnecessary noise generation, and to minimize the potential for both on-site and off-site harmful noise levels. The Project Manager and SHSO will establish noise reduction procedures (as appropriate to the Site and the work) to meet these requirements.

8.0 PERSONNEL TRAINING

Work zones that will accomplish the general objective stated above will be established by the Project Manager and the SHSO. Site access will be monitored by the SHSO, who will maintain a log-in sheet for personnel that will include, at the minimum, personnel on the Site, their arrival and departure times and their destination on the Site. All workers will be properly trained in accordance with OSHA requirements (29 CFR 1910). Personnel exiting the work zone(s) will be decontaminated prior to exiting the Site.

Site-specific training will be provided to each employee. Personnel will be briefed by the SHSO as to the potential hazards to be encountered. Topics will include:

- Availability of this HASP;
- General site hazards and specific hazards in the work areas, including those attributable to known or suspected on-site contaminants;
- Selection, use, testing, and care of the body, eye, hand, and foot protection being worn, with the limitations of each;
- Decontamination procedures for personnel, their personal protective equipment, and other equipment used on the Site;

- Emergency response procedures and requirements;
- Emergency alarm systems and other forms of notification, and evacuation routes to be followed; and,
- Methods to obtain emergency assistance and medical attention.

9.0 DECONTAMINATION

The SHSO will establish a decontamination system and decontamination procedures (appropriate to the Site and the work) that will prevent potentially hazardous materials from leaving the Site. Vehicles will be brushed to remove materials adhering to surfaces. Sampling equipment will be segregated and, after decontamination, stored separately from PPE. All decontaminated or clean sampling equipment not in use will be protected and stored in a designated, controlled storage area.

10.0 EMERGENCY RESPONSE

10.1 Notification of Site Emergencies

In the event of an emergency, the SHSO will be immediately notified of the nature and extent of the emergency (the names and contact information for key site safety and management personnel, as well as other site safety contact telephone numbers, shall be posted at the Site).

Table 1 in this HASP contains Emergency Response Telephone Numbers, and immediately following is a map detailing the directions to the nearest hospital emergency room. This information will be maintained at the work Site by the SHSO. The location of the nearest telephone will be determined prior to the initiation of on-site activities. In addition to any permanent phone lines, a cellular phone will be in the possession of the SHSO, or an authorized designee, at all times.

10.2 Responsibilities

Prior to the initiation of on-site work activities, the SHSO will:

- Notify individuals, authorities and/or health care facilities of the potentially hazardous activities and potential wastes that may develop as a result of the remedial activities;
- Confirm that first aid supplies and a fire extinguisher are available on-site;
- Have a working knowledge of safety equipment available; and,
- Confirm that a map detailing the most direct route to the hospital is prominently posted with the emergency telephone numbers.

The SHSO will be responsible for directing notification, response and follow-up actions and for contacting outside response personnel (ambulance, fire department, or others). In the case of an evacuation, the SHSO will account for personnel. A log of individuals entering and leaving the Site will be kept so that everyone can be accounted for in an emergency.

Upon notification of an exposure incident, the SHSO will contact the appropriate emergency response personnel for recommended medical diagnosis and, if necessary, treatment. The SHSO will determine whether and at what levels exposure actually occurred, the cause of such exposure, and the means to prevent similar incidents from occurring.

10.3 Accidents and Injuries

In the event of an accident or injury, measures will be taken to assist those who have been injured or exposed and to protect others from hazards. If an individual is transported to a hospital or doctor, a copy of the HASP will accompany the individual.

The SHSO will be notified and respond according to the severity of the incident. The SHSO will investigate the incident and prepare a signed and dated report documenting the investigation. An exposure-incident report will also be completed by the SHSO and the exposed individual. The form will be filed with the employee's medical and safety records to serve as documentation of the incident and the actions taken.

10.4 Communication

No special hand signals will be utilized within the work zone. Field personnel will utilize standard hand signals during the operation of heavy equipment.

10.5 Safe Refuge

Vehicles and on-site structures will serve as the immediate place of refuge in the event of an emergency. If evacuation from the area is necessary, project vehicles will be used to transport on-site personnel to safety.

10.6 Site Security and Control

Site security and control during emergencies, accidents and incidents will be monitored by the SHSO. The SHSO is responsible for limiting access to the Site to authorized personnel and for oversight of reaction activities.

10.7 Emergency Evacuation

In case of an emergency, personnel will evacuate to the safe refuge identified by the SHSO, both for their personal safety and to prevent the hampering of response/rescue efforts.

10.8 Resuming Work

A determination that it is safe to return to work will be made by the SHSO and/or any personnel assisting in the emergency, e.g., fire department, police department, utility company, etc. No personnel will be allowed to return to the work areas until a full determination has been made by the above-identified personnel that all field activities can continue unobstructed. Such a determination will depend upon the nature of the emergency (e.g., downed power lines --

removal of all lines from the property; fire -- extinguished fire; injury -- safe transport of the injured party to a medical facility with either assurance of acceptable medical care present or completion of medical care; etc.). Before on-site work is resumed following an emergency, necessary emergency equipment will be recharged, refilled or replaced. Government agencies will be notified as appropriate. An Incident Report Form will be filed.

10.9 Fire Fighting Procedures

A fire extinguisher will be available in the work zone during on-site activities. This extinguisher is intended for small fires. When a fire cannot be controlled with the extinguisher, the area will be evacuated immediately. The SHSO will be responsible for directing notification, response and follow-up actions and for contacting ambulance and fire department personnel.

10.10 Emergency Decontamination Procedure

The extent of emergency decontamination depends on the severity of the injury or illness and the nature of the contamination. Whenever possible, minimum decontamination will consist of washing, rinsing and/or removal of contaminated outer clothing and equipment. If time does not permit decontamination, the person will be given first aid treatment and then wrapped in plastic or a blanket prior to transport.

10.11 Emergency Equipment

The SHSO will maintain a dedicated vehicle containing the following on-site equipment for safety and emergency response: fire extinguisher; first-aid kit; and, extra copy of this HASP.

11.0 SPECIAL PRECAUTIONS AND PROCEDURES

The activities associated with this remediation may involve potential risks of exposure to both chemical and physical hazards. The potential for chemical exposure to hazardous or regulated substances will be significantly reduced through the use of monitoring, personal protective clothing, engineering controls, and implementation of safe work practices.

11.1 Heat/Cold Stress

Training in prevention of heat/cold stress will be provided as part of the site-specific training. The timing of this project is such that heat/cold stress may pose a threat to the health and safety of personnel. Work/rest regimens will be employed, as necessary, so that personnel do not suffer adverse effects from heat/cold stress. Special clothing and appropriate diet and fluid intake regimens will be recommended to personnel to further reduce this temperature-related hazard. Rest periods will be recommended in the event of high/low temperatures and/or humidity to counter the negative effects of heat/cold stress.

11.2 Heavy Equipment

Working in the vicinity of heavy equipment is the primary safety hazard at the Site. Physical hazards in working near heavy construction equipment include the following: overhead hazards, slips/trip/falls, hand and foot injuries, moving part hazards, improper lifting/back injuries and noise. All workers will be properly trained in accordance with OSHA requirements (29 CFR 1910). No workers will be permitted within any excavated areas without proper personal protective equipment (PPE), including, as warranted, any necessary Level C equipment (e.g., respirators and protective suits). Air monitoring in excavation areas will be conducted for VOCs in accordance with Section 5.0.

11.3 Additional Safety Practices

The following are important safety precautions to be enforced during the remedial activities.

Medicine and alcohol can aggravate the effect of exposure to certain compounds. Controlled substances and alcoholic beverages will not be consumed during remedial activities. Consumption of prescribed drugs will only be at the discretion of a physician familiar with the person's work.

Eating, drinking, chewing gum or tobacco, smoking, or other practices that increase the probability of hand-to-mouth transfer and ingestion of material is prohibited except in areas designated by the SHSO.

Contact with potentially contaminated surfaces will be avoided whenever possible. Workers will not unnecessarily walk through puddles, mud or other discolored surfaces; kneel on the ground; or lean, sit, or place equipment on drums, containers, vehicles, or the ground.

Personnel and equipment in the work areas will be minimized, consistent with site operations.

Unsafe equipment left unattended will be identified by a "DANGER, DO NOT OPERATE" tag.

Work areas for various operational activities will be established.

11.4 Daily Log Contents

The SHSO will establish a system appropriate to the Site, the work and the work zones that will record, at a minimum, the following information:

- Personnel on the Site (arrival and departure times) and their destination on the Site;
- Incidents and unusual activities Site such as (but not limited to) accidents, spills, breaches of security, injuries, equipment failures and weather-related problems;
- Changes to the HASP; and,
- Daily information, such as: changes to work and health and safety plans, work accomplished and the current Site status, and monitoring results.

Daily logs will be provided in periodic reports to NYSDEC and NYSDOH, as specified in the RIWP.

12.0 EMERGENCY INFORMATION

12.1 Emergency Contact Information

The following table indicates emergency contact information. This table should be copied and freely distributed and/or posted at the Site to ensure ready access.

Emergency Contact Information

Emergency Agencies	Phone Numbers
EMERGENCY	911
HOSPITAL New York Presbyterian Hospital 463 Fashion Avenue, Manhattan, New York	(212) 632-7400 or 911
POLICE 215 West 49 th Street, New York, NY	(212) 391-0883 or 911
FIRE	911
City of New York – General Information	311
Water and Sewer (NYCDEP)	311
Site Health and Safety Officer, Richard Hooker, GBTS	(845) 452-1658

12.2 Directions to Hospital

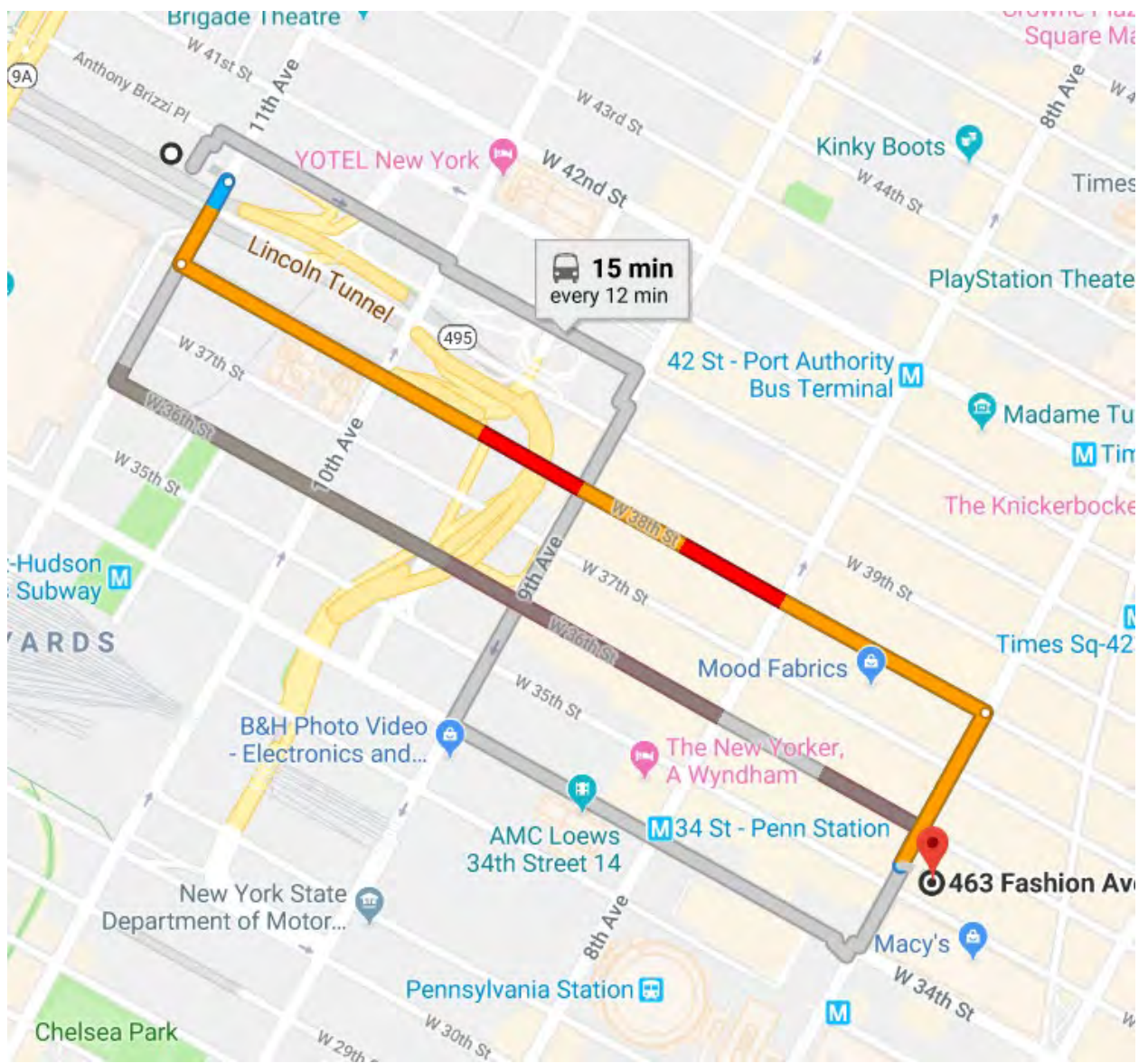
Head south on 11th Avenue

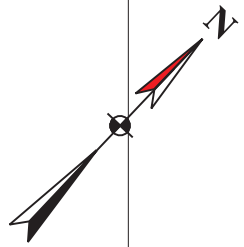
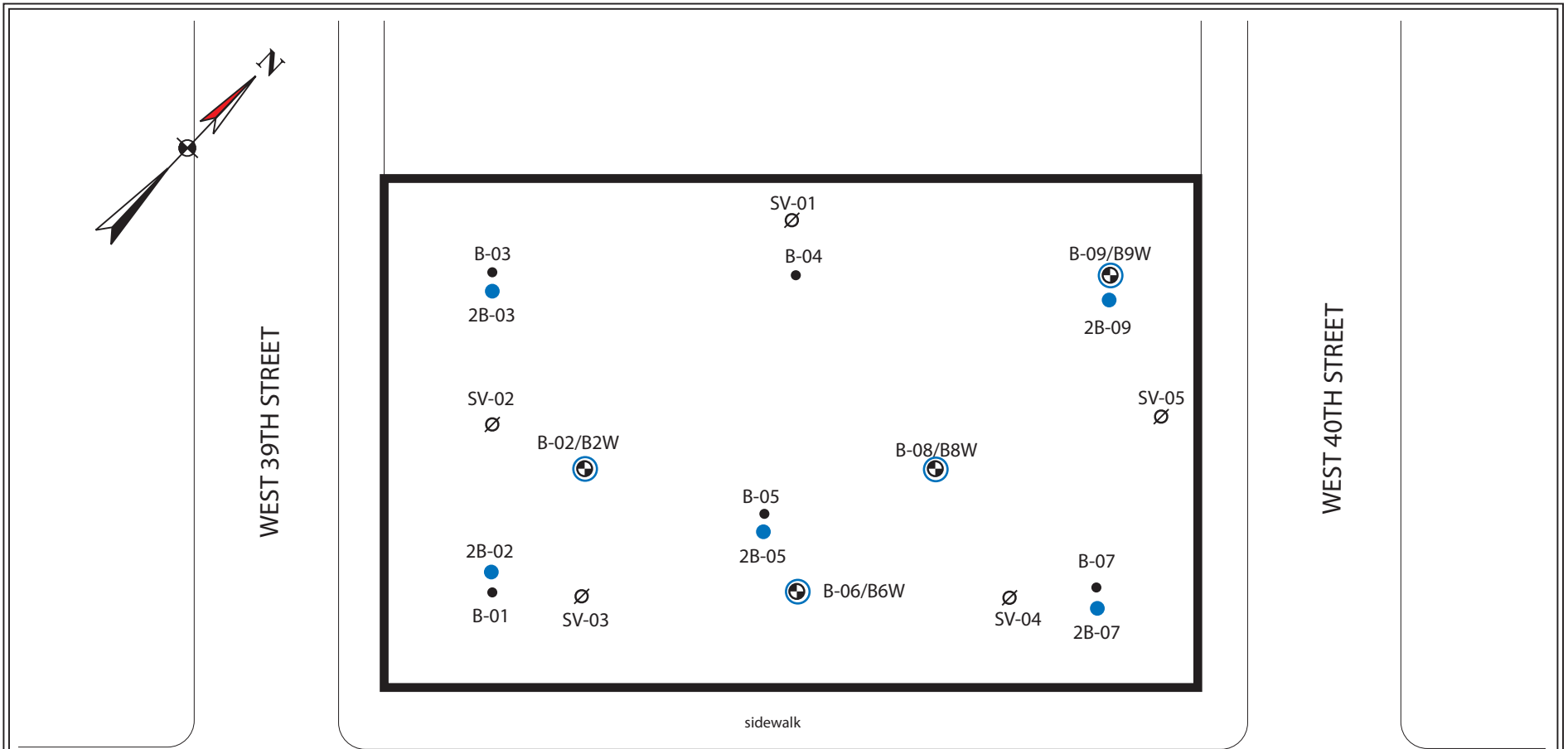
Left turn onto W 38th Street

Right turn onto Fashion Avenue

Hospital on LEFT, 463 Fashion Avenue

12.3 Map to Hospital





WEST 39TH STREET

WEST 40TH STREET

11th AVENUE



Base map provided by HAKS Engineers, Architects & Land Surveyors PC survey dated 11/5/14. All feature locations are approximate. This map is intended as a schematic to be used in conjunction with the associated report, and it should not be relied upon as a survey for planning or other activities.

Proposed Fieldwork Map
495 11th Avenue
Borough of Manhattan, New York

- Legend:
- subject property border
 - previous boring location
 - previous soil vapor location
 - previous boring location and existing monitoring well location
 - proposed boring location
 - existing monitoring well to be sampled

File: 22003-0076
July 2023
Scale as shown
Figures

ATTACHMENT - Quality Assurance Project Plan



TECHNICAL
SERVICES

**SITE INVESTIGATION
QUALITY ASSURANCE PROJECT PLAN**

495 Eleventh

**495 11th Avenue
Manhattan, New York
NYSDEC BCP Site: C231141**

July 2023

**GBTS Project:
22003-0076**

SITE INVESTIGATION QUALITY ASSURANCE PROJECT PLAN

July 2023

GBTS Project: 22003-0076

Prepared By:

Gallagher Bassett Technical Services
22 IBM Road, Suite 101
Poughkeepsie, New York 12601

Prepared For:

495 11th Avenue Owner Realty LLC
PO Box 234550
Great Neck, New York, 11023

The undersigned have reviewed this Site Investigation Quality Assurance Project Plan and certify to 495 11th Avenue Owner Realty LLC and to the New York State Department of Environmental Conservation that the information provided in this document is accurate as of the date of issuance by this office.



Scott Spitzer
Gallagher Bassett Technical Services
Technical Director – Environmental Consulting



Richard Hooker
Gallagher Bassett Technical Services
Manager – Environmental Consulting

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1.0 PROJECT MANAGEMENT

1.1 Project/Task Organization

Major participants in the project are shown below along with their specific responsibilities and authorities. Resumes for Gallagher Bassett Technical Services (GBTS) personnel and for the Data Validator are provided in Attachment D this Quality Assurance Project Plan (QAPP).

Ronnie Lee New York State Department of Environmental Conservation (NYSDEC)

Ronnie Lee is the project manager for the NYSDEC and is responsible for review and approval of all project submittals.

Victoria Panico Project Manager, GBTS

Victoria Panico, CHMM is the Qualified Environmental Professional (QEP) for the project, responsible for overview of all project activities. Ms. Panico has authority over all GBTS personnel and subcontractors and will be responsible for final review and approval of all project submittals prior to submission to the NYSDEC.

Scott Spitzer Technical Director, Environmental Consulting, GBTS

Scott Spitzer will be the Project Manager, responsible for directing and coordinating all project activities, reviewing all project documents, and ensuring that project plans are followed. Mr. Spitzer has authority to direct the activities of the field team (OSC and subcontractors).

Richard Hooker Quality Assurance Officer, GBTS

Richard Hooker, PhD will be responsible for reviewing all sampling procedures and certifying that the data was collected and analyzed using the appropriate procedures, and will assist in the development of the sampling and analytical portion of a site-specific quality assurance project plan (QAPP).

Erick Salazar On-Site Coordinator (OSC), GBTS

The OSC will be responsible for the completion of all on-site fieldwork, collection of all samples, completion of the field log, and chains of custody. The OSC will have authority over all on-site subcontractors.

Laboratory York Analytical Laboratories

York Analytical Laboratories, will be responsible for analysis of samples, and is New York State Department of Health (NYSDOH) Environmental Laboratory Approved Program (ELAP) certified in the appropriate categories, including PFAS by EPA Method 1633.

Subcontractors *To be determined*

Subcontractors will be responsible for the operation of special equipment and providing technical assistance as needed.

1.2 Principal Data Users

The principal users of the generated data in this project are listed below.

1. Residents of the Hells Kitchen neighborhood of Manhattan, especially those residing near the Site
2. 495 11th Avenue Owner Realty LLC
3. NYSDEC

1.3 Problem Definition/Background

Site remediation is planned under the NYSDEC Brownfields Cleanup Program (BCP ID: C231141) in accordance with the NYSDEC-approved Remedial Action Work Plan (RAWP; June 2022). The requirements, procedures and protocols of the RAWP are wholly incorporated by reference into this QAPP. The RAWP describes the tasks required to remediate on-Site contamination. The Remedial Investigation Report (RIR) prepared for the Site did not include laboratory analysis of soil or groundwater for per- and polyfluoroalkyl Substances (PFAS). The RAWP specifies that PFAS data be documented in accordance with a Pre-Design Investigation Work Plan (PDIWP).

1.4 Project/Task Description

The project will meet this objective through compliance with NYSDEC DER-10 Technical Guidance for Site Investigation.

1.5 Quality Objectives and Criteria

The data collected in this project will be used to document Site environmental conditions. In order to meet the data quality objectives of precision, accuracy, representation, comparability, and completeness the following actions will be taken:

- Media samples will be collected based on the procedures in the RAWP and this QAPP, to ensure data consistency.
- Data generated from media sampling will be submitted for review by an independent third party (see Section 3.4.1, below).

Prior to field activities, the QEP, Project Manager and the OSC will review the PDIWP to ensure that the data quality objectives of precision, accuracy, representation, comparability, and completeness will be met during the field activities.

At the completion of field activities, the Project Manager will review field logs and chains of custody to ensure that field activities met the intent of the PDIWP. If a problem is identified, Mr. Richard Hooker and the Project Manager will meet to determine corrective measures necessary to meet data quality objectives.

1.6 Documents and Records

Electronic and paper copies of all fieldwork observations and measurements will be retained by GBTS.

2.0 SAMPLING AND ANALYSIS PLAN

Sample collection, handling and laboratory analysis is summarized below. Figures indicating Site location and areas of soil excavation are provided as Attachment A.

2.1 Sampling Overview

Soil samples will be collected from soil borings, and groundwater samples will be collected from the existing monitoring well network.

2.2 Fieldwork and Sampling Methodology

All fieldwork activities, including collection and handling of media samples, will be in accordance with the Standard Operating Procedures (SOPs) provided in Attachment B. Sampling will occur for PFAS, and guidelines for such sampling will be strictly followed by all field and laboratory personnel. Basic SOP components are summarized below.

2.2.1 General Fieldwork

The OSC will be responsible for compliance with the SOPs, including:

- Documentation of all fieldwork activities in logbooks for inclusion in final reports;
- Assessment of media characteristics (soil type, presence or absence of foreign materials, field indications of contamination), and instrument readings using properly calibrated and operated precision instruments;
- Identification of materials requiring special handling (media that may contain elevated concentrations of contaminants or is grossly contaminated, hazardous materials, etc.) and ensuring proper secure on-site storage, pending characterization and disposition;
- Ensuring that unforeseen environmental conditions are managed in accordance with applicable federal and state regulations;
- Sample collection, including procedures to minimize potential cross-contamination; and,
- Implementation of decontamination procedures.

Sample collection and laboratory analysis for PFAS will comply with *Sampling, Analysis, and Assessment of Per-And Polyfluoroalkyl Substances Under NYSDEC’s Part 375 Remedial Programs*, April 2023 (NYSDEC PFAS Guidance), provided in Attachment B (SOPs), which includes the target list of 40 PFAS compounds required by USEPA Method 1633.

Detailed PFAS sampling protocols, including lists of prohibited behaviors and materials, are provided in the SOP.

2.2.2 Soil Sampling

It is anticipated that five (5) soil samples will be collected from borings (additional samples may be collected based on any field evidence of contamination). Samples will be collected directly from exposed soil in the sampling instrument using dedicated clean equipment.

2.2.3 Other Materials

Any non-soil solid materials requiring laboratory analysis will be placed into laboratory supplied glassware when possible, or will alternatively be placed into double locking plastic bags and then boxed in order to prevent a tear or other breach in the bags. Liquid samples from excavations, collection pits, or drums/tanks, etc., will be sampled using a dedicated disposal sampling device.

Any required sampling for other media will be in compliance with fieldwork protocols specified in the RAWP

2.3 Sample Handling and Custody

2.3.1 Sample Containers

The following laboratory-supplied containers will be used for sample collection (as applicable):

Media	Analyte Class	Collection Container (subject to laboratory requirements)	Preservation
Soil	PFAS	1, 250-ml HDPE plastic (fill halfway)	4° C
Soil	PFAS MS/MSD	1, 250-ml HDPE plastic (fill halfway), (may use soil from a sample container)	4° C
Water	PFAS	2, 250-ml HDPE plastic (fill to neck)	4° C
Water	Trip blank (PFAS)	2, 250-ml HDPE plastic (fill to neck)	4° C
Water	Field blank (PFAS)	1, 250-ml HDPE plastic (fill to neck)	4° C

2.3.2 Sampling Frequency

Soil and groundwater samples will be analyzed for PFAS in accordance with the NYSDEC PFAS Guidance.

Sampling requirements and USEPA Methods are outlined below.

Media /QC Parameter	Number of Samples ^a	Analytes (USEPA Method) ^{b, c}
Soil	5	PFAS: NYSDEC target list (1633)
Groundwater	4	PFAS: NYSDEC target list (1633)
Trip Blank	1 per sample cooler (each day of sampling)	PFAS NYSDEC target list (1633)
Field Blank (PFAS)	1 per sample day	PFAS NYSDEC target list (1633)
Equipment Blank (PFAS)	1 per sampling day (non-dedicated)	PFAS NYSDEC target list (1633)
Duplicates, MS/MSD	1 for every 20 samples (minimum 1/week)	As per sample collection requirements; PFAS soil MS/MSD may be from same container as sample
Notes a Equipment blanks (when required) to be collected at a minimum of one per day for each matrix. b PFAS will be analyzed using methodologies based on EPA Method 1633; additional laboratory methods may include Synthetic Precipitation Leaching Procedure (SPLP, by 1312, and/or Total Oxidizable Precursor Assay (TOP Assay)		

2.3.3 Sample Custody

Samples will be handled by the OSC and maintained at cold temperatures (4 +/- 2 °C), as warranted. Upon the completion of each day of sample collection activities, all samples will be shipped via either courier or overnight delivery (per laboratory requirements) to a NYSDOH ELAP certified laboratory under proper chain of custody. Laboratory personnel will record the cooler temperature upon receipt and analyze the samples prior to the expiration of the hold times as specified in the NYSDEC Analytical Services Protocols (ASP).

2.4 Analytical Methods

Media samples will be analyzed as indicated in Section 2.3.2, above. Analytical methods for the samples will be implemented as follows:

Matrix	Sample Analysis (Holding Time)	USEPA Analytical Method
Soil	PFAS (14 days)	1633 (reporting limit 1 µg/kg)
Water	PFAS (14 days)	1633, modified (reporting limit 2 ng/L)

2.5 Quality Control

Accuracy and precision will be determined by repeated analysis of laboratory standards, and matrix effects and recovery will be determined through use of spiked samples. The laboratory will run standards, blanks, and spiked samples during sample analysis. Duplicate sampling, and matrix spike (MS)/matrix spike duplicate (MSD) analyses, will be performed in accordance with Section 2.3.2. For each day of sampling, a trip blank will be included with each sample cooler and be analyzed for PFAS.

Samples will be identified using a unique ID number. This ID will be recorded on the sampling log and/or field record and the sampling container (samples for each fieldwork day will be assigned to a Sample Delivery Group [SDG] by the laboratory). In accordance with current best fieldwork practices, permanent marker will not be utilized to label samples planned for analysis for PFAS. Samples for each day of fieldwork will be shipped via courier to the laboratory under proper chain of custody procedures.

2.6 Quality Assurance

2.6.1 Instrument/Equipment, Testing, Inspection, and Maintenance

Field measurements will be conducted using monitoring equipment specialized for each task, including use of a photoionization detector (PID) during fieldwork to screen for volatile organic vapors. All equipment will be properly stored (within buildings or construction trailers when not in use) and calibrated (as warranted) in accordance with the manufacturer's instructions (malfunctions are normally apparent during calibration). In the event of malfunction, equipment will be cleaned and tested. Equipment testing, inspection and maintenance will be the responsibility of the Project Manager and OSC. Any other equipment selected for field measurements will be similarly managed.

2.6.2 Inspection/Acceptance of Supplies and Consumables

All supplies and consumables will be inspected and tested (if necessary) by the Project Manager or OSC. The following supplies and consumables will be used (as applicable) for each sample:

- Laboratory-supplied sampling containers, as specified in Section 2.3.1
- Laboratory-supplied materials for PFAS sampling, including trip blanks and PFAS-free cooler
- Plastic tubing for groundwater sampling
- PFAS-free water for decontamination
- Disposable gloves (nitrile or equivalent)

2.6.3 Data Management

For the purpose of data management, the data can be divided into field and laboratory data.

Field data will be recorded at the time of measurement on written field logs. Laboratory data will be reviewed upon receipt and summarized in data summary tables. The NYSDEC electronic data deliverable format for the analytical data will be requested from the testing laboratory. NYSDEC ASP Category B Data Deliverables will be requested from the testing laboratory and forwarded to an independent third party data validator for the development of Data Usability Summary Report (DUSR).

3.0 DATA REVIEW, VALIDATION AND USABILITY

3.1 Field Measurements

If field instruments are determined to be functioning correctly through calibration and measurements of standards, and if there are no inconsistencies between written records and data recorded in the meters, the data will be assumed to be valid and will be accepted as an indication of field conditions. If instruments malfunction prior to field measurement, they will be restored to proper function prior to re-use. If malfunctions occur immediately after field measurements are taken, the measurements will be retaken as soon as possible. Inconsistencies between written records and recorded meter data will be resolved by re-testing the material, if possible. If re-testing is not possible, (i.e. the sample has been shipped to the laboratory), the inconsistency will be described in appropriate subsequent reporting and the laboratory analysis will be utilized to classify the material. In addition, all field data will be reviewed by the Project Manager for consistency and plausibility.

3.2 Laboratory Analysis

A NYSDOH ELAP-certified laboratory will provide a NYSDEC ASP Category B data package and NYSDEC Electronic Data Deliverable format for the determinative sample analyses.

3.3 Standards, Criteria and Guidance (SCG)

Soil and groundwater for PFOA and PFOS are compared to SCG provided in the NYSDEC PFAS Guidance.

3.4 Verification and Validation Methods

3.4.1 Verification Method

Once collected, all data will go to the Project Manager for review and verification. Review will involve determining that data has been collected at the proper locations by the proper persons and that all field and laboratory logs are complete.

A Data Usability Summary Report (DUSR) will be prepared by a third, independent party. Data Validation will be performed by Tony Zoccolillo of ZDataReports, Data Management and Validation Service, of Syracuse, New York. A resume outlining the education and data validation experience of Mr. Zoccolillo is provided in Attachment D.

3.4.2 Authority for Verification

Authority for verification, validation, and resolution of data issues will be distributed among the investigators. Authority to resolve issues regarding verification of field measurements will rest with the QEP, Project Manager and Mr. Richard Hooker.

4.0 REPORTING REQUIREMENTS

Following review, validation, and verification, all data will be conveyed to users via a Remedial Investigation Report (RIR) in accordance with the requirements of NYSDEC DER-10 Section 3.1.4. The RIR will summarize all data collected during implementation of the RIWP (and any additional work), and will include, at a minimum:

- Descriptions of fieldwork activities and observations;
- Summaries of laboratory analytical results from sampling events, described in the report text and provided in data summary tables, as well as DUSRs for all data;
- Characterization of contamination sources (including environmental fate and transport);
- A qualitative human exposure assessment;
- Accounts of any deviations from RIWP procedural requirements; and,
- Conclusions drawn from applicable, available data.

ATTACHMENT A

Figures



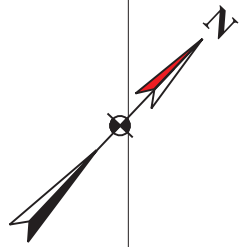
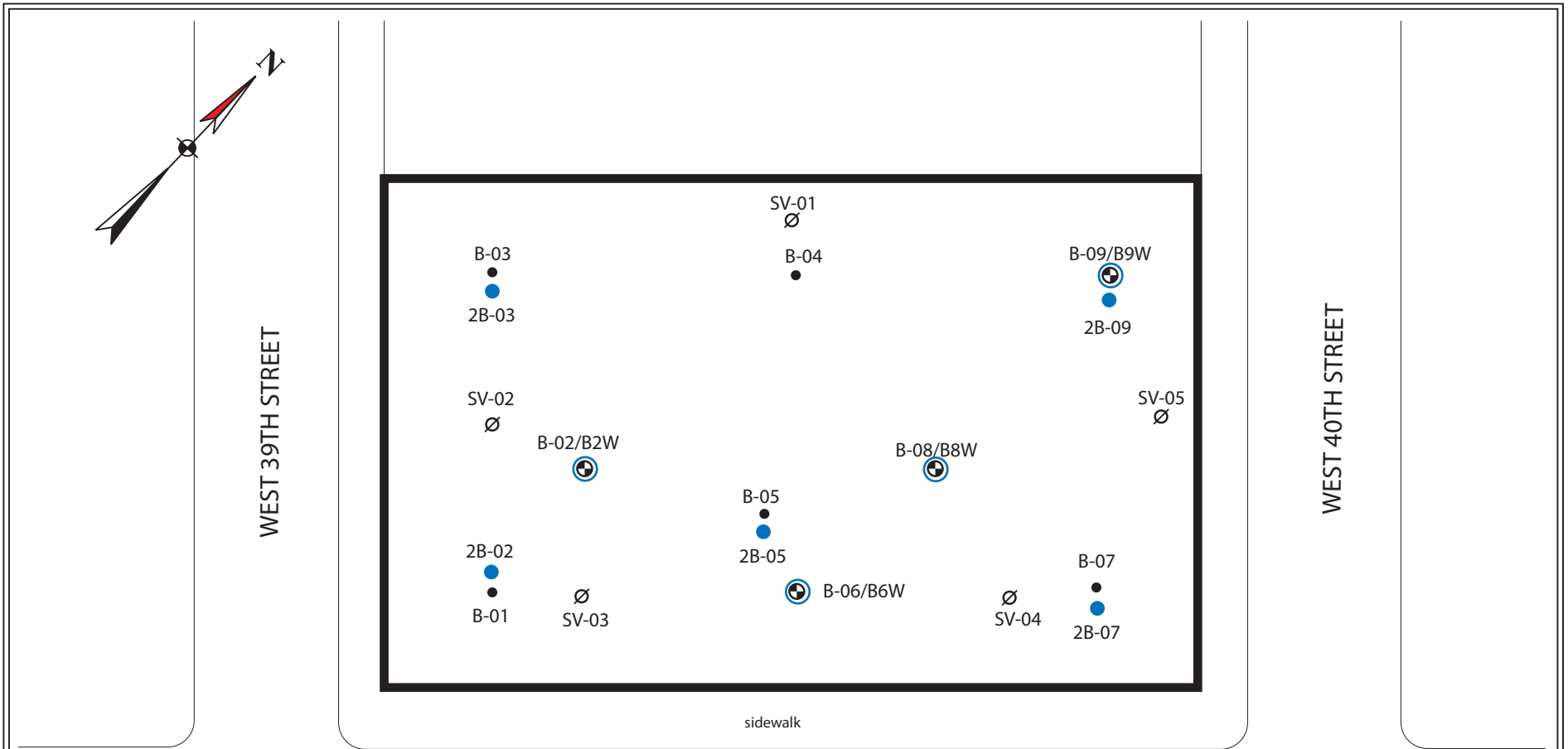
Site Location Map
BCP Site: C231141
495 11th Avenue
Borough of Manhattan

Legend:
— subject property

File: 22003-0076

July 2023

Figures



WEST 39TH STREET

WEST 40TH STREET

11th AVENUE

sidewalk



Base map provided by HAKS Engineers, Architects & Land Surveyors PC survey dated 11/5/14. All feature locations are approximate. This map is intended as a schematic to be used in conjunction with the associated report, and it should not be relied upon as a survey for planning or other activities.

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- Legend:
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 - previous boring location and existing monitoring well location
 - proposed boring location
 - existing monitoring well to be sampled

File: 22003-0076
July 2023
Scale as shown
Figures

ATTACHMENT B

Standard Operating Procedures



TECHNICAL
SERVICES

STANDARD OPERATING PROCEDURES

Fieldwork Sampling and Decontamination

Updated July 2023

22 IBM Road, Suite 101, Poughkeepsie, NY 12601
(845)-452-1658 www.gallagherbassett.com

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SUPPLEMENTAL DOCUMENTATION

Supplement A	Model Fieldwork Logs
Supplement B	USEPA Groundwater Sampling
Supplement C	Decontamination
Supplement D	PFAS Sampling Guidance

I. INTRODUCTION

This document provides Standard Operating Procedures (SOPs) for use by Gallagher Bassett Technical Services (GBTS) personnel during fieldwork events that require the collection of soil, groundwater, soil vapor and/or air samples. General procedures are presented below; detailed protocols, as available, are provided in the supplemental documentation. Equipment checklists, forms and calibration documents are maintained at GBTS offices. All SOPs and supporting documentation are periodically updated.

II. FIELDWORK SAMPLING

Fieldwork sampling procedures are described below (model field logs are provided in Supplement A). Selection of field equipment will be based on anticipated site conditions (updated check-lists of equipment and supplies required for sampling activities are maintained at the local field office). All equipment operations will be in accordance with applicable operating manuals and specifications, and will be conducted (as needed) by an experienced subcontractor holding applicable permits/licenses. Decontamination procedures will be implemented as warranted during all fieldwork activities. Special requirements for PFAS sampling are noted in Section III.

A. Procedures to be Conducted Prior to Fieldwork

Prior to the initiation of any ground-intrusive fieldwork, a request for a complete utility markout of the fieldwork site will be submitted to an appropriate service, as required by state regulations. Confirmation of underground utility locations will be secured and a field check of the utility markout will be conducted prior to the extension of soil borings¹.

A Fieldwork Map and Work Plan, indicating sampling locations and objectives, will be prepared prior to fieldwork activities, and sampling locations will be confirmed and located prior to starting work.

B. General Fieldwork Methodology

At the start of the work day, all on-site personnel, including environmental subcontractors and observers, will be briefed on planned activities and the contents of the site-specific Health and Safety Plan (HASP). Independent field logs will be utilized to document relevant information, including arrival and departure times of on-site personnel, safety meetings, basic weather conditions, and detailed notes and drawings documenting all fieldwork activities and/or any other relevant events and conditions.

On-site personnel will be properly dressed for the intended activities² and the anticipated weather conditions, including use of personnel protective equipment in accordance with the HASP.

Sampling locations will be determined in the field, measured to the nearest 0.5-foot relative to a fixed on-site marker, and will be recorded in logbooks for inclusion in all final maps.

¹ Markout requirements apply to any ground intrusive methodologies, including the extension of test pits.

² Special care is required when for sampling of PFAS; see Section IV

Media will be collected in accordance with the Quality Assurance Project Plan (QAPP) and in a manner consistent with NYSDEC and/or NYSDOH requirements, including protocols for handling and custody. New, dedicated disposable nitrile gloves will be worn at each sampling location, and will be changed frequently based on field conditions. Fieldwork personnel will assess media characteristics (e.g., soil type, presence of debris, indications of contamination, etc.) and record all observations in log books.

On-site senior personnel will be responsible for: a) identifying any materials that require special handling, such as media that may contain high levels of contaminants or is grossly contaminated or likely to be hazardous; b) ensuring that identified materials are properly securely stored on-site (stockpiled on plastic and covered, or placed in approved containers), pending characterization and proper disposition; and, c) ensuring that unforeseen environmental conditions are managed in accordance with applicable federal and state regulations.

Sample collection from recovered media will be performed without unnecessary delay. Samples will be placed into labeled containers provided by the laboratory, stored in dedicated coolers kept at 4 (+/-2) °C and handled under proper chain of custody. All samples will be shipped to a NYSDOH ELAP certified laboratory via laboratory courier (either upon completion of each day of sample collection activities, or the following day after overnight storage in a dedicated sample refrigerator).

C. Extension of Soil Borings

Soil borings will be extended using either hand-held or mechanized equipment, based on site conditions and Work Plan requirements. Mechanized equipment includes using either direct push technology (DPT) or rotary methods, including hollow stem auger (HSA) and sonic drilling. The small size of DPT rigs allows for sampling in tight spaces and areas that are sensitive to the use of heavy equipment. DPT can be used in overburden soils where the soil texture allows for direct push of sampling equipment. A HSA or sonic rig will be utilized if significant subsurface obstructions (e.g., large cobbles, boulders, concrete, etc) are (or are expected to be) encountered.

Hand borings will be extended (as warranted) using manual DPT equipment (e.g., Geoprobe), which includes a collection barrel lined with disposable acetate sleeves, extension rods and a slide hammer. The barrel will collect samples from discreet intervals of 2 feet. Hand boring methods are generally restricted to shallow soil sampling (0 to 6' below grade) and may be employed/attempted if access by mechanized equipment is not practical.

DPT will typically be utilized during the extension of borings in overburden soils. The DPT rig will be equipped with a macro-core sampling barrel (minimum diameter 4") lined with disposable acetate sleeves. The barrel will collect samples from discreet intervals of either 4 or 5 feet. HSA rigs will use a continuous hollow stem auger with a split-spoon (collection interval of 2 feet) or other collection device. This system drives drill cuttings to the surface as drilling progresses, which will require management. Sonic drill rigs will utilize coring barrels of various lengths lined with plastic tubing.

Bore hole openings will be periodically screened with a photoionization detector (PID).

D. Installation and Development of Monitoring wells

Groundwater monitoring wells will be installed by the drilling subcontractor. Unless otherwise specified, monitoring wells will be constructed of two-inch PVC casing with a ten-foot length of 0.01-inch slotted PVC well screening across the water table. No glue will be used to thread the casing lengths. A minimum of 2 feet of screening will extend above the water table, with approximately 8 feet below the water level (depth to water will be inferred based on saturated soils encountered during installation, or from data from existing groundwater monitoring wells).

The annular space between the well screen and the borehole will be backfilled with clean silica sand to approximately two feet above the screen. A seal consisting of at least 12 inches of hydrated bentonite clay will be placed above the sand pack and the remaining annular space will be grouted with cement.

A locked cap with vent will be installed at the top of the PVC riser (well protection will be in accordance with the Work Plan, including use of secure “drive-over” metal cover or stick-up metal outer casing). A surveyor’s transit level will be used to determine the elevation of the top of the PVC well riser, relative to a permanent on-site marker, for use in determining relative groundwater elevations. Well locations and relative elevations will be recorded in field logs and indicated on all fieldwork maps.

The wells will be developed one week following installation. The wells will be developed with a properly decontaminated mechanical pump and dedicated polyethylene tubing in order to clear fine-grained material that may have settled around the well screen and to enhance the natural hydraulic connection between the well screen and the surrounding soils. Well development will begin at the top of the screened interval to prevent clogging of the pump within the well casing. Well development will be discontinued when the discharge water is free of obvious sediment, turbidity is below 50 NTUs and indicator parameters (e.g., dissolved oxygen, temperature, etc.) have stabilized. Upon completion, the pump assembly will be removed from the well while the pump is still running to avoid discharge of purged water back into the well. Development water will be securely stored on-site pending laboratory analysis.

E. Soil Sampling

Recovered sampling equipment will be placed on a clean surface (folding table, plastic sheeting, etc.) and opened (liners will be sliced with a clean razor knife). Recovered soils will be observed for potential contamination through observation and use of properly calibrated field instruments, e.g., PID. Samples will be collected directly from the sampling device. The volume of material collected will be sufficient for the required analyses and for reasonably anticipated potential additional analyses. Soil to be analyzed for volatile organic compounds (VOCs) will be collected following USEPA Method 5035 protocols, using laboratory sampling kits. Samples to be analyzed for parameters other than VOCs will be collected as either grab or composite samples, using disposable plastic trowels or properly decontaminated stainless steel instruments, or directly by the fieldwork technician using dedicated, fresh disposable nitrile gloves.

F. Groundwater Sampling

Groundwater sampling will be conducted using USEPA “Low-Stress” protocols (detailed in Supplement B). Sampling will be conducted using the following general procedures:

1. Groundwater sampling will begin at the potentially least contaminated well (as determined from well location and/or previous data) and proceed to the potentially most contaminated well. The field technician will check and record the condition of all monitoring wells for damage or evidence of tampering before initiating sampling. Plastic will be placed around wells to minimize potential contamination of sampling equipment from the ground surface, and all monitoring, purging and sampling equipment will be placed on the sheeting.
2. The protective casing on the well will be unlocked, the air in the well head will be screened with a PID, and static water level (from the top of the casing) will be measured with a decontaminated water-level meter. A peristaltic pump with plastic tubing, or a submersible pump attached to tubing (if required by Site conditions, e.g., well depth) will be used for sampling. The tubing (or pump attached to tubing) will be slowly lowered until reaching two to three feet off of the well bottom to prevent disturbance and re-suspension of any remaining sediment.
3. Depth to water will be measured to nearest 0.01 feet, relative to a reference measuring point on the well casing (if no pre-existing reference point is found, a reference point will be marked on the inner casing and noted in the field logbook). The water level will be measured before the pump is started and at intervals of every three to five minutes. Pumping rates will be reduced (as needed) to the minimum capabilities of the pump to ensure stabilization of the water level (drawdown of 0.3 feet or less).
4. During pumping, field indicator parameters (turbidity, temperature, specific conductance, pH, redox potential, and dissolved oxygen) will be monitored and recorded approximately every five minutes. The well will be considered stabilized when the indicator parameters have stabilized for three consecutive readings (the minimum purge interval will be at least 15 minutes).
5. All groundwater samples will be collected in a manner consistent with the QAPP.
6. The protective cap on the well will be replaced and locked following sampling, and the field sampling crew will move to the next most contaminated well and the process will be repeated.

III. GENERAL DECONTAMINATION PROTOCOL

Consistent decontamination methods will be used to reduce or eliminate contamination and cross-contamination of samples by field equipment, other samples or personnel, and to minimize potential exposures caused by the spread of contaminants. Decontamination will occur any time a sampling tool or instrument used in field investigations contacts sampled media or personnel using the equipment. These procedures will be used in conjunction with all non-dedicated (i.e. reusable) equipment used during the

handling, sampling or measuring of environmental media, and will be implemented primarily on-site at the point of use or at a designated equipment decontamination station at the project site.

Types of equipment usually requiring decontamination include pumps, gauges, augers and sampling barrels. Drilling equipment, water level meters, submersible pumping equipment, and any other non-dedicated monitoring and sampling equipment will be decontaminated prior to the start of fieldwork, after the collection of each media sample, and between boring intervals and/or sampling locations. Water quality parameter sensors and flow-through cell will be cleaned between sampling locations in accordance with the manufacturer's recommendations.

Materials and methods for decontamination are provided in Supplement D.

IV. PFAS SAMPLING - SPECIAL REQUIREMENTS

Special requirements apply to all fieldwork procedures during sampling for per- and polyfluoroalkyl substances (PFAS). Because of the potential presence of PFAS in common consumer products and in equipment typically used to collect media and the need for very low reporting limits, special handling and care must be taken when collecting samples for PFAS analysis to avoid sample contamination. There is only limited research regarding how the use of various procedures and materials affect sample results, and this SOP therefore represent a conservative approach. Field personnel should take precautions to avoid items that are likely to contain PFAS at the sampling site as well as avoid specific items during the sampling event, and must frequently check for updates to this SOP. The most recent NYSDEC guidance document (April 2023), as well as a *PFAS Sampling Quick Reference Field Guide* (provided by Michigan Department of Environment, Great Lakes, and Energy), are provided in Supplement E.

A. EQUIPMENT AND SUPPLIES

Avoid personal protective equipment (PPE, including clothing chemically treated for UV protection) and field supplies that may include PFAS and which could cross-contaminate field samples. Personal body products such as shampoos, moisturizers and cosmetics may contain PFAS and should be used with care the day of sampling. Sunblock and insect repellent ingredients need to be verified to ensure that they do not contain PFAS before use in the field.

Food and food packaging should not enter the sampling zone.

Water resistant, waterproof, stain-treated, clothing recently washed with fabric softeners, and new clothing should be avoided. If sampling in inclement weather a canopy tent may be a good option (note, however, that water resistant/waterproof material likely contains PFAS and disposable gloves should be worn when putting up and/or moving the tent.

Waterproof field books may contain PFAS and should not be used. Documentation of field activities should be on loose paper on an aluminum clipboard or in a waterproof field book that does not use PFAS. Field notes should be taken with a ball point pen (avoid large felt tip markers; fine and ultra-fine point Sharpie®

markers are acceptable). Sticky notes, etc., may contain PFAS and should be avoided (pre-printed labels should be verified PFAS-free).

Disposable, powderless, nitrile gloves must be worn during PFAS sampling and handling activities and should be changed frequently during and between sampling activities.

Sealed laboratory-supplied sampling containers may be placed into LDPE resealable storage bags (e.g., Ziploc®) that will not contact the sample media.

Chemical ice packs should not be used unless it is verified that they are PFAS-free. Samples for PFAS analysis should be placed on water ice immediately and should ideally be received by the laboratory at a temperature less than 6° Celsius.

B. GENERAL SAMPLING PROCEDURES

Sampling must be conducted in accordance with the project-specific QAPP, including use of laboratory-supplied sample containers.

If non-dedicated non-disposable equipment is used for sampling, proper decontamination is necessary. Decontamination reagents should be checked to ensure that they do not contain PFAS before use. Similarly, water used for decontamination should be checked (i.e. field equipment blanks) to verify that it does not contain PFAS. It may be necessary to collect samples of decontamination water prior to use to ensure that water being used for decontamination does not contain PFAS.

Soil samples should be collected using stainless steel, acetate, or polypropylene constructed equipment. Liners for soil sampling should not contain PFAS.

If a monitoring well has dedicated tubing that may contain PFAS, the dedicated tubing should be removed, and silicone or HDPE tubing should be used to sample for PFAS following at least one well volume purge prior to sampling for PFAS. The recommended length of time that dedicated tubing should be removed, and the recommended amount of purging conducted prior to sampling where dedicated tubing has been present is variable. If it is anticipated that dedicated tubing may be a source of PFAS cross contamination extra precaution, such as removal of the tubing 14 days prior to sampling or purging of three well volumes, should be considered.

Care should be taken to not cross contaminate PFAS samples if samples for non-PFAS analyses are being collected. For example, if VOCs and PFAS water samples are being collected, the VOCs would be collected using a peristaltic pump with HDPE and silicone tubing, and then a second set of samples would be collected for PFAS after changing gloves and switching sample container sets.

If transfer bottles are necessary for surface water sample collection, they should be PFAS-free and made of the same material as the laboratory provided sample containers.

If a water supply is to be sampled, both a pre- and post-treatment sample may be necessary. Carbon filtration, reverse osmosis, and other filter media may bias laboratory results for PFAS. Water should be

allowed to run freely until water quality parameter stabilization has occurred, typically between 3 and 5 minutes. Water flow rate should be reduced for minimal aeration.

Do not filter samples for PFAS analysis.

C. DECONTAMINATION OF PFAS SAMPLING EQUIPMENT

Special requirements apply to decontaminating non-dedicated equipment used for PFAS sampling. Laboratory supplied PFAS-free deionized water is preferred for decontamination (commercially available deionized water in an HDPE container, and municipal drinking water, may be used for decontamination if verified to be PFAS-free. Sampling equipment can be scrubbed using a polyethylene or polyvinyl chloride (PVC) brush to remove particulates. Decontamination procedures should include triple rinsing with PFAS-free water. Note that a QAPP prepared for NYSDEC program sites prohibits use of Liquinox®.

V. INVESTIGATION DERIVED WASTES

Disposal of any waste materials will be in accordance with provisions of the applicable site-specific Work Plan. If not otherwise specified: 1) discarded personal protective equipment and other fieldwork supplies not significantly impacted by free petroleum or other gross contaminants will be disposed as municipal solid waste; and, 2) well development purge water, spent absorbents or other significantly contaminated materials, and/or any recovered free-petroleum, will be properly stored on-site, in properly labeled and secured containers, pending final off-site disposal at a permitted facility.

Supplement A - Model Fieldwork Logs

Soil Boring Log



TECHNICAL SERVICES

Boring ID	Site ID:							GBTS PROJECT
PAGE ____ OF ____	DATE:		DRILLER (RIG)					
	GBTS STAFF:		WEATHER:					
BORING INTERVAL (RECOVERY)	SURFACE MATERIAL:		MOISTURE	PID (PPM)	ODORS	STAINING	NAPL	SAMPLES COLLECTED
	SOIL / MATERIAL DESCRIPTION							
____ - ____ (____ %)								
____ - ____ (____ %)								
____ - ____ (____ %)								
____ - ____ (____ %)								
____ - ____ (____ %)								
____ - ____ (____ %)								
Notes Fill, water conditions, field evidence of contamination, well installation details, etc								

ND (non-detect) **PID** (photoionization detector) **ppm** (parts per million) **NAPL** (non-aqueous phase liquid)
F (fine) **M** (medium) **C** (coarse) **P** (plastic) **LP** (low plastic) **NP** (non-plastic)

GROUNDWATER MONITORING WELL PURGE DATA SHEET



Project Name: _____
GBTS PROJECT #: _____
Date: _____
Field Personnel: _____
Weather: _____

Well ID: _____
PID Reading: _____
Depth of well: _____
Depth to water: _____
Pump type: _____

Time	Temp (°C)	pH	ORP (mv)	Specific Conductivity (ms/cm)	Turbidity (NTU)	Dissolved Oxygen (mg/L)	Depth to Water (ft)	Purge Rate (mL/m)	Comments (e.g. color/clarity)

***** STABILIZATION CRITERIA *****

Temp +/- 3% pH +/- 0.1 ORP +/- 10 Spec Cond +/- 3% Turb +/- 10% DO +/- 10%

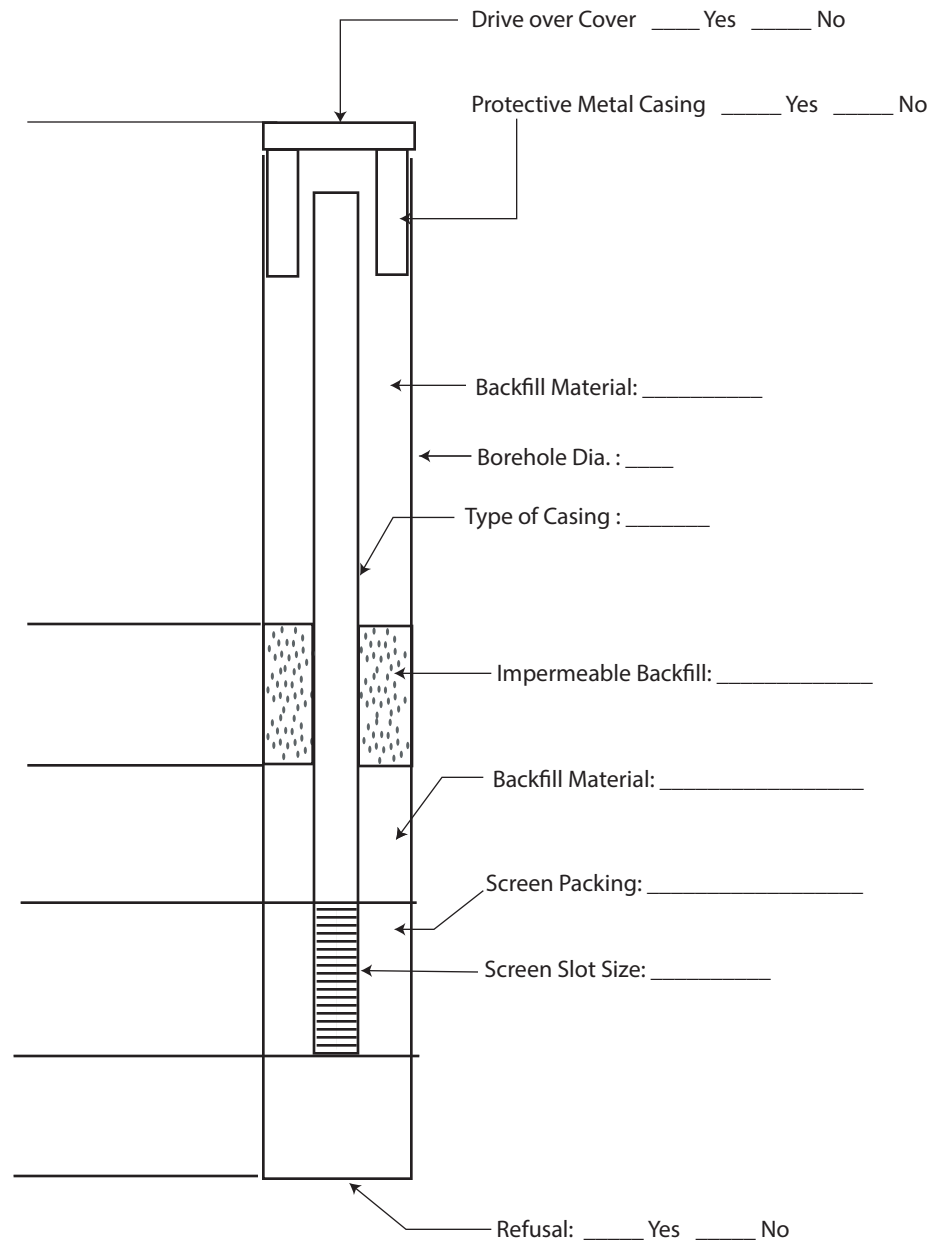
NOTES:

*****PURGED WATER DETAILS*****

Start/End time: _____
Total purge time: _____
Total volume: _____
Purge rate: _____

CHARACTERISTICS:

Odor: none | slight | moderate | strong
Sheen: none | slight | moderate | strong
L/DNAPL: Yes | No **L/DNAPL thickness (in.):** _____



Materials Used:
 Screen (PVC)
 Riser (PVC)
 Plug (PVC)
 Silica Sand
 Bentonite clay

Monitor Well Construction Log - Well ID:
 Site:

Project:

Date:

Appendix:

Supplement B - USEPA Groundwater Sampling

U.S. ENVIRONMENTAL PROTECTION AGENCY REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUNDWATER SAMPLES FROM MONITORING WELLS

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Approved by: _____
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Revision Page

Date	Rev #	Summary of changes	Sections
7/30/96	1	Finalized	
01/19/10	2	Updated	All sections
3/23/17	3	Updated	All sections
9/20/17	4	Updated	Section 7.0

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1.0 USE OF TERMS

Equipment blank: The equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank needs only to include the pump in subsequent sampling rounds. If the pump and tubing are dedicated to the well, the equipment blank is collected prior to its placement in the well. If the pump and tubing will be used to sample multiple wells, the equipment blank is normally collected after sampling from contaminated wells and not after background wells.

Field duplicates: Field duplicates are collected to determine precision of the sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

Indicator field parameters: This SOP uses field measurements of turbidity, dissolved oxygen, specific conductance, temperature, pH, and oxidation/reduction potential (ORP) as indicators of when purging operations are sufficient and sample collection may begin.

Matrix Spike/Matrix Spike Duplicates: Used by the laboratory in its quality assurance program. Consult the laboratory for the sample volume to be collected.

Potentiometric Surface: The level to which water rises in a tightly cased well constructed in a confined aquifer. In an unconfined aquifer, the potentiometric surface is the water table.

QAPP: Quality Assurance Project Plan

SAP: Sampling and Analysis Plan

SOP: Standard operating procedure

Stabilization: A condition that is achieved when all indicator field parameter measurements are sufficiently stable (as described in the "Monitoring Indicator Field Parameters" section) to allow sample collection to begin.

Temperature blank: A temperature blank is added to each sample cooler. The blank is measured upon receipt at the laboratory to assess whether the samples were properly cooled during transit.

Trip blank (VOCs): Trip blank is a sample of analyte-free water taken to the sampling site and returned to the laboratory. The trip blanks (one pair) are added to each sample cooler that contains VOC samples.

2.0 SCOPE & APPLICATION

The goal of this groundwater sampling procedure is to collect water samples that reflect the total mobile organic and inorganic loads (dissolved and colloidal sized fractions) transported through the subsurface under ambient flow conditions, with minimal physical and chemical alterations from sampling operations. This standard operating procedure (SOP) for collecting groundwater samples will help ensure that the project's data quality objectives (DQOs) are met under certain low-flow conditions.

The SOP emphasizes the need to minimize hydraulic stress at the well-aquifer interface by maintaining low water-level drawdowns, and by using low pumping rates during purging and sampling operations. Indicator field parameters (e.g., dissolved oxygen, pH, etc.) are monitored during purging in order to determine when sample collection may begin. Samples properly collected using this SOP are suitable for analysis of groundwater contaminants (volatile and semi-volatile organic analytes, dissolved gases, pesticides, PCBs, metals and other inorganics), or naturally occurring analytes. This SOP is based on Puls, and Barcelona (1996).

This procedure is designed for monitoring wells with an inside diameter (1.5-inches or greater) that can accommodate a positive lift pump with a screen length or open interval ten feet or less and with a water level above the top of the screen or open interval (Hereafter, the "screen or open interval" will be referred to only as "screen interval"). This SOP is not applicable to other well-sampling conditions.

While the use of dedicated sampling equipment is not mandatory, dedicated pumps and tubing can reduce sampling costs significantly by streamlining sampling activities and thereby reducing the overall field costs.

The goal of this procedure is to emphasize the need for consistency in deploying and operating equipment while purging and sampling monitoring wells during each sampling event. This will help to minimize sampling variability.

This procedure describes a general framework for groundwater sampling. Other site specific information (hydrogeological context, conceptual site model (CSM), DQOs, etc.) coupled with systematic planning must be added to the procedure in order to develop an appropriate site specific SAP/QAPP. In addition, the site specific SAP/QAPP must identify the specific equipment that will be used to collect the groundwater samples.

This procedure does not address the collection of water or free product samples from wells containing free phase LNAPLs and/or DNAPLs (light or dense non-aqueous phase

liquids). For this type of situation, the reader may wish to check: Cohen, and Mercer (1993) or other pertinent documents.

This SOP is to be used when collecting groundwater samples from monitoring wells at all Superfund, Federal Facility and RCRA sites in Region 1 under the conditions described herein. Request for modification of this SOP, in order to better address specific situations at individual wells, must include adequate technical justification for proposed changes. All changes and modifications must be approved and included in a revised SAP/QAPP before implementation in field.

3.0 BACKGROUND FOR IMPLEMENTATION

It is expected that the monitoring well screen has been properly located (both laterally and vertically) to intercept existing contaminant plume(s) or along flow paths of potential contaminant migration. Problems with inappropriate monitoring well placement or faulty/improper well installation cannot be overcome by even the best water sampling procedures. This SOP presumes that the analytes of interest are moving (or will potentially move) primarily through the more permeable zones intercepted by the screen interval.

Proper well construction, development, and operation and maintenance cannot be overemphasized. The use of installation techniques that are appropriate to the hydrogeologic setting of the site often prevent "problem well" situations from occurring. During well development, or redevelopment, tests should be conducted to determine the hydraulic characteristics of the monitoring well. The data can then be used to set the purging/sampling rate, and provide a baseline for evaluating changes in well performance and the potential need for well rehabilitation. Note: if this installation data or well history (construction and sampling) is not available or discoverable, for all wells to be sampled, efforts to build a sampling history should commence with the next sampling event.

The pump intake should be located within the screen interval and at a depth that will remain under water at all times. It is recommended that the intake depth and pumping rate remain the same for all sampling events. The mid-point or the lowest historical midpoint of the saturated screen length is often used as the location of the pump intake. For new wells, or for wells without pump intake depth information, the site's SAP/QAPP must provide clear reasons and instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected. If the depths to top and bottom of the well screen are not known, the SAP/QAPP will need to describe how the sampling depth will be determined and how the data can be used.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU, and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection

may still take place provided the indicator field parameter criteria in this procedure are met. If after 2 hours of purging indicator field parameters have not stabilized, one of three optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization), c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may reflect a sampling bias and therefore, the data may not meet the data quality objectives of the sampling event).

It is recommended that low-flow sampling be conducted when the air temperature is above 32°F (0°C). If the procedure is used below 32°F, special precautions will need to be taken to prevent the groundwater from freezing in the equipment. Because sampling during freezing temperatures may adversely impact the data quality objectives, the need for water sample collection during months when these conditions are likely to occur should be evaluated during site planning and special sampling measures may need to be developed. Ice formation in the flow-through-cell will cause the monitoring probes to act erratically. A transparent flow-through-cell needs to be used to observe if ice is forming in the cell. If ice starts to form on the other pieces of the sampling equipment, additional problems may occur.

4.0 HEALTH & SAFETY

When working on-site, comply with all applicable OSHA requirements and the site's health/safety procedures. All proper personal protection clothing and equipment are to be worn. Some samples may contain biological and chemical hazards. These samples should be handled with suitable protection to skin, eyes, etc.

5.0 CAUTIONS

The following cautions need to be considered when planning to collect groundwater samples when the below conditions occur.

If the groundwater degasses during purging of the monitoring well, dissolved gases and VOCs will be lost. When this happens, the groundwater data for dissolved gases (e.g., methane, ethene, ethane, dissolved oxygen, etc.) and VOCs will need to be qualified. Some conditions that can promote degassing are the use of a vacuum pump (e.g., peristaltic pumps), changes in aperture along the sampling tubing, and squeezing/pinching the pump's tubing which results in a pressure change.

When collecting the samples for dissolved gases and VOCs analyses, avoid aerating the groundwater in the pump's tubing. This can cause loss of the dissolved gases and VOCs in

the groundwater. Having the pump's tubing completely filled prior to sampling will avoid this problem when using a centrifugal pump or peristaltic pump.

Direct sun light and hot ambient air temperatures may cause the groundwater in the tubing and flow-through-cell to heat up. This may cause the groundwater to degas which will result in loss of VOCs and dissolved gases. When sampling under these conditions, the sampler will need to shade the equipment from the sunlight (e.g., umbrella, tent, etc.). If possible, sampling on hot days, or during the hottest time of the day, should be avoided. The tubing exiting the monitoring well should be kept as short as possible to avoid the sun light or ambient air from heating up the groundwater.

Thermal currents in the monitoring well may cause vertical mixing of water in the well bore. When the air temperature is colder than the groundwater temperature, it can cool the top of the water column. Colder water which is denser than warm water sinks to the bottom of the well and the warmer water at the bottom of the well rises, setting up a convection cell. "During low-flow sampling, the pumped water may be a mixture of convecting water from within the well casing and aquifer water moving inward through the screen. This mixing of water during low-flow sampling can substantially increase equilibration times, can cause false stabilization of indicator parameters, can give false indication of redox state, and can provide biological data that are not representative of the aquifer conditions" (Vroblesky 2007).

Failure to calibrate or perform proper maintenance on the sampling equipment and measurement instruments (e.g., dissolved oxygen meter, etc.) can result in faulty data being collected.

Interferences may result from using contaminated equipment, cleaning materials, sample containers, or uncontrolled ambient/surrounding air conditions (e.g., truck/vehicle exhaust nearby).

Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and/or proper planning to avoid ambient air interferences. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

Clean and decontaminate all sampling equipment prior to use. All sampling equipment needs to be routinely checked to be free from contaminants and equipment blanks collected to ensure that the equipment is free of contaminants. Check the previous equipment blank data for the site (if they exist) to determine if the previous cleaning procedure removed the contaminants. If contaminants were detected and they are a concern, then a more vigorous cleaning procedure will be needed.

6.0 PERSONNEL QUALIFICATIONS

All field samplers working at sites containing hazardous waste must meet the requirements of the OSHA regulations. OSHA regulations may require the sampler to take the 40 hour OSHA health and safety training course and a refresher course prior to engaging in any field activities, depending upon the site and field conditions.

The field samplers must be trained prior to the use of the sampling equipment, field instruments, and procedures. Training is to be conducted by an experienced sampler before initiating any sampling procedure.

The entire sampling team needs to read, and be familiar with, the site Health and Safety Plan, all relevant SOPs, and SAP/QAPP (and the most recent amendments) before going onsite for the sampling event. It is recommended that the field sampling leader attest to the understanding of these site documents and that it is recorded.

7.0 EQUIPMENT AND SUPPLIES

A. Informational materials for sampling event

A copy of the current Health and Safety Plan, SAP/QAPP, monitoring well construction data, location map(s), field data from last sampling event, manuals for sampling, and the monitoring instruments' operation, maintenance, and calibration manuals should be brought to the site.

B. Well keys.

C. Extraction device

Adjustable rate, submersible pumps (e.g., centrifugal, bladder, etc.) which are constructed of stainless steel or polytetrafluoroethylene (PTFE, i.e. Teflon®) are preferred. PTFE, however, should not be used when sampling for per- and polyfluoroalkyl substances (PFAS) as it is likely to contain these substances.

Note: If extraction devices constructed of other materials are to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

If bladder pumps are selected for the collection of VOCs and dissolved gases, the pump setting should be set so that one pulse will deliver a water volume that is sufficient to fill a 40 mL VOC vial. This is not mandatory, but is considered a “best practice”. For the proper operation, the bladder pump will need a minimum amount of water above the pump; consult the manufacturer for the recommended submergence. The pump’s recommended submergence value should be determined during the planning stage, since it may influence well construction and placement of dedicated pumps where water-level fluctuations are significant.

Adjustable rate, peristaltic pumps (suction) are to be used with caution when collecting samples for VOCs and dissolved gases (e.g., methane, carbon dioxide, etc.) analyses. Additional information on the use of peristaltic pumps can be found in Appendix A. If peristaltic pumps are used, the inside diameter of the rotor head tubing needs to match the inside diameter of the tubing installed in the monitoring well.

Inertial pumping devices (motor driven or manual) are not recommended. These devices frequently cause greater disturbance during purging and sampling, and are less easily controlled than submersible pumps (potentially increasing turbidity and sampling variability, etc.). This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

D. Tubing

PTFE (Teflon®) or PTFE-lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. As discussed in the previous section, PTFE tubing should not be used when sampling for PFAS. In this case, a suitable alternative such as high-density polyethylene tubing should be used.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for metal and other inorganics analyses.

Note: If tubing constructed of other materials is to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

The use of 1/4 inch or 3/8 inch (inside diameter) tubing is recommended. This will help ensure that the tubing remains liquid filled when operating at very low pumping rates when using centrifugal and peristaltic pumps.

Silastic tubing should be used for the section around the rotor head of a peristaltic pump. It should be less than a foot in length. The inside diameter of the tubing used at the pump rotor head must be the same as the inside diameter of tubing placed in the well. A tubing connector is used to connect the pump rotor head tubing to the well tubing. Alternatively, the two pieces of tubing can be connected to each other by placing the one end of the tubing inside the end of the other tubing. The tubing must not be reused.

E. The water level measuring device

Electronic "tape", pressure transducer, water level sounder/level indicator, etc. should be capable of measuring to 0.01 foot accuracy. Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each sampling event.

F. Flow measurement supplies

Graduated cylinder (size according to flow rate) and stopwatch usually will suffice.

Large graduated bucket used to record total water purged from the well.

G. Interface probe

To be used to check on the presence of free phase liquids (LNAPL, or DNAPL) before purging begins (as needed).

H. Power source (generator, nitrogen tank, battery, etc.)

When a gasoline generator is used, locate it downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate samples.

I. Indicator field parameter monitoring instruments

Use of a multi-parameter instrument capable of measuring pH, oxidation/reduction potential (ORP), dissolved oxygen (DO), specific conductance, temperature, and coupled with a flow-through-cell is required when measuring all indicator field parameters, except turbidity. Turbidity is collected using a separate instrument. Record equipment/instrument identification (manufacturer, and model number).

Transparent, small volume flow-through-cells (e.g., 250 mLs or less) are preferred. This allows observation of air bubbles and sediment buildup in the cell, which can interfere with the operation of the monitoring instrument probes, to be easily detected. A small volume

cell facilitates rapid turnover of water in the cell between measurements of the indicator field parameters.

It is recommended to use a flow-through-cell and monitoring probes from the same manufacturer and model to avoid incompatibility between the probes and flow-through-cell.

Turbidity samples are collected before the flow-through-cell. A “T” connector coupled with a valve is connected between the pump’s tubing and flow-through-cell. When a turbidity measurement is required, the valve is opened to allow the groundwater to flow into a container. The valve is closed and the container sample is then placed in the turbidimeter.

Standards are necessary to perform field calibration of instruments. A minimum of two standards are needed to bracket the instrument measurement range for all parameters except ORP which use a Zobell solution as a standard. For dissolved oxygen, a wet sponge used for the 100% saturation and a zero dissolved oxygen solution are used for the calibration.

Barometer (used in the calibration of the Dissolved Oxygen probe) and the conversion formula to convert the barometric pressure into the units of measure used by the Dissolved Oxygen meter are needed.

J. Decontamination supplies

Includes (for example) non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.

K. Record keeping supplies

Logbook(s), well purging forms, chain-of-custody forms, field instrument calibration forms, etc.

L. Sample bottles

M. Sample preservation supplies (as required by the analytical methods)

N. Sample tags or labels

O. PID or FID instrument

If appropriate, to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

P. Miscellaneous Equipment

Equipment to keep the sampling apparatus shaded in the summer (e.g., umbrella) and from freezing in the winter. If the pump's tubing is allowed to heat up in the warm weather, the cold groundwater may degas as it is warmed in the tubing.

8.0 EQUIPMENT/INSTRUMENT CALIBRATION

Prior to the sampling event, perform maintenance checks on the equipment and instruments according to the manufacturer's manual and/or applicable SOP. This will ensure that the equipment/instruments are working properly before they are used in the field.

Prior to sampling, the monitoring instruments must be calibrated and the calibration documented. The instruments are calibrated using U.S Environmental Protection Agency Region 1 *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity)*, March 23, 2017, or latest version or from one of the methods listed in 40CFR136, 40CFR141 and SW-846.

The instruments shall be calibrated at the beginning of each day. If the field measurement falls outside the calibration range, the instrument must be re-calibrated so that all measurements fall within the calibration range. At the end of each day, a calibration check is performed to verify that instruments remained in calibration throughout the day. This check is performed while the instrument is in measurement mode, not calibration mode. If the field instruments are being used to monitor the natural attenuation parameters, then a calibration check at mid-day is highly recommended to ensure that the instruments did not drift out of calibration. Note: during the day if the instrument reads zero or a negative number for dissolved oxygen, pH, specific conductance, or turbidity (negative value only), this indicates that the instrument drifted out of calibration or the instrument is malfunctioning. If this situation occurs the data from this instrument will need to be qualified or rejected.

9.0 PRELIMINARY SITE ACTIVITIES (as applicable)

Check the well for security (damage, evidence of tampering, missing lock, etc.) and record pertinent observations (include photograph as warranted).

If needed, lay out a sheet of clean polyethylene for monitoring and sampling equipment, unless equipment is elevated above the ground (e.g., on a table, etc.).

Remove well cap and if appropriate measure VOCs at the rim of the well with a PID or FID instrument and record reading in field logbook or on the well purge form.

If the well casing does not have an established reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook (consider a photographic record as well). All water level measurements must be recorded relative to this reference point (and the altitude of this point should be determined using techniques that are appropriate to site's DQOs).

If water-table or potentiometric surface map(s) are to be constructed for the sampling event, perform synoptic water level measurement round (in the shortest possible time) before any purging and sampling activities begin. If possible, measure water level depth (to 0.01 ft.) and total well depth (to 0.1 ft.) the day before sampling begins, in order to allow for re-settlement of any particulates in the water column. This is especially important for those wells that have not been recently sampled because sediment buildup in the well may require the well to be redeveloped. If measurement of total well depth is not made the day before, it should be measured after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe may not be necessary unless analytical data or field analysis signal a worsening situation. This SOP cannot be used in the presence of LNAPLs or DNAPLs. If NAPLs are present, the project team must decide upon an alternate sampling method. All project modifications must be approved and documented prior to implementation.

If available check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). If changes are made in the intake depth or extraction rate(s) used during previous sampling event(s), for either portable or dedicated extraction devices, record new values, and explain reasons for the changes in the field logbook.

10.0 PURGING AND SAMPLING PROCEDURE

Purging and sampling wells in order of increasing chemical concentrations (known or anticipated) are preferred.

The use of dedicated pumps is recommended to minimize artificial mobilization and entrainment of particulates each time the well is sampled. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

A. Initial Water Level

Measure the water level in the well before installing the pump if a non-dedicated pump is being used. The initial water level is recorded on the purge form or in the field logbook.

B. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the appropriate depth (may not be the mid-point of the screen/open interval). The Sampling and Analysis Plan/Quality Assurance Project Plan should specify the sampling depth (used previously), or provide criteria for selection of intake depth for each new well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well.

Pump tubing lengths, above the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating may cause the groundwater to degas, which is unacceptable for the collection of samples for VOC and dissolved gases analyses.

C. Measure Water Level

Before starting pump, measure water level. Install recording pressure transducer, if used to track drawdowns, to initialize starting condition.

D. Purge Well

From the time the pump starts purging and until the time the samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged. This information is recorded on the purge form or in the field logbook.

Start the pump at low speed and slowly increase the speed until discharge occurs. Check water level. Check equipment for water leaks and if present fix or replace the affected equipment. Try to match pumping rate used during previous sampling event(s). Otherwise, adjust pump speed until there is little or no water level drawdown. If the

minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging.

Monitor and record the water level and pumping rate every five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" somewhat as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. If the initial water level is above the top of the screen do not allow the water level to fall into the well screen. The final purge volume must be greater than the stabilized drawdown volume plus the pump's tubing volume. If the drawdown has exceeded 0.3 feet and stabilizes, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are collected.

Avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This will cause the groundwater to degas and result in a loss of VOCs and dissolved gasses in the groundwater samples.

Note: the flow rate used to achieve a stable pumping level should remain constant while monitoring the indicator parameters for stabilization and while collecting the samples.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (e.g., bladder, peristaltic), and/or the use of dedicated equipment. For new monitoring wells, or wells where the following situation has not occurred before, if the recovery rate to the well is less than 50 mL/min., or the well is being essentially dewatered during purging, the well should be sampled as soon as the water level has recovered sufficiently to collect the volume needed for all anticipated samples. The project manager or field team leader will need to make the decision when samples should be collected, how the sample is to be collected, and the reasons recorded on the purge form or in the field logbook. A water level measurement needs to be performed and recorded before samples are collected. If the project manager decides to collect the samples using the pump, it is best during this recovery period that the pump intake tubing not be removed, since this will aggravate any turbidity problems. Samples in this specific situation may be collected without stabilization of indicator field parameters. Note that field conditions and efforts to overcome problematic situations must be recorded in order to support field decisions to deviate from normal procedures described in this SOP. If this type of problematic situation persists in a well, then water sample collection should be

changed to a passive or no-purge method, if consistent with the site's DQOs, or have a new well installed.

E. Monitor Indicator Field Parameters

After the water level has stabilized, connect the "T" connector with a valve and the flow-through-cell to monitor the indicator field parameters. If excessive turbidity is anticipated or encountered with the pump startup, the well may be purged for a while without connecting up the flow-through-cell, in order to minimize particulate buildup in the cell (This is a judgment call made by the sampler). Water level drawdown measurements should be made as usual. If possible, the pump may be installed the day before purging to allow particulates that were disturbed during pump insertion to settle.

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, ORP, DO) at a frequency of five minute intervals or greater. The pump's flow rate must be able to "turn over" at least one flow-through-cell volume between measurements (for a 250 mL flow-through-cell with a flow rate of 50 mLs/min., the monitoring frequency would be every five minutes; for a 500 mL flow-through-cell it would be every ten minutes). If the cell volume cannot be replaced in the five minute interval, then the time between measurements must be increased accordingly. Note: during the early phase of purging, emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments followed by stabilization of indicator parameters. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings are within the following limits:

Turbidity (10% for values greater than 5 NTU; if three Turbidity values are less than 5 NTU, consider the values as stabilized),

Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),

Specific Conductance (3%),

Temperature (3%),

pH (± 0.1 unit),

Oxidation/Reduction Potential (± 10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Samples for turbidity measurements are obtained before water enters the flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and

continue monitoring activities. Record start and stop times and give a brief description of cleaning activities.

The flow-through-cell must be designed in a way that prevents gas bubble entrapment in the cell. Placing the flow-through-cell at a 45 degree angle with the port facing upward can help remove bubbles from the flow-through-cell (see Appendix B Low-Flow Setup Diagram). Throughout the measurement process, the flow-through-cell must remain free of any gas bubbles. Otherwise, the monitoring probes may act erratically. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must remain submerged in water at all times.

F. Collect Water Samples

When samples are collected for laboratory analyses, the pump's tubing is disconnected from the "T" connector with a valve and the flow-through-cell. The samples are collected directly from the pump's tubing. Samples must not be collected from the flow-through-cell or from the "T" connector with a valve.

VOC samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the pump's flow rate is too high to collect the VOC/dissolved gases samples, collect the other samples first. Lower the pump's flow rate to a reasonable rate and collect the VOC/dissolved gases samples and record the new flow rate.

During purging and sampling, the centrifugal/peristaltic pump tubing must remain filled with water to avoid aeration of the groundwater. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help ensure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use the following procedure to collect samples: collect non-VOC/dissolved gases samples first, then increase flow rate slightly until the water completely fills the tubing, collect the VOC/dissolved gases samples, and record new drawdown depth and flow rate.

For bladder pumps that will be used to collect VOC or dissolved gas samples, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 mL VOC vial.

Use pre-preserved sample containers or add preservative, as required by analytical methods, to the samples immediately after they are collected. Check the analytical methods

(e.g. EPA SW-846, 40 CFR 136, water supply, etc.) for additional information on preservation.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter (transparent housing preferred) is required, and the filter size (0.45 μm is commonly used) should be based on the sampling objective. Pre-rinse the filter with groundwater prior to sample collection. Make sure the filter is free of air bubbles before samples are collected. Preserve the filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health or ecological risk calculations.

Label each sample as collected. Samples requiring cooling will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

G. Post Sampling Activities

If a recording pressure transducer is used to track drawdown, re-measure water level with tape.

After collection of samples, the pump tubing may be dedicated to the well for re-sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth annually is usually sufficient after the initial low stress sampling event. However, a greater frequency may be needed if the well has a “silting” problem or if confirmation of well identity is needed.

Secure the well.

11.0 DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well, and then following sampling of each subsequent well. Pumps should not be removed between purging and sampling operations. The pump, tubing, support cable and electrical wires which were in contact with the well should be decontaminated by one of the procedures listed below.

The use of dedicated pumps and tubing will reduce the amount of time spent on decontamination of the equipment. If dedicated pumps and tubing are used, only the initial sampling event will require decontamination of the pump and tubing.

Note if the previous equipment blank data showed that contaminant(s) were present after using the below procedure or the one described in the SAP/QAPP, a more vigorous procedure may be needed.

Procedure 1

Decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump and tubing. The pump may be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Optional - flush with isopropyl alcohol (pesticide grade; must be free of ketones {e.g., acetone}) or with methanol. This step may be required if the well is highly contaminated or if the equipment blank data from the previous sampling event show that the level of contaminants is significant.

Flush with distilled/deionized water. This step must remove all traces of alcohol (if used) from the equipment. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

12.0 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. Quality control samples include field duplicates, equipment blanks, matrix spike/matrix spike duplicates, trip blanks (VOCs), and temperature blanks.

13.0 FIELD LOGBOOK

A field log shall be kept to document all groundwater field monitoring activities (see Appendix C, example table), and record the following for each well:

Site name, municipality, state.

Well identifier, latitude-longitude or state grid coordinates.

Measuring point description (e.g., north side of PVC pipe).

Well depth, and measurement technique.

Well screen length.

Pump depth.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and detection method.

Pumping rate, drawdown, indicator parameters values, calculated or measured total volume pumped, and clock time of each set of measurements.

Type of tubing used and its length.

Type of pump used.

Clock time of start and end of purging and sampling activity.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analyses.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions, including approximate ambient air temperature.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling/monitoring equipment used, including trade names, model number, instrument identification number, diameters, material composition, etc.

14.0 DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, field indicator parameters measured during purging, field instrument calibration information, and whatever other field logbook information is needed to allow for a full evaluation of data usability.

Note: the use of trade, product, or firm names in this sampling procedure is for descriptive purposes only and does not constitute endorsement by the U.S. EPA.

15.0 REFERENCES

Cohen, R.M. and J.W. Mercer, 1993, *DNAPL Site Evaluation*; C.K. Smoley (CRC Press), Boca Raton, Florida.

Robert W. Puls and Michael J. Barcelona, *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*, April 1996 (EPA/540/S-95/504).

U.S. Environmental Protection Agency, 1992, *RCRA Ground-Water Monitoring: Draft Technical Guidance*; Washington, DC (EPA/530-R-93-001).

U.S. Environmental Protection Agency, 1987, *A Compendium of Superfund Field Operations Methods*; Washington, DC (EPA/540/P-87/001).

U.S. Environmental Protection Agency, Region 1, *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity)*, March 23, 2017 or latest version.

U.S. Environmental Protection Agency, EPA SW-846.

U.S. Environmental Protection Agency, 40 CFR 136.

U.S. Environmental Protection Agency, 40 CFR 141.

Vroblesky, Don A., Clifton C. Casey, and Mark A. Lowery, Summer 2007, Influence of Dissolved Oxygen Convection on Well Sampling, *Ground Water Monitoring & Remediation* 27, no. 3: 49-58.

APPENDIX A

PERISTALTIC PUMPS

Before selecting a peristaltic pump to collect groundwater samples for VOCs and/or dissolved gases, (e.g., methane, carbon dioxide, etc.) consideration should be given to the following:

- The decision of whether or not to use a peristaltic pump is dependent on the intended use of the data.
- If the additional sampling error that may be introduced by this device is NOT of concern for the VOC/dissolved gases data's intended use, then this device may be acceptable.
- If minor differences in the groundwater concentrations could affect the decision, such as to continue or terminate groundwater cleanup or whether the cleanup goals have been reached, then this device should NOT be used for VOC/dissolved gases sampling. In these cases, centrifugal or bladder pumps are a better choice for more accurate results.

EPA and USGS have documented their concerns with the use of the peristaltic pumps to collect water sample in the below documents.

- "Suction Pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.
- "The agency does not recommend the use of peristaltic pumps to sample ground water particularly for volatile organic analytes" *RCRA Ground-Water Monitoring Draft Technical Guidance*, EPA Office of Solid Waste, November 1992.
- "The peristaltic pump is limited to shallow applications and can cause degassing resulting in alteration of pH, alkalinity, and volatiles loss", *Low-flow (Minimal drawdown) Ground-Water Sampling Procedures*, by Robert Puls & Michael Barcelona, April 1996, EPA/540/S-95/504.
- "Suction-lift pumps, such as peristaltic pumps, can operate at a very low pumping rate; however, using negative pressure to lift the sample can result in the loss of volatile analytes", USGS Book 9 Techniques of Water-Resources Investigation, Chapter A4. (Version 2.0, 9/2006).

APPENDIX B

SUMMARY OF SAMPLING INSTRUCTIONS

These instructions are for using an adjustable rate, submersible pump or a peristaltic pump with the pump's intake placed at the midpoint of a 10 foot or less well screen or an open interval. The water level in the monitoring well is above the top of the well screen or open interval, the ambient temperature is above 32°F, and the equipment is not dedicated. Field instruments are already calibrated. The equipment is setup according to the diagram at the end of these instructions.

1. Review well installation information. Record well depth, length of screen or open interval, and depth to top of the well screen. Determine the pump's intake depth (e.g., mid-point of screen/open interval).
2. On the day of sampling, check security of the well casing, perform any safety checks needed for the site, lay out a sheet of polyethylene around the well (if necessary), and setup the equipment. If necessary a canopy or an equivalent item can be setup to shade the pump's tubing and flow-through-cell from the sun light to prevent the sun light from heating the groundwater.
3. Check well casing for a reference mark. If missing, make a reference mark. Measure the water level (initial) to 0.01 ft. and record this information.
4. Install the pump's intake to the appropriate depth (e.g., midpoint) of the well screen or open interval. Do not turn-on the pump at this time.
5. Measure water level and record this information.
6. Turn-on the pump and discharge the groundwater into a graduated waste bucket. Slowly increase the flow rate until the water level starts to drop. Reduce the flow rate slightly so the water level stabilizes. Record the pump's settings. Calculate the flow rate using a graduated container and a stop watch. Record the flow rate. Do not let the water level drop below the top of the well screen.

If the groundwater is highly turbid or discolored, continue to discharge the water into the bucket until the water clears (visual observation); this usually takes a few minutes. The turbid or discolored water is usually from the well-being disturbed during the pump installation. If the water does not clear, then you need to make a choice whether to continue purging the well (hoping that it will clear after a reasonable time) or continue to

the next step. Note, it is sometimes helpful to install the pump the day before the sampling event so that the disturbed materials in the well can settle out.

If the water level drops to the top of the well screen during the purging of the well, stop purging the well, and do the following:

Wait for the well to recharge to a sufficient volume so samples can be collected. This may take a while (pump may be removed from well, if turbidity is not a problem). The project manager will need to make the decision when samples should be collected and the reasons recorded in the site's log book. A water level measurement needs to be performed and recorded before samples are collected. When samples are being collected, the water level must not drop below the top of the screen or open interval. Collect the samples from the pump's tubing. Always collect the VOCs and dissolved gases samples first. Normally, the samples requiring a small volume are collected before the large volume samples are collected just in case there is not sufficient water in the well to fill all the sample containers. All samples must be collected, preserved, and stored according to the analytical method. Remove the pump from the well and decontaminate the sampling equipment.

If the water level has dropped 0.3 feet or less from the initial water level (water level measure before the pump was installed); proceed to Step 7. If the water level has dropped more than 0.3 feet, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are be collected.

7. Attach the pump's tubing to the "T" connector with a valve (or a three-way stop cock). The pump's tubing from the well casing to the "T" connector must be as short as possible to prevent the groundwater in the tubing from heating up from the sun light or from the ambient air. Attach a short piece of tubing to the other end of the end of the "T" connector to serve as a sampling port for the turbidity samples. Attach the remaining end of the "T" connector to a short piece of tubing and connect the tubing to the flow-through-cell bottom port. To the top port, attach a small piece of tubing to direct the water into a calibrated waste bucket. Fill the cell with the groundwater and remove all gas bubbles from the cell. Position the flow-through-cell in such a way that if gas bubbles enter the cell they can easily exit the cell. If the ports are on the same side of the cell and the cell is cylindrical shape, the cell can be placed at a 45-degree angle with the ports facing upwards; this position should keep any gas bubbles entering the cell away from the monitoring probes and allow the gas bubbles to exit the cell easily (see Low-Flow Setup Diagram). Note:

make sure there are no gas bubbles caught in the probes' protective guard; you may need to shake the cell to remove these bubbles.

8. Turn-on the monitoring probes and turbidity meter.

9. Record the temperature, pH, dissolved oxygen, specific conductance, and oxidation/reduction potential measurements. Open the valve on the "T" connector to collect a sample for the turbidity measurement, close the valve, do the measurement, and record this measurement. Calculate the pump's flow rate from the water exiting the flow-through-cell using a graduated container and a stop watch, and record the measurement. Measure and record the water level. Check flow-through-cell for gas bubbles and sediment; if present, remove them.

10. Repeat Step 9 every 5 minutes or as appropriate until monitoring parameters stabilized. Note: at least one flow-through-cell volume must be exchanged between readings. If not, the time interval between readings will need to be increased. Stabilization is achieved when three consecutive measurements are within the following limits:

Turbidity (10% for values greater than 5 NTUs; if three Turbidity values are less than 5 NTUs, consider the values as stabilized),

Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),

Specific Conductance (3%),

Temperature (3%),

pH (± 0.1 unit),

Oxidation/Reduction Potential (± 10 millivolts).

If these stabilization requirements do not stabilize in a reasonable time, the probes may have been coated from the materials in the groundwater, from a buildup of sediment in the flow-through-cell, or a gas bubble is lodged in the probe. The cell and the probes will need to be cleaned. Turn-off the probes (not the pump), disconnect the cell from the "T" connector and continue to purge the well. Disassemble the cell, remove the sediment, and clean the probes according to the manufacturer's instructions. Reassemble the cell and connect the cell to the "T" connector. Remove all gas bubbles from the cell, turn-on the probes, and continue the measurements. Record the time the cell was cleaned.

11. When it is time to collect the groundwater samples, turn-off the monitoring probes, and disconnect the pump's tubing from the "T" connector. If you are using a centrifugal or peristaltic pump check the pump's tubing to determine if the tubing is completely filled with water (no air space).

All samples must be collected and preserved according to the analytical method. VOCs and dissolved gases samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

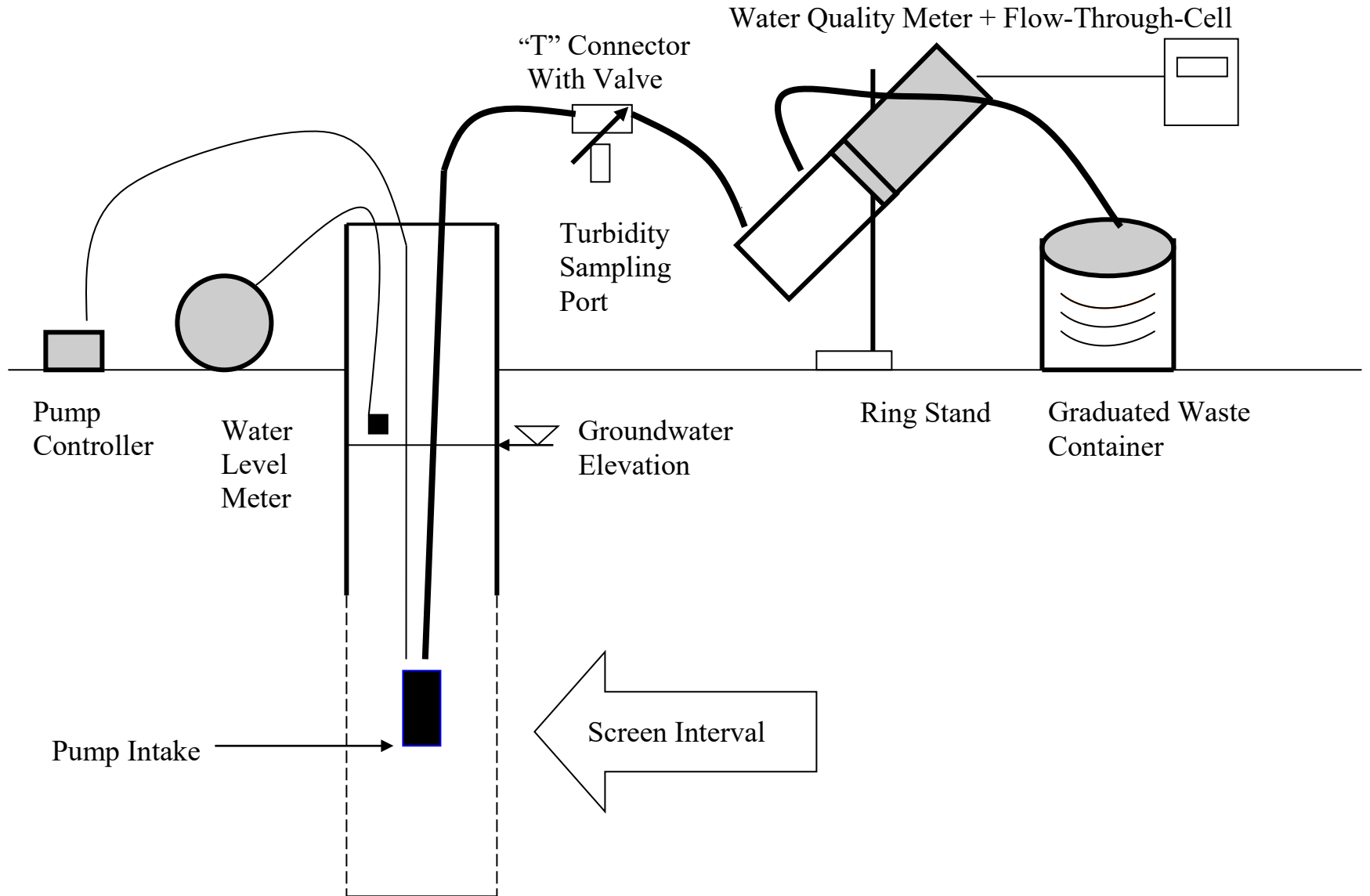
If the pump's tubing is not completely filled with water and the samples are being collected for VOCs and/or dissolved gases analyses using a centrifugal or peristaltic pump, do the following:

All samples must be collected and preserved according to the analytical method. The VOCs and the dissolved gases (e.g., methane, ethane, ethene, and carbon dioxide) samples are collected last. When it becomes time to collect these samples increase the pump's flow rate until the tubing is completely filled. Collect the samples and record the new flow rate.

12. Store the samples according to the analytical method.

13. Record the total purged volume (graduated waste bucket). Remove the pump from the well and decontaminate the sampling equipment.

Low-Flow Setup Diagram



EXAMPLE (Minimum Requirements)
WELL PURGING-FIELD WATER QUALITY MEASUREMENTS FORM

Location (Site/Facility Name) _____						Depth to _____ / _____ of screen					
Well Number _____				Date _____		(below MP) top bottom			Pump Intake at (ft. below MP) _____		
Field Personnel _____						Purging Device; (pump type) _____					
Sampling Organization _____						Total Volume Purged _____					
Identify MP _____											

Clock Time 24 HR	Water Depth below MP ft	Pump Dial ¹	Purge Rate ml/min	Cum. Volume Purged liters	Temp. "C	Spec. Cond. ² µS/cm	pH	ORP ³ mv	DO mg/L	Tur- bidity NTU	Comments

Stabilization Criteria 3% 3% ±0.1 ±10 mv 10% 10%

- 1. Pump dial setting (for example: hertz, cycles/min, etc).
- 2. µSiemens per cm(same as µmhos/cm)at 25°C.
- 3. Oxidation reduction potential (ORP)

Supplement C - Decontamination

SOP SUPPLEMENT: DECONTAMINATION

1.0 Objectives

Decontamination will occur any time a sampling tool or instrument used in field investigations contacts sampled media or personnel using the equipment. This procedure will be used in conjunction with all non-dedicated (i.e. reusable) equipment used during the handling, sampling or measuring of media. Special precautions are required when sampling for PFAS.

These procedures will be implemented primarily on-site at the point of use or at a designated equipment decontamination station at the project site. Examples of equipment usually requiring decontamination include pumps, depth gauges, hand augers, macro-core sampling barrels and other related equipment used for the collection of samples or the measurement of field parameters.

2.0 Required Materials

The equipment and supplies required for this SOP include the following:

- Plastic sheeting for the decontamination area
- Properly labeled drums to hold waste decontamination solutions and expendable supplies
- Plastic bags and/or aluminum foil to keep decontaminated equipment clean until the next use
- Gloves, aprons, safety glasses, and any other PPE required in the Site HASP
- Disposable towels and wipes
- Clean buckets or tubs to hold wash and rinse solutions, of a size appropriate to the equipment to be decontaminated
- Long-handled brushes for scrubbing and flat-bladed scrapers
- Dispensing bottles
- Tap water
- Deionized or distilled water (grade determined by project requirements)
- Non-phosphate detergent such as Alconox
- Methanol, nitric acid, etc. as required by the project work plan or quality assurance plan

Some Work Plans may include additional equipment rinses based on the contaminants being investigated. Examples of this are 0.1N nitric acid when cross-contamination from metals is a concern, and solvents such as methanol, isopropanol, or hexane, when cross-contamination from organics is a concern. If these are required, labeled inert dispensing bottles and Safety Data Sheets (SDS) for these rinses will be filed on site.

Supplement D - PFAS Sampling Guidance



Department of
Environmental
Conservation

SAMPLING, ANALYSIS, AND ASSESSMENT OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS)

Under NYSDEC's Part 375 Remedial Programs

April 2023



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ERRATA SHEET for

SAMPLING, ANALYSIS, AND ASSESSMENT OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS) Under NYSDEC’s Part 375 Remedial Programs Issued January 17, 2020

Citation and Page Number	Current Text	Corrected Text	Date
Title of Appendix I, page 32	Appendix H	Appendix I	2/25/2020
Document Cover, page 1	Guidelines for Sampling and Analysis of PFAS	Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC’s Part 375 Remedial Programs	9/15/2020
Data Assessment and Application to Site Cleanup Page 3	Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published	Until such time as Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published	3/28/2023
Water Sample Results Page 3	PFOA and PFOS should be further assessed and considered as potential contaminants of concern in groundwater or surface water if PFOA or PFOS is detected in any water sample at or above 10 ng/L (ppt) and is determined to be attributable to the site, either by a comparison of upgradient and downgradient levels, or the presence of soil source areas, as defined below.	NYSDEC has adopted ambient water quality guidance values for PFOA and PFOS. Groundwater samples should be compared to the human health criteria of 6.7 ng/l (ppt) for PFOA and 2.7 ng/l (ppt) for PFOS. These guidance values also include criteria for surface water for PFOS applicable for aquatic life, which may be applicable at some sites. Drinking water sample results should be compared to the NYS maximum contaminant level (MCL) of 10 ng/l (ppt). Analysis to determine if PFOA and PFOS concentrations are attributable to the site should include a comparison between upgradient and downgradient levels, and the presence of soil source areas, as defined below.	3/28/2023
Soil Sample Results Page 3	Soil cleanup objectives for PFOA and PFOS have been proposed in an upcoming revision to 6 NYCRR Part 375-6. Until SCOs are in effect, the following are to be used as guidance values:	NYSDEC will delay adding soil cleanup objectives for PFOA and PFOS to 6 NYCRR Part 375-6 until the PFAS rural soil background study has been completed. Until SCOs are in effect, the following are to be used as guidance values:	3/28/2023
Protection of Groundwater Page 3	PFOA (ppb) 1.1 PFOS (ppb) 3.7	PFOA (ppb) 0.8 PFOS (ppb) 1.0	3/28/2023

Citation and Page Number	Current Text	Corrected Text	Date
Footnote 2 Page 3	The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).	The Protection of Groundwater values are based on the above referenced ambient groundwater guidance values. Details on that calculation are available in the following document, prepared for the February 2022 proposed changes to Part 375 (https://www.dec.ny.gov/docs/remediation_hudson_pdf/part375techsupport.pdf). The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).	3/28/2023
Testing for Imported Soil Page 4	If the concentrations of PFOA and PFOS in leachate are at or above 10 ppt (the Maximum Contaminant Levels established for drinking water by the New York State Department of Health), then the soil is not acceptable.	If the concentrations of PFOA and PFOS in leachate are at or above the ambient water quality guidance values for groundwater, then the soil is not acceptable.	3/28/2023
Routine Analysis, page 9	“However, laboratories analyzing environmental samples...PFOA and PFOS in drinking water by EPA Method 537, 537.1 or ISO 25101.”	“However, laboratories analyzing environmental samples...PFOA and PFOS in drinking water by EPA Method 537, 537.1, ISO 25101, or Method 533.”	9/15/2020
Additional Analysis, page 9, new paragraph regarding soil parameters	None	“In cases where site-specific cleanup objectives for PFOA and PFOS are to be assessed, soil parameters, such as Total Organic Carbon (EPA Method 9060), soil pH (EPA Method 9045), clay content (percent), and cation exchange capacity (EPA Method 9081), should be included in the analysis to help evaluate factors affecting the leachability of PFAS in site soils.”	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Data Assessment and Application to Site Cleanup Page 10	Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFAS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Target levels for cleanup of PFAS in other media, including biota and sediment, have not yet been established by the DEC.	Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Preliminary target levels for cleanup of PFOA and PFOS in other media, including biota and sediment, have not yet been established by the DEC.	9/15/2020
Water Sample Results Page 10	<p>PFAS should be further assessed and considered as a potential contaminant of concern in groundwater or surface water (...)</p> <p>If PFAS are identified as a contaminant of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10.</p>	<p>PFOA and PFOS should be further assessed and considered as potential contaminants of concern in groundwater or surface water (...)</p> <p>If PFOA and/or PFOS are identified as contaminants of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10.</p>	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Soil Sample Results, page 10	<p>“The extent of soil contamination for purposes of delineation and remedy selection should be determined by having certain soil samples tested by Synthetic Precipitation Leaching Procedure (SPLP) and the leachate analyzed for PFAS. Soil exhibiting SPLP results above 70 ppt for either PFOA or PFOS (individually or combined) are to be evaluated during the cleanup phase.”</p>	<p>“Soil cleanup objectives for PFOA and PFOS will be proposed in an upcoming revision to 6 NYCRR Part 375-6. Until SCOs are in effect, the following are to be used as guidance values. “</p> <p>[Interim SCO Table]</p> <p>“PFOA and PFOS results for soil are to be compared against the guidance values listed above. These guidance values are to be used in determining whether PFOA and PFOS are contaminants of concern for the site and for determining remedial action objectives and cleanup requirements. Site-specific remedial objectives for protection of groundwater can also be presented for evaluation by DEC. Development of site-specific remedial objectives for protection of groundwater will require analysis of additional soil parameters relating to leachability. These additional analyses can include any or all the parameters listed above (soil pH, cation exchange capacity, etc.) and/or use of SPLP.</p> <p>As the understanding of PFAS transport improves, DEC welcomes proposals for site-specific remedial objectives for protection of groundwater. DEC will expect that those may be dependent on additional factors including soil pH, aqueous pH, % organic carbon, % Sand/Silt/Clay, soil cations: K, Ca, Mg, Na, Fe, Al, cation exchange capacity, and anion exchange capacity. Site-specific remedial objectives should also consider the dilution attenuation factor (DAF). The NJDEP publication on DAF can be used as a reference: https://www.nj.gov/dep/srp/guidance/rs/daf.pdf. ”</p>	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
<p>Testing for Imported Soil Page 11</p>	<p>Soil imported to a site for use in a soil cap, soil cover, or as backfill is to be tested for PFAS in general conformance with DER-10, Section 5.4(e) for the PFAS Analyte List (Appendix F) using the analytical procedures discussed below and the criteria in DER-10 associated with SVOCs.</p> <p>If PFOA or PFOS is detected in any sample at or above 1 µg/kg, then soil should be tested by SPLP and the leachate analyzed for PFAS. If the SPLP results exceed 10 ppt for either PFOA or PFOS (individually) then the source of backfill should be rejected, unless a site-specific exemption is provided by DER. SPLP leachate criteria is based on the Maximum Contaminant Levels proposed for drinking water by New York State’s Department of Health, this value may be updated based on future Federal or State promulgated regulatory standards. Remedial parties have the option of analyzing samples concurrently for both PFAS in soil and in the SPLP leachate to minimize project delays. Category B deliverables should be submitted for backfill samples, though a DUSR is not required.</p>	<p>Testing for PFAS should be included any time a full TAL/TCL analyte list is required. Results for PFOA and PFOS should be compared to the applicable guidance values. If PFOA or PFOS is detected in any sample at or above the guidance values then the source of backfill should be rejected, unless a site-specific exemption is provided by DER based on SPLP testing, for example. If the concentrations of PFOA and PFOS in leachate are at or above 10 ppt (the Maximum Contaminant Levels established for drinking water by the New York State Department of Health), then the soil is not acceptable.</p> <p>PFOA, PFOS and 1,4-dioxane are all considered semi-volatile compounds, so composite samples are appropriate for these compounds when sampling in accordance with DER-10, Table 5.4(e)10. Category B deliverables should be submitted for backfill samples, though a DUSR is not required.</p>	<p>9/15/2020</p>

Citation and Page Number	Current Text	Corrected Text	Date
Footnotes	None	<p>¹ TOP Assay analysis of highly contaminated samples, such as those from an AFFF (aqueous film-forming foam) site, can result in incomplete oxidation of the samples and an underestimation of the total perfluoroalkyl substances.</p> <p>² The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the soil cleanup objective for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).</p>	9/15/2020
Additional Analysis, page 9	In cases... soil parameters, such as Total Organic Carbon (EPA Method 9060), soil...	In cases... soil parameters, such as Total Organic Carbon (Lloyd Kahn), soil...	1/8/2021
Appendix A, General Guidelines, fourth bullet	List the ELAP-approved lab(s) to be used for analysis of samples	List the ELAP- certified lab(s) to be used for analysis of samples	1/8/2021
Appendix E, Laboratory Analysis and Containers	Drinking water samples collected using this protocol are intended to be analyzed for PFAS by ISO Method 25101.	Drinking water samples collected using this protocol are intended to be analyzed for PFAS by EPA Method 537, 537.1, 533, or ISO Method 25101	1/8/2021
Water Sample Results Page 9	<p>“In addition, further assessment of water may be warranted if either of the following screening levels are met:</p> <p>a. any other individual PFAS (not PFOA or PFOS) is detected in water at or above 100 ng/L; or</p> <p>b. total concentration of PFAS (including PFOA and PFOS) is detected in water at or above 500 ng/L”</p>	Deleted	6/15/2021

Citation and Page Number	Current Text	Corrected Text	Date
Routine Analysis, Page XX	Currently, New York State Department of Health’s Environmental Laboratory Approval Program (ELAP)... criteria set forth in the DER’s laboratory guidelines for PFAS in non-potable water and solids (Appendix H - Laboratory Guidelines for Analysis of PFAS in Non-Potable Water and Solids).	Deleted	5/31/2022
Analysis and Reporting, Page XX	As of October 2020, the United States Environmental Protection Agency (EPA) does not have a validated method for analysis of PFAS for media commonly analyzed under DER remedial programs (non-potable waters, solids). DER has developed the following guidelines to ensure consistency in analysis and reporting of PFAS.	Deleted	5/31/2022
Routine Analysis, Page XX	LC-MS/MS analysis for PFAS using methodologies based on EPA Method 537.1 is the procedure to use for environmental samples. Isotope dilution techniques should be utilized for the analysis of PFAS in all media.	EPA Method 1633 is the procedure to use for environmental samples.	
Soil Sample Results, Page XX	Soil cleanup objectives for PFOA and PFOS will be proposed in an upcoming revision to 6 NYCRR Part 375-6	Soil cleanup objectives for PFOA and PFOS have been proposed in an upcoming revision to 6 NYCRR Part 375-6	
Appendix A	“Include in the text... LC-MS/MS for PFAS using methodologies based on EPA Method 537.1”	“Include in the textEPA Method 1633”	
Appendix A	“Laboratory should have ELAP certification for PFOA and PFOS in drinking water by EPA Method 537, 537.1, EPA Method 533, or ISO 25101”	Deleted	
Appendix B	“Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1”	“Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633”	

Citation and Page Number	Current Text	Corrected Text	Date
Appendix C	“Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1”	“Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633”	
Appendix D	“Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1”	“Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633”	
Appendix G		Updated to include all forty PFAS analytes in EPA Method 533	
Appendix H		Deleted	
Appendix I	Appendix I	Appendix H	
Appendix H	“These guidelines are intended to be used for the validation of PFAS analytical results for projects within the Division of Environmental Remediation (DER) as well as aid in the preparation of a data usability summary report.”	“These guidelines are intended to be used for the validation of PFAS using EPA Method 1633 for projects within the Division of Environmental Remediation (DER).”	
Appendix H	“The holding time is 14 days...”	“The holding time is 28 days...”	
Appendix H, Initial Calibration	“The initial calibration should contain a minimum of five standards for linear fit...”	“The initial calibration should contain a minimum of six standards for linear fit...”	
Appendix H, Initial Calibration	Linear fit calibration curves should have an R ² value greater than 0.990.	Deleted	
Appendix H, Initial Calibration Verification	Initial Calibration Verification Section	Deleted	
Appendix H	secondary Ion Monitoring Section	Deleted	
Appendix H	Branched and Linear Isomers Section	Deleted	

Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs

Objective

New York State Department of Environmental Conservation's Division of Environmental Remediation (DER) performs or oversees sampling of environmental media and subsequent analysis of PFAS as part of remedial programs implemented under 6 NYCRR Part 375. To ensure consistency in sampling, analysis, reporting, and assessment of PFAS, DER has developed this document which summarizes currently accepted procedures and updates previous DER technical guidance pertaining to PFAS.

Applicability

All work plans submitted to DEC pursuant to one of the remedial programs under Part 375 shall include PFAS sampling and analysis procedures that conform to the guidelines provided herein.

As part of a site investigation or remedial action compliance program, whenever samples of potentially affected media are collected and analyzed for the standard Target Analyte List/Target Compound List (TAL/TCL), PFAS analysis should also be performed. Potentially affected media can include soil, groundwater, surface water, and sediment. Based upon the potential for biota to be affected, biota sampling and analysis for PFAS may also be warranted as determined pursuant to a Fish and Wildlife Impact Analysis. Soil vapor sampling for PFAS is not required.

Field Sampling Procedures

DER-10 specifies technical guidance applicable to DER's remedial programs. Given the prevalence and use of PFAS, DER has developed "best management practices" specific to sampling for PFAS. As specified in DER-10 Chapter 2, quality assurance procedures are to be submitted with investigation work plans. Typically, these procedures are incorporated into a work plan, or submitted as a stand-alone document (e.g., a Quality Assurance Project Plan). Quality assurance guidelines for PFAS are listed in Appendix A - Quality Assurance Project Plan (QAPP) Guidelines for PFAS.

Field sampling for PFAS performed under DER remedial programs should follow the appropriate procedures outlined for soils, sediments, or other solids (Appendix B), non-potable groundwater (Appendix C), surface water (Appendix D), public or private water supply wells (Appendix E), and fish tissue (Appendix F).

QA/QC samples (e.g. duplicates, MS/MSD) should be collected as specified in DER-10, Section 2.3(c). For sampling equipment coming in contact with aqueous samples only, rinsate or equipment blanks should be collected. Equipment blanks should be collected at a minimum frequency of one per day per site or one per twenty samples, whichever is more frequent.

Analysis and Reporting

The investigation work plan should describe analysis and reporting procedures, including laboratory analytical procedures for the methods discussed below. As specified in DER-10 Section 2.2, laboratories should provide a full Category B deliverable. In addition, a Data Usability Summary Report (DUSR) should be prepared by an independent, third-party data validator. Electronic data submissions should meet the requirements provided at: <https://www.dec.ny.gov/chemical/62440.html>.

DER has developed a *PFAS Analyte List* (Appendix G) for remedial programs to understand the nature of contamination at sites. It is expected that reported results for PFAS will include, at a minimum, all the compounds listed. If lab and/or matrix specific issues are encountered for any analytes, the DER project manager, in consultation with the DER chemist, will make case-by-case decisions as to whether certain analytes may be temporarily or permanently discontinued from analysis at each site. As with other contaminants that are analyzed for at a site, the *PFAS Analyte List* may be refined for future sampling events based on investigative findings.

Routine Analysis

EPA Method 1633 is the procedure to use for environmental samples. Reporting limits for PFOA and PFOS in aqueous samples should not exceed 2 ng/L. Reporting limits for PFOA and PFOS in solid samples should not exceed 0.5 µg/kg. Reporting limits for all other PFAS in aqueous and solid media should be as close to these limits as possible. If laboratories indicate that they are not able to achieve these reporting limits for the entire *PFAS Analyte List*, site-specific decisions regarding acceptance of elevated reporting limits for specific PFAS can be made by the DER project manager in consultation with the DER chemist. Data review guidelines were developed by DER to ensure data comparability and usability (Appendix H - Data Review Guidelines for Analysis of PFAS in Non-Potable Water and Solids).

Additional Analysis

Additional laboratory methods for analysis of PFAS may be warranted at a site, such as the Synthetic Precipitation Leaching Procedure (SPLP) and Total Oxidizable Precursor Assay (TOP Assay).

In cases where site-specific cleanup objectives for PFOA and PFOS are to be assessed, soil parameters, such as Total Organic Carbon (Lloyd Kahn), soil pH (EPA Method 9045), clay content (percent), and cation exchange capacity (EPA Method 9081), should be included in the analysis to help evaluate factors affecting the leachability of PFAS in site soils.

SPLP is a technique used to determine the mobility of chemicals in liquids, soils and wastes, and may be useful in determining the need for addressing PFAS-containing material as part of the remedy. SPLP by EPA Method 1312 should be used unless otherwise specified by the DER project manager in consultation with the DER chemist.

Impacted materials can be made up of PFAS that are not analyzable by routine analytical methodology. A TOP Assay can be utilized to conceptualize the amount and type of oxidizable PFAS which could be liberated in the environment, which approximates the maximum concentration of perfluoroalkyl substances that could be generated if all polyfluoroalkyl substances were oxidized. For example, some polyfluoroalkyl substances may degrade or transform to form perfluoroalkyl substances (such as PFOA or PFOS), resulting in an increase in perfluoroalkyl substance concentrations as contaminated groundwater moves away from a source. The TOP Assay converts, through oxidation, polyfluoroalkyl substances (precursors) into perfluoroalkyl substances that can be detected by routine analytical methodology.¹

¹ TOP Assay analysis of highly contaminated samples, such as those from an AFFF (aqueous film-forming foam) site, can result in incomplete oxidation of the samples and an underestimation of the total perfluoroalkyl substances.

Commercial laboratories have adopted methods which allow for the quantification of targeted PFAS in air and biota. The EPA’s Office of Research and Development (ORD) is currently developing methods which allow for air emissions characterization of PFAS, including both targeted and non-targeted analysis of PFAS. Consult with the DER project manager and the DER chemist for assistance on analyzing biota/tissue and air samples.

Data Assessment and Application to Site Cleanup

Until such time as Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Preliminary target levels for cleanup of PFOA and PFOS in other media, including biota and sediment, have not yet been established by the DEC.

Water Sample Results

NYSDEC has adopted ambient water quality guidance values for PFOA and PFOS. Groundwater samples should be compared to the human health criteria of 6.7 ng/l (ppt) for PFOA and 2.7 ng/l (ppt) for PFOS. These human health criteria should also be applied to surface water that is used as a water supply. This guidance also includes criteria for surface water for PFOS applicable for aquatic life, which may be applicable at some sites. Drinking water sample results should be compared to the NYS maximum contaminant level (MCL) of 10 ng/l (ppt). Analysis to determine if PFOA and PFOS concentrations are attributable to the site should include a comparison between upgradient and downgradient levels, and the presence of soil source areas, as defined below.

If PFOA and/or PFOS are identified as contaminants of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10.

Soil Sample Results

NYSDEC will delay adding soil cleanup objectives for PFOA and PFOS to 6 NYCRR Part 375-6 until the PFAS rural soil background study has been completed. Until SCOs are in effect, the following are to be used as guidance values:

Guidance Values for Anticipated Site Use	PFOA (ppb)	PFOS (ppb)
Unrestricted	0.66	0.88
Residential	6.6	8.8
Restricted Residential	33	44
Commercial	500	440
Industrial	600	440
Protection of Groundwater ²	0.8	1.0

PFOA and PFOS results for soil are to be compared against the guidance values listed above. These guidance values are to be used in determining whether PFOA and PFOS are contaminants of concern for the site and for determining remedial action objectives and cleanup requirements. Site-specific remedial objectives for protection of groundwater can also be presented for evaluation by DEC. Development of site-specific remedial objectives for protection of groundwater will require analysis of additional soil parameters relating to leachability. These

² The Protection of Groundwater values are based on the above referenced ambient groundwater guidance values. Details on that calculation are available in the following document, prepared for the February 2022 proposed changes to Part 375 (https://www.dec.ny.gov/docs/remediation_hudson_pdf/part375techsupport.pdf). The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).

additional analyses can include any or all the parameters listed above (soil pH, cation exchange capacity, etc.) and/or use of SPLP.

As the understanding of PFAS transport improves, DEC welcomes proposals for site-specific remedial objectives for protection of groundwater. DEC will expect that those may be dependent on additional factors including soil pH, aqueous pH, % organic carbon, % Sand/Silt/Clay, soil cations: K, Ca, Mg, Na, Fe, Al, cation exchange capacity, and anion exchange capacity. Site-specific remedial objectives should also consider the dilution attenuation factor (DAF). The NJDEP publication on DAF can be used as a reference:
<https://www.nj.gov/dep/srp/guidance/rs/daf.pdf>.

Testing for Imported Soil

Testing for PFAS should be included any time a full TAL/TCL analyte list is required. Results for PFOA and PFOS should be compared to the applicable guidance values. If PFOA or PFOS is detected in any sample at or above the guidance values then the source of backfill should be rejected, unless a site-specific exemption is provided by DER based on SPLP testing, for example. If the concentrations of PFOA and PFOS in leachate are at or above the ambient water quality guidance values for groundwater, then the soil is not acceptable.

PFOA, PFOS and 1,4-dioxane are all considered semi-volatile compounds, so composite samples are appropriate for these compounds when sampling in accordance with DER-10, Table 5.4(e)10. Category B deliverables should be submitted for backfill samples, though a DUSR is not required.

Appendix A - Quality Assurance Project Plan (QAPP) Guidelines for PFAS

The following guidelines (general and PFAS-specific) can be used to assist with the development of a QAPP for projects within DER involving sampling and analysis of PFAS.

General Guidelines in Accordance with DER-10

- Document/work plan section title – Quality Assurance Project Plan
- Summarize project scope, goals, and objectives
- Provide project organization including names and resumes of the project manager, Quality Assurance Officer (QAO), field staff, and Data Validator
 - The QAO should not have another position on the project, such as project or task manager, that involves project productivity or profitability as a job performance criterion
- List the ELAP certified lab(s) to be used for analysis of samples
- Include a site map showing sample locations
- Provide detailed sampling procedures for each matrix
- Include Data Quality Usability Objectives
- List equipment decontamination procedures
- Include an “Analytical Methods/Quality Assurance Summary Table” specifying:
 - Matrix type
 - Number or frequency of samples to be collected per matrix
 - Number of field and trip blanks per matrix
 - Analytical parameters to be measured per matrix
 - Analytical methods to be used per matrix with minimum reporting limits
 - Number and type of matrix spike and matrix spike duplicate samples to be collected
 - Number and type of duplicate samples to be collected
 - Sample preservation to be used per analytical method and sample matrix
 - Sample container volume and type to be used per analytical method and sample matrix
 - Sample holding time to be used per analytical method and sample matrix
- Specify Category B laboratory data deliverables and preparation of a DUSR

Specific Guidelines for PFAS

- Include in the text that sampling for PFAS will take place
- Include in the text that PFAS will be analyzed by EPA Method 1633
- Include the list of PFAS compounds to be analyzed (*PFAS Analyte List*)
- Include the laboratory SOP for PFAS analysis
- List the minimum method-achievable Reporting Limits for PFAS
 - Reporting Limits should be less than or equal to:
 - Aqueous – 2 ng/L (ppt)
 - Solids – 0.5 µg/kg (ppb)
- Include the laboratory Method Detection Limits for the PFAS compounds to be analyzed
-
- Include detailed sampling procedures
 - Precautions to be taken
 - Pump and equipment types
 - Decontamination procedures
 - Approved materials only to be used
- Specify that regular ice only will be used for sample shipment
- Specify that equipment blanks should be collected at a minimum frequency of 1 per day per site for each matrix

Appendix B - Sampling Protocols for PFAS in Soils, Sediments and Solids

General

The objective of this protocol is to give general guidelines for the collection of soil, sediment and other solid samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Containers

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

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in to contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel spoon
- stainless steel bowl
- steel hand auger or shovel without any coatings

Equipment Decontamination

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

Sampling Techniques

Sampling is often conducted in areas where a vegetative turf has been established. In these cases, a pre-cleaned trowel or shovel should be used to carefully remove the turf so that it may be replaced at the conclusion of sampling. Surface soil samples (e.g. 0 to 6 inches below surface) should then be collected using a pre-cleaned, stainless steel spoon. Shallow subsurface soil samples (e.g. 6 to ~36 inches below surface) may be collected by digging a hole using a pre-cleaned hand auger or shovel. When the desired subsurface depth is reached, a pre-cleaned hand auger or spoon shall be used to obtain the sample.

When the sample is obtained, it should be deposited into a stainless steel bowl for mixing prior to filling the sample containers. The soil should be placed directly into the bowl and mixed thoroughly by rolling the material into the middle until the material is homogenized. At this point the material within the bowl can be placed into the laboratory provided container.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at $4 \pm 2^\circ$ Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A soil log or sample log shall document the location of the sample/borehole, depth of the sample, sampling equipment, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

Appendix C - Sampling Protocols for PFAS in Monitoring Wells

General

The objective of this protocol is to give general guidelines for the collection of groundwater samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container

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

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including plumbers tape and sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel inertia pump with HDPE tubing
- peristaltic pump equipped with HDPE tubing and silicone tubing
- stainless steel bailer with stainless steel ball
- bladder pump (identified as PFAS-free) with HDPE tubing

Equipment Decontamination

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

Sampling Techniques

Monitoring wells should be purged in accordance with the sampling procedure (standard/volume purge or low flow purge) identified in the site work plan, which will determine the appropriate time to collect the sample. If sampling using standard purge techniques, additional purging may be needed to reduce turbidity levels, so samples contain a limited amount of sediment within the sample containers. Sample containers that contain sediment may cause issues at the laboratory, which may result in elevated reporting limits and other issues during the sample preparation that can compromise data usability. Sampling personnel should don new nitrile gloves prior to sample collection due to the potential to contact PFAS containing items (not related to the sampling equipment) during the purging activities.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at $4 \pm 2^\circ$ Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Collect one equipment blank per day per site and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Additional equipment blank samples may be collected to assess other equipment that is utilized at the monitoring well
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A purge log shall document the location of the sample, sampling equipment, groundwater parameters, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

Appendix D - Sampling Protocols for PFAS in Surface Water

General

The objective of this protocol is to give general guidelines for the collection of surface water samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container

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

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel cup

Equipment Decontamination

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

Sampling Techniques

Where conditions permit, (e.g. creek or pond) sampling devices (e.g. stainless steel cup) should be rinsed with site medium to be sampled prior to collection of the sample. At this point the sample can be collected and poured into the sample container.

If site conditions permit, samples can be collected directly into the laboratory container.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at $4 \pm 2^\circ$ Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Collect one equipment blank per day per site and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A sample log shall document the location of the sample, sampling equipment, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

Appendix E - Sampling Protocols for PFAS in Private Water Supply Wells

General

The objective of this protocol is to give general guidelines for the collection of water samples from private water supply wells (with a functioning pump) for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container

Drinking water samples collected using this protocol are intended to be analyzed for PFAS by EPA Method 537, 537.1, 533, or ISO Method 25101. The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials (e.g. plumbers tape), including sample bottle cap liners with a PTFE layer.

Equipment Decontamination

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

Sampling Techniques

Locate and assess the pressure tank and determine if any filter units are present within the building. Establish the sample location as close to the well pump as possible, which is typically the spigot at the pressure tank. Ensure sampling equipment is kept clean during sampling as access to the pressure tank spigot, which is likely located close to the ground, may be obstructed and may hinder sample collection.

Prior to sampling, a faucet downstream of the pressure tank (e.g., washroom sink) should be run until the well pump comes on and a decrease in water temperature is noted which indicates that the water is coming from the well. If the homeowner is amenable, staff should run the water longer to purge the well (15+ minutes) to provide a sample representative of the water in the formation rather than standing water in the well and piping system including the pressure tank. At this point a new pair of nitrile gloves should be donned and the sample can be collected from the sample point at the pressure tank.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at $4 \pm 2^\circ$ Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- If equipment was used, collect one equipment blank per day per site and a minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers.
- A field reagent blank (FRB) should be collected at a rate of one per 20 samples. The lab will provide a FRB bottle containing PFAS free water and one empty FRB bottle. In the field, pour the water from the one bottle into the empty FRB bottle and label appropriately.
- Request appropriate data deliverable (Category B) and an electronic data deliverable
- For sampling events where multiple private wells (homes or sites) are to be sampled per day, it is acceptable to collect QC samples at a rate of one per 20 across multiple sites or days.

Documentation

A sample log shall document the location of the private well, sample point location, owner contact information, sampling equipment, purge duration, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate and available (e.g. well construction, pump type and location, yield, installation date). Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appendix F - Sampling Protocols for PFAS in Fish

This appendix contains a copy of the current SOP developed by the Division of Fish and Wildlife (DFW) entitled “General Fish Handling Procedures for Contaminant Analysis” (Ver. 8). This SOP should be followed when collecting fish for contaminant analysis. Note, however, that the Bureau of Ecosystem Health will not be supplying bags or tags. All supplies are the responsibility of the collector

Procedure Name: General Fish Handling Procedures for Contaminant Analysis

Number: FW-005

Purpose: This procedure describes data collection, fish processing and delivery of fish collected for contaminant monitoring. It contains the chain of custody and collection record forms that should be used for the collections.

Organization: Environmental Monitoring Section
Bureau of Ecosystem Health
Division of Fish and Wildlife (DFW)
New York State Department of Environmental Conservation (NYSDEC)
625 Broadway
Albany, New York 12233-4756

Version: 8

Previous Version Date: 21 March 2018

Summary of Changes to this Version: Updated bureau name to Bureau of Ecosystem Health. Added direction to list the names of all field crew on the collection record. Minor formatting changes on chain of custody and collection records.

Originator or Revised by: Wayne Richter, Jesse Becker

Date: 26 April 2019

Quality Assurance Officer and Approval Date: Jesse Becker, 26 April 2019

**NEW YORK STATE
DEPARTMENT OF ENVIRONMENTAL CONSERVATION**

GENERAL FISH HANDLING PROCEDURES FOR CONTAMINANT ANALYSES

- A. Original copies of all continuity of evidence (i.e., Chain of Custody) and collection record forms must accompany delivery of fish to the lab. A copy shall be directed to the Project Leader or as appropriate, Wayne Richter. All necessary forms will be supplied by the Bureau of Ecosystem Health. Because some samples may be used in legal cases, it is critical that each section is filled out completely. Each Chain of Custody form has three main sections:
1. The top box is to be filled out **and signed** by the person responsible for the fish collection (e.g., crew leader, field biologist, researcher). This person is responsible for delivery of the samples to DEC facilities or personnel (e.g., regional office or biologist).
 2. The second section is to be filled out **and signed** by the person responsible for the collections while being stored at DEC, before delivery to the analytical lab. This may be the same person as in (1), but it is still required that they complete the section. Also important is the **range of identification numbers** (i.e., tag numbers) included in the sample batch.
 3. Finally, the bottom box is to record any transfers between DEC personnel and facilities. Each subsequent transfer should be **identified, signed, and dated**, until laboratory personnel take possession of the fish.
- B. The following data are required on each **Fish Collection Record** form:
1. Project and Site Name.
 2. DEC Region.
 3. All personnel (and affiliation) involved in the collection.
 4. Method of collection (gill net, hook and line, etc.)
 5. Preservation Method.
- C. The following data are to be taken on each fish collected and recorded on the **Fish Collection Record** form:
1. Tag number - Each specimen is to be individually jaw tagged at time of collection with a unique number. Make sure the tag is turned out so that the number can be read without opening the bag. Use tags in sequential order. For small fish or composite samples place the tag inside the bag with the samples. The Bureau of Ecosystem Health can supply the tags.
 2. Species identification (please be explicit enough to enable assigning genus and species). Group fish by species when processing.
 3. Date collected.
 4. Sample location (waterway and nearest prominent identifiable landmark).
 5. Total length (nearest mm or smallest sub-unit on measuring instrument) and weight (nearest g or

smallest sub-unit of weight on weighing instrument). Take all measures as soon as possible with calibrated, protected instruments (e.g. from wind and upsets) and prior to freezing.

6. Sex - fish may be cut enough to allow sexing or other internal investigation, but do not eviscerate. Make any incision on the right side of the belly flap or exactly down the midline so that a left-side fillet can be removed.

D. General data collection recommendations:

1. It is helpful to use an ID or tag number that will be unique. It is best to use metal striped bass or other uniquely numbered metal tags. If uniquely numbered tags are unavailable, values based on the region, water body and year are likely to be unique: for example, R7CAY11001 for Region 7, Cayuga Lake, 2011, fish 1. If the fish are just numbered 1 through 20, we have to give them new numbers for our database, making it more difficult to trace your fish to their analytical results and creating an additional possibility for errors.
 2. Process and record fish of the same species sequentially. Recording mistakes are less likely when all fish from a species are processed together. Starting with the bigger fish species helps avoid missing an individual.
 3. If using Bureau of Ecosystem Health supplied tags or other numbered tags, use tags in sequence so that fish are recorded with sequential Tag Numbers. This makes data entry and login at the lab and use of the data in the future easier and reduces keypunch errors.
 4. Record length and weight as soon as possible after collection and before freezing. Other data are recorded in the field upon collection. An age determination of each fish is optional, but if done, it is recorded in the appropriate "Age" column.
 5. For composite samples of small fish, record the number of fish in the composite in the Remarks column. Record the length and weight of each individual in a composite. All fish in a composite sample should be of the same species and members of a composite should be visually matched for size.
 6. Please submit photocopies of topographic maps or good quality navigation charts indicating sampling locations. GPS coordinates can be entered in the Location column of the collection record form in addition to or instead for providing a map. These records are of immense help to us (and hopefully you) in providing documented location records which are not dependent on memory and/or the same collection crew. In addition, they may be helpful for contaminant source trackdown and remediation/control efforts of the Department.
 7. When recording data on fish measurements, it will help to ensure correct data recording for the data recorder to call back the numbers to the person making the measurements.
- E. Each fish is to be placed in its own individual plastic bag. For small fish to be analyzed as a composite, put all of the fish for one composite in the same bag but use a separate bag for each composite. It is important to individually bag the fish to avoid difficulties or cross contamination when processing the fish for chemical analysis. Be sure to include the fish's tag number inside the bag, preferably attached to the fish with the tag number turned out so it can be read. Tie or otherwise secure the bag closed. **The Bureau of Ecosystem Health will supply the bags.** If necessary, food grade bags may be procured from a suitable vendor (e.g., grocery store). It is preferable to redundantly label each bag with a manila tag tied between the knot and the body of the bag. This tag should be labeled with the project name, collection location, tag number, collection date, and fish species. If scales are collected, the scale envelope should be labeled with

the same information.

- F. Groups of fish, by species, are to be placed in one large plastic bag per sampling location. **The Bureau of Ecosystem Health will supply the larger bags.** Tie or otherwise secure the bag closed. Label the site bag with a manila tag tied between the knot and the body of the bag. The tag should contain: project, collection location, collection date, species and **tag number ranges**. Having this information on the manila tag enables lab staff to know what is in the bag without opening it.
- G. Do not eviscerate, fillet or otherwise dissect the fish unless specifically asked to. If evisceration or dissection is specified, the fish must be cut along the exact midline or on the right side so that the left side fillet can be removed intact at the laboratory. If filleting is specified, the procedure for taking a standard fillet (SOP PREPLAB 4) must be followed, including removing scales.
- H. Special procedures for PFAS: Unlike legacy contaminants such as PCBs, which are rarely found in day to day life, PFAS are widely used and frequently encountered. Practices that avoid sample contamination are therefore necessary. While no standard practices have been established for fish, procedures for water quality sampling can provide guidance. The following practices should be used for collections when fish are to be analyzed for PFAS:
- No materials containing Teflon.
 - No Post-it notes.
 - No ice packs; only water ice or dry ice.
 - Any gloves worn must be powder free nitrile.
 - No Gore-Tex or similar materials (Gore-Tex is a PFC with PFOA used in its manufacture).
 - No stain repellent or waterproof treated clothing; these are likely to contain PFCs.
 - Avoid plastic materials, other than HDPE, including clipboards and waterproof notebooks.
 - Wash hands after handling any food containers or packages as these may contain PFCs.
 - Keep pre-wrapped food containers and wrappers isolated from fish handling.
 - Wear clothing washed at least six times since purchase.
 - Wear clothing washed without fabric softener.
 - Staff should avoid cosmetics, moisturizers, hand creams and similar products on the day of sampling as many of these products contain PFCs (Fujii et al. 2013). Sunscreen or insect repellent should not contain ingredients with “fluor” in their name. Apply any sunscreen or insect repellent well downwind from all materials. Hands must be washed after touching any of these products.
- I. All fish must be kept at a temperature $<45^{\circ}\text{F}$ ($<8^{\circ}\text{C}$) immediately following data processing. As soon as possible, freeze at $-20^{\circ}\text{C} \pm 5^{\circ}\text{C}$. Due to occasional freezer failures, daily freezer temperature logs are required. The freezer should be locked or otherwise secured to maintain chain of custody.
- J. In most cases, samples should be delivered to the Analytical Services Unit at the Hale Creek field station. Coordinate delivery with field station staff and send copies of the collection records, continuity of evidence forms and freezer temperature logs to the field station. For samples to be analyzed elsewhere, non-routine collections or other questions, contact Wayne Richter, Bureau of Ecosystem Health, NYSDEC, 625 Broadway, Albany, New York 12233-4756, 518-402-8974, or the project leader about sample transfer. Samples will then be directed to the analytical facility and personnel noted on specific project descriptions.
- K. A recommended equipment list is at the end of this document.

**NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
DIVISION OF FISH AND WILDLIFE
FISH COLLECTION RECORD**

page _____ of _____

Project and Site Name _____ DEC Region _____

Collections made by (include all crew) _____

Sampling Method: Electrofishing Gill netting Trap netting Trawling Seining Angling Other _____

Preservation Method: Freezing Other _____ Notes (SWFDB survey number): _____

FOR LAB USE ONLY- LAB ENTRY NO.	COLLECTION OR TAG NO.	SPECIES	DATE TAKEN	LOCATION	AGE	SEX &/OR REPROD. CONDIT	LENGTH ()	WEIGHT ()	REMARKS

richter: revised 2011, 5/7/15, 10/4/16, 3/20/17; becker: 3/23/17, 4/26/19

**NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
CHAIN OF CUSTODY**

I, _____, of _____ collected the
(Print Name) (Print Business Address)

following on _____, 20____ from _____
(Date) (Water Body)

in the vicinity of _____
(Landmark, Village, Road, etc.)

Town of _____, in _____ County.

Item(s) _____

Said sample(s) were in my possession and handled according to standard procedures provided to me prior to collection. The sample(s) were placed in the custody of a representative of the New York State Department of Environmental Conservation on _____, 20____.

Signature Date

I, _____, received the above mentioned sample(s) on the date specified and assigned identification number(s) _____ to the sample(s). I have recorded pertinent data for the sample(s) on the attached collection records. The sample(s) remained in my custody until subsequently transferred, prepared or shipped at times and on dates as attested to below.

Signature Date

SECOND RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
THIRD RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
FOURTH RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
RECEIVED IN LABORATORY BY (Print Name)	TIME & DATE	REMARKS
SIGNATURE	UNIT	
LOGGED IN BY (Print Name)	TIME & DATE	ACCESSION NUMBERS
SIGNATURE	UNIT	

NOTICE OF WARRANTY

By signature to the chain of custody (reverse), the signatory warrants that the information provided is truthful and accurate to the best of his/her ability. The signatory affirms that he/she is willing to testify to those facts provided and the circumstances surrounding the same. Nothing in this warranty or chain of custody negates responsibility nor liability of the signatories for the truthfulness and accuracy of the statements provided.

HANDLING INSTRUCTIONS

On day of collection, collector(s) name(s), address(es), date, geographic location of capture (attach a copy of topographic map or navigation chart), species, number kept of each species, and description of capture vicinity (proper noun, if possible) along with name of Town and County must be indicated on reverse.

Retain organisms in manila tagged plastic bags to avoid mixing capture locations. Note appropriate information on each bag tag.

Keep samples as cool as possible. Put on ice if fish cannot be frozen within 12 hours. If fish are held more than 24 hours without freezing, they will not be retained or analyzed.

Initial recipient (either DEC or designated agent) of samples from collector(s) is responsible for obtaining and recording information on the collection record forms which will accompany the chain of custody. This person will seal the container using packing tape and writing his signature, the time and the date across the tape onto the container with indelible marker. Any time a seal is broken, for whatever purpose, the incident must be recorded on the Chain of Custody (reason, time, and date) in the purpose of transfer block. Container then is resealed using new tape and rewriting signature, with time and date.

EQUIPMENT LIST

Scale or balance of appropriate capacity for the fish to be collected.

Fish measuring board.

Plastic bags of an appropriate size for the fish to be collected and for site bags.

Individually numbered metal tags for fish.

Manila tags to label bags.

Small envelopes, approximately 2" x 3.5", if fish scales are to be collected.

Knife for removing scales.

Chain of custody and fish collection forms.

Clipboard.

Pens or markers.

Paper towels.

Dish soap and brush.

Bucket.

Cooler.

Ice.

Duct tape.

Appendix G – PFAS Analyte List

Group	Chemical Name	Abbreviation	CAS Number
Perfluoroalkyl sulfonic acids	Perfluorobutanesulfonic acid	PFBS	375-73-5
	Perfluoropentanesulfonic 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
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	PFTTrDA	72629-94-8
	Perfluorotetradecanoic acid	PFTeDA	376-06-7
Per- and Polyfluoroether carboxylic acids	Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
	4,8-Dioxa-3H-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
Fluorotelomer sulfonic acids	4:2 Fluorotelomer sulfonic acid	4:2-FTS	757124-72-4
	6:2 Fluorotelomer sulfonic acid	6:2-FTS	27619-97-2
	8:2 Fluorotelomer sulfonic acid	8:2-FTS	39108-34-4
Fluorotelomer carboxylic acids	3:3 Fluorotelomer carboxylic acid	3:3 FTCA	356-02-5
	5:3 Fluorotelomer carboxylic acid	5:3 FTCA	914637-49-3
	7:3 Fluorotelomer carboxylic acid	7:3 FTCA	812-70-4
Perfluorooctane sulfonamides	Perfluorooctane sulfonamide	PFOSA	754-91-6
	N-methylperfluorooctane sulfonamide	NMeFOSA	31506-32-8
	N-ethylperfluorooctane sulfonamide	NEtFOSA	4151-50-2
Perfluorooctane sulfonamidoacetic acids	N-methylperfluorooctane sulfonamidoacetic acid	N-MeFOSAA	2355-31-9
	N-ethylperfluorooctane sulfonamidoacetic acid	N-EtFOSAA	2991-50-6
Perfluorooctane sulfonamide ethanols	N-methylperfluorooctane sulfonamidoethanol	MeFOSE	24448-09-7
	N-ethylperfluorooctane sulfonamidoethanol	EtFOSE	1691-99-2

Group	Chemical Name	Abbreviation	CAS Number
Ether sulfonic acids	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (F-53B Major)	9Cl-PF3ONS	756426-58-1
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor)	11Cl-PF3OUdS	763051-92-9
	Perfluoro(2-ethoxyethane) sulfonic acid	PFEESA	113507-82-7

Appendix H - Data Review Guidelines for Analysis of PFAS in Non-Potable Water and Solids

General

These guidelines are intended to be used for the validation of PFAS using EPA Method 1633 for projects within the Division of Environmental Remediation (DER). Data reviewers should understand the methodology and techniques utilized in the analysis. Consultation with the end user of the data may be necessary to assist in determining data usability based on the data quality objectives in the Quality Assurance Project Plan. A familiarity with the laboratory’s Standard Operating Procedure may also be needed to fully evaluate the data. If you have any questions, please contact DER’s Quality Assurance Officer, Dana Barbarossa, at dana.barbarossa@dec.ny.gov.

Preservation and Holding Time

Samples should be preserved with ice to a temperature of less than 6°C upon arrival at the lab. The holding time is 28 days to extraction for aqueous and solid samples. The time from extraction to analysis for aqueous samples is 28 days and 40 days for solids.

Temperature greatly exceeds 6°C upon arrival at the lab*	Use professional judgement to qualify detects and non-detects as estimated or rejected
Holding time exceeding 28 days to extraction	Use professional judgement to qualify detects and non-detects as estimated or rejected if holding time is grossly exceeded

*Samples that are delivered to the lab immediately after sampling may not meet the thermal preservation guidelines. Samples are considered acceptable if they arrive on ice or an attempt to chill the samples is observed.

Initial Calibration

The initial calibration should contain a minimum of six standards for linear fit and six standards for a quadratic fit. The relative standard deviation (RSD) for a quadratic fit calibration should be less than 20%.

The low-level calibration standard should be within 50% - 150% of the true value, and the mid-level calibration standard within 70% - 130% of the true value.

%RSD >20%	J flag detects and UJ non detects
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Continuing Calibration Verification

Continuing calibration verification (CCV) checks should be analyzed at a frequency of one per ten field samples. If CCV recovery is very low, where detection of the analyte could be in question, ensure a low level CCV was analyzed and use to determine data quality.

CCV recovery <70 or >130%	J flag results
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Blanks

There should be no detections in the method blanks above the reporting limits. Equipment blanks, field blanks, rinse blanks etc. should be evaluated in the same manner as method blanks. Use the most contaminated blank to evaluate the sample results.

Blank Result	Sample Result	Qualification
Any detection	<Reporting limit	Qualify as ND at reporting limit
Any detection	>Reporting Limit and >10x the blank result	No qualification
>Reporting limit	>Reporting limit and <10x blank result	J+ biased high

Field Duplicates

A blind field duplicate should be collected at rate of one per twenty samples. The relative percent difference (RPD) should be less than 30% for analyte concentrations greater than two times the reporting limit. Use the higher result for final reporting.

RPD >30%	Apply J qualifier to parent sample
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Lab Control Spike

Lab control spikes should be analyzed with each extraction batch or one for every twenty samples. In the absence of lab derived criteria, use 70% - 130% recovery criteria to evaluate the data.

Recovery <70% or >130% (lab derived criteria can also be used)	Apply J qualifier to detects and UJ qualifier to non detects
--	--

Matrix Spike/Matrix Spike Duplicate

One matrix spike and matrix spike duplicate should be collected at a rate of one per twenty samples. Use professional judgement to reject results based on out of control MS/MSD recoveries.

Recovery <70% or >130% (lab derived criteria can also be used)	Apply J qualifier to detects and UJ qualifier to non detects of parent sample only
RPD >30%	Apply J qualifier to detects and UJ qualifier to non detects of parent sample only

Extracted Internal Standards (Isotope Dilution Analytes)

Problematic analytes (e.g. PFBA, PFPeA, fluorotelomer sulfonates) can have wider recoveries without qualification. Qualify corresponding native compounds with a J flag if outside of the range.

Recovery <50% or >150%	Apply J qualifier
Recovery <25% or >150% for poor responding analytes	Apply J qualifier
Isotope Dilution Analyte (IDA) Recovery <10%	Reject results

Signal to Noise Ratio

The signal to noise ratio for the quantifier ion should be at least 3:1. If the ratio is less than 3:1, the peak is discernable from the baseline noise and symmetrical, the result can be reported. If the peak appears to be baseline noise and/or the shape is irregular, qualify the result as tentatively identified.

Reporting Limits

If project-specific reporting limits were not met, please indicate that in the report along with the reason (e.g. over dilution, dilution for non-target analytes, high sediment in aqueous samples).

Peak Integrations

Target analyte peaks should be integrated properly and consistently when compared to standards. Ensure branched isomer peaks are included for PFAS where standards are available. Inconsistencies should be brought to the attention of the laboratory or identified in the data review summary report.

EGLE PFAS SAMPLING QUICK REFERENCE FIELD GUIDE¹

All Items Used During Sampling Event

● Prohibited
<ul style="list-style-type: none"> Items or materials that contain fluoropolymers such as <ul style="list-style-type: none"> Polytetrafluoroethylene (PTFE), that includes the trademarks Teflon® and Hostaflon® Polyvinylidene fluoride (PVDF), that includes the trademark Kynar® Polychlorotrifluoroethylene (PCTFE), that includes the trademark Neoflon® Ethylene-tetrafluoro-ethylene (ETFE), that includes the trademark Tefzel® Fluorinated ethylene propylene (FEP), that includes the trademarks Teflon® FEP and Hostaflon® FEP Items or materials that contain any other fluoropolymer

Pumps, Tubing, and Sampling Equipment

● Prohibited	■ Allowable	▲ Needs Screening ²
<ul style="list-style-type: none"> Items or materials containing any fluoropolymer (potential items include tubing, valves, or pipe thread seal tape) 	<ul style="list-style-type: none"> High-density polyethylene (HDPE) Low-density polyethylene (LDPE) tubing Polypropylene Silicone Stainless-steel Any items used to secure sampling bottles made from: <ul style="list-style-type: none"> Natural rubber Nylon (cable ties) Uncoated metal springs Polyethylene 	<ul style="list-style-type: none"> Any items or materials that will come into direct contact with the sample that have not been verified to be PFAS-free <ul style="list-style-type: none"> Do not assume that any sampling items or materials are PFAS-free based on composition alone

Sample Storage and Preservation

● Prohibited	■ Allowable	▲ Needs Screening ²
<ul style="list-style-type: none"> Polytetrafluoroethylene (PTFE): Teflon® lined bottles or caps 	<ul style="list-style-type: none"> Glass jars⁴ Laboratory-provided PFAS-Free bottles: <ul style="list-style-type: none"> HDPE or polypropylene Regular wet ice Thin HDPE sheeting LDPE resealable storage bags (i.e. Ziploc®) that will not contact the sample media⁶ 	<ul style="list-style-type: none"> Aluminium foil⁴ Chemical or blue ice⁵ Plastic storage bags other than those listed as ■ Allowable Low-density polyethylene (LDPE) bottles

Field Documentation

● Prohibited	■ Allowable	▲ Needs Screening ²
<ul style="list-style-type: none"> Clipboards coated with PFAS Notebooks made with PFAS treated paper PFAS treated loose paper PFAS treated adhesive paper products 	<ul style="list-style-type: none"> Loose paper (non-waterproof, non-recycled) Rite in the Rain® notebooks Aluminium, polypropylene, or Masonite field clipboards Ballpoint pens, pencils, and Fine or Ultra-Fine Point Sharpie® markers 	<ul style="list-style-type: none"> Plastic clipboards, binders, or spiral hard cover notebooks All markers not listed as ■ Allowable Post-It® Notes or other adhesive paper products Waterproof field books

Decontamination

● Prohibited	■ Allowable	▲ Needs Screening ²
<ul style="list-style-type: none"> Decon 90® PFAS treated paper towel 	<ul style="list-style-type: none"> Alconox®, Liquinox®, or Citranox® Triple rinse with PFAS-free deionized water Cotton cloth or untreated paper towel 	<ul style="list-style-type: none"> Municipal water Recycled paper towels or chemically treated paper towels

Clothing, Boots, Rain Gear, and PPE

● Prohibited	■ Allowable	▲ Needs Screening ²
<ul style="list-style-type: none"> • New or unwashed clothing • Anything made of or with: <ul style="list-style-type: none"> ○ Gore-Tex™ or other water-resistant synthetics • Anything applied with or recently washed with: <ul style="list-style-type: none"> ○ Fabric softeners ○ Fabric protectors, including UV protection ○ Insect resistant chemicals ○ Water, dirt, and/or stain resistant chemicals 	<ul style="list-style-type: none"> • Powderless nitrile gloves • Well-laundered synthetic or 100% cotton clothing, with most recent launderings not using fabric softeners • Made of or with: <ul style="list-style-type: none"> ○ Polyurethane ○ Polyvinyl chloride (PVC) ○ Wax coated fabrics ○ Rubber / Neoprene ○ Uncoated Tyvek® 	<ul style="list-style-type: none"> • Latex gloves • Water and/or dirt resistant leather gloves • Any special gloves required by a HASP • Tyvek® suits, clothing that contains Tyvek®, or coated Tyvek®

Food and Beverages

● Prohibited	■ Allowable
<ul style="list-style-type: none"> • No food should be consumed in the staging or sampling areas, including pre-packaged food or snacks. <ul style="list-style-type: none"> ■ If consuming food on-site becomes necessary, move to the staging area and remove PPE. After eating, wash hands thoroughly and put on new PPE. 	<ul style="list-style-type: none"> • Brought and consumed only outside the vicinity of the sampling area: <ul style="list-style-type: none"> ○ Bottled water ○ Hydration drinks (i.e. Gatorade®, Powerade®)

Personal Care Products (PCPs) - for day of sample collection⁶

● Prohibited	■ Allowable	▲ Needs Screening ²
<ul style="list-style-type: none"> • Any PCPs⁶, sunscreen, and insect repellent applied in the sampling area. 	<p>PCPs⁶, sunscreens, and insect repellents applied in the staging area, away from sampling bottles and equipment followed by thoroughly washing hands:</p> <p>PCPs⁶:</p> <ul style="list-style-type: none"> • Cosmetics, deodorants/antiperspirants, moisturizers, hand creams, and other PCPs⁶ <p>Sunscreens:</p> <ul style="list-style-type: none"> • Banana Boat® for Men Triple Defense Continuous Spray Sunscreen SPF 30 • Banana Boat® Sport Performance Coolzone Broad Spectrum SPF 30 • Banana Boat® Sport Performance Sunscreen Lotion Broad Spectrum SPF 30 • Banana Boat® Sport Performance Sunscreen Stick SPF 50 • Coppertone® Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50 • Coppertone® Sport High Performance AccuSpray Sunscreen SPF 30 • Coppertone® Sunscreen Stick Kids SPF 55 • L'Oréal® Silky Sheer Face Lotion 50 • Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 50 • Meijer® Sunscreen Continuous Spray Broad Spectrum SPF 30 • Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50 • Meijer® Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70 • Neutrogena® Beach Defense Water+Sun Barrier Lotion SPF 70 • Neutrogena® Beach Defense Water+Sun Barrier Spray Broad Spectrum SPF 30 • Neutrogena® Pure & Free Baby Sunscreen Broad Spectrum SPF 60+ • Neutrogena® UltraSheer Dry-Touch Sunscreen Broad Spectrum SPF 30 <p>Insect Repellents:</p> <ul style="list-style-type: none"> • OFF® Deep Woods • Sawyer® Permethrin 	<ul style="list-style-type: none"> • Products other than those listed as <ul style="list-style-type: none"> ■ Allowable

¹ This table is not considered to be a complete listing of prohibited or allowable materials. All materials should be evaluated prior to use during sampling. The manufacturers of various products should be contacted in order to determine if PFAS was used in the production of any particular product.

² Equipment blank samples should be taken to verify these products are PFAS-free prior to use during sampling.

³ **For surface water foam samples:** LDPE storage bags may be used in the sampling of foam on surface waters. In this instance, it is allowable for the LDPE bag to come into direct contact with the sample media.

⁴ **For fish and other wildlife samples:** Depending on the project objectives, glass jars and aluminum foil might be used for PFAS sampling. PFAS has been found to bind to glass and if the sample is stored in a glass jar, a rinse of the jar is required during the sample analysis. PFAS are sometimes used as a protective layer for some aluminum foils. An equipment blank sample should be collected prior to any aluminum foil use.

⁵ Regular ice is recommended as there are concerns that chemical and blue ice may not cool and maintain the sample at or below 42.8°F (6°C) (as determined by EPA 40 CFR 136 – NPDES) during collection and through transit to the laboratory.

⁶ Based on evidence, avoidance of PCPs is considered to be precautionary because none have been documented as having cross-contaminated samples due to their use. However, if used, application of PCPs must be done at the staging area and away from sampling bottles and equipment, and hands must be thoroughly washed after the use of any PCPs prior to sampling.



TECHNICAL
SERVICES

STANDARD OPERATING PROCEDURES

Laboratory Supplied Documents for PFAS

Updated September 2022

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PFAS Target compounds

Analytical Method Information

Printed: 05/23/2023 9:34 am

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---		--Blank Spike / LCS--	
					%Rec	RPD	%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 (PFEEESA)	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

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(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---		--Blank Spike / LCS--	
					%Rec	RPD	%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 (PFEEESA)	0.500	3.56 ng/L		30	25-150	30	50-150	30
Perfluoro-3,6-dioxahexanoic 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

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(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
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 – PFAS 1633

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

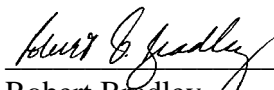
Approvals

Laboratory Director/QA Officer



Krys Trafalski

Vice President/Chief Scientific Officer



Robert Bradley

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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
Perfluoropentanesulfonic 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 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorohexane sulfonic acid	4:2FTS	757124-72-4
1 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorooctane sulfonic acid	6:2FTS	27619-97-2
1 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorodecane sulfonic acid	8:2FTS	39108-34-4
Perfluorooctane sulfonamides		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2
Perfluorooctane sulfonamidoacetic acids		
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
Perfluorooctane sulfonamide ethanols		
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2
Per- and Polyfluoroether carboxylic acids		
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
4,8-Dioxa-3 <i>H</i> -perfluorononanoic acid	ADONA	919005-14-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Ether sulfonic acids		
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	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 <i>H</i> ,2 <i>H</i> ,3 <i>H</i> ,3 <i>H</i> -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 is 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 °C from the time of collection until shipped to the laboratory. Samples must be shipped as soon as practical with sufficient ice to maintain the sample temperature below 6 °C during transport. Sample are to be received by the laboratory within 48 hours of collection. The laboratory must confirm that the sample temperature is 0 - 6 °C upon receipt. Once received by the laboratory, the samples may be stored at ≤ -20 °C, or at 0 - 6 °C, until sample preparation. However, the allowable holding time for samples depends on the storage temperature, as described below:
- 5.3.1 Aqueous samples** may be held in the laboratory for up to 90 days from collection, when stored at ≤ -20 °C and protected from the light. When stored at 0 - 6 °C and protected from the light, aqueous samples may be held for up to **28 days**, with the caveat that issues were observed with certain perfluorooctane sulfonamide ethanols and perfluorooctane sulfonamidoacetic acids after **7 days**. These issues are more likely to elevate the observed concentrations of other PFAS compounds via the transformation of these precursors if they are present in the sample.
- 5.3.2 Solid samples (soils and sediments)** may be held for up to 90 days, if stored in the dark at either 0 - 6 °C or ≤ -20 °C, with the caveat that samples may need to be extracted as soon as possible if NFDHA is an important analyte.
- 5.4 **SAMPLE EXTRACT HOLDING TIMES** – Store sample extracts in the dark at less than 0 - 4 °C until analyzed. If stored in the dark at less than 0 - 4 °C, sample extracts may be stored for up to 90 days, with the caveat that issues were observed for some ether sulfonates after 28 days. These issues may elevate the observed concentrations of the ether sulfonates in the extract over time. Samples may need to be extracted as soon as possible if NFDHA is an important analyte.

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

- 6.1 250-500 mL polypropylene bottles with polypropylene caps. VWR Scientific or equivalent: Part no. 414004-125, 12 pk. Alternate: White PP unlined lid L238WH and 16oz. clarified PP single wall jar 70-400 neck, item J066-Containers and Packaging.com or equivalent.
- 6.2 Transport Tube: Virgin Polypropylene, White, Plastic, 10 mL Capacity, 16 mm OD, 93 mm Overall Lg, Self-Standing, 250 PK, Item 710Z420, Gamut.com (Grainger), with PP cap or equivalent.
- 6.3 Graduated cylinders, 50, 100, 250, 500 and 1000mL, Polypropylene, VWR Scientific or equivalent
- 6.4 Analytical Balance, 0.0001g., checked for accuracy each day of use with Class S weights, certified annually by an outside service
- 6.5 Extract concentrators: Organomation Model N-EVAP 112, 24 position concentrator with water batch control and nitrogen supply controls or equivalent
- 6.6 3.1 Micron in-line filters, Promochrom only
- 6.7 1.0-2.0 mL polypropylene snap cap vials, Agilent part no. 5182-0567 or equivalent
- 6.8 Snap caps, polypropylene or olefin, 11 mm, 11/9k, Agilent Part no. 5182-0542
- 6.9 Solid Phase Extraction Tubes: for EPA 1633: WAX (weak anion exchange mixed mode polymeric sorbent – Phenomenex No. 8B-S038-HCH 200 mg or Waters Oasis 150 mg Cat. # 186002493. Must have a pKa > 8 to remain positively charged during the extraction. Alternate is Agilent Bond Elute WAX 200 mg-cat. No. 5610-2151
- 6.10 Syringes, Hamilton or equivalent 5.0 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, MPFH_xA, MPFH_xS 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.:

<u>Description</u>	<u>Part nos.</u>	<u>Comes in</u>
40 Compound Target 1633 list targets	PFAC-MXJ PFAS-MXI PFAC-MXH PFAC-MXG PFAC-MXF	4 Days – 1.2 mL
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 are 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 refig. Adjacent to QQQ1 N2 generator in box labeled EPA 1633 standards- all are opened, labeled and good to use.

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

* 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	
<p><u>As Needed:</u> 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</p>	<p><u>Daily (When in use)</u> 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</p>
<p><u>Semi-Annually</u> Replace oil mist and odor elements. Replace activated alumina filter if applicable</p>	<p><u>Annually</u> Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.</p>

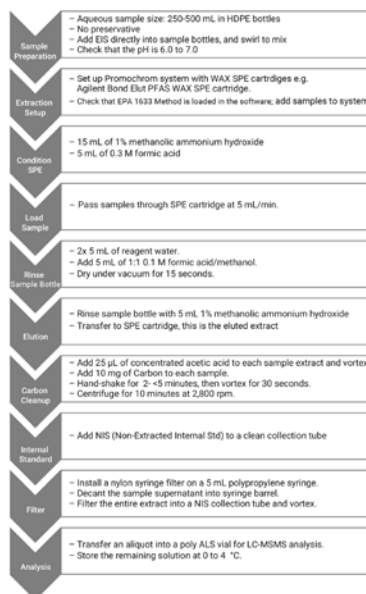
8.2 Sample Preparation (Extraction, Clean-up and Concentration)-Aqueous Matrices

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 ± 0.02 mL through a tared 0.2 um PP filter. Dry filter ≥ 12 hours @ 105C, cool in dessicator. Calc % TSS
2. Check pH with short range pH paper to insure pH = 6.5 ± 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 ± 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 <55°C.. 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 <6°C.
19. Sample is ready for analysis.

Figure 1.0 Aqueous Sample Preparation Steps



- 8.2.1 To measure sample initial volume for aqueous samples, remove the cap and weight the bottle and record the weight in the sample weight. For MBLK, LLOPR and OPR use 250-500 mL volumes). After SPE processing, be sure the empty bottle is dry and weight to determine the amount of sample in grams (essentially equal to volume in mL). Use that number for the initial volume in Element LIMS.
- 8.2.2 For every 20 field samples (Field blanks are considered field samples in as they are treated as such), a blank (MBLK), blank spikes, (2 levels-LLOPR and OPR as BS1 and BS2 respectively). A matrix spike is not necessary since isotope dilution is used. If an MS/MSD is required by a specific project, spike 100 µL 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 µL of EIS (isotopic surrogates) (250/5000 ng/mL) to each sample and QC sample, recap and invert to mix well.
- 8.2.5 Add, 5µL (low level spike), 50 µL (mid-level spike)
- 8.2.6 Using the Promochrom automated system, run a cleaning run. Be sure the reservoirs of LC/MS grade methanol and HPLC plus grade water or equivalent are full. Prime all lines and align all components.
- 8.2.7. Load in the EPA1633 method and adjust the sample volume to 10 ml more than the highest volume container measured by visual comparison to a calibrated bottle of the same size.
- 8.2.8 The SPE method solvents for extractions are as follows:

- Solvent 1 = MeOH
 - Solvent 2 = H₂O
 - 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°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°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 than 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 $\pm 30\%$ 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:

$$\text{MDLb} = \text{Average of Blank Detections} + (3.143 \times \text{Std. Dev.})$$

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 in 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) <i>Section 9.1.5</i>	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 2 nd 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™ 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.
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Acetic Acid, Glacial	-Flammable liquid and vapor. -Irritation	10 ppm TWA; 25 mg/m ³ 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/m ³ ; 50 ppm OSHA TWA: 18 mg/m ³ ; 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/m ³ 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)



Analytical Standard Record

Standard ID: **Y22B197**

Description:	MPPAC-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 (ml):	1	Department:	PFAS
Vials:	1	Lot No.:	MPPACHIFIS0921
Vendor:	Wellington Laboratories		
Comments:	Stock ISTD for EPA method 1633		

Analyte	CAS Number	Concentration	Units
M3PFBA		1	ug/mL
MPPFDA		0.25	ug/mL
MPPHxA		0.5	ug/mL
MPPHxS		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 (¹³C) perfluoroalkylcarboxylic acids (C₄, C₆, C₈-C₁₀) and two mass-labelled (¹⁸O and ¹³C) perfluoroalkanesulfonates (C₆ and C₈). The components and their concentrations are given in Table A.

The individual mass-labelled perfluoroalkylcarboxylic acids and mass-labelled perfluoroalkanesulfonates all have chemical purities of >98% and isotopic purities of ≥99% per ¹³C or >94% per ¹⁸O.

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.

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**

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, \dots, x_n on which it depends is:

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

QUALITY MANAGEMENT:

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

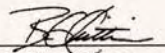


For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at www.well-labs.com or contact us directly at info@well-labs.com

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

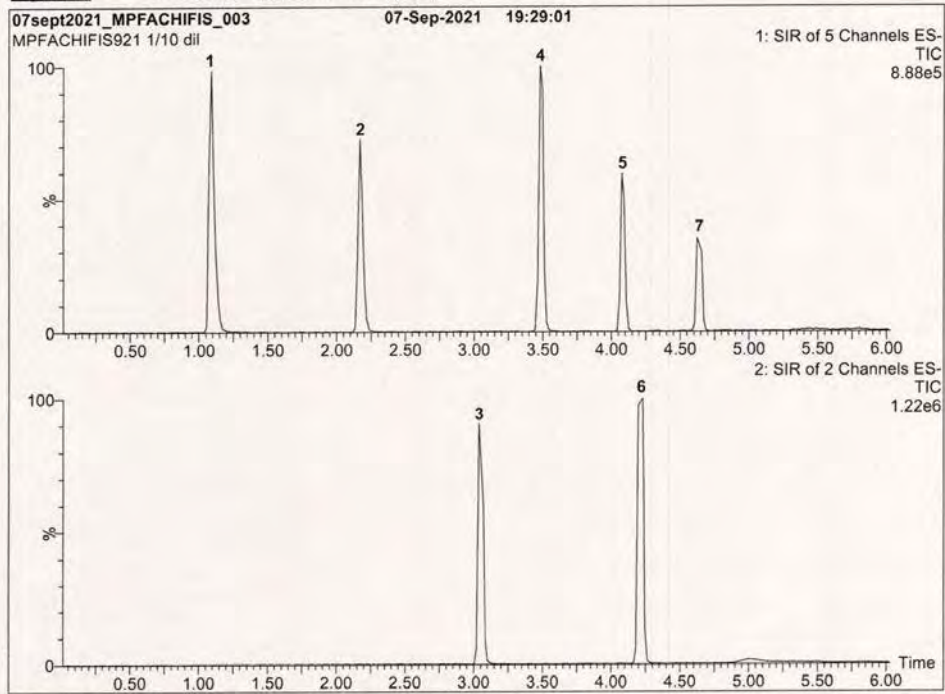
Compound	Acronym	Concentration (ng/mL)		Peak Assignment in Figure 1
		as the salt	as the acid	
Perfluoro-n-(2,3,4- ¹³ C ₃)butanoic acid	M3PFBA	1000		1
Perfluoro-n-(1,2- ¹³ C ₂)hexanoic acid	MPFHxA	500		2
Perfluoro-n-(1,2,3,4- ¹³ C ₄)octanoic acid	MPFOA	500		4
Perfluoro-n-(1,2,3,4,5- ¹³ C ₅)nonanoic acid	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: 
B.G. Chittim, General Manager

Date: 10/13/2021
(mm/dd/yyyy)

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 RP₁₈
1.7 μm, 2.1 x 100 mm

Mobile phase: Gradient

Start: 50% H₂O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH₄OAc buffer)
Ramp to 90% organic over 9 min and hold for
2 min before returning to initial conditions in 1 min.
Time: 15 min

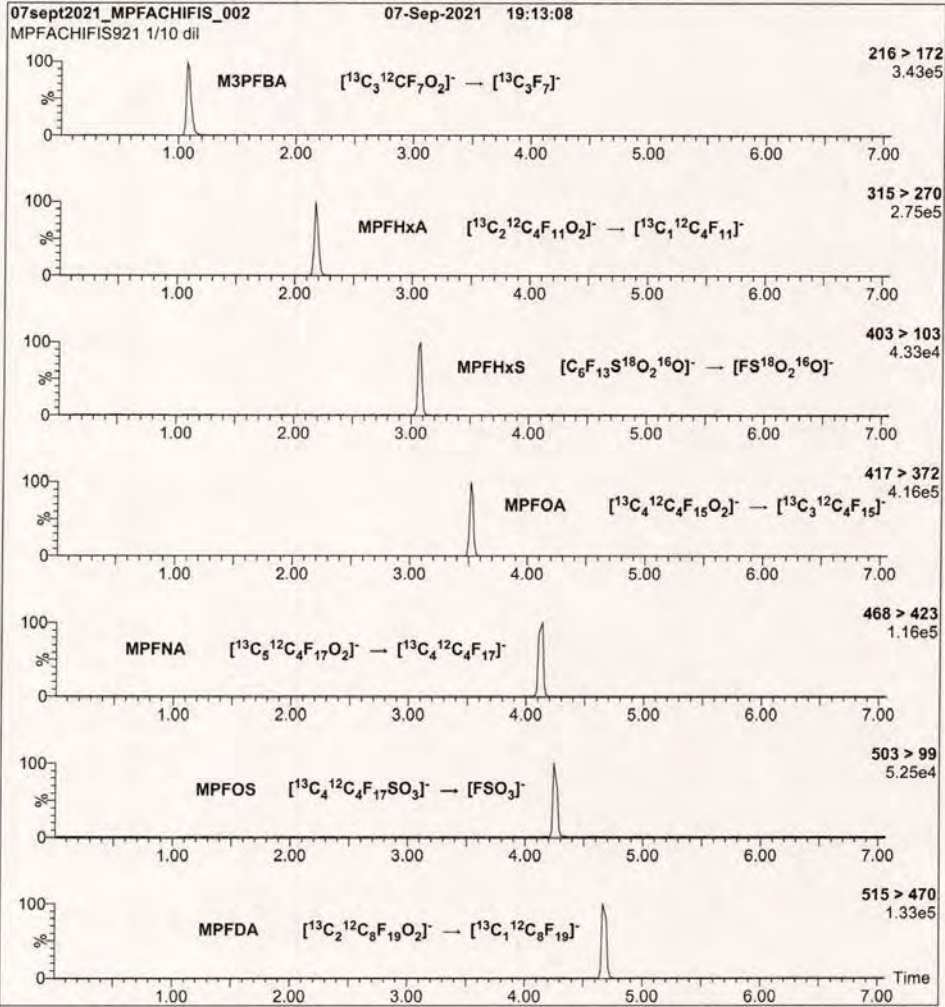
Flow: 300 μL/min

MS Parameters:

Experiment: SIR

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = variable (2-8)
Desolvation Temperature (°C) = 350
Desolvation Gas Flow (L/hr) = 1000

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 $\mu\text{L}/\text{min}$

MS Parameters:

Collision Gas (mbar) = 3.18e-3

Collision Energy (eV) = 4-64 (variable)

Attachment 2 – Extracted Internal Standards (EIS)



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/TPA/1% H2O	Prepared By:	Robert Q. Bradley
Final Volume (mL):	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 (¹³C) perfluoroalkylcarboxylic acids (C₄-C₁₂, C₁₄), three mass-labelled (¹³C) perfluoroalkanesulfonates (C₄, C₆, and C₈), three mass-labelled (one ¹³C and two ²H) perfluoro-1-octanesulfonamides, three mass-labelled (¹³C) fluorotelomer sulfonates (4:2, 6:2, and 8:2), two mass-labelled (²H) perfluorooctanesulfonamidoacetic acids, two mass-labelled (²H) perfluorooctanesulfonamidoethanols, and mass-labelled (¹³C) hexafluoropropylene oxide dimer acid. The components and their concentrations are given in Table A.

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

The individual mass-labelled perfluorooctanesulfonamidoacetic acids, mass-labelled perfluorooctanesulfonamidoethanols, and two mass-labelled (²H) perfluoro-1-octanesulfonamides all have chemical purities of >98% and isotopic purities of ≥98%.

DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture
Figure 1: LC/MS Data (SIR)
Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

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

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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, \dots, x_n on which it depends is:

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

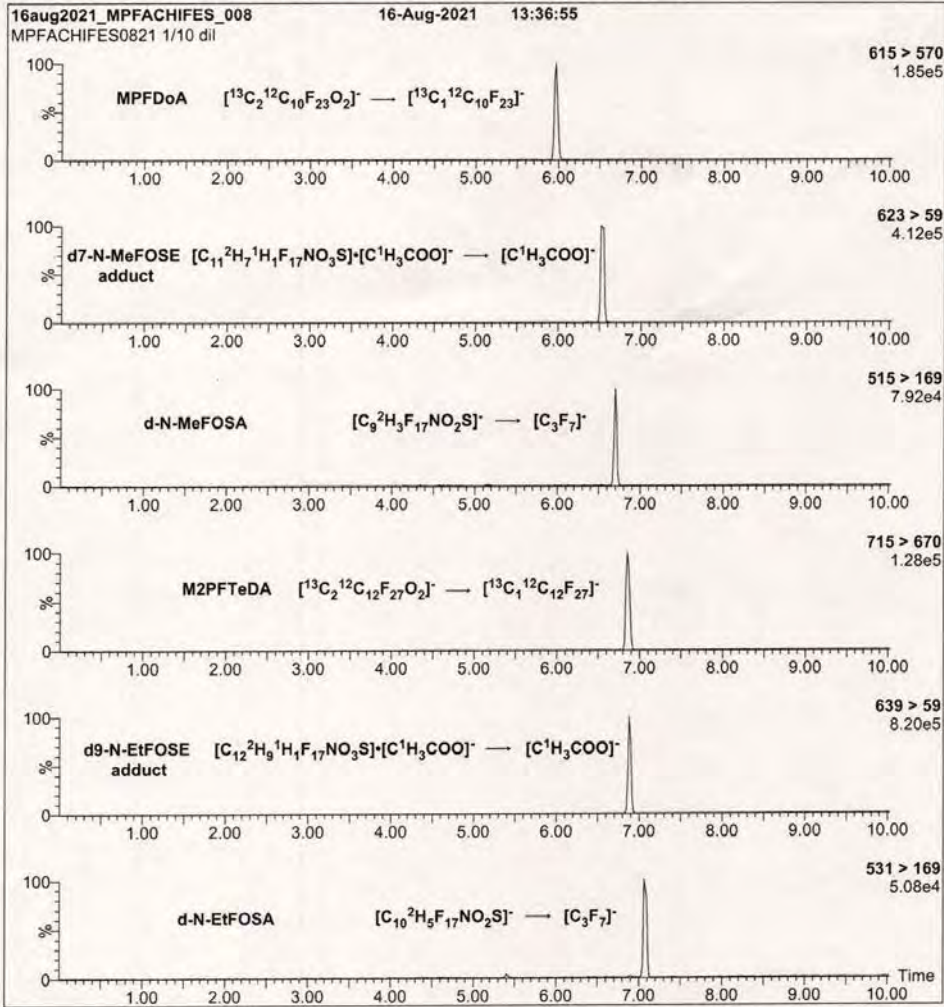
QUALITY MANAGEMENT:

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



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

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)

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

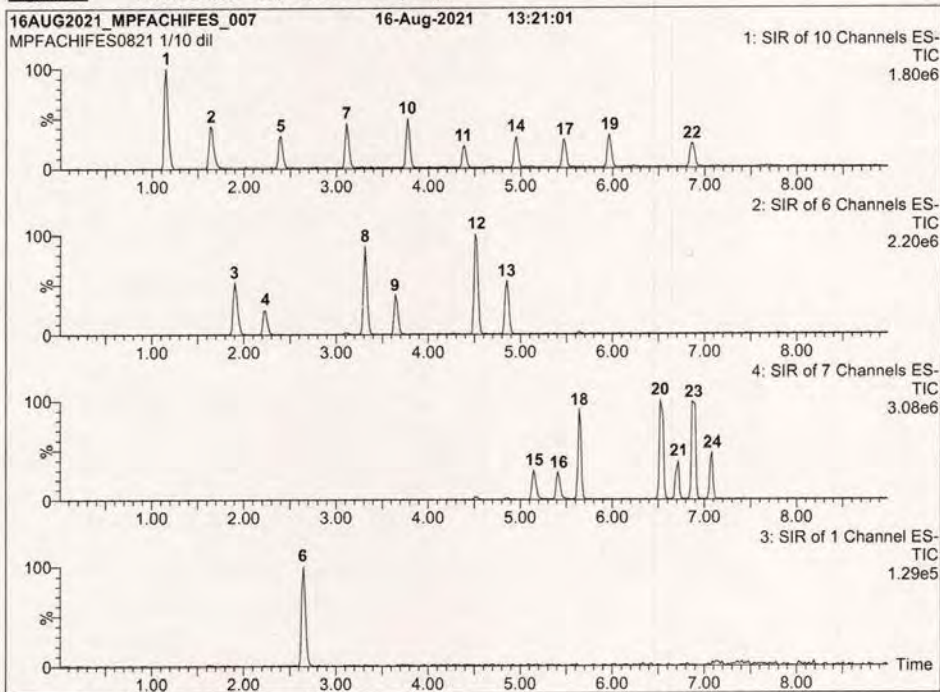
Compound	Acronym	Concentration (ng/mL)		Peak Assignment in Figure 1
		as the salt	as the acid	
Perfluoro-n-(¹³ C) ₄ butanoic acid	MPFBA	2000		1
Perfluoro-n-(¹³ C) ₅ pentanoic acid	M5PFPeA	1000		2
Perfluoro-n-(1,2,3,4,6- ¹³ C) ₆ hexanoic acid	M5PFHxA	500		5
Perfluoro-n-(1,2,3,4- ¹³ C) ₇ heptanoic acid	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) ₁₀ decanoic acid	M6PFDA	250		14
Perfluoro-n-(1,2,3,4,5,6,7- ¹³ C) ₁₁ undecanoic acid	M7PFUDA	250		17
Perfluoro-n-(1,2- ¹³ C) ₁₂ dodecanoic acid	MPFDoA	250		19
Perfluoro-n-(1,2- ¹³ C) ₁₄ tetradecanoic acid	M2PFTeDA	250		22
Perfluoro-1-(¹³ C) ₈ octanesulfonamide	M8FOSA	500		18
N-methyl-d ₂ -perfluoro-1-octanesulfonamide	d-N-MeFOSA	500		21
N-ethyl-d ₅ -perfluoro-1-octanesulfonamide	d-N-EtFOSA	500		24
N-methyl-d ₂ -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 ₂ -perfluoro-1-octanesulfonamido)ethan-d ₂ -ol	d7-N-MeFOSE	5000		20
2-(N-ethyl-d ₅ -perfluoro-1-octanesulfonamido)ethan-d ₅ -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) ₄ butanesulfonate	M3PFBS	500	466	3
Sodium perfluoro-1-(1,2,3- ¹³ C) ₆ 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) ₆ hexanesulfonate	M2-4:2FTS	1000	938	4
Sodium 1H,1H,2H,2H-perfluoro-(1,2- ¹³ C) ₈ octanesulfonate	M2-6:2FTS	1000	951	9
Sodium 1H,1H,2H,2H-perfluoro-(1,2- ¹³ C) ₁₀ decanesulfonate	M2-8:2FTS	1000	960	13

* Concentrations have been rounded to three significant figures.

Certified By: 
 B.G. Chittim, General Manager

Date: 10/13/2021
(mm/dd/yyyy)

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 RP₁₈
 1.7 μ m, 2.1 x 100 mm

Mobile phase: Gradient
 Start: 50% H₂O / 50% (80:20 MeOH:ACN)
 (both with 10 mM NH₄OAc buffer)
 Ramp to 90% organic over 9 min and hold for
 2 min before returning to initial conditions in 1 min.
 Time: 15 min

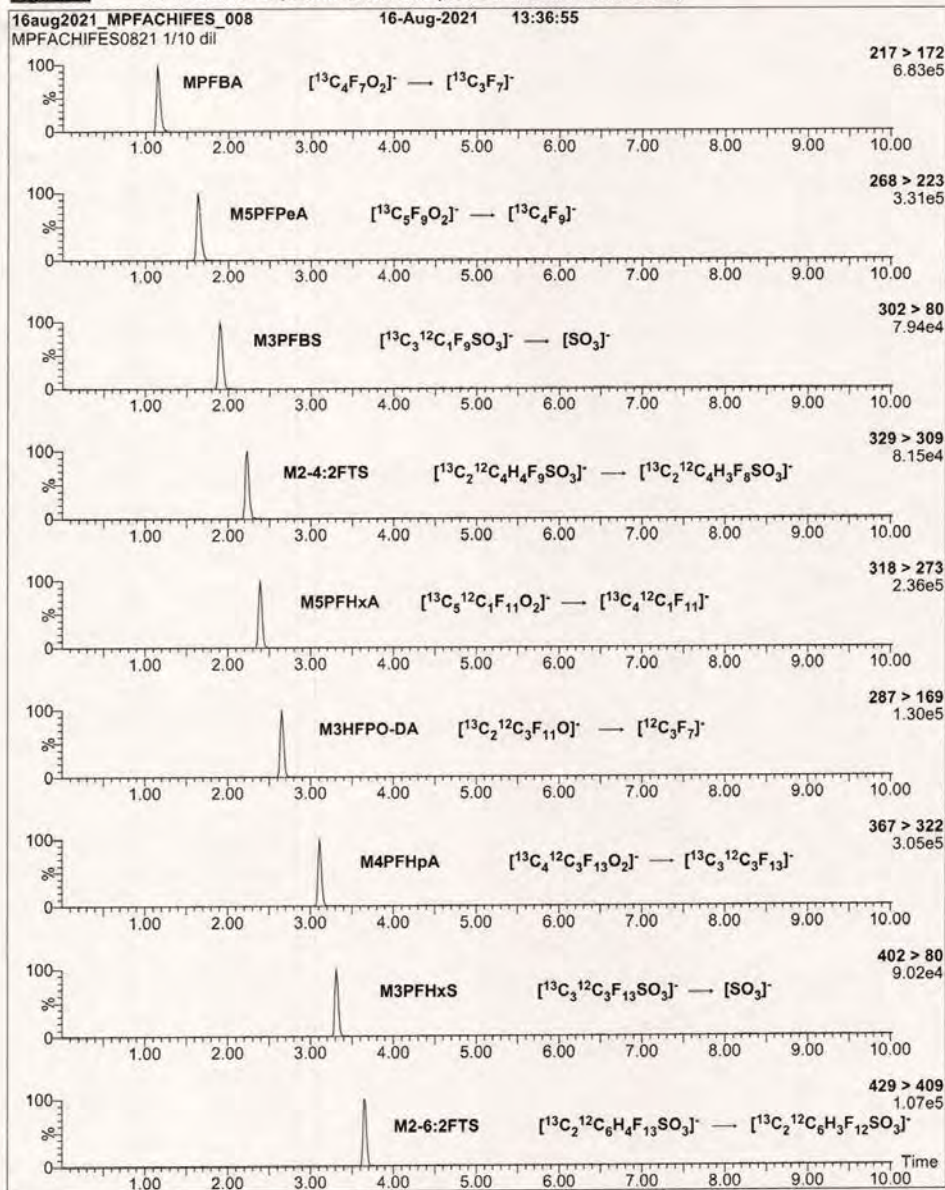
Flow: 300 μ L/min

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

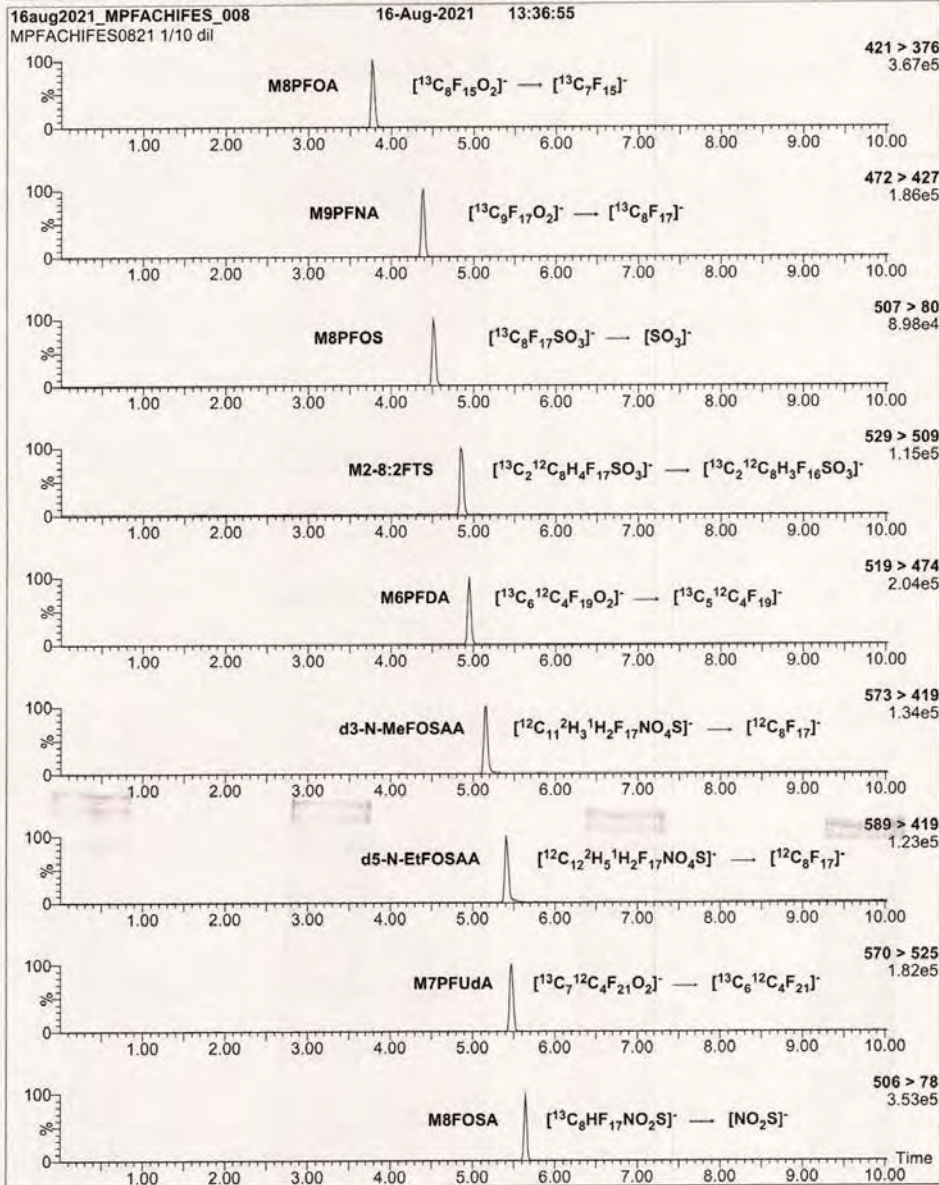
Figure 2: MPFAC-HIF-ES; LC/MS/MS Data (Selected MRM Transitions)



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)

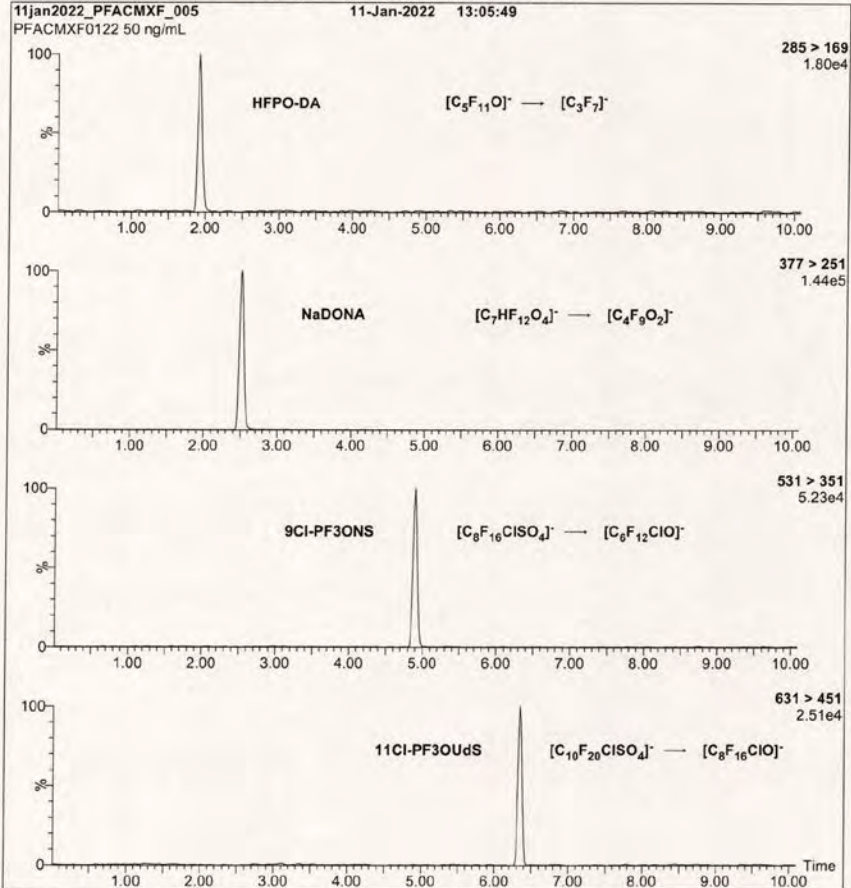


Form: 13, Issued 2004-11-10
Revision: 9, Revised 2020-12-23

MPFACHIFES0821 (6 of 7)
rev1

Attachment 3 – Target Analyte Mixtures

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 μ L/min

MS Parameters:

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

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, \dots, x_n on which it depends is:

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

QUALITY MANAGEMENT:

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



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

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

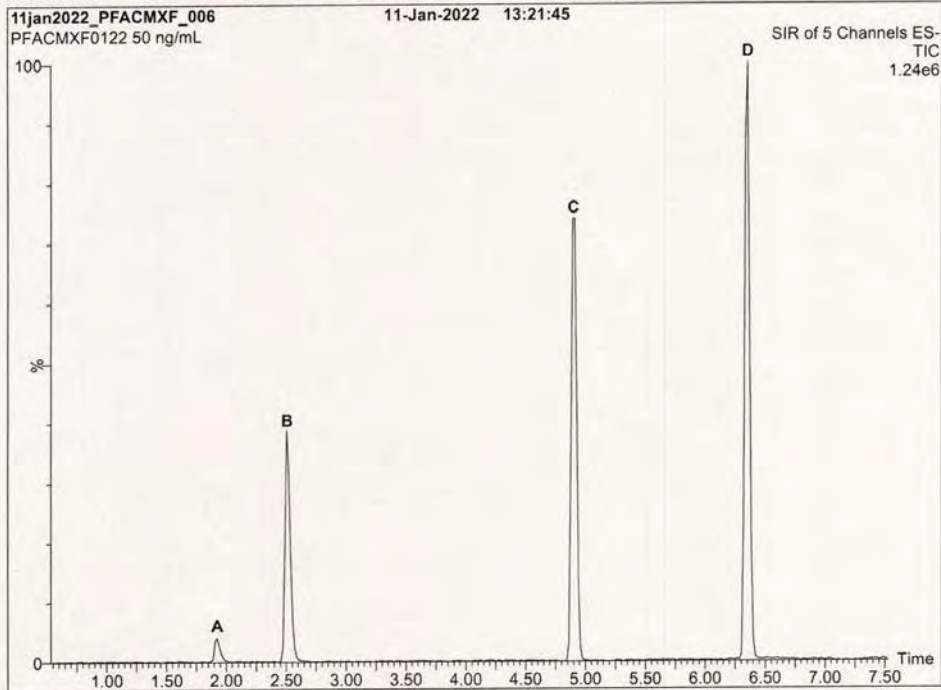
Compound	Acronym	Concentration* (ng/mL)		Peak Assignment in Figure 1
		as the salt	as the acid	
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid	HFPO-DA	2000		A
Sodium dodecafluoro-3H-4,8-dioxananoate	NaDONA	2000	1890	B
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	9Cl-PF3ONS	2000	1870	C
Potassium 11-chloroicosafafluoro-3-oxaundecane-1-sulfonate	11Cl-PF3OUdS	2000	1890	D

* Concentrations have been rounded to three significant figures.

Certified By: 
B.G. Chittim, General Manager

Date: 01/12/2022
(mm/dd/yyyy)

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 RP₁₈
 1.7 μm, 2.1 x 100 mm

Mobile phase: Gradient

Start: 45% H₂O / 55% (80:20 MeOH:ACN)
 (both with 10 mM NH₄OAc 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

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/H2O	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:



**WELLINGTON
LABORATORIES**

**CERTIFICATE OF ANALYSIS
DOCUMENTATION**

PFAC-MXF

**Native Replacement PFAS
Solution/Mixture**

<u>PRODUCT CODE:</u>	PFAC-MXF
<u>LOT NUMBER:</u>	PFACMXF0122
<u>SOLVENT(S):</u>	Methanol / Water (<1%)
<u>DATE PREPARED:</u> (mm/dd/yyyy)	01/10/2022
<u>LAST TESTED:</u> (mm/dd/yyyy)	01/11/2022
<u>EXPIRY DATE:</u> (mm/dd/yyyy)	01/11/2025
<u>RECOMMENDED STORAGE:</u>	Refrigerate ampoule

DESCRIPTION:

PFAC-MXF is a solution/mixture of sodium dodecafluoro-3H-4,8-dioxanonoate (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:

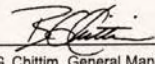
- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acid to the methyl ester.

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519-822-2436 • Fax: 519-822-2849 • info@well-labs.com**

Table A: PFAC-MXI; Components and Concentrations ($\mu\text{g/mL}$; $\pm 5\%$ in methanol)

Compound	Acronym	Concentration ($\mu\text{g/mL}$)	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

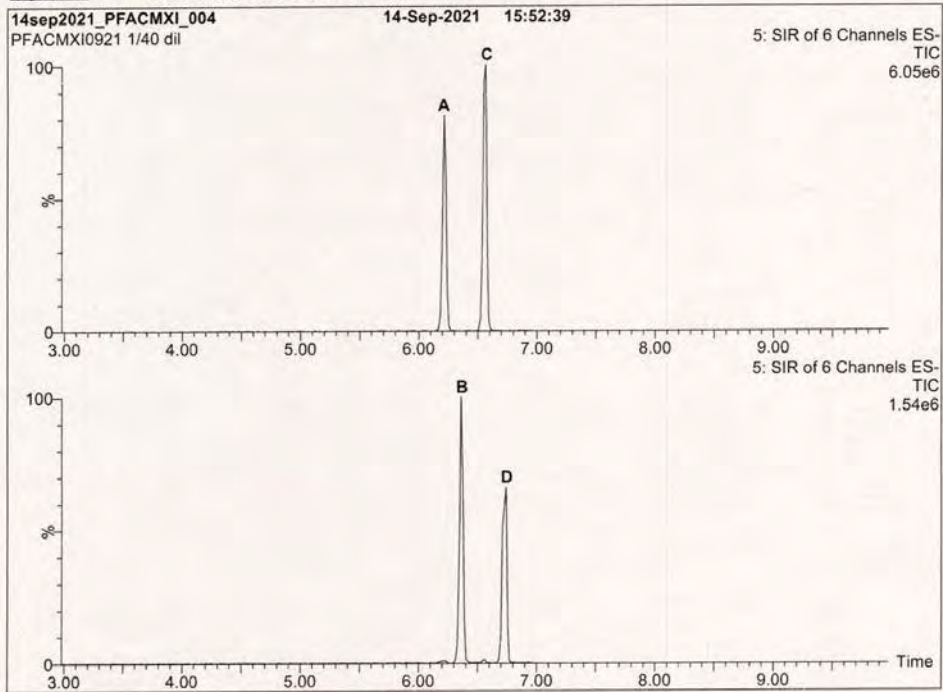
Certified By: 

B.G. Chittim, General Manager

Date: 09/23/2021

(mm/dd/yyyy)

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 RP₁₈
1.7 μ m, 2.1 x 100 mm

Mobile phase: Gradient

Start: 50% H₂O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH₄OAc buffer)
Ramp to 90% organic over 9 min and hold for
2 min before returning to initial conditions in 1 min.
Time: 15 min

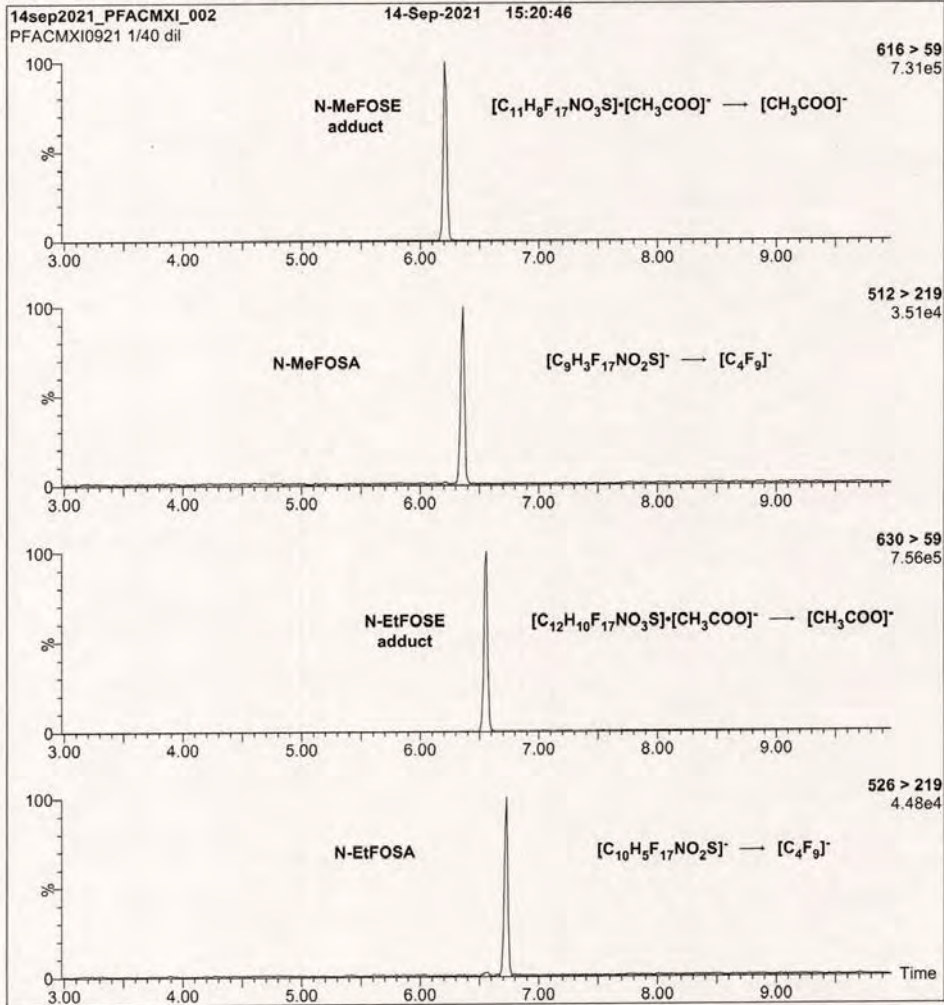
Flow: 300 μ L/min

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

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 μ L/min

MS Parameters:

Collision Gas (mbar) = 3.31e-3

Collision Energy (eV) = 6-60 (variable)

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-EFOSA	4151-50-2	1	ug/mL
N-EFOSE	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: _____



**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.

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

PFACMXI0921 (1 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:

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, \dots, x_n on which it depends is:

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

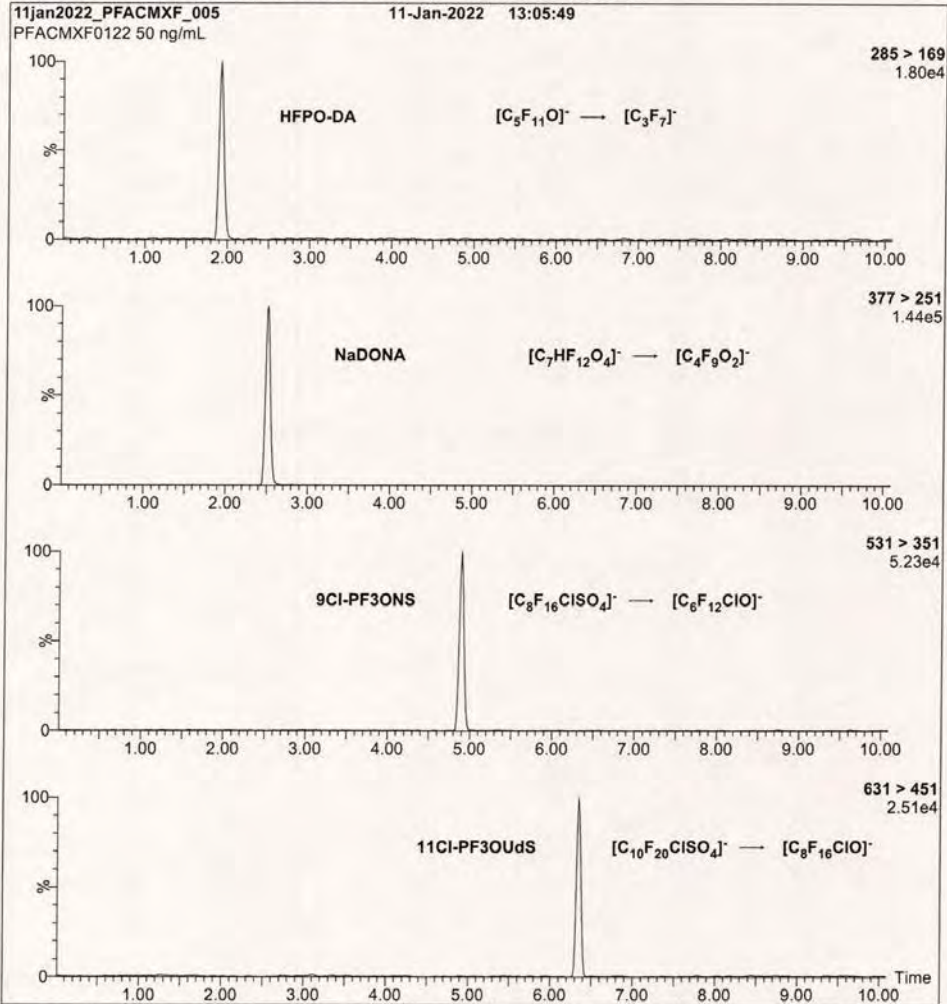
QUALITY MANAGEMENT:

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



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

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 μ L/min

MS Parameters:

Collision Gas (mbar) = 3.43e-3

Collision Energy (eV) = 6-60 (variable)

INTENDED USE:

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HANDLING:

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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.

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UNCERTAINTY:

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

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

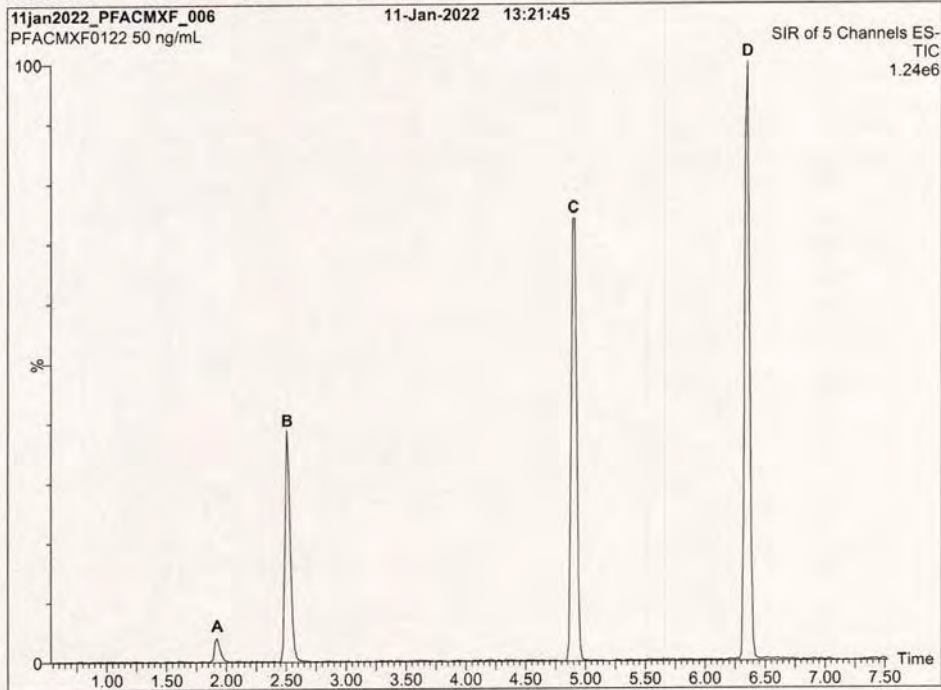
Compound	Acronym	Concentration* (ng/ml)		Peak Assignment in Figure 1
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid	HFPO-DA	2000		A
Compound	Acronym	Concentration* (ng/mL)		Peak Assignment in Figure 1
		as the salt	as the acid	
Sodium dodecafluoro-3H-4,8-dioxananoate	NaDONA	2000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

* Concentrations have been rounded to three significant figures.

Certified By: 
B.G. Chittim, General Manager

Date: 01/12/2022
(mm/dd/yyyy)

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 RP₁₈
 1.7 μ m, 2.1 x 100 mm

Mobile phase: Gradient

Start: 45% H₂O / 55% (80:20 MeOH:ACN)
 (both with 10 mM NH₄OAc 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 μ L/min

MS Parameters:

Experiment: SIR

Source: Electrospray (negative)
 Capillary Voltage (kV) = 2.00
 Cone Voltage (V) = variable (15-74)
 Desolvation Temperature ($^{\circ}$ C) = 325
 Desolvation Gas Flow (L/hr) = 1000

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/H2O	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:



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-dioxanonoate (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:

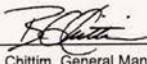
- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acid to the methyl ester.

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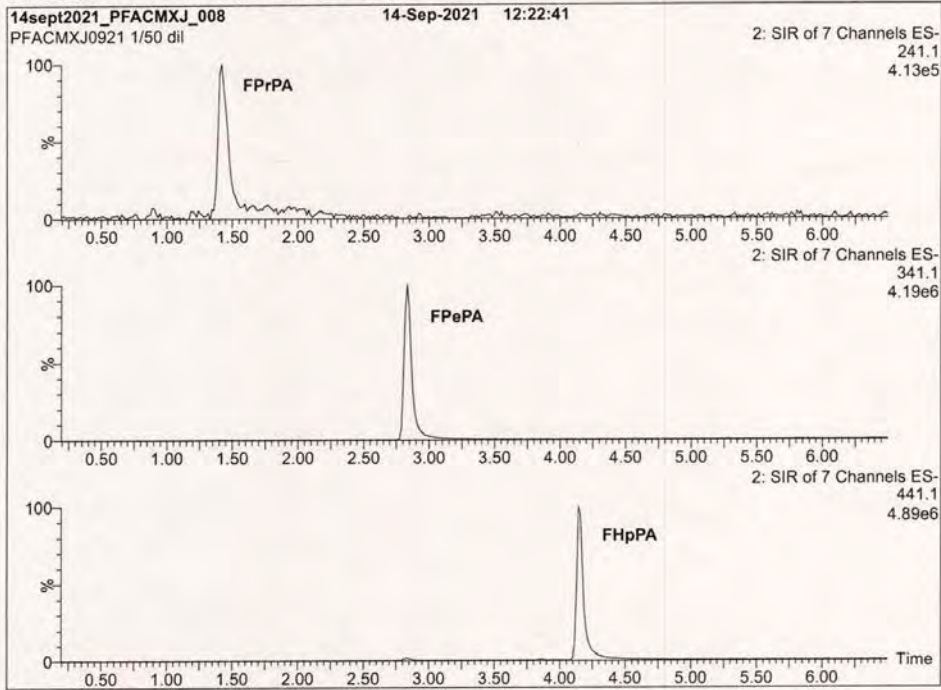
Table A: PFAC-MXJ; Components and Concentrations (µg/mL; ± 5% in methanol)

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

Certified By: 
 B.G. Chittim, General Manager

Date: 10/02/2021
(mm/dd/yyyy)

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 RP₁₈
 1.7 μ m, 2.1 x 100 mm

Mobile phase: Gradient

Start: 50% H₂O / 50% (80:20 MeOH:ACN)
 (both with 10 mM NH₄OAc buffer)
 Ramp to 90% organic over 9 min and hold for
 2 min before returning to initial conditions in 1 min.
 Time: 15 min

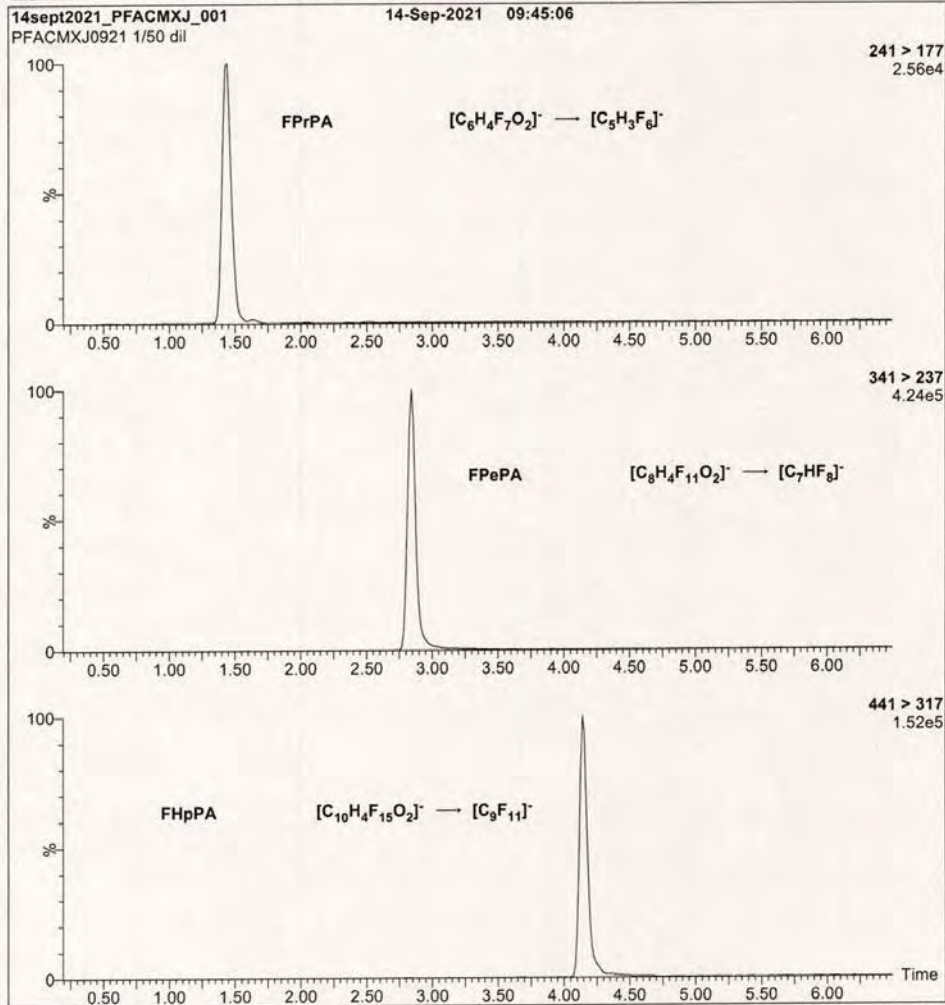
Flow: 300 μ L/min

MS Parameters:

Experiment: SIR

Source: Electrospray (negative)
 Capillary Voltage (kV) = 2.50
 Cone Voltage (V) = variable (2-74)
 Desolvation Temperature ($^{\circ}$ C) = 350
 Desolvation Gas Flow (L/hr) = 1000

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 μ L/min

MS Parameters:

Collision Gas (mbar) = 3.31e-3

Collision Energy (eV) = 6-60 (variable)



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 (mL):	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 (FPPrPA)	356-02-2	4	ug/mL

Reviewed By

Date



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

PFACMXJ0921 (1 of 5)
rev1

INTENDED USE:

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HANDLING:

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HOMOGENEITY:

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UNCERTAINTY:

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

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

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

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TRACEABILITY:

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EXPIRY DATE / PERIOD OF VALIDITY:

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LIMITED WARRANTY:

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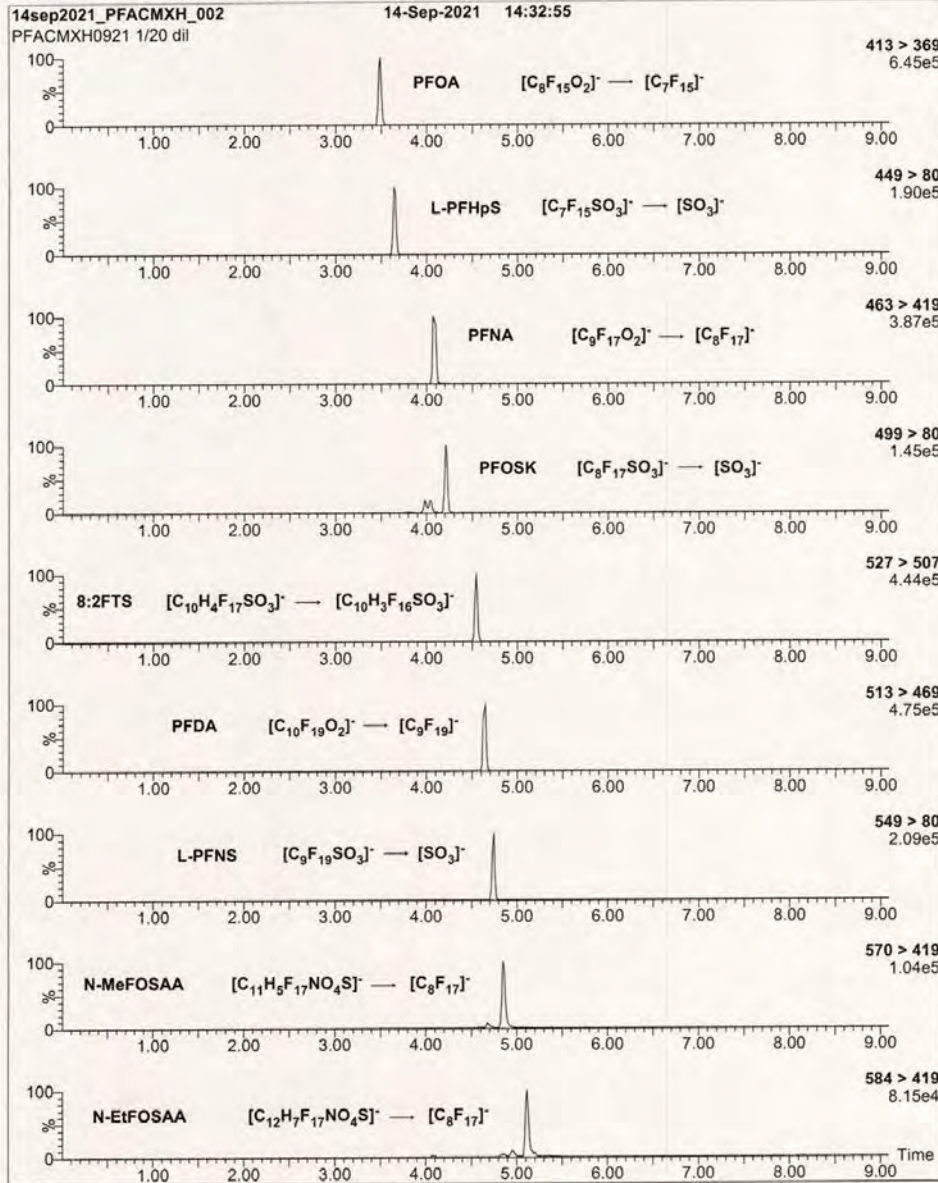
QUALITY MANAGEMENT:

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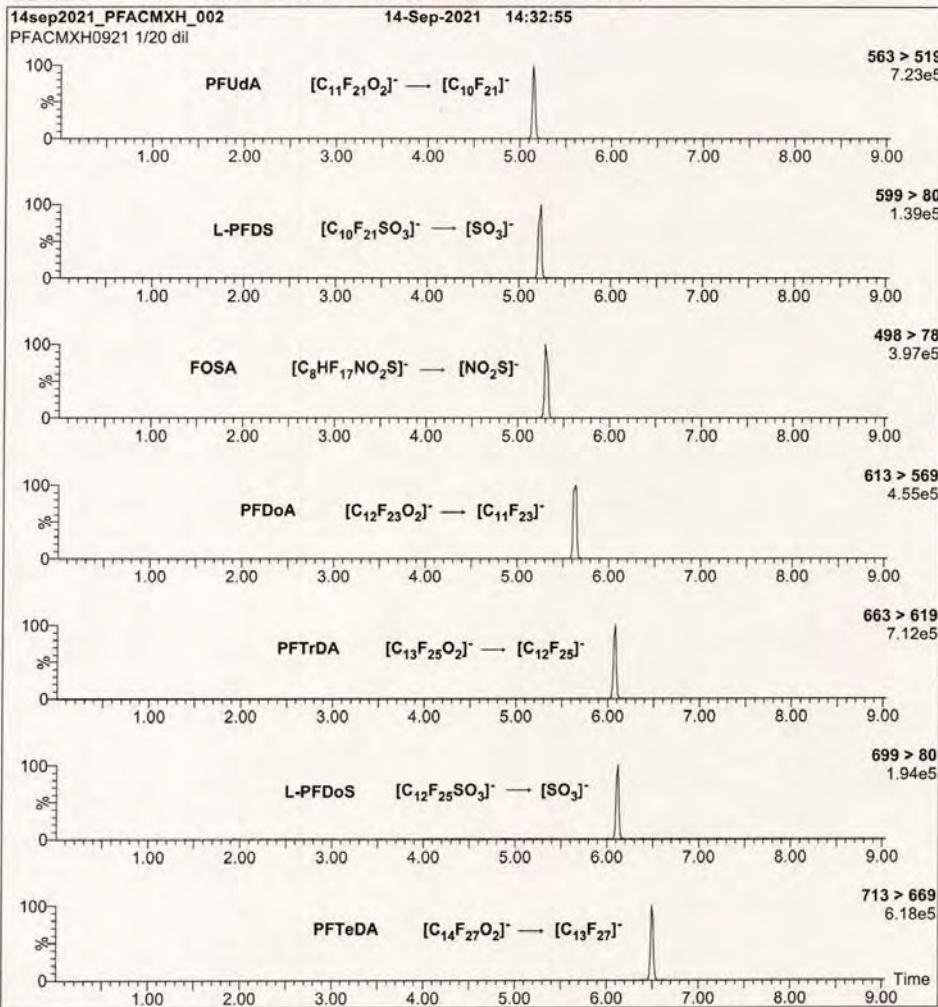
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Figure 2: PFAC-MXH; LC/MS/MS Data (Selected MRM Transitions)



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

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 μ L/min

MS Parameters:

Collision Gas (mbar) = 3.31e-3

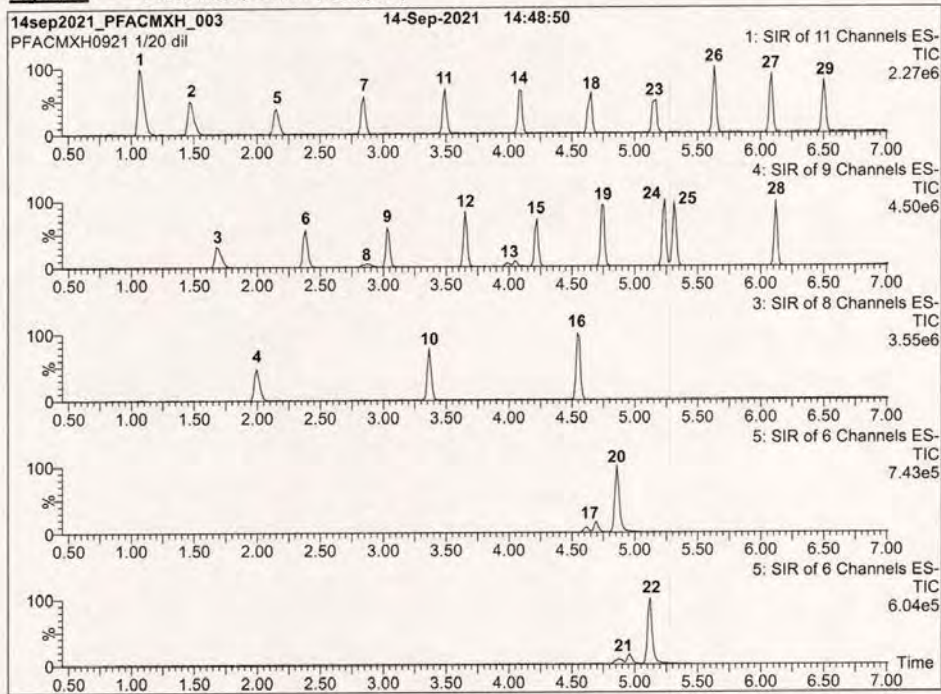
Collision Energy (eV) = 6-60 (variable)

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

Isomer	Compound	Structure	Percent Composition by ¹⁹ F-NMR	
1	Potassium perfluoro-1-octanesulfonate	CF ₃ CF ₂ CF ₂ CF ₂ CF ₂ CF ₂ CF ₂ CF ₂ SO ₃ ⁻ K ⁺	78.8	78.8
2	Potassium 1-trifluoromethylperfluoroheptanesulfonate**	CF ₃ CF ₂ CF ₂ CF ₂ CF ₂ CF ₂ CF(SO ₃ ⁻)K ⁺ CF ₃	1.2	21.1
3	Potassium 2-trifluoromethylperfluoroheptanesulfonate	CF ₃ CF ₂ CF ₂ CF ₂ CF ₂ CF(CF ₃)SO ₃ ⁻ K ⁺ CF ₃	0.6	
4	Potassium 3-trifluoromethylperfluoroheptanesulfonate	CF ₃ CF ₂ CF ₂ CF ₂ CF(CF ₃)CF ₂ SO ₃ ⁻ K ⁺ CF ₃	1.9	
5	Potassium 4-trifluoromethylperfluoroheptanesulfonate	CF ₃ CF ₂ CF ₂ CF(CF ₃)CF ₂ CF ₂ SO ₃ ⁻ K ⁺ CF ₃	2.2	
6	Potassium 5-trifluoromethylperfluoroheptanesulfonate	CF ₃ CF ₂ CF(CF ₃)CF ₂ CF ₂ CF ₂ SO ₃ ⁻ K ⁺ CF ₃	4.5	
7	Potassium 6-trifluoromethylperfluoroheptanesulfonate	CF ₃ CF(CF ₃)CF ₂ CF ₂ CF ₂ CF ₂ SO ₃ ⁻ K ⁺ CF ₃	10.0	
8	Potassium 5,5-di(trifluoromethyl)perfluorohexanesulfonate	CF ₃ CF ₃ CCF ₂ CF ₂ CF ₂ CF ₂ SO ₃ ⁻ K ⁺ CF ₃	0.2	
9	Potassium 4,4-di(trifluoromethyl)perfluorohexanesulfonate	CF ₃ CF ₃ CF ₂ CCF ₂ CF ₂ CF ₂ SO ₃ ⁻ K ⁺ CF ₃	0.03	
10	Potassium 4,5-di(trifluoromethyl)perfluorohexanesulfonate	CF ₃ CF ₃ CF(CF ₃)CF ₂ CF ₂ CF ₂ SO ₃ ⁻ K ⁺ CF ₃	0.4	
11	Potassium 3,5-di(trifluoromethyl)perfluorohexanesulfonate	CF ₃ CF ₃ CF(CF ₃)CF ₂ CF(CF ₃)CF ₂ SO ₃ ⁻ K ⁺ CF ₃	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 RP₁₈
1.7 μm, 2.1 x 100 mm

Mobile phase: Gradient

Start: 50% H₂O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH₄OAc buffer)
Ramp to 90% organic over 9 min and hold for 2 min
before returning to initial conditions in 1 min.
Time: 15 min

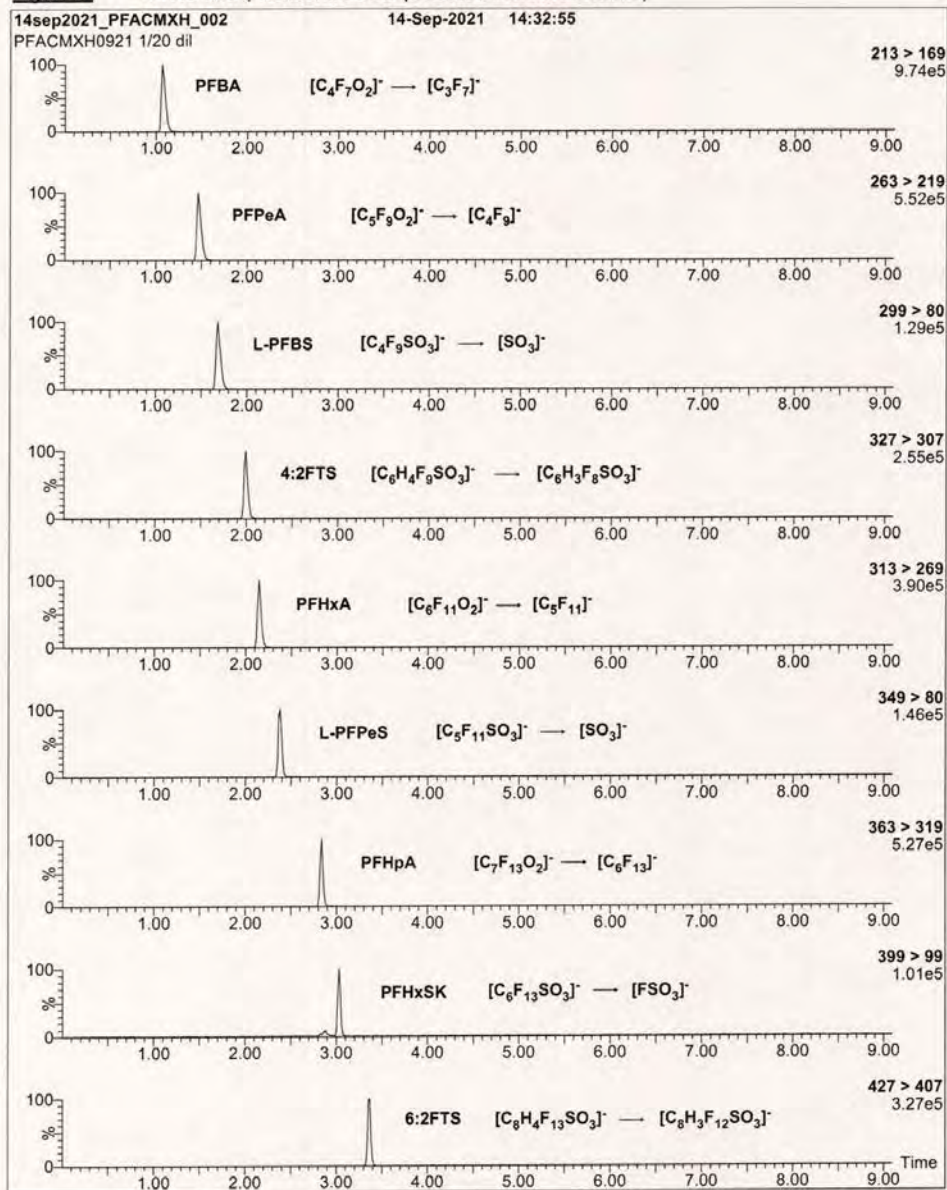
Flow: 300 μL/min

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

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



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

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rev0

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

Isomer	Compound	Structure	Percent Composition by ¹⁹ F-NMR	
1	N-methylperfluoro-1-octanesulfonamidoacetic acid	$\text{CF}_3(\text{CF}_2)_7\text{SO}_2\text{NCH}_2\text{CO}_2\text{H}$	76.0	76.0
2	N-methylperfluoro-3-methylheptanesulfonamidoacetic acid	$\text{CF}_3(\text{CF}_2)_3\text{CF}(\text{CF}_2)_2\text{SO}_2\text{NCH}_2\text{CO}_2\text{H}$	0.7	24.0
3	N-methylperfluoro-4-methylheptanesulfonamidoacetic acid	$\text{CF}_3(\text{CF}_2)_2\text{CF}(\text{CF}_2)_3\text{SO}_2\text{NCH}_2\text{CO}_2\text{H}$	2.0	
4	N-methylperfluoro-5-methylheptanesulfonamidoacetic acid	$\text{CF}_3\text{CF}_2\text{CF}(\text{CF}_2)_4\text{SO}_2\text{NCH}_2\text{CO}_2\text{H}$	6.0	
5	N-methylperfluoro-6-methylheptanesulfonamidoacetic acid	$\text{CF}_3\text{CF}(\text{CF}_2)_5\text{SO}_2\text{NCH}_2\text{CO}_2\text{H}$	14.0	
6	N-methylperfluoro-5,5-dimethylhexanesulfonamidoacetic acid	$\text{CF}_3\text{C}(\text{CF}_3)_2(\text{CF}_2)_4\text{SO}_2\text{NCH}_2\text{CO}_2\text{H}$	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 ¹⁹F-NMR)*

Isomer	Compound	Structure	Percent Composition by ¹⁹ F-NMR	
1	N-ethylperfluoro-1-octanesulfonamidoacetic acid	$\begin{array}{c} \text{CF}_3(\text{CF}_2)_7\text{SO}_2\text{NCH}_2\text{CO}_2\text{H} \\ \\ \text{C}_2\text{H}_5 \end{array}$	77.5	77.5
2	N-ethylperfluoro-3-methylheptanesulfonamidoacetic acid	$\begin{array}{c} \text{CF}_3(\text{CF}_2)_3\text{CF}(\text{CF}_2)_2\text{SO}_2\text{NCH}_2\text{CO}_2\text{H} \\ \quad \quad \quad \\ \text{CF}_3 \quad \quad \quad \text{C}_2\text{H}_5 \end{array}$	2.3	22.5
3	N-ethylperfluoro-4-methylheptanesulfonamidoacetic acid	$\begin{array}{c} \text{CF}_3(\text{CF}_2)_2\text{CF}(\text{CF}_2)_3\text{SO}_2\text{NCH}_2\text{CO}_2\text{H} \\ \quad \quad \quad \\ \text{CF}_3 \quad \quad \quad \text{C}_2\text{H}_5 \end{array}$	2.2	
4	N-ethylperfluoro-5-methylheptanesulfonamidoacetic acid	$\begin{array}{c} \text{CF}_3\text{CF}_2\text{CF}(\text{CF}_2)_4\text{SO}_2\text{NCH}_2\text{CO}_2\text{H} \\ \quad \quad \quad \\ \text{CF}_3 \quad \quad \quad \text{C}_2\text{H}_5 \end{array}$	5.4	
5	N-ethylperfluoro-6-methylheptanesulfonamidoacetic acid	$\begin{array}{c} \text{CF}_3\text{CF}(\text{CF}_2)_5\text{SO}_2\text{NCH}_2\text{CO}_2\text{H} \\ \quad \quad \quad \\ \text{CF}_3 \quad \quad \quad \text{C}_2\text{H}_5 \end{array}$	10.4	
6	N-ethylperfluoro-5,5-dimethylhexanesulfonamidoacetic acid	$\begin{array}{c} \text{CF}_3 \\ \\ \text{CF}_3\text{C}(\text{CF}_2)_4\text{SO}_2\text{NCH}_2\text{CO}_2\text{H} \\ \\ \text{CF}_3 \quad \quad \quad \\ \quad \quad \quad \quad \quad \text{C}_2\text{H}_5 \end{array}$	0.3	
7	N-ethylperfluoro-4,5-dimethylhexanesulfonamidoacetic acid	$\begin{array}{c} \text{CF}_3 \\ \\ \text{CF}_3\text{CF}(\text{CF}_2)_3\text{SO}_2\text{NCH}_2\text{CO}_2\text{H} \\ \quad \quad \quad \\ \text{CF}_3 \quad \quad \quad \text{C}_2\text{H}_5 \end{array}$	0.3	
8	N-ethylperfluoro-3,5-dimethylhexanesulfonamidoacetic acid	$\begin{array}{c} \text{CF}_3 \\ \\ \text{CF}_3\text{CF}(\text{CF}_2)_2\text{CF}(\text{CF}_2)_2\text{SO}_2\text{NCH}_2\text{CO}_2\text{H} \\ \quad \quad \quad \\ \text{CF}_3 \quad \quad \quad \text{C}_2\text{H}_5 \end{array}$	0.3	
9	Other Unidentified Isomers		1.3	

* Percent of total N-ethylperfluorooctanesulfonamidoacetic acid isomers only.

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

Isomer	Compound	Structure	Percent Composition by ¹⁹ F-NMR	
1	Potassium perfluoro-1-hexanesulfonate	CF ₃ CF ₂ CF ₂ CF ₂ CF ₂ CF ₂ SO ₃ ⁻ K ⁺	81.1	81.1
2	Potassium 1-trifluoromethylperfluoropentanesulfonate**	CF ₃ CF ₂ CF ₂ CF ₂ CF(SO ₃ ⁻)K ⁺ CF ₃	2.9	18.9
3	Potassium 2-trifluoromethylperfluoropentanesulfonate	CF ₃ CF ₂ CF ₂ CF(CF ₃)SO ₃ ⁻ K ⁺ CF ₃	1.4	
4	Potassium 3-trifluoromethylperfluoropentanesulfonate	CF ₃ CF ₂ CF(CF ₃)CF ₂ SO ₃ ⁻ K ⁺ CF ₃	5.0	
5	Potassium 4-trifluoromethylperfluoropentanesulfonate	CF ₃ CF(CF ₃)CF ₂ CF ₂ SO ₃ ⁻ K ⁺ CF ₃	8.9	
6	Potassium 3,3-di(trifluoromethyl)perfluorobutanesulfonate	CF ₃ CF ₃ CCF ₂ CF ₂ SO ₃ ⁻ K ⁺ CF ₃	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 (C₇-C₁₇), eight native perfluoroalkanesulfonates (C₄, C₆, C₇, C₈, C₁₀, and C₁₂ linear; C₆ and C₈ 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

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

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

QUALITY MANAGEMENT:

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



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

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
		as the salt	as the acid	
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 *	N-MeFOSAA: linear isomer	0.760		20
	N-MeFOSAA: Σ branched isomers	0.240		17
N-ethylperfluorooctanesulfonamidoacetic acid *	N-EtFOSAA: linear isomer	0.775		22
	N-EtFOSAA: Σ branched isomers	0.225		21
Compound	Acronym	Concentration* (µg/mL)		Peak Assignment in Figure 1
Potassium perfluoro-1-butanedisulfonate	L-PFBS	1.00	0.887	
Sodium perfluoro-1-pentanesulfonate	L-PFPeS	1.00	0.941	6
Potassium perfluorohexanesulfonate *	PFHxSK: linear isomer	0.811	0.741	9
	PFHxSK: Σ branched isomers	0.189	0.173	8
Sodium perfluoro-1-heptanesulfonate	L-PFHpS	1.00	0.953	12
Potassium perfluorooctanesulfonate *	PFOSK: linear isomer	0.788	0.732	15
	PFOSK: Σ branched isomers	0.211	0.196	13
Sodium perfluoro-1-nonanesulfonate	L-PFNs	1.00	0.962	19
Sodium perfluoro-1-decanesulfonate	L-PFDS	1.00	0.965	24
Sodium perfluoro-1-dodecanesulfonate	L-PFDoS	1.00	0.970	28
Sodium 1H,1H,2H,2H-perfluorohexanesulfonate	4:2FTS	4.00	3.75	4
Sodium 1H,1H,2H,2H-perfluorooctanesulfonate	6:2FTS	4.00	3.80	10
Sodium 1H,1H,2H,2H-perfluorodecanesulfonate	8:2FTS	4.00	3.84	16

* See Table B for percent composition of linear and branched N-MeFOSAA isomers.
 * See Table C for percent composition of linear and branched N-EtFOSAA isomers.
 * See Table D for percent composition of linear and branched PFHxSK isomers.
 * See Table E for percent composition of linear and branched PFOSK isomers.

* Concentrations have been rounded to three significant figures.

Certified By: 
 B.G. Chittim, General Manager

Date: 09/23/2021
(mm/dd/yyyy)

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.:	PEACMXH0921
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-EFOSAA	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:

Attachment 4 – Calibration Concentrations, nominal

Calibration Solutions (ng/mL) Compound								
CSI (LOQ)	CS2	Perfluoropalkyl carboxylic	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	
PFDaA	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	
PENS	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	
NEFOSA	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	
NEFOSAA	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	
NEFOSE	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-PF3OUs	0.8	2	5	10	20	50	250	
PFEESA	0.4	1	2.5	5	10	25	125	

Calibration Solutions (ng/mL) Compound							
CSI (LOQ)	CS2 Fluorotelomer carboxylic acids	CS3	CS4 (CV ¹)	CS5	CS6	CS7 ²	
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 ₁ -PFBA	10	10	10	10	10	10	10
¹³ C ₃ -PFPeA	5	5	5	5	5	5	5
¹³ C ₃ -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 ₆ -PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₇ -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 ₁ -NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D ₁ -NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D ₁ -NMeFOSAA	5	5	5	5	5	5	5
D ₁ -NEtFOSAA	5	5	5	5	5	5	5
D ₇ -NMeFOSE	25	25	25	25	25	25	25
D ₆ -NEtFOSE	25	25	25	25	25	25	25
¹³ C ₇ -HFPO-DA	10	10	10	10	10	10	10
Non-extracted Internal Standard (NIS) Analytes							
¹³ C ₁ -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 ₃ -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
¹⁶ O ₂ -PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₁ -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 Injection
 Compress A Compressibility Mode A Compressibility Value Set Injection with needle wash
Compressibility A 70 10e-6/bar 3.00 µL
 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

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



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-CF ₃ PF ₃ OdS	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 2F TS)	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 2F TS)	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-decanesulfonate (8 2F TS)	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 2F TS)	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-hexanesulfonate (4 2F TS)	No	427	Unit/Enh (6490)	407	Unit/Enh (6490)	Yes	No	162	24	4	6.188	3	Negative
1H,1H,2H,2H-perfluoro-1-octanesulfonate (6 2F TS)	No	427	Unit/Enh (6490)	79.7	Unit/Enh (6490)	Yes	No	162	48	4	6.188	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



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-CI-PF3ONS ADONA	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-MeFOSA	No	572.99	Unit/Enh (6490)	418.8	Unit/Enh (6490)	Yes	No	130	20	4	7.17	3	Negative
d5-NEFOSA	No	531	Unit/Enh (6490)	219	Unit/Enh (6490)	Yes	No	150	20	4	8.52	3	Negative
d5-NEFOSA	No	531	Unit/Enh (6490)	169	Unit/Enh (6490)	Yes	No	150	20	4	8.52	3	Negative
d5-N-EIFOSAA	No	589.02	Unit/Enh (6490)	530.9	Unit/Enh (6490)	Yes	No	130	20	4	7.36	3	Negative
d5-N-EIFOSAA	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-NEFOSE	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
M2PF TeD A	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

Acquisition Method Report



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
NEFOSA	No	526	Unit/Enh (6490)	219	Unit/Enh (6490)	Yes	No	120	20	4	8.528	3	Negative
NEFOSA	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
NEFOSE	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 (MBFOSA)	No	506	Unit/Enh (6490)	78	Unit/Enh (6490)	Yes	No	162	48	4	7.59	3	Negative
Perfluoro-1 - [13C8]octanesulfonic acid (MBPFOS)	No	507	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	174	48	4	6.59	3	Negative
Perfluoro-1 - [13C8]octanesulfonic acid (MBPFOS)	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



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
Perfluorodecanoic acid (PFDoA)	No	613	Unit/Enh (6490)	568.8	Unit/Enh (6490)	Yes	No	90	12	4	7.876	3	Negative
Perfluorodecanoic acid (PFDoA)	No	613	Unit/Enh (6490)	168.7	Unit/Enh (6490)	Yes	No	90	28	4	7.876	3	Negative
Perfluorooheptanoic acid (PFHpA)	No	363	Unit/Enh (6490)	318.8	Unit/Enh (6490)	Yes	No	90	8	4	5.601	3	Negative
Perfluorooheptanoic acid (PFHpA)	No	363	Unit/Enh (6490)	168.9	Unit/Enh (6490)	Yes	No	90	16	4	5.601	3	Negative
Perfluorooxanesulfonic acid (PFHxS)	No	398.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	40	4	5.685	3	Negative
Perfluorooxanesulfonic acid (PFHxS)	No	398.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	44	4	5.685	3	Negative
Perfluorooxanoic acid (PFHxA)	No	313	Unit/Enh (6490)	268.9	Unit/Enh (6490)	Yes	No	70	4	4	4.856	3	Negative
Perfluorooxanoic acid (PFHxA)	No	313	Unit/Enh (6490)	119	Unit/Enh (6490)	Yes	No	70	20	4	4.856	3	Negative
Perfluoron-1,2-dodecanoic acid (MPF DoA)	No	615	Unit/Enh (6490)	570	Unit/Enh (6490)	Yes	No	53	8	4	7.71	3	Negative
Perfluoron-1,2-dodecanoic acid (MPF DoA)	No	217	Unit/Enh (6490)	172	Unit/Enh (6490)	Yes	No	59	4	4	1.22	3	Negative
Perfluoron-1,2-dodecanoic acid (MPF DoA)	No	268	Unit/Enh (6490)	223	Unit/Enh (6490)	Yes	No	62	4	4	3.44	3	Negative
Perfluoron-1,2-dodecanoic acid (MPF DoA)	No	421	Unit/Enh (6490)	376	Unit/Enh (6490)	Yes	No	59	4	4	6.05	3	Negative
Perfluoron-1,2-dodecanoic acid (MPF DoA)	No	421	Unit/Enh (6490)	172	Unit/Enh (6490)	Yes	No	59	16	4	6.05	3	Negative

Acquisition Method Report



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]non anoic acid (M9PFNA)	No	472	Unit/Enh (6490)	427	Unit/Enh (6490)	Yes	No	59	8	4	6.56	3	Negative
Perfluoro- n- [13C9]non anoic 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)	168.9	Unit/Enh (6490)	Yes	No	70	4	4	1.246	3	Negative
Perfluoron onanesulfo nate (L- PFNS)	No	548.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	159	48	4	7.174	3	Negative
Perfluoron onanesulfo nate (L- PFNS)	No	548.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	159	48	4	7.174	3	Negative
Perfluoron onanoic acid (PFNA)	No	463	Unit/Enh (6490)	418.8	Unit/Enh (6490)	Yes	No	90	8	4	6.718	3	Negative
Perfluoron onanoic 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
Perfluoro ctanesulfo nic acid (PFOS)	No	498.9	Unit/Enh (6490)	98.9	Unit/Enh (6490)	Yes	No	150	44	4	6.743	3	Negative
Perfluoro ctanesulfo nic acid (PFOS)	No	498.9	Unit/Enh (6490)	79.9	Unit/Enh (6490)	Yes	No	150	84	4	6.743	3	Negative
Perfluoro ctanoic acid (PFOA)	No	413	Unit/Enh (6490)	368.8	Unit/Enh (6490)	Yes	No	90	8	4	6.202	3	Negative
Perfluoro ctanoic acid (PFOA)	No	413	Unit/Enh (6490)	168.9	Unit/Enh (6490)	Yes	No	90	16	4	6.202	3	Negative
Perfluoro tridecanoic acid (PFTA)	No	713	Unit/Enh (6490)	669	Unit/Enh (6490)	Yes	No	110	12	4	8.414	3	Negative
Perfluoro tridecanoic acid (PFTA)	No	713	Unit/Enh (6490)	168.8	Unit/Enh (6490)	Yes	No	110	28	4	8.414	3	Negative
Perfluoro tridecanoic acid (PFTA)	No	663	Unit/Enh (6490)	618.8	Unit/Enh (6490)	Yes	No	90	12	4	8.164	3	Negative
Perfluoro undecanoic acid (PFUDA)	No	563	Unit/Enh (6490)	519	Unit/Enh (6490)	Yes	No	90	8	4	7.538	3	Negative
Perfluoro undecanoic acid (PFUDA)	No	563	Unit/Enh (6490)	169	Unit/Enh (6490)	Yes	No	90	24	4	7.538	3	Negative
PFEEA	No	315	Unit/Enh (6490)	135	Unit/Enh (6490)	Yes	No	112	26	4	4.464	3	Negative
PFEEA	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



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
---------------	-----

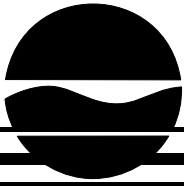
Name: Binary Pump

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/min ²
Max. Flow Ramp Down	100.000 mL/min ²
Expected Mixer	No check

ATTACHMENT C

SCO Tables



Department of Environmental Conservation

Division of Environmental Remediation

6 NYCRR PART 375
Environmental Remediation Programs
Subparts 375-1 to 375-4 & 375-6

Effective December 14, 2006

New York State Department of Environmental Conservation

375-6.8

Soil cleanup objective tables.

(a) Unrestricted use soil cleanup objectives.

Table 375-6.8(a):Unrestricted Use Soil Cleanup Objectives

Contaminant	CAS Number	Unrestricted Use
Metals		
Arsenic	7440-38-2	13 ^c
Barium	7440-39-3	350 ^c
Beryllium	7440-41-7	7.2
Cadmium	7440-43-9	2.5 ^c
Chromium, hexavalent ^e	18540-29-9	1 ^b
Chromium, trivalent ^e	16065-83-1	30 ^c
Copper	7440-50-8	50
Total Cyanide ^{e, f}		27
Lead	7439-92-1	63 ^c
Manganese	7439-96-5	1600 ^c
Total Mercury		0.18 ^c
Nickel	7440-02-0	30
Selenium	7782-49-2	3.9 ^c
Silver	7440-22-4	2
Zinc	7440-66-6	109 ^c
PCBs/Pesticides		
2,4,5-TP Acid (Silvex) ^f	93-72-1	3.8
4,4'-DDE	72-55-9	0.0033 ^b
4,4'-DDT	50-29-3	0.0033 ^b
4,4'-DDD	72-54-8	0.0033 ^b
Aldrin	309-00-2	0.005 ^c
alpha-BHC	319-84-6	0.02
beta-BHC	319-85-7	0.036
Chlordane (alpha)	5103-71-9	0.094

Table 375-6.8(a):Unrestricted Use Soil Cleanup Objectives

Contaminant	CAS Number	Unrestricted Use
delta-BHC ^g	319-86-8	0.04
Dibenzofuran ^f	132-64-9	7
Dieldrin	60-57-1	0.005 ^c
Endosulfan I ^{d, f}	959-98-8	2.4
Endosulfan II ^{d, f}	33213-65-9	2.4
Endosulfan sulfate ^{d, f}	1031-07-8	2.4
Endrin	72-20-8	0.014
Heptachlor	76-44-8	0.042
Lindane	58-89-9	0.1
Polychlorinated biphenyls	1336-36-3	0.1
Semivolatile organic compounds		
Acenaphthene	83-32-9	20
Acenaphthylene ^f	208-96-8	100 ^a
Anthracene ^f	120-12-7	100 ^a
Benz(a)anthracene ^f	56-55-3	1 ^c
Benzo(a)pyrene	50-32-8	1 ^c
Benzo(b)fluoranthene ^f	205-99-2	1 ^c
Benzo(g,h,i)perylene ^f	191-24-2	100
Benzo(k)fluoranthene ^f	207-08-9	0.8 ^c
Chrysene ^f	218-01-9	1 ^c
Dibenz(a,h)anthracene ^f	53-70-3	0.33 ^b
Fluoranthene ^f	206-44-0	100 ^a
Fluorene	86-73-7	30
Indeno(1,2,3-cd)pyrene ^f	193-39-5	0.5 ^c
m-Cresol ^f	108-39-4	0.33 ^b
Naphthalene ^f	91-20-3	12
o-Cresol ^f	95-48-7	0.33 ^b

Table 375-6.8(a):Unrestricted Use Soil Cleanup Objectives

Contaminant	CAS Number	Unrestricted Use
p-Cresol ^f	106-44-5	0.33 ^b
Pentachlorophenol	87-86-5	0.8 ^b
Phenanthrene ^f	85-01-8	100
Phenol	108-95-2	0.33 ^b
Pyrene ^f	129-00-0	100
Volatile organic compounds		
1,1,1-Trichloroethane ^f	71-55-6	0.68
1,1-Dichloroethane ^f	75-34-3	0.27
1,1-Dichloroethene ^f	75-35-4	0.33
1,2-Dichlorobenzene ^f	95-50-1	1.1
1,2-Dichloroethane	107-06-2	0.02 ^c
cis -1,2-Dichloroethene ^f	156-59-2	0.25
trans-1,2-Dichloroethene ^f	156-60-5	0.19
1,3-Dichlorobenzene ^f	541-73-1	2.4
1,4-Dichlorobenzene	106-46-7	1.8
1,4-Dioxane	123-91-1	0.1 ^b
Acetone	67-64-1	0.05
Benzene	71-43-2	0.06
n-Butylbenzene ^f	104-51-8	12
Carbon tetrachloride ^f	56-23-5	0.76
Chlorobenzene	108-90-7	1.1
Chloroform	67-66-3	0.37
Ethylbenzene ^f	100-41-4	1
Hexachlorobenzene ^f	118-74-1	0.33 ^b
Methyl ethyl ketone	78-93-3	0.12
Methyl tert-butyl ether ^f	1634-04-4	0.93
Methylene chloride	75-09-2	0.05

Table 375-6.8(a): Unrestricted Use Soil Cleanup Objectives

Contaminant	CAS Number	Unrestricted Use
n - Propylbenzene ^f	103-65-1	3.9
sec-Butylbenzene ^f	135-98-8	11
tert-Butylbenzene ^f	98-06-6	5.9
Tetrachloroethene	127-18-4	1.3
Toluene	108-88-3	0.7
Trichloroethene	79-01-6	0.47
1,2,4-Trimethylbenzene ^f	95-63-6	3.6
1,3,5-Trimethylbenzene ^f	108-67-8	8.4
Vinyl chloride ^f	75-01-4	0.02
Xylene (mixed)	1330-20-7	0.26

All soil cleanup objectives (SCOs) are in parts per million (ppm).

Footnotes

^a The SCOs for unrestricted use were capped at a maximum value of 100 ppm. See [Technical Support Document \(TSD\)](#), section 9.3.

^b For constituents where the calculated SCO was lower than the contract required quantitation limit (CRQL), the CRQL is used as the Track 1 SCO value.

^c For constituents where the calculated SCO was lower than the rural soil background concentration, as determined by the Department and Department of Health rural soil survey, the rural soil background concentration is used as the Track 1 SCO value for this use of the site.

^d SCO is the sum of endosulfan I, endosulfan II and endosulfan sulfate.

^e The SCO for this specific compound (or family of compounds) is considered to be met if the analysis for the total species of this contaminant is below the specific SCO.

^f Protection of ecological resources SCOs were not developed for contaminants identified in Table 375-6.8(b) with “NS”. Where such contaminants appear in Table 375-6.8(a), the applicant may be required by the Department to calculate a protection of ecological resources SCO according to the TSD.

(b) Restricted use soil cleanup objectives.

Table 375-6.8(b): Restricted Use Soil Cleanup Objectives

Contaminant	CAS Number	Protection of Public Health				Protection of Ecological Resources	Protection of Ground-water
		Residential	Restricted-Residential	Commercial	Industrial		
Metals							
Arsenic	7440-38-2	16 ^f	16 ^f	16 ^f	16 ^f	13 ^f	16 ^f
Barium	7440-39-3	350 ^f	400	400	10,000 ^d	433	820
Beryllium	7440-41-7	14	72	590	2,700	10	47
Cadmium	7440-43-9	2.5 ^f	4.3	9.3	60	4	7.5
Chromium, hexavalent ^h	18540-29-9	22	110	400	800	1 ^e	19
Chromium, trivalent ^h	16065-83-1	36	180	1,500	6,800	41	NS
Copper	7440-50-8	270	270	270	10,000 ^d	50	1,720
Total Cyanide ^h		27	27	27	10,000 ^d	NS	40
Lead	7439-92-1	400	400	1,000	3,900	63 ^f	450
Manganese	7439-96-5	2,000 ^f	2,000 ^f	10,000 ^d	10,000 ^d	1600 ^f	2,000 ^f
Total Mercury		0.81 ^j	0.81 ^j	2.8 ^j	5.7 ^j	0.18 ^f	0.73
Nickel	7440-02-0	140	310	310	10,000 ^d	30	130
Selenium	7782-49-2	36	180	1,500	6,800	3.9 ^f	4 ^f
Silver	7440-22-4	36	180	1,500	6,800	2	8.3
Zinc	7440-66-6	2200	10,000 ^d	10,000 ^d	10,000 ^d	109 ^f	2,480
PCBs/Pesticides							
2,4,5-TP Acid (Silvex)	93-72-1	58	100 ^a	500 ^b	1,000 ^c	NS	3.8
4,4'-DDE	72-55-9	1.8	8.9	62	120	0.0033 ^e	17
4,4'-DDT	50-29-3	1.7	7.9	47	94	0.0033 ^e	136
4,4'-DDD	72-54-8	2.6	13	92	180	0.0033 ^e	14
Aldrin	309-00-2	0.019	0.097	0.68	1.4	0.14	0.19
alpha-BHC	319-84-6	0.097	0.48	3.4	6.8	0.04 ^g	0.02
beta-BHC	319-85-7	0.072	0.36	3	14	0.6	0.09
Chlordane (alpha)	5103-71-9	0.91	4.2	24	47	1.3	2.9

Table 375-6.8(b): Restricted Use Soil Cleanup Objectives

Contaminant	CAS Number	Protection of Public Health				Protection of Ecological Resources	Protection of Ground-water
		Residential	Restricted-Residential	Commercial	Industrial		
delta-BHC	319-86-8	100 ^a	100 ^a	500 ^b	1,000 ^c	0.04 ^g	0.25
Dibenzofuran	132-64-9	14	59	350	1,000 ^c	NS	210
Dieldrin	60-57-1	0.039	0.2	1.4	2.8	0.006	0.1
Endosulfan I	959-98-8	4.8 ⁱ	24 ⁱ	200 ⁱ	920 ⁱ	NS	102
Endosulfan II	33213-65-9	4.8 ⁱ	24 ⁱ	200 ⁱ	920 ⁱ	NS	102
Endosulfan sulfate	1031-07-8	4.8 ⁱ	24 ⁱ	200 ⁱ	920 ⁱ	NS	1,000 ^c
Endrin	72-20-8	2.2	11	89	410	0.014	0.06
Heptachlor	76-44-8	0.42	2.1	15	29	0.14	0.38
Lindane	58-89-9	0.28	1.3	9.2	23	6	0.1
Polychlorinated biphenyls	1336-36-3	1	1	1	25	1	3.2
Semivolatiles							
Acenaphthene	83-32-9	100 ^a	100 ^a	500 ^b	1,000 ^c	20	98
Acenaphthylene	208-96-8	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	107
Anthracene	120-12-7	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	1,000 ^c
Benz(a)anthracene	56-55-3	1 ^f	1 ^f	5.6	11	NS	1 ^f
Benzo(a)pyrene	50-32-8	1 ^f	1 ^f	1 ^f	1.1	2.6	22
Benzo(b)fluoranthene	205-99-2	1 ^f	1 ^f	5.6	11	NS	1.7
Benzo(g,h,i)perylene	191-24-2	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	1,000 ^c
Benzo(k)fluoranthene	207-08-9	1	3.9	56	110	NS	1.7
Chrysene	218-01-9	1 ^f	3.9	56	110	NS	1 ^f
Dibenz(a,h)anthracene	53-70-3	0.33 ^e	0.33 ^e	0.56	1.1	NS	1,000 ^c
Fluoranthene	206-44-0	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	1,000 ^c
Fluorene	86-73-7	100 ^a	100 ^a	500 ^b	1,000 ^c	30	386
Indeno(1,2,3-cd)pyrene	193-39-5	0.5 ^f	0.5 ^f	5.6	11	NS	8.2
m-Cresol	108-39-4	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	0.33 ^e
Naphthalene	91-20-3	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	12

Table 375-6.8(b): Restricted Use Soil Cleanup Objectives

Contaminant	CAS Number	Protection of Public Health				Protection of Ecological Resources	Protection of Ground-water
		Residential	Restricted-Residential	Commercial	Industrial		
o-Cresol	95-48-7	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	0.33 ^e
p-Cresol	106-44-5	34	100 ^a	500 ^b	1,000 ^c	NS	0.33 ^e
Pentachlorophenol	87-86-5	2.4	6.7	6.7	55	0.8 ^e	0.8 ^e
Phenanthrene	85-01-8	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	1,000 ^c
Phenol	108-95-2	100 ^a	100 ^a	500 ^b	1,000 ^c	30	0.33 ^e
Pyrene	129-00-0	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	1,000 ^c
Volatiles							
1,1,1-Trichloroethane	71-55-6	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	0.68
1,1-Dichloroethane	75-34-3	19	26	240	480	NS	0.27
1,1-Dichloroethene	75-35-4	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	0.33
1,2-Dichlorobenzene	95-50-1	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	1.1
1,2-Dichloroethane	107-06-2	2.3	3.1	30	60	10	0.02 ^f
cis-1,2-Dichloroethene	156-59-2	59	100 ^a	500 ^b	1,000 ^c	NS	0.25
trans-1,2-Dichloroethene	156-60-5	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	0.19
1,3-Dichlorobenzene	541-73-1	17	49	280	560	NS	2.4
1,4-Dichlorobenzene	106-46-7	9.8	13	130	250	20	1.8
1,4-Dioxane	123-91-1	9.8	13	130	250	0.1 ^e	0.1 ^e
Acetone	67-64-1	100 ^a	100 ^b	500 ^b	1,000 ^c	2.2	0.05
Benzene	71-43-2	2.9	4.8	44	89	70	0.06
Butylbenzene	104-51-8	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	12
Carbon tetrachloride	56-23-5	1.4	2.4	22	44	NS	0.76
Chlorobenzene	108-90-7	100 ^a	100 ^a	500 ^b	1,000 ^c	40	1.1
Chloroform	67-66-3	10	49	350	700	12	0.37
Ethylbenzene	100-41-4	30	41	390	780	NS	1
Hexachlorobenzene	118-74-1	0.33 ^e	1.2	6	12	NS	3.2
Methyl ethyl ketone	78-93-3	100 ^a	100 ^a	500 ^b	1,000 ^c	100 ^a	0.12

Table 375-6.8(b): Restricted Use Soil Cleanup Objectives

Contaminant	CAS Number	Protection of Public Health				Protection of Ecological Resources	Protection of Groundwater
		Residential	Restricted-Residential	Commercial	Industrial		
Methyl tert-butyl ether	1634-04-4	62	100 ^a	500 ^b	1,000 ^c	NS	0.93
Methylene chloride	75-09-2	51	100 ^a	500 ^b	1,000 ^c	12	0.05
n-Propylbenzene	103-65-1	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	3.9
sec-Butylbenzene	135-98-8	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	11
tert-Butylbenzene	98-06-6	100 ^a	100 ^a	500 ^b	1,000 ^c	NS	5.9
Tetrachloroethene	127-18-4	5.5	19	150	300	2	1.3
Toluene	108-88-3	100 ^a	100 ^a	500 ^b	1,000 ^c	36	0.7
Trichloroethene	79-01-6	10	21	200	400	2	0.47
1,2,4-Trimethylbenzene	95-63-6	47	52	190	380	NS	3.6
1,3,5- Trimethylbenzene	108-67-8	47	52	190	380	NS	8.4
Vinyl chloride	75-01-4	0.21	0.9	13	27	NS	0.02
Xylene (mixed)	1330-20-7	100 ^a	100 ^a	500 ^b	1,000 ^c	0.26	1.6

All soil cleanup objectives (SCOs) are in parts per million (ppm).

NS=Not specified. See [Technical Support Document \(TSD\)](#).

Footnotes

^a The SCOs for residential, restricted-residential and ecological resources use were capped at a maximum value of 100 ppm. See TSD section 9.3.

^b The SCOs for commercial use were capped at a maximum value of 500 ppm. See TSD section 9.3.

^c The SCOs for industrial use and the protection of groundwater were capped at a maximum value of 1000 ppm. See TSD section 9.3.

^d The SCOs for metals were capped at a maximum value of 10,000 ppm. See TSD section 9.3.

^e For constituents where the calculated SCO was lower than the contract required quantitation limit (CRQL), the CRQL is used as the SCO value.

^f For constituents where the calculated SCO was lower than the rural soil background concentration as determined by the Department and Department of Health rural soil survey, the rural soil background concentration is used as the Track 2 SCO value for this use of the site.

^g This SCO is derived from data on mixed isomers of BHC.

^h The SCO for this specific compound (or family of compounds) is considered to be met if the analysis for the total species of this contaminant is below the specific SCO.

ⁱ This SCO is for the sum of endosulfan I, endosulfan II, and endosulfan sulfate.

^j This SCO is the lower of the values for mercury (elemental) or mercury (inorganic salts). See TSD Table 5.6-1.

CP-51 / Soil Cleanup Guidance

New York State Department of Environmental Conservation

DEC Policy

Issuing Authority: Alexander B. Grannis, Commissioner

Date Issued: October 21, 2010

Latest Date Revised:

I. Summary

This policy provides the framework and procedures for the selection of soil cleanup levels appropriate for each of the remedial programs in the New York State Department of Environmental Conservation (DEC) Division of Environmental Remediation (DER). This policy applies to the Inactive Hazardous Waste Disposal Site Remedial Program, known as the State Superfund Program (SSF); Brownfield Cleanup Program (BCP); Voluntary Cleanup Program (VCP); Environmental Restoration Program (ERP); Spill Response Program - Navigation Law (NL) section 176 (SRP); and the Resource Conservation and Recovery Act (RCRA) Corrective Action Program. It replaces *Technical and Administrative Guidance Memorandum (TAGM) 4046: Determination of Soil Cleanup Objectives and Cleanup Levels* (January 24, 1994); the *Petroleum Site Inactivation and Closure Memorandum* (February 23, 1998); and Sections III and IV of *Spill Technology and Remediation Series (STARS) #1* (August 1992).

This document is used in conjunction with the applicable statutes, regulations and guidance. Site-specific soil cleanup levels, determined in accordance with this guidance, are only applied after:

- the site, or area of concern, is fully investigated to determine the nature and extent of contamination;
- all sources of contamination are addressed consistent with the hierarchy provided in 6 NYCRR 375-1.8(c) or consistent with the RCRA Corrective Action Program (as appropriate);
- groundwater, if contaminated, has been evaluated for appropriate remedial actions consistent with 6 NYCRR 375-1.8(d) or consistent with the RCRA Corrective Action Program (as appropriate); and
- impacts on adjacent residential properties, surface water, aquatic ecological resources are evaluated, as well as indoor air, soil vapor, vapor intrusion and other appropriate media.

II. Policy

It is DEC's policy, consistent with applicable statutes and regulations, that all remedies will be protective of public health and the environment. DEC's preference is that remedial programs, including the selection of soil cleanup levels, be designed such that the performance standard results in the implementation of a permanent remedy resulting in no future land use restrictions. However, some of

Table 1

**Supplemental Soil Cleanup Objectives
(ppm)**

Contaminant	CAS Number	Residential	Restricted Residential	Commercial	Industrial	Protection of Ecological Resources	Protection of Groundwater
METALS							
Aluminum	7429-90-5					10,000 ^{a,b}	
Antimony	7440-36-0					12 ^c	
Boron	7440-42-8					0.5	
Calcium	7440-70-2					10,000 ^{a,b}	
Cobalt	7440-48-4	30				20	
Iron	7439-89-6	2,000					
Lithium	7439-93-2					2	
Molybdenum	7439-98-7					2	
Technetium	7440-26-8					0.2	
Thallium	7440-28-0					5 ^c	
Tin	7440-31-5					50	
Uranium	7440-61-1					5	
Vanadium	7440-62-2	100 ^a				39 ^b	
PESTICIDES							
Biphenyl	92-52-4					60	
Chlordecone (Kepone)	143-50-0					0.06	
Dibenzofuran	132-64-9						6.2
2,4-D (2,4-Dichloro-phenoxyacetic acid)	94-75-7	100 ^a					0.5
Furan	110-00-9					600	
Gamma Chlordane	5103-74-2	0.54					14
Heptachlor Epoxide	1024-57-3	0.077					0.02
Methoxychlor	72-43-5	100 ^a				1.2	900

Contaminant	CAS Number	Residential	Restricted Residential	Commercial	Industrial	Protection of Ecological Resources	Protection of Ground-water
Parathion	56-38-2	100 ^a					1.2
2,4,5-T	93-76-5	100 ^a					1.9
2,3,7,8-TCDD	1746-01-6					0.000001	
2,3,7,8-TCDF	51207-31-9					0.000001	
SEMIVOLATILE ORGANIC COMPOUNDS							
Aniline	62-53-3	48	100 ^a	500 ^a	1000 ^a		0.33 ^b
Bis(2-ethylhexyl) phthalate	117-81-7	50				239	435
Benzoic Acid	65-85-0	100 ^a					2.7
Butylbenzyl-phthalate	85-68-7	100 ^a					122
4-Chloroaniline	106-47-8	100 ^a					0.22
Chloroethane	75-00-3						1.9
2-Chlorophenol	95-57-8	100 ^a				0.8	
3-Chloroaniline	108-42-9					20	
3-Chlorophenol	108-43-0					7	
Di-n-butyl-phthalate	84-74-2	100 ^a				0.014	8.1
2,4-Dichlorophenol	120-83-2	100 ^a				20	0.40
3,4-Dichlorophenol	95-77-2					20	
Diethylphthalate	84-66-2	100 ^a				100	7.1
Di-n-hexyl-phthalate	84-75-3					0.91	
2,4-Dinitrophenol	51-28-5	100 ^a				20	0.2
Dimethylphthlate	131-11-3	100 ^a				200	27
Di-n-octylphthlate	117-84-0	100 ^a					120
1,2,3,6,7,8-HCDF	57117-44-9					0.00021	
Hexachloro-benzene	118-74-1	0.41					1.4
2,6-Dinitrotoluene	606-20-2	1.03					1.0
Isophorone	78-59-1	100 ^a					4.4

Contaminant	CAS Number	Residential	Restricted Residential	Commercial	Industrial	Protection of Ecological Resources	Protection of Ground-water
4-methyl-2-pentanone	108-10-1						1.0
2-methyl-naphthalene	91-57-6	0.41					36.4
2-Nitroaniline	88-74-4						0.4
3-Nitroaniline	99-09-2						0.5
Nitrobenzene	98-95-3	3.7	15	69	140	40	0.17 ^b
2-Nitrophenol	88-75-5					7	0.3
4-Nitrophenol	100-02-7					7	0.1
Pentachloroaniline	527-20-8					100	
2,3,5,6-Tetrachloroaniline	3481-20-7					20	
2,3,4,5-Tetrachlorophenol	4901-51-3					20	
2,4,5-Trichloroaniline	636-30-6					20	
2,4,5-Trichlorophenol	95-95-4	100 ^a				4	0.1
2,4,6-Trichlorophenol	88-06-2					10	
VOLATILE ORGANIC COMPOUNDS							
2-Butanone	78-93-3	100 ^a					0.3
Carbon Disulfide	75-15-0	100 ^a					2.7
Chloroacetamide	79-07-2					2	
Dibromochloromethane	124-48-1					10	
2,4-Dichloro aniline	554-00-7					100	
3,4-Dichloroaniline	95-76-1					20	
1,2-Dichloropropane	78-87-5					700	
1,3-Dichloropropane	142-28-9						0.3
2,6-Dinitrotoluene	606-20-2	1.03					0.17 ^b
Ethylacetate	141-78-6					48	

Contaminant	CAS Number	Residential	Restricted Residential	Commercial	Industrial	Protection of Ecological Resources	Protection of Ground-water
4-methyl-2-pentanone	108-10-1						1.0
113 Freon (1,1,2- TFE)	76-13-1	100 ^a					6
isopropylbenzene	98-82-8	100 ^a					2.3
p-isopropyltoluene	99-87-6						10
Hexachlorocyclopentadiene	77-47-4					10	
Methanol	67-56-1					6.5	
N-nitrosodiphenylamine	86-30-6					20	
Pentachlorobenzene	608-93-5					20	
Pentachloronitrobenzene	82-68-8					10	
Styrene	100-42-5					300	
1,2,3,4-Tetrachlorobenzene	634-66-2					10	
1,1,2,2-Tetrachloroethane	79-34-5	35					0.6
1,1,2,2-Tetrachloroethylene	127-18-4					2	
1,2,3-Trichlorobenzene	87-61-6					20	
1,2,4-Trichlorobenzene	120-82-1					20	3.4
1,2,3-Trichloropropane	96-18-4	80					0.34

^a SCOs for organic contaminants (volatile organic compounds, semivolatile organic compounds, and pesticides) are capped at 100 ppm for residential use, 500 ppm for commercial use, 1000 ppm for industrial use. SCOs for metals are capped at 10,000 ppm.

^b Based on rural background study

^c SCO limited by contract required quantitation limit.

Table 2**Soil Cleanup Levels for Gasoline Contaminated Soils**

Contaminant	CAS Registry Number	Soil Cleanup Level (ppm)
Benzene	71-43-2	0.06
n-Butylbenzene	104-51-8	12.0
sec-Butylbenzene	135-98-8	11.0
Ethylbenzene	100-41-4	1.0
Isopropylbenzene	98-82-8	2.3
p-Isopropyltoluene	99-87-6	10.0
Methyl-Tert-Butyl-Ether	1634-04-4	0.93
Naphthalene	91-20-3	12.0
n-Propylbenzene	103-65-1	3.9
Tert-Butylbenzene	98-06-6	5.9
Toluene	108-88-3	0.7
1,2,4-Trimethylbenzene	95-63-6	3.6
1,3,5-Trimethylbenzene	108-67-8	8.4
Xylene (Mixed)	1330-20-7	0.26

Table 3**Soil Cleanup Levels for Fuel Oil Contaminated Soil**

Contaminant	CAS Registry Number	Soil Cleanup Level (ppm)
Acenaphthene	83-32-9	20
Acenaphthylene	208-96-8	100
Anthracene	120-12-7	100
Benz(a)Anthracene	56-55-3	1.0
Dibenzo(a,h)Anthracene	53-70-3	0.33
Benzene	71-43-2	0.06
n-Butylbenzene	104-51-8	12.0
sec-Butylbenzene	135-98-8	11.0
Tert-Butylbenzene	98-06-6	5.9
Chrysene	218-01-9	1.0
Ethylbenzene	100-41-4	1.0
Fluoranthene	206-44-0	100
Benzo(b)Fluoranthene	205-99-2	1.0
Benzo(k)Fluoranthene	207-08-9	0.8
Fluorene	86-73-7	30
Isopropylbenzene	98-82-8	2.3
p-Isopropyltoluene	99-87-6	10.0
Naphthalene	91-20-3	12.0
n-Propylbenzene	103-65-1	3.9
Benzo(g,h,i)Perylene	191-24-2	100
Phenanthrene	85-01-8	100
Pyrene	129-00-0	100
Benzo(a)Pyrene	50-32-8	1.0
Indeno(1,2,3-cd)Pyrene	193-39-5	0.5
1,2,4-Trimethylbenzene	95-63-6	3.6
1,3,5-Trimethylbenzene	108-67-8	8.4
Toluene	108-88-3	0.7
Xylene (Mixed)	1330-20-7	0.26

ATTACHMENT D

Resumes

CURRENT POSITION: ASSISTANT PROJECT MANAGER

PROFESSIONAL SUMMARY

Erick Salazar serves as Assistant Project Manager for environmental site assessments and Phase II technical environmental investigations. His responsibilities include: investigating site histories, conducting facility inspections, reviewing regulatory agency records, documenting facility compliance with relevant State and Federal regulations, and preparing reports. He assists with Phase II technical environmental investigations and fieldwork including implementation of community air monitoring plans (CAMP), collecting soil and water samples and tank removal oversight.

Mr. Salazar has experience in the implementation of CAMP monitoring, personal sampling for lead and dust of workers, coordinating pre-demolition C&D waste inventory as part of Sandy relief work on Staten Island, and providing oversight of site remedial activities on rural properties.

PROFESSIONAL EXPERIENCE

Mr. Salazar has experience in the implementation of CAMP monitoring, personal sampling for lead and dust of workers, coordinating pre-demolition C&D waste inventory as part of Sandy relief work on Staten Island, and providing oversight of site remedial activities on rural properties.

Mr. Salazar's experience with Health and Safety services include:

- Complete OSHA training and three years' experience of Sites handling regulated materials as well as hazardous and non-hazardous wastes.
- Preparation of Environmental Health & Safety Plan for (EHASP) for debris removal and soil sampling project in Ulster County, New York.
- Assistance in the preparation of EHASPs for NYSDEC sites in Dutchess and Westchester Counties.
- Implementation of CAMP at sites in Dutchess, Ulster, Bronx and Queens Counties, including preparation of status reports, preparation of incident reports, and communication with involved regulatory agencies.
- Collection/analysis of media samples (air, water and soil) per requirements of the EHASP and/or remedial work plans.

EDUCATION:

- BS, Biology, State University at New Paltz, NY

REGISTRATIONS / CERTIFICATIONS:

- OSHA, 40-hr Hazardous Waste Operations & Emergency Response Health & Safety Certification
- OSHA, 10-hr General Construction Industry Training & Certification

CURRENT POSITION: MANAGER, ENVIRONMENTAL CONSULTING

PROFESSIONAL SUMMARY

Richard Hooker serves as Senior Project Manager for investigative and remedial projects including NYSDEC and OER Brownfields sites, Phase II investigations, and environmental management of construction projects. He also prepares and evaluates interdisciplinary, comprehensive environmental impact assessment reviews (NEPA, SEQR and CEQR) and has a particular expertise in noise issues. Mr. Hooker develops investigative and remedial work plans, health and safety plans, performs fieldwork, and prepares technical reports. He works with regulatory authorities and subcontractors including construction personnel, waste repositories and haulage contractors, laboratories and drillers. His responsibilities include: designing noise studies, investigating site histories, and document reviews, cost benefit analysis of remedial alternatives, overseeing excavations and in situ remediation, sampling, sample data evaluation, report preparation, and obtaining regulatory closure. He has extensive experience of sampling and sample collection protocols for soil, vapor, indoor air, sediment, and groundwater and has worked to remediate a wide range of environmental contaminants including petroleum, heavy metals, PCBs, and solvents.

Mr. Hooker holds a Ph.D. from the University of St. Andrews, St. Andrews, Scotland and a BA from Staffordshire University, Stoke-on-Trent, England. Prior to relocating to the Hudson Valley, he served as an Assistant Professor at the University of Glasgow, Scotland.

PROFESSIONAL EXPERIENCE

3475 Third Avenue, Bronx, NY—Investigated and remediated this former manufacturing facility to NYSDEC Brownfields to Track 1 cleanup standards. This site was the first project in the OER Jumpstart program established to assist cleanup on government supported affordable and supportive housing projects in NYC. Under this program OER sponsored enrollment in the NYS Brownfield Cleanup Program. Work on this trailblazing project required liaising with OER and NYSDEC Region 2 to ensure documentation met the requirements of both agencies. Certificate of Completion secured in 2016.

Former A.C. Dutton Lumber Yard, Dutchess County, NY—Documented hazardous concentrations of arsenic and chromium in soils and concrete surfaces at this NYSDEC Brownfields site contaminated by the historical pressure treatment of lumber. Developed a Workplan for site remediation and directed environmental restoration activities, including: characterization, excavation and removal of hazardous soils, scarification concrete warehouse floors, removal aboveground and underground chemical and petroleum storage tanks.

Lincoln Place, Brooklyn, NY—performed CEQR, SEQR and NEPA reviews including shadow and noise studies for this site prior to development. Prepared Remedial Workplan and oversaw remediation of metals-contaminated soils during construction and implemented remedy for the site including SSDS system installation, vapor barrier, and installation of composite cover system. Prepared FER and obtained NYCHPD and NYCDEP closeout for the site.

Grace Terrace, Mount Vernon, NY—oversaw remediation and obtained NYSDEC Spill file closure after a previously unknown UST and associated petroleum contaminated soil were encountered during construction

excavations. Coordinated with the GC to ensure appropriate cleanup was performed without delaying the construction schedule. Remedial actions included characterization and appropriate off-site disposition of petroleum contaminated soil and groundwater, application of chemical oxidation treatment, installation vapor barrier and active SSSS system.

Former Fur Processing Facility, Bronx, NY—Documented the presence of chlorinated hydrocarbon, petroleum, and metals contamination beneath and/or near a former industrial structure. Coordinated the sampling and removal of multiple drums of hazardous and non-hazardous material from the structure and secured NYCDEP approval. Developed a Workplan for site remediation and directed environmental restoration activities, including: excavation and removal of both aboveground and underground storage tanks, removal of contaminated soils, installation of a barrier layer soil cap, and pre-demolition removal of asbestos materials.

Jamaica Hospital Medical Center, Queens, NY—Coordinated and supervised the removal of two, large underground storage tanks and documented site conditions through soil and groundwater sampling. Secured NYSDEC approval of PBS tank closure and registration requirements.

EDUCATION:

- Ph.D., University of St. Andrews, Scotland
- BA, Staffordshire University, England

REGISTRATIONS / CERTIFICATIONS

- OSHA-40 Hazwoper
- OSHA-10 Construction

CURRENT POSITION: DIRECTOR OF ENVIRONMENTAL INVESTIGATIONS

PROFESSIONAL SUMMARY

Scott Spitzer serves as Director of Environmental Investigations, overseeing the technical elements of Phase I and Phase II technical environmental investigations and remedial projects, including Brownfield sites. Mr. Spitzer supervises all GBTS field staff and reviews all documents prepared by GBTS to ensure consistency and technical accuracy.

His responsibilities associated with the preparation of site assessments include: investigating site histories, conducting facility inspections, reviewing regulatory agency records, documenting facility compliance with relevant State and Federal regulations, and preparing reports. As project manager for complex technical environmental investigations (including sites currently on the NYSDEC Registry of Inactive Hazardous Waste Sites), Mr. Spitzer is involved with: coordinating subcontractors; overseeing fieldwork; designing and implementing material, soil, and water sampling plans, preparing technical reports and interfacing with regulatory agency personnel.

Mr. Spitzer has 15 years' experience in the preparation of Phase I, Phase II and Brownfields investigations and in the management of complex remediation projects. He is knowledgeable in both New York State and Federal environmental regulations and has an understanding of a broad range of remedial technologies. Mr. Spitzer studied environmental science at SUNY Purchase and holds a BS in Biology from SUNY at Stony Brook, Stony Brook, New York.

PROFESSIONAL EXPERIENCE

Former NuHart Plastics Manufacturing Site, Brooklyn, NY: GBTS conducted a complex remedial investigation of a NYSDEC Class 2 Inactive Hazardous Waste Disposal ("Superfund") site, where a plume of liquid phthalates and chlorinated solvents had impacted groundwater. Extensive sampling was conducted of both on- and off-site soil, soil vapor and groundwater, and phthalates were removed from recovery wells as an interim remedial measure. A Remedial Investigation Report was completed, allowing the site owner to move create a Remedial Design Document.

Scenic Hudson Land Trust, Inc., Beacon Waterfront Project, Beacon, NY: GBTS conducted soil and groundwater investigations on a former MOSF and adjacent scrap yard. Projects involved soil remediation of both petroleum and PCB-contaminated soils and long-term groundwater monitoring. Both projects were classified as Voluntary Clean-Up projects by the NYSDEC and closure status was attained.

Sakmann Restaurant Corporation Site, Fort Montgomery, NY: Conducted Phase I Environmental Site Assessment and Phase II Subsurface Investigations for former filling station and automotive repair garage contaminated by solvent and waste-oil discharges to an on-site drywell. Designed and implemented a sampling plan for soils impacted by chlorinated hydrocarbons, petroleum, and metals. Created Work Plan (in coordination with the NYSDEC Voluntary Cleanup Program) for remediation of on-site contamination and long-term sampling of on-site groundwater monitoring wells.

Staten Island Marina Site, Staten Island, NY: Conducted Phase I Environmental Site Assessment and Phase II Subsurface Investigation for an active marine facility engaged in boat painting and engine maintenance activities. Coordinated the delineation of metals contamination over a three-acre area and analyzed potential impacts from onsite fill materials. Submitted remedial and budgetary analysis in support of regulatory agency approval for conversion of boatyard into a public park.

Octagon House Development Site, Roosevelt Island, NY: Conducted Phase I Environmental Site Assessment and Phase II Subsurface Investigations at the former site of a large, urban hospital. Interpreted the results of geotechnical studies, extended test pits, and conducted extensive soil sampling, to document subsurface soil conditions in support of client's application to the U.S. Housing and Urban Development Agency (HUD). Created Work Plan (in coordination with the NYCDEP Office of Environmental Planning and Assessment) for site-wide remediation of contaminated soils and secured NYCDEP approval for site remediation as required by HUD.

Camp Glen Gray Boy Scout Facility, Mahwah, NJ: Conducted Phase I Environmental Site Assessment and Phase II Subsurface Investigations at an approximately 800-acre campground containing numerous structures. Documented subsurface soil conditions at the locations of aboveground and underground storage tanks, and delineated lead contamination at a former firing range. Assisted in design and implementation of remediation plans for removal of petroleum and lead contaminated soils, and obtained NJDEP approvals.

EDUCATION:

- BS, Biology, SUNY at Stony Brook, NY

REGISTRATIONS / CERTIFICATIONS

- OSHA, 40-hr. Hazardous Waste Operations & Emergency Response Health & Safety Certification
- OSHA, 10-hr. General Construction Industry Training and Certification

CURRENT POSITION: ASSISTANT PROJECT MANAGER

PROFESSIONAL SUMMARY

Victoria Panico serves as an Assistant Project Manager for environmental site assessments, Phase II environmental investigations and NYC OER remediation projects. Ms. Panico develops investigative and remedial work plans, performs fieldwork, prepares technical reports, and coordinates with subcontractors including construction personnel, laboratories and drillers. Her responsibilities include: investigating site histories, conducting facility inspections, reviewing regulatory agency records, communications with stakeholders (client, construction manager, and regulatory agencies), preparation, submission and approval of reports, and obtain regulatory closure. She conducts Phase II technical environmental investigations and fieldwork including implementation of community air monitoring plans (CAMP), and sampling of soil, soil vapor and groundwater.

Ms. Panico has experience preparing Remedial Action Reports, conducting Site Management Plan annual inspections and reporting, evaluating the effectiveness of SSD systems and providing oversight of site remedial activities on rural properties.

PROFESSIONAL EXPERIENCE

Gallagher Bassett Technical Services

Phase I Environmental Site Assessments (ESAs)

Completed over 75 Phase I ESAs including residential, commercial, industrial and agricultural properties. Responsibilities include: investigating site histories, conducting facility inspections, reviewing regulatory agency records, documenting facility compliance with relevant State and Federal regulations, and preparing reports.

Phase II ESAs and Site Investigations

Completed/assisted with over 15 Phase II sub-surface investigations. Experience sampling and sample collection for soil, soil vapor and groundwater. Ms. Panico works with regulatory authorities and subcontractors including construction personnel, waste repositories, laboratories and drillers. She has also experience conducting waste characterization sampling and collection of end-point samples.

NYC Voluntary Cleanup Program (VCP) Sites

Serves as Assistant Project Manager for NYC Voluntary Cleanup Program (VCP) remediation and redevelopment projects, which includes assisting in the design of remedial actions, oversight of remedial activities, and implementing remedies including installation of SSD systems, vapor barriers, and composite cover systems. Responsibilities include: preparation of RIRs and RAWPs, on-going project management and remedial oversight, ensuring compliance with the remedial action, communications with stakeholders (client, construction manager, and regulatory agencies), facilitation of spill closure, preparation and submission of daily, weekly, and monthly status reports, client updates and satisfaction, preparation of RARs, and obtaining regulatory closure.

EDUCATION:

- BS, Environmental Science, Science Emphasis, Marist College, Poughkeepsie, NY

REGISTRATIONS / CERTIFICATIONS

- 40-hour OSHA HAZWOPER & annual refresher training
- 30-hour OSHA Construction
- 8-hour Fall Prevention
- 4-hour Supported Scaffold User & refresher

Daniel A. Bellucci, P.E.

30 South Main Street, Unit 204 • Ipswich, Massachusetts 01938 • (845) 803-4347 • dan@coredowndrilling.com

Education & Certifications

B.S. Environmental Engineering University of New Hampshire 2011
Professional Engineer (Environmental) MA, NY, NH
40 Hour HAZWOPER Training Certification- 29 CFR 1910.120
Understanding the MCP Course – UMass Lowell (Spring 2019)
ASTM E 1527 Phase I Environmental Site Assessment (ESA) Training, 2012
National Groundwater Association - Certified Well Driller (CWD)
Dig Safe Certified Excavator in Safe Digging Practices, 2019-2024

Employment History

Owner/ Partner Bellucci Engineering, PLLC (November 2019)

Owner/ Partner Core Down Drilling LLC, Brewster, NY (January 2013 to present)

Environmental Engineer, P.E. EBI Consulting, Site Investigation and Remediation (SIR) Group, Burlington, MA (October 2012 to July 2019)

- Project Management including site investigation proposal preparation, management of junior field staff and client correspondence, project invoicing (fixed fee)
- Extensive field experience conducting hundreds of Phase II Subsurface Investigations throughout Eastern US
- Sub-slab depressurization system pilot testing, design, installation, system startup and ongoing operation, maintenance and sampling
- Field and project management experience associated with the following: soil, groundwater, soil vapor, and surface water sampling / monitoring, UST closure, monitoring well elevation surveys, monitoring well installation and abandonments, operation and maintenance of groundwater pump and treat systems; construction site air monitoring, soil management plans, site plan creation using AutoCAD
- Annual US Airforce base stormwater and wastewater sampling
- Broad spectrum of Real Estate Due Diligence projects including Phase I Environmental Site Assessments (ESAs), State File Reviews, Telecommunications ESAs, Environmental Protection Plans, Soil Management Plans, Release Abatement Measure Plans and Spill Prevention Control and Countermeasure Plans
- Experience with Massachusetts Contingency Plan including sampling protocol, RAM Plans, RAM Completion Reports, Permanent Solution Reports, LRA Reports, Downgradient Property Status Reports and RAM Status Reports and eDEP file submittals

Environmental Engineer HydroEnvironmental Solutions Inc., Somers, NY (November 2011 to October 2012)

- Oversight and project management of remediation projects including petroleum release sites, industrial redevelopment soil characterization and disposal, and construction dewatering discharge permitting
- Detailed field reporting, soil and water sampling and analysis of laboratory data
- Operator: Geoprobe[®] Direct Push macro core sampling drill rig
- Invoice review, cost estimates, spill closure report compilation, review and output
- O&M of pumps, carbon filtration units, and oil/water separators
- Spill Prevention, Control and Countermeasure Plan compilation and review

Field Technician Tri-State Environmental Services Inc., Hawthorne, NY (September 2011 to November 2011)

- Hazardous materials handling, transportation, and disposal procedures
-

Related Educational Experience

- Senior Research Presentation on Microbial Degradation of PCB's in the Hudson River
 - Advanced Water Treatment Facility Full Scale Team Design
 - Membrane filtration design to optimize pathogen removal prior to disinfection, decreasing disinfection byproducts
-

Engineering & Business Software Experience

AutoCAD, Win Log/ Strata Explorer, Microsoft Office, Sales Force/ Financial Force

Anthony J. Zoccolillo

Objective

Combine my existing strengths based on 25+ years of environmental and manufacturing information management experience to implement enterprise data management and business intelligence and to continue to expand my knowledgebase.

Education

1980 - 1987 Syracuse University Syracuse, New York

Bachelor of Science in Chemistry

- Additional coursework in the System Information Science program including Basic, Pascal and Scheme programming languages.

1977 - 1980 Rochester Institute of Technology Rochester, New York

Major: Chemistry

- Includes coursework in the programming languages APL and FORTRAN.

Professional experience

2011 - Present Oneida Nation Enterprises Verona, New York

Database Administrator

- Responsible for administrative support of Oracle (10g and 11g) and SQL Server (2005, 2008, 2008 R2, 2012, 2014) databases including database structural design, procedural scripting and developing BI analysis tools. I design data warehouse structures in support of multidimensional cubes in Microsoft SQL Server Analytical Services (SSAS), develop ETL packages in SQL Server Integrated Services (SSIS) and I have extensive reporting design experience with SQL Server Reporting Services (SSRS). I have developed numerous database front-end applications in VB.NET and MVC. Additionally, I have training and experience in report design with Cognos 10 and Microstrategy 9.4 and 10.

2010 - Present ZDataReports Syracuse, New York

Data Validation

- Performed environmental data validation audits in accordance with USEPA National Functional Guidelines on laboratory analyses for volatiles, semivolatiles, metals, pesticides, herbicides, PCBs, and dioxin/furans in water, soil and air samples.

2006 - 2011 TRW Automotive, Inc. Auburn, New York

Computer Integrated Manufacturing Engineer II

- Responsible for development and maintenance of Automotive Manufacturing Traceability System (AMTS) consisting of the management of 30+ SQL Server databases, design and modification of VB6 and VB.NET HMI desktop applications, SQL Server Reporting services, and integration with SAP system. I was involved in installation and design of a VMWare VSphere 4 environment in the early stages of database server virtualization process.

2003 - 2006 Tetra Tech EM, Inc. Rockaway, New Jersey

IT Systems Specialist

- Designed ESRI ArcPad 6/7 PDA applications and ArcIMS/ArcSDE web sites. Experience with Visual Studio.NET, ESRI ArcSDE, ArcIMS, ArcGIS and ArcPad, Oracle 10g and SQL Server 2000 and 2005, Tomcat 4/5, javascript, ASP, ASP.NET, Cold Fusion, Macromedia Web products, and others.
- Major projects include the design and development of a management database/pda application Unocal oil pipelines in Alaska. I also build an ArcPad application to manage site excavation, transport and mass balance for Maxwell House. I built a reusable, database driven, ArcIMS GIS viewer for the US Air Force Reserve command which included multiple map overlay viewing, overlay printing of D and E size figures, and Smart Card security modeling,

1999 - 2003 Ecology & Environment. Inc. Lancaster, New York

Chief/Senior Programmer

- Designed relational databases and programmed distributed database applications in ASP, Visual Basic, Visual Basic for Applications, Embedded Tools, and SQL Server (7 and 2000).
- Major projects include the development of a system for the management, presentation, and statistical reporting and charting of analytical data for the People's Republic of China as part of the Huangpu River Environmental Monitoring Information System. I also developed a web-based n-tier project management tool compatible with MS Project data structure.

1993 - 1999 Blasland, Bouck & Lee, Inc. Syracuse, New York

Senior Project Scientist I/Information Management Specialist

- Responsibilities include the development and maintenance of numerous environmental analytical databases, data validation in accordance with USEPA guidelines, and other related data analysis techniques.
- Major projects include data validation, database design, database programming and management for such firms as Tenneco, Amoco, Lockheed Martin, General Electric and other PRP groups responsible for work at multiple sites along the Kalamazoo (MI), Fox (WI) and Passaic (NJ) river systems.

1988 - 1993 Upstate Laboratories. Inc. Syracuse, New York

GC/MS Supervisor

- Responsibilities include supervision of an environmental laboratory gas chromatography/mass spectroscopy (GC/MS) department consisting of 5 instruments and 2 subordinates in the analyses of all variety of USEPA volatile and semi-volatile methodologies. I also provided technical supervision and method development for all High Performance Liquid Chromatography (HPLC) analytical techniques.

1980 - 1988 Onondaga County Health Department Syracuse, New York

Laboratory Technician I

- As an analyst with the Forensic Toxicology Laboratory, I was responsible for all aspects of chemical analyses including blood alcohol, common drugs of abuse, lead toxicity, and gunshot residue utilizing an array of chromatographic techniques. I also developed procedural manuals for the laboratory, and developed an information system for logging, tracking and reporting of the forensic sample data using Enable Office Automation software.

Project Highlights

- **Manufacturing QA Analysis, ONE** – Designed a SQL Server database for storage of manufacturing facility QA/QC testing results including a Microsoft Access front-end for data input and SSRS reports for Process QC Charting. In Phase II of this project Mr. Zoccolillo added a data warehouse database including ETL packages and SSAS Cubes used in multidimensional analysis. A Microsoft Excel Pivot table was constructed for BI Analysis of the data along with SSRS reports.
- **Finance Audit Office Automation, ONE** – Designed a system in Microsoft Office to build daily financial balance sheets and summary dashboards using Microsoft Excel and VBA scripting in conjunction with PL/SQL procedures and packages in an Oracle data warehouse.
- **Automated Materials Replenishment System, TRW** - Was responsible for the design and implementation of automating the replenishment of manufacturing line-side inventories by monitoring usage as reported through process databases, query SAP for appropriate lots and locations of replacement materials and notify the warehouse of those needs. The system utilized ASP.NET web applications for manual requests and reporting, and Windows Services for automated process execution.
- **SAP Midline Back Flush and Assembly Scrap Reporting Automated Services, TRW** - Designed several web-based applications and Windows Services utilizing ASP.NET 2005 and SAP .NET Tools used to integrate the real-time production environment with the SAP ERP system for optimization of JIT processes.
- **Design of Data Storage System of Very Large Databases for Quality Testing, TRW** - Mr. Zoccolillo designed and implemented a SQL Server data storage model utilizing advanced partitioning, replication and mirroring techniques to build a data storage and reporting structure used for high speed, high

availability data collection of product testing equipment.

- **GEOBASE Portal/GIS Viewer Design for US Air Force Reserve Command, Warner Robins, GA** — Mr. Zoccolillo was lead developer for the GEOBASE Portal redesign and the multi-based, multi-service GIS viewer. This viewer features a SQL Server database driven interface to an ESRI multi-service GIS viewer. The viewer we interface is built on ASP, ASP.NET and ESRI ArcIMS technologies. The viewer is able to display multiple services simultaneously and is able to print the overlaid maps to large size plotters.
- **Oil Field Database with ESRI ArcPad Field Application for Unocal, Anchorage, AK** — A custom database was designed, initially in MS Visio and ported to MS Access and Oracle 9i, for management of oil well, well pad, reserve pit, and pipeline data including field surveys, photos, spills, abandonment, etc. The database has the ability to export data to the ArcPad 7 PDA application. Data could be viewed, added, edited and deleted in ArcPad and the data is imported back into the database along with spatial data in the form of shapefiles.
- **mFRIS Application, Forest Technology Group, Charleston, SC** — mFRIS is the mobile forest resource information system designed by FTG as a field data collection component for their larger internet based webFRIS system. Mr. Zoccolillo was specifically recruited by FTG based on his role with ESRI ArcPad 6 beta tester and expertise within the developer's online forum. This project, budget at \$35,000, resulted in the development of the first handheld application for forestry management. It was developed using ESRI ArcPad 6 and runs on Windows CE devices. mFRIS functions as a data collection tool for shapes and associated data and included many features absent in standard Arcpad 6, such as buffering, custom queries, multi-selection and statistical summaries.
- **Mobile Device Applications for UN Gulf War Reparations, Saudi Arabia and Kuwait** — Mr. Zoccolillo was lead developer for all ArcPad and Windows CE embedded development for the multiphase site characterization of the Persian Gulf coast as part of the United Nations Gulf War Reparations Act. Using ArcPad 6.0 beta and Microsoft Embedded Tools 3.0, he developed custom applications for Windows CE devices that were used to collect shoreline and terrestrial data that made use of GPS for navigation and sample locations.
- **HRBEMIS World Bank Project, Shanghai, China** — The Huang Pu River Environmental Management Information System was a 1.5 million dollar project for the Peoples Republic of China in conjunction with the World Bank. The HRBEMIS system contained two major parts; a hydrochemical river modeling system and an analytical database and statistical reporting system. The river modeling system consisted of a FORTRAN based, 2 dimensional hydrolic river model interfaced though a custom GIS application designed in Visual Basic 6/Map Objects 2. The database reporting system was built with Microsoft Access, Visual Basic and Crystal Reports and consisted of three primary databases for point source, precipitation and water quality data. Various statistical reports and charts could be generated from each of the databases and the data could be queried from the GIS application. Mr. Zoccolillo functioned as the senior programmer and later as project manager.

Publications

- ***Dynamic Web-Based GIS Browsing and Plotting for Multiple Bases and Map Services***, GIS in the Defense and Intelligence Communities, Volume 2, ESRI, 2005, pp. 10-13.

References

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George Cherian, (315) 750-5436, george@cherians.net
Michael Fifield, (315) 725-8321, fifieldm@gmail.com

ATTACHMENT - Community Air Monitoring Plan



TECHNICAL
SERVICES

COMMUNITY AIR MONITORING PLAN

495 ELEVENTH

495 Eleventh Avenue
Manhattan, New York

NYSDEC BCP Site: C231141

July 2023

GBTS Project: 22003-0076

[Technical Services Division](#)

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COMMUNITY AIR MONITORING PLAN

July 2023

GBTS Project:

22003-0076

Prepared By:

**Gallagher Bassett Technical Services
22 IBM Road, Suite 101
Poughkeepsie, New York 12601**

Prepared For:

**495 11th Avenue Owner Realty LLC
PO Box 234550
Great Neck, New York, 11023**

The undersigned have reviewed this Community Air Monitoring Plan and certify to 495 11th Avenue Owner Realty LLC and to the New York State Department of Environmental Conservation that the information provided in this document is accurate as of the date of issuance by this office.



Scott Spitzer
Gallagher Bassett Technical Services
Technical Director – Environmental Consulting



Richard Hooker
Gallagher Bassett Technical Services
Manager – Environmental Consulting

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ATTACHMENTS:

Figure: Proposed Fieldwork Map

NYSDOH Generic CAMP

1.0 INTRODUCTION

1.1 Purpose

This Community Air Monitoring Plan (CAMP) has been developed to provide the requirements and general procedures to be followed by Gallagher Bassett Technical Services (GBTS) and on-Site subcontractors while performing investigation services at the 495 Eleventh BCP Site (ID: C231141) located in Manhattan, New York.

This CAMP requires real-time monitoring for volatile organic compounds (VOCs) and particulates (i.e., dust) at the perimeter of each designated work area and is intended to provide protection for the downwind receptors, including off-Site properties and on-site workers not directly involved in the handling of contaminated materials. Implementation of the CAMP helps to confirm that work activities did not spread contamination off-site through the air.

The Project Manager or Site Health and Safety Officer (SHSO) may impose other requirements necessary for safe Site operations and protection of potential receptors.

1.2 Site Location and Description

The Site is defined as the property located at 495 Eleventh Avenue, Manhattan, City of New York, New York. A figure illustrating the Site configuration and areas of proposed investigation activities is included as an Attachment to this CAMP.

1.3 Work Activities

Investigation activities are detailed in the NYSDEC-approved Pre-Design Investigation Work Plan (PDIWP) dated July 2023. The specific tasks detailed in the PDIWP are wholly incorporated by reference into this HASP. The PDIWP describes the tasks required to investigate environmental contamination at the Site.

The Remedial Investigation Report prepared for the Site documented the presence of fill materials and groundwater with elevated levels of metals and polycyclic aromatic hydrocarbons (PAHs). Low-level soil vapor contamination, typical of urban settings, is present at the Site.

1.4 Health and Safety Hazards

Elevated concentrations of organic compounds (PAHs) and metals are present in Site soil and groundwater, and low-grade volatile compounds are present in soil vapor. The possibility exists for on-site personnel to have contact with contaminated soils, groundwater and/or vapor during investigative activities. Contact with contaminated substances may present a skin contact, inhalation and/or ingestion hazard.

2.0 AIR MONITORING

2.1 General Requirements

The implementation of the CAMP will document the presence or absence of VOCs and dust in the air surrounding the work zone, which may migrate off-Site due to fieldwork activities. Monitoring will be conducted at all times that fieldwork activities which are likely to generate emissions are occurring. This plan provides guidance on the need for implementing more stringent dust and emission controls based on air quality data.

2.1.1 Continuous Monitoring

Real-time air monitoring for VOCs and particulate levels at the perimeter of the exclusion zone or work area will be performed according to the NYSDOH Generic Community Air Monitoring Plan (provided as an Attachment), and in accordance with the special requirements presented below, during all ground intrusive activities and any other fieldwork that is reasonably likely to generate significant dust or vapors from known or suspected contaminated soils. Ground intrusive activities include, but are not limited to, soil/waste excavation and handling, test pit excavation or trenching, and the installation of soil borings or monitoring wells.

2.1.2 Periodic Monitoring

Periodic monitoring for VOCs will be performed during non-intrusive activities such as the collection of soil samples or the collection of groundwater samples from existing monitoring wells. Periodic monitoring during sample collection, for instance, will consist of taking a reading upon arrival at a sample location, monitoring while opening a well cap or overturning soil, monitoring during well baling/purging, and taking a reading prior to leaving a sample location. Depending upon the proximity of potentially exposed individuals, continuous monitoring may be performed during sampling activities. Examples of such situations include groundwater sampling at wells on the curb of a busy urban street, in the midst of a public park, or adjacent to a school or residence.

2.1.3 Health and Safety

Photoionization detector (PID) and dust readings consistently in excess of CAMP limits will be used as an indication of the need to initiate personnel monitoring, increase worker protective measures, and/or modify or cease on-site operations in order to mitigate off-site community exposure. PID readings that consistently exceed background in the breathing zone (during any proposed tasks) will necessitate moving away from the source or implementing a higher level of personal protective equipment (concentrations of VOCs in the air are expected to be below the OSHA Permissible Exposure Limits [PELs]).

2.1.4 VOC Monitoring, Response Levels, and Actions

VOCs will be monitored at the downwind perimeter of the immediate work area (i.e., the exclusion zone) on a continuous basis during invasive work. Upwind concentrations will be measured at the start of each workday and periodically thereafter to establish background conditions. Monitoring work will be performed using equipment appropriate to measure the types of contaminants known or suspected to be present.

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.

If the ambient air concentration of total organic vapors at the downwind perimeter of the work area or exclusion zone exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities will be temporarily halted and monitoring continued. If the total organic 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 of the work area or exclusion zone persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities will be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities will resume provided that the total organic vapor level 200 feet downwind of the exclusion zone or half the distance to the nearest potential receptor or occupied 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 will be shut down.

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

2.1.4 Particulate Monitoring, Response Levels, and Actions

Particulate concentrations will be monitored continuously at the upwind and downwind perimeters of the exclusion zone at temporary particulate monitoring stations. 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 equipped with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration should be visually assessed during all work activities.

If the downwind PM-10 particulate level is 100 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) greater than background (upwind perimeter) for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques will be employed and work will continue provided that downwind PM-10 particulate levels do not exceed $150 \mu\text{g}/\text{m}^3$ above the upwind level and provided that no visible dust is migrating from the work area.

If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than $150 \mu\text{g}/\text{m}^3$ above the upwind level, work will be stopped and a re-evaluation of activities initiated. Work will resume provided that dust suppression measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within $150 \mu\text{g}/\text{m}^3$ of the upwind level and in preventing visible dust migration.

All readings will be recorded and will be available for NYSDEC personnel to review.

2.2 Special Requirements

2.2.1 Work within 20 Feet of Potential Receptors

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 should be considered to prevent exposures related to the work activities and to control dust and odors. Consideration should be given to implementing the planned activities when potentially exposed populations are at a minimum, such as during weekends or evening hours in non-residential settings.

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). Depending upon the nature of contamination, chemical-specific colorimetric tubes of sufficient sensitivity may be necessary for comparing the exposure point concentrations with appropriate pre-determined response levels (response actions should also be predetermined). Background readings in the occupied spaces must be taken and discussed with NYSDOH prior to commencement of the work.

If total particulate concentrations opposite the walls of occupied structures or next to intake vents exceed $150 \text{mcg}/\text{m}^3$, work activities should be suspended until controls are implemented and are successful in reducing the total particulate concentration to $150 \text{mcg}/\text{m}^3$ or less at the monitoring point.

Depending upon the nature of contamination and remedial activities, other parameters (e.g., explosivity, oxygen, hydrogen sulfide, and carbon monoxide) may also need to be monitored. Response levels and actions should be pre-determined, as necessary, for each site.

2.2.2 Special Requirements for Indoor Work

Unless a self-contained, negative-pressure enclosure with proper emission controls will encompass the work area, all individuals not directly involved with the planned work must be absent from the room in which the work will occur. Monitoring requirements shall be as stated above under Section 2.2.1, except that in this instance “nearby/occupied structures” would be adjacent occupied rooms. Additionally, the location of all exhaust vents in the room and their discharge points, as well as potential vapor pathways (openings, conduits, etc.) relative to adjoining rooms, should be understood and the monitoring locations established accordingly. In these situations, it is strongly recommended that exhaust fans or other engineering controls be used to create negative air pressure within the work area during remedial activities. Additionally, it is strongly recommended that the planned work be implemented during hours (e.g., weekends or evenings) when building occupancy is at a minimum.

2.3 Contaminant Control

Mitigation measures may be required to control the generation of vapors and/or dust.

2.3.1 Dust Control

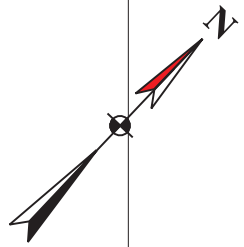
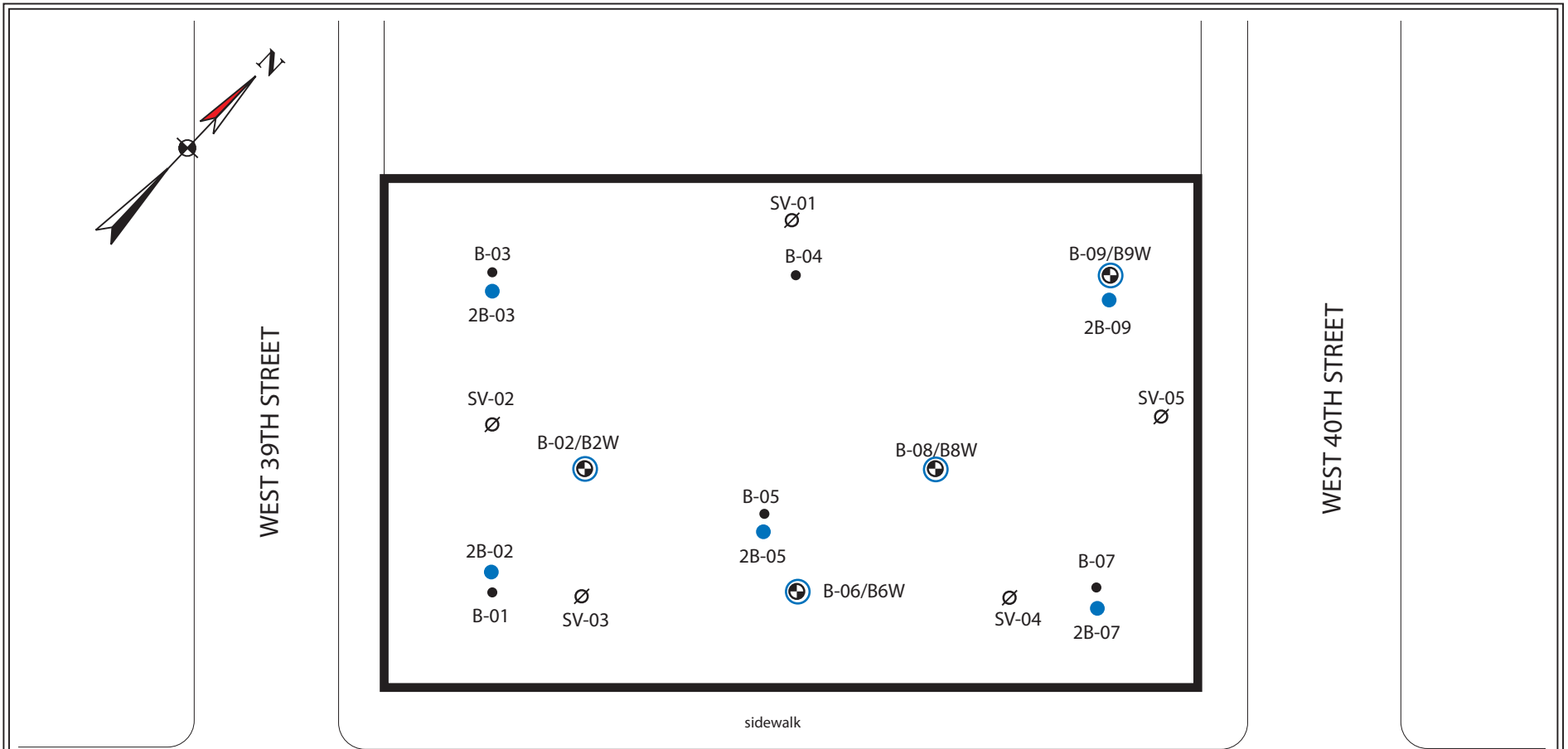
Mitigation measures may include reducing the surface area of contaminated soil being disturbed at one time, wetting heavy equipment and exposed soils to reduce fugitive dust, using covered stockpiles/trucks, or stopping excavation and/or other soil disturbing activities. Dust suppression will be conducted during construction activities that will disturb on-Site soils and may include misting, reduction in soil movement, reducing vehicle speeds, or cessation of excavation.

2.3.2 Vapor Control

Mitigation measures include all controls used for dust suppression, as well as specific techniques for reducing releases of vapors, such as spraying commercial odor control products (e.g., BioSolve) or limiting work to cooler or less windy times of day.

3.0 QUALITY ASSURANCE

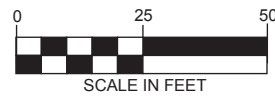
All instruments will be properly calibrated before the start of fieldwork, with periodic calibration checks as necessary. All equipment will be operated in accordance with the manufacturer's recommendations and the operator's manual. The fieldwork manager will review all data and take appropriate actions based on the requirements in Section 2 of this CAMP. A record of all calibration events, and any unusual occurrence that affect CAMP data, will be recorded in the project field log book. Instrument calibration shall be documented in the designated field logbook. Exceedances of action levels observed during performance of the CAMP will be reported to the NYSDEC Project Manager and included in the Daily Report.



WEST 39TH STREET

WEST 40TH STREET

11th AVENUE



Base map provided by HAKS Engineers, Architects & Land Surveyors PC survey dated 11/5/14. All feature locations are approximate. This map is intended as a schematic to be used in conjunction with the associated report, and it should not be relied upon as a survey for planning or other activities.

Proposed Fieldwork Map
495 11th Avenue
Borough of Manhattan, New York

- Legend:
- subject property border
 - previous boring location
 - previous soil vapor location
 - ⊕ previous boring location and existing monitoring well location
 - proposed boring location
 - ⊕ existing monitoring well to be sampled

File: 22003-0076
July 2023
Scale as shown
Figures

Appendix 1A

New York State Department of Health Generic Community Air Monitoring Plan

Overview

A Community Air Monitoring Plan (CAMP) requires real-time monitoring for volatile organic compounds (VOCs) and particulates (i.e., dust) at the downwind perimeter of each designated work area when certain activities are in progress at contaminated sites. The CAMP is not intended for use in establishing action levels for worker respiratory protection. Rather, its intent is to provide a measure of protection for the downwind community (i.e., off-site receptors including residences and businesses and on-site workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative and remedial work activities. The action levels specified herein require increased monitoring, corrective actions to abate emissions, and/or work shutdown. Additionally, the CAMP helps to confirm that work activities did not spread contamination off-site through the air.

The generic CAMP presented below will be sufficient to cover many, if not most, sites. Specific requirements should be reviewed for each situation in consultation with NYSDOH to ensure proper applicability. In some cases, a separate site-specific CAMP or supplement may be required. Depending upon the nature of contamination, chemical-specific monitoring with appropriately-sensitive methods may be required. Depending upon the proximity of potentially exposed individuals, more stringent monitoring or response levels than those presented below may be required. Special requirements will be necessary for work within 20 feet of potentially exposed individuals or structures and for indoor work with co-located residences or facilities. These requirements should be determined in consultation with NYSDOH.

Reliance on the CAMP should not preclude simple, common-sense measures to keep VOCs, dust, and odors at a minimum around the work areas.

Community Air Monitoring Plan

Depending upon the nature of known or potential contaminants at each site, real-time air monitoring for VOCs and/or particulate levels at the perimeter of the exclusion zone or work area will be necessary. Most sites will involve VOC and particulate monitoring; sites known to be contaminated with heavy metals alone may only require particulate monitoring. If radiological contamination is a concern, additional monitoring requirements may be necessary per consultation with appropriate DEC/NYSDOH staff.

Continuous monitoring will be required for all ground intrusive activities and during the demolition of contaminated or potentially contaminated structures. Ground intrusive activities include, but are not limited to, soil/waste excavation and handling, test pitting or trenching, and the installation of soil borings or monitoring wells.

Periodic monitoring for VOCs will be required during non-intrusive activities such as the collection of soil and sediment samples or the collection of groundwater samples from existing monitoring wells. "Periodic" monitoring during sample collection might reasonably consist of taking a reading upon arrival at a sample location, monitoring while opening a well cap or

overturning soil, monitoring during well baling/purging, and taking a reading prior to leaving a sample location. In some instances, depending upon the proximity of potentially exposed individuals, continuous monitoring may be required during sampling activities. Examples of such situations include groundwater sampling at wells on the curb of a busy urban street, in the midst of a public park, or adjacent to a school or residence.

VOC Monitoring, Response Levels, and Actions

Volatile organic compounds (VOCs) must be monitored at the downwind perimeter of the immediate work area (i.e., the exclusion zone) on a continuous basis or as otherwise specified. Upwind concentrations should be measured at the start of each workday and periodically thereafter to establish background conditions, particularly if wind direction changes. The monitoring work should be performed using equipment appropriate to measure the types of contaminants known or suspected to be present. The equipment should be calibrated at least daily for the contaminant(s) of concern or for an appropriate surrogate. The equipment should be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

1. If the ambient air concentration of total organic vapors at the downwind perimeter of the work area or exclusion zone exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities must be temporarily halted and monitoring continued. If the total organic vapor level readily decreases (per instantaneous readings) below 5 ppm over background, work activities can resume with continued monitoring.

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

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

4. All 15-minute readings must be recorded and be available for State (DEC and NYSDOH) personnel to review. Instantaneous readings, if any, used for decision purposes should also be recorded.

Particulate Monitoring, Response Levels, and Actions

Particulate concentrations should be monitored continuously at the upwind and downwind perimeters of the exclusion zone at temporary particulate monitoring stations. The particulate monitoring should be performed using real-time monitoring equipment capable of measuring particulate matter less than 10 micrometers in size (PM-10) and capable of integrating over a period of 15 minutes (or less) for comparison to the airborne particulate action level. The equipment must be equipped with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration should be visually assessed during all work activities.

1. If the downwind PM-10 particulate level is 100 micrograms per cubic meter (mcg/m^3) greater than background (upwind perimeter) for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques must be employed. Work may continue with dust suppression techniques provided that downwind PM-10 particulate levels do not exceed $150 \text{ mcg}/\text{m}^3$ above the upwind level and provided that no visible dust is migrating from the work area.

2. If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than $150 \text{ mcg}/\text{m}^3$ above the upwind level, work must be stopped and a re-evaluation of activities initiated. Work can resume provided that dust suppression measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within $150 \text{ mcg}/\text{m}^3$ of the upwind level and in preventing visible dust migration.

3. All readings must be recorded and be available for State (DEC and NYSDOH) and County Health personnel to review.

December 2009