Quality Assurance Project Plan/Field Sampling Plan (QAPP/FSP)

3728 Park Avenue
Bronx, New York 10456

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Prepared for:

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1. Introduction

Roux Environmental Engineering and Geology, D.P.C. (Roux), on behalf of Sol on Park LLC (Sol on Park), has prepared this Quality Assurance Project Plan/Field Sampling Plan (QAPP/FSP) to describe the measures that will be taken to ensure that the data generated during performance of the Remedial Action (RA) at the Sol on Park Site located at 3728 Park Avenue, Bronx, New York (Site) are of quality sufficient to meet project-specific data quality objectives (DQOs). This QAPP/FSP also includes field sampling procedures.

This QAPP/FSP was prepared in accordance with the guidance provided in NYSDEC Technical Guidance DER-10 Technical Guidance for Site Investigation and Remediation (DER-10), the NYSDEC BCP Guide, and the United States Environmental Protection Agency's (USEPA's) Guidance for the Data Quality Objectives Process (EPA QA/G 4) dated February 2006.

1.1 Purpose

The QAPP/FSP describes in detail the field sampling and quality assurance/quality control (QA/QC) methods to be used during soil tasks performed during the RA.

This QAPP/FSP was prepared in accordance with the NYSDEC's DER-10 and provides guidelines and procedures to be followed by field personnel during performance of sampling during the RA. Information contained in this QAPP/FSP relates to:

- Sampling objectives (Section 2);
- Project organization (Section 3);
- Sample media, sampling locations, analytical suites, sampling frequencies and analytical laboratory (Section 4);
- Field sampling procedures (Section 5);
- Sample handling, sample analysis, and QA/QC (Section 6); and
- Site control procedures and decontamination (Section 7).

2. Sampling Objectives

The sampling program is designed to meet the data quality objectives (DQOs) set forth in the DER-10. Specifically, analytical parameters selected for each sample, as described in Section 4, are comprehensive, and are intended to meet the following objectives:

- Analyze endpoint bottom and sidewall samples; and
- Analyze offsite backfill/on-Site reuse soil to evaluate its suitability for use as backfill that meets the lower of the Restricted Residential Soil Cleanup Objectives (RRSCOs) or Protection of Groundwater Cleanup Objectives (PGWSCOs) presented in Table 6.8 (b) of the latest revision of Title 6 of New York Code of Rules and Regulations (NYCRR) Part 375 (Part 375).

Sampling procedures are discussed in Section 5 of this QAPP/FSP. A discussion of the DQOs and quality assurance/quality control is provided in Section 6.

3. Project Organization

A general and generic summary of the overall management structure and responsibilities of project team members are presented below. Professional profiles for the team are provided in Attachment 1.

Project Principal

Charlie McGuckin, P.E. will serve as Project Principal. The Project Principal is responsible for defining project objectives and bears ultimate responsibility for the successful completion of the investigation.

Project Manager

Dana Hignell of Roux will serve as Project Manager. The Project Manager is responsible for defining project objectives and bears ultimate responsibility for the successful completion of the work. This individual will provide overall management for the implementation of the scope of work and will coordinate all field activities. The Project Manager is also responsible for data review/interpretation and report preparation.

Field Team Leader

The Field Team Leader will be determined prior to the start of work. The Field Team Leader bears the responsibility for the successful execution of the field program. The Field Team Leader will direct the activities of the technical staff in the field, as well as all subcontractors. The Field Team Leader will also assist in the interpretation of data and in report preparation. The Field Team Leader reports to the Project Manager.

Laboratory Project Manager

Laboratory analysis will be completed by Eurofins of Edison, New Jersey, Sacramento, California, and Burlington, Vermont, New York State Department of Health (NYSDOH) Environmental Laboratory Approval Program (ELAP)-certified laboratories (11452, 11666, and 10391, respectively). The Laboratory Project Manager is Melissa Haas. The Laboratory Project Manager is responsible for sample container preparation, sample custody in the laboratory, and completion of the required analysis through oversight of the laboratory staff. The Laboratory Project Manager will ensure that QA procedures are followed and that an acceptable laboratory report is prepared and submitted. The Laboratory Project Manager reports to the Project Principal and Project Manager.

Quality Assurance Officer

Brooke Hildebrand of Roux will serve as the Quality Assurance Officer (QAO) for this project. The QAO is responsible for conducting reviews, inspections, and audits to ensure that the data collection is conducted in accordance with the FSP and QAPP. The QAO's responsibilities range from ensuring effective field equipment decontamination procedures and proper sample collection to the review of all laboratory analytical data for completeness and usefulness. The QAO reports to the Project Manager and makes independent recommendations to the Field Team Leader.

4. Sample Media, Locations, Analytical Suites, and Frequency

The media to be sampled during the remedial action may include soil and fill materials. Sampling locations, analytical suites, and frequency is provided below and shown in Tables 1 and 2. Specifics regarding the collection of samples at each location and for each task are provided in Section 5 of this QAPP/FSP.

4.1 Endpoint Soil Sampling

The Documentation samples for the Remedial Action are described in the RAWP and include the proposed samples to be collected during the remedial construction. The proposed samples to be collected are described below.

Bottom Confirmation Sampling

Bottom confirmation sampling will be conducted at a frequency of one sample per approximately 900 square feet of excavation area (35 samples total). The frequency of bottom sampling proposed for this alternative is consistent with the guidance provided in NYSDEC DER-10 5.4. Confirmation samples will be collected following completion of onsite excavation for remedial and redevelopment purposes in order to ensure RRSCOs have been met and to properly document the remaining onsite soil quality. Confirmation samples will be analyzed for the full suite of parameters (VOCs, SVOCs, metals, pesticides, PCBs, and PFAS). Locations of endpoint samples are shown in Figure 13 of the RAWP.

Sidewall Confirmation Sampling

As part of the remedial action, sidewall confirmation endpoint samples will be collected to properly confirm the remaining sidewall soil quality. Sidewall samples will be collected at a frequency of one sidewall sample per 30 linear feet. The frequency of sidewall sampling proposed for this alternative is consistent with the guidance provided in NYSDEC DER-10 5.4. Locations of endpoint samples are shown in Figure 13 of the RAWP.

A total of 35 bottom confirmation samples (BCS) and 11 sidewall confirmation samples (SCS), will be collected at the Site within the Track 2 area. Samples will be distributed throughout the Site, in a grid-like fashion, and will be biased toward impacts, if any are observed.

The bottom and sidewall confirmation samples will be tested for the TCL/Part 375 / TAL (TCL) list of parameters including:

- TCL/Part 375 VOC;
- TCL/Part 375 Base neutral acids (BNA)/SVOCs (including 1,4-Dioxane);
- TCL/Part 375 Pesticides;
- TCL/Part 375 PCBs;
- TAL/Part 375 Metals (including hexavalent chromium);
- Total Cyanide; and
- PFAS*

*Per- and Polyfluoroalkyl Substances (PFAS) including the 40 compounds listed in the most recent April 2023 NYSDEC guidance Sampling, Analysis, and Assessment of Per-and Polufluoroalkyl Substances (PFAS) Under NYSDEC's Part 375

Remedial Programs (NYSDEC April 2023 Guidance) is included as Attachment 2 to this QAPP/FSP. Soil samples placed on hold at the laboratory will not be analyzed for PFAS. PFAS in soil will be analyzed via USEPA EPA Method 1633A. The 40 PFAS are:

- Perfluorobutanesulfonic acid
- Perfluoropentansulfonic acid
- Perfluorohexanesulfonic acid
- Perfluoroheptanesulfonic acid
- Perfluorooctanesulfonic acid
- Perfluorononanesulfonic acid
- Perfluorodecanesulfonic acid
- Perfluorododecanesulfonic acid
- Perfluorobutanoics acid
- Perfluoropentanoic acid
- Perfluorohexanoic acid
- Perfluoroheptanoic acid
- Perfluorooctanoic acid
- Perfluorononanoic acid
- Perfluorodecanoic acid
- Perfluoroundecanoic acid
- Perfluorododecanoic acid
- Perfluorotridecanoic acid
- Perfluorotetradecanoic acid
- 1H,1H, 2H, 2H-Perfluorohexane sulfonic acid
- 1H,1H, 2H, 2H-Perfluorooctane sulfonic acid
- 1H,1H, 2H, 2H-Perfluorodecane sulfonic acid
- Perfluroroctanesulfonamide
- N-methyl perfluorooctanesulfonamide
- N-ethyl perfluorooctanesulfonamide
- N-methyl perfluorooctanesulfonamidoacetic acid
- N-ethyl perfluorooctanesulfonamidoacetic acid
- N-methyl perfluorooctanesulfonamidoethanol
- N-ethyl perfluorooctanesulfonamidoethanol
- Hexafluoropropylene oxide dimer acid
- 4,8-Dioxa-3H-perfluorononanoic acid
- Perfluoro-3-methoxypropanoic acid
- Perfluoro-4-methoxybutanoic acid
- Nonafluoro-3,6-dioxaheptanoic acid
- 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid
- 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid
- Perfluoro(2-ethoxyethane)sulfonic acid
- 3-Perfluoropropyl propanoic acid
- 2H,2H,3H,3H-Perfluorooctanoic acid
- 3-Perfluoroheptyl propanoic acid

All PFAS compounds, listed above, in soil will be analyzed and reported to 0.5 microgram per kilogram (ug/kg).

4.2 Offsite Backfill/On-Site Reuse Soil Sampling

Although not planned, onsite soil may be reused for backfill. For offsite fill/on-Site reuse soil materials requiring chemical testing, the following samples shall be collected:

- One sample for pre-qualification chemical testing at the source location, including the following:
    1. Chemical testing shall be performed at a minimum for the parameters listed in Table 375-6.8(b) of the latest revision of Part 375. Samples will be analyzed for the same parameters and by the same methods listed in Section 4.1.
    2. Backfill/on-Site reuse soil materials excluding those materials that do not require sampling as described below, shall meet criteria presented in Section 2.0.
    3. Backfill/on-Site reuse soil materials that exceed the criteria presented in Section 2.0 shall not be imported to the Site without prior approval of the NYSDEC.
    4. The backfill/on-Site reuse soil material will be free of extraneous debris or solid waste.
    5. If the NYSDEC agrees that the material originated from a virgin source, then a minimum of one sample (i.e., the pre-qualification sample) will be collected and analyzed per source.

- If the source is not virgin, the sampling frequency will comply with DER-10 Table 5.4(e)10 shown below:

Table 5.4(e)10 Recommended Number of Soil Samples for Soil Imported To or Exported From a Site			
Contaminant	**VOCs**	**SVOCs, Inorganics & PCBs/Pesticides**	
Soil Quantity (cubic yards)	**Discrete Samples**	**Composite**	**Discrete Samples/Composite**
0-50	1	1	3-5 discrete samples from different locations in the fill being provided will comprise a composite sample for analysis
50-100	2	1	
100-200	3	1	
200-300	4	1	
300-400	4	2	
400-500	5	2	
500-800	6	2	
800-1000	7	2	
➢ 1000	Add an additional 2 VOC and 1 composite for each additional 1000 Cubic yards or consult with DER		

The source of the offsite fill/on-Site reuse soil must be documented by the supplier, including the location where the fill/on-Site reuse soil was obtained and a brief history of the site that is the source of the fill.

Samples of offsite backfill/on-Site reuse soil will be analyzed for the same list of parameters in Section 4.1.

All PFAS compounds, listed above, in soil will be analyzed and reported to 0.5 ug/kg.

The following materials may be imported, without chemical testing, to be used as backfill beneath pavement, buildings or as part of the site cover, provided that it contains less than 10 percent by weight material that would pass through a size 100 sieve and consists of:

- gravel, rock or stone, consisting of virgin material from a permitted mine or quarry; or
- recycled concrete or brick from a NYSDEC registered construction and demolition debris processing facility if the material conforms to the requirements of Section 304 of the 2002 New York State Department of Transportation Standard Construction and Materials Volume 1.

5. Field Sampling Procedures

This section provides a detailed discussion of the field procedures to be used during sampling of the various media being evaluated as part of the RA implementation (i.e., soil and fill material). Additional information including intervals to be sampled and sample rationale is provided in the RAWP. Additional details regarding sampling procedures and protocols are described in Roux's relevant Standard Operating Procedures (SOPs), which are provided in Attachment 3.

5.1 Soil Sampling

Details for the collection of soil samples is provided below. Additional necessary precautions will be taken when sampling for ECs in the field, including but not limited to:

- Using the proper field clothing or personal protective equipment (i.e. no materials will contain Gore-Tex or Tyvek);
- Avoid sampling equipment components/containers making contact with aluminum foil, low density polyethylene (LDPE), glass, or polytetrafluoroethylene materials;
- Following PFAS field sampling guidelines (i.e., using sampling materials made from high density polyethylene [HDPE], silicon, or stainless steel and avoid using equipment containing Teflon and using sharpies, permanent markers, adhesives, and waterproof/plastic clipboards and notebooks); and
- Utilizing regular ice cubes for sample presevation and only Alconox for decontamination.

5.1.1 Bottom and Sidewall Confirmation Sampling

Bottom and sidewall confirmation soil samples will be collected from the interval beneath the terminal extent of the remedial excavation. The soil will be observed for lithology and evidence of contamination (e.g., staining, odors, and/or visible free product) and placed immediately thereafter into large Ziploc® bags for recording headspace using a PID. After a minimum of 15 minutes for equilibration with the headspace in the Ziploc® bag, each sample will be screened for organic vapors using a PID equipped with a 10.6 eV lamp. Samples for possible VOC analysis will be placed in a laboratory-supplied jar or encore sampler prior to screening, due to the potential for loss of VOCs through volatilization. These samples will be placed in the laboratory-supplied containers and shipped to the laboratory under chain of custody procedures in accordance with Roux's SOPs in Attachment 3.

5.2 Offsite Backfill/On-Site Reuse Soil Sampling

Although not planned, onsite soil may be reused for backfill. All imported fill/on-Site reuse soil material samples will be collected using pre-cleaned stainless steel sampling tools (i.e., trowels, spatulas, etc.) or new Ziploc® bags. In general, where composite samples are required for offsite fill/on-Site reuse soil material samples, composite samples will be collected from a minimum of three locations across the stockpiled materials. The exception is for VOC samples, which will be collected as grab samples.

6. Sample Handling and Analysis

To ensure quality data acquisition and collection of representative samples, there are selective procedures to minimize sample degradation or contamination. These include procedures for preservation of the samples, as well as sample packaging, shipping procedures, and QA/QC.

6.1 Field Sample Handling

A discussion of the proposed number and types of samples to be collected during each task, as well as the analyses to be performed, can be found in Section 4 of this QAPP/FSP. The types of containers, volumes, and preservation techniques for the aforementioned testing parameters are presented in Table 3.

6.2 Sample Custody Documentation

The purpose of documenting sample custody is to ensure that the integrity and handling of the samples is not subject to question. Sample custody will be maintained from the point of sampling through the analysis (and return of unused sample portion, if applicable).

Each individual collecting samples is personally responsible for the care and custody of the samples. All sample labels should be pre printed or filled out using waterproof ink. The technical staff will review all field activities with the Field Team Leader to determine whether proper custody procedures were followed during the field work and to decide if additional samples are required.

All samples being shipped offsite for analysis must be accompanied by a properly completed chain of custody form. The sample numbers will be listed on the chain of custody form. When transferring the possession of samples, individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to/from a secure storage area, and to the laboratory.

Samples will be packaged for shipment and dispatched to the appropriate laboratory for analysis with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and/or secured with strapping tape in at least two locations for shipment to the laboratory.

6.3 Sample Shipment

Samples are anticipated to be transported to the laboratory via courier, however, if sample shipment is necessary, sample packaging and shipping procedures are based upon USEPA specifications, as well as DOT regulations. The procedures vary according to potential sample analytes, concentration, and matrix and are designed to provide optimum protection for the samples and the public. Sample packaging and shipment must be performed using the general outline described below.

All samples will be shipped within 24 hours of collection and will be preserved appropriately from the time of sample collection. A description of the sample packing and shipping procedures is presented below:

1. Prepare cooler(s) for shipment:
    - tape drain(s) of cooler shut;
    - affix “This Side Up” arrow labels and “Fragile” labels on each cooler; and
    - place mailing label with laboratory address on top of cooler(s).

2. Arrange sample containers in groups by sample number.
3. Ensure that all bottle labels are completed correctly. Place clear tape over bottle labels to prevent moisture accumulation from causing the label to peel off.
4. Arrange containers in front of assigned coolers.
5. Place packaging material approximately at the bottom of the cooler to act as a cushion for the sample containers.
6. Arrange containers in the cooler so that they are not in contact with the cooler or other samples.
7. Fill remaining spaces with packaging material.
8. Ensure all containers are firmly packed in packaging material.
9. If ice is required to preserve the samples, ice cubes should be repackaged in Ziploc® bags and placed on top of the packaging material.
10. Sign chain of custody form (or obtain signature) and indicate the time and date it was relinquished to courier as appropriate.
11. Separate chain of custody forms. Seal proper copies within a large Ziploc® bag and tape to inside cover of cooler. Retain copies of all forms.
12. Close lid and latch.
13. Secure each cooler using custody seals.
14. Tape cooler shut on both ends.
15. Relinquish overnight delivery service as appropriate. Retain air bill receipt for project records. (Note: All samples will be shipped for "NEXT A.M." delivery).

6.4 Quality Assurance/Quality Control

Brooke Hildebrand will review the analytical data for quality assurance and quality control in accordance with NYSDEC standards. The professional profile for Brooke Hildebrand is provided in Attachment 1. A laboratory SOP for analysis of PFAS is included in Attachment 4.

The primary intended use for the Remedial Action data is to confirm if remediation has been performed in accordance with the RAWP at the Site. The primary DQO of the soil program, therefore, is that data be accurate and precise, thus, representative of the actual Site conditions. Accuracy refers to the ability of the laboratory to obtain a true value (i.e., compared to a standard) and is assessed through the use of laboratory quality control (QC) samples, including laboratory control samples and matrix spike samples, as well as through the use of surrogates, which are compounds not typically found in the environment that are injected into the samples prior to analysis. Precision refers to the ability to replicate a value and is assessed through both field and laboratory duplicate samples.

Sensitivity is also a critical issue in generating representative data. Laboratory equipment must be of sufficient sensitivity to detect target compounds and analytes at levels below NYSDEC standards and guidelines whenever possible. Equipment sensitivity can be decreased by field or laboratory contamination of samples, and by sample matrix effects. Assessment of instrument sensitivity is performed through the analysis of reagent blanks, near-detection-limit standards, and response factors. Potential field and/or laboratory contamination is assessed through use of trip blanks, method blanks, and equipment rinse blanks (also called "field blanks"). Field blanks for PFAS will be collected at a minimum frequency of one per day.

Table 1 lists the requirements for field and laboratory QC samples that will be analyzed to assess data accuracy and precision, as well as to determine if equipment sensitivity has been compromised. Table 2 lists the number/type of field and QA/QC samples that will be collected during the Remedial Action. Table 3 lists the preservation, holding times and sample container information.

All RAWP "assessment" analyses (i.e., TCL/Part 375 / TAL, ECs) will be performed in accordance with the NYSDEC Analytical Services Protocol (ASP), using USEPA SW 846 methods.

All laboratory data are to be reported in NYSDEC ASP Category B deliverables and will be delivered to NYSDEC in electronic data deliverable (EDD) format as described on NYSDEC's website (http://www.dec.ny.gov/chemical/62440.html) and recent updated procedures enacted in November 2018. A Data Usability Report will be prepared meeting the requirements in Section 2.2(a)1.ii and Appendix 2B of DER-10 for all data packages generated for the RAWP. The DUSR will be prepared by Judy Harry, a third-party data validator. Validator resume is included in Attachment 1.

7. Site Control Procedures and Decontamination

Site control procedures, including decontamination and waste handling and disposal, are discussed below. Site control procedures have been developed to minimize both the risk of exposure to contamination and the spread of contamination during field activities at the Site. All personnel who come into designated work areas, including contractors and observers, will be required to adhere strictly to the conditions imposed herein and to the provisions of a Site-Specific Health and Safety Plan (HASP). The HASP is included as Appendix K to the RAWP.

7.1 Decontamination

In an attempt to avoid the spread of contamination, all drilling and sampling equipment must be decontaminated at a reasonable frequency in a properly designed and located decontamination area. Detailed procedures for the decontamination of field and sampling equipment are included in Roux's SOPs for the Decontamination of Field Equipment located in Attachment 3. The location of the decontamination area will be determined prior to the start of field operations. The decontamination area will be constructed to ensure that all wash water generated during decontamination can be collected and containerized for proper disposal.

Only "PFAS-free" water will be used for decontamination of sample equipment onsite. Only Alconox will be used as decontamination detergent (Liquinox shall not be used).

7.2 Waste Handling and Disposal

All waste materials (drill cuttings, decontamination water, etc.) generated during sampling will be consolidated, and stored in appropriate labeled bulk containers (drums, etc.), and temporarily staged at an investigation derived waste storage area onsite. Roux will then coordinate waste characterization and disposal by appropriate means.

TABLES

1. Field and Laboratory QC Summary
2. Remedial Action Sampling Summary
3. Preservation, Holding Times, and Sample Containers

Table 1. Field and Laboratory QC Summary

QC Check Type	Minimum Frequency	Use
Field QC		
Duplicate	1 per matrix per 20 samples or SDG*	Precision
Trip Blank	1 per VOC cooler	Sensitivity
Field Blank (non-PFAS)	1 per matrix per 20 samples	Sensitivity
Field Blank (PFAS)	1 per matrix per day	Sensitivity
Laboratory QC		
Laboratory Control Sample	1 per matrix per SDG	Accuracy
Matrix Spike/Matrix Spike Duplicate/Matrix Duplicate**	1 per matrix per SDG	Accuracy/Precision
Surrogate Spike	All organics samples	Accuracy
Laboratory Duplicate	1 per matrix per SDG	Precision
Method Blank	1 per matrix per SDG	Sensitivity

Notes:

* SDG - Sample Delivery Group - Assumes a single extraction or preparation

** Provided to lab by field sampling personnel

PFAS - Per- and Polyfluoroalkyl Substances

Table 2. Remedial Action Sampling Summary

Sample Medium	Target Analytes	Field Samples	Replicates[1]	Trip Blanks[2]	Field Blanks[1]	Matrix Spikes[1]	Spike Duplicates[1]	Total No. of Samples
Bottom Confirmation Soil Samples	TCL/Part 375 VOCs	35	2	10	2	2	2	53
	TCL/Part 375 SVOCs	35	2	0	2	2	2	43
	TCL/Part 375 Pesticides	35	2	0	2	2	2	43
	TCL/Part 375 PCBs	35	2	0	2	2	2	43
	TAL Metals	35	2	0	2	2	2	43
	PFAS	35	2	0	10	2	2	51
Sidewall Confirmation Soil Samples	TCL/Part 375 VOCs	22	2	10	2	2	2	40
	TCL/Part 375 SVOCs	22	2	0	2	2	2	30
	TCL/Part 375 Pesticides	22	2	0	2	2	2	30
	TCL/Part 375 PCBs	22	2	0	2	2	2	30
	TAL Metals	22	2	0	2	2	2	30
	PFAS	22	2	0	10	2	2	38

Totals are estimated based on scope of work as written, actual sample quantities may vary based on field conditions. QA/QC sample quantities will be adjusted accordingly.

[1] Based on 1 per day for PFAS or 1 per 20 samples or 1 per Sample Delivery Group for all other parameters.

[2] Based on 1 cooler per day. Assumes up to 10 sampling events conducted over time as the excavation progress.

TCL/Part 375 - USEPA Contract Laboratory Program Target Compound List

USEPA - United States Environmental Protection Agency

VOCs - Volatile Organic Compounds

SVOCs - Semivolatile Organic Compounds

PCBs - Polychlorinated Biphenyls

PFAS - Per- and Polyfluoroalkyl Substances

TAL - USEPA Contract Laboratory Program Target Analyte List

Table 3. Preservation, Holding Times and Sample Containers

Analysis	Matrix	Bottle Type	Preservation(a)	Holding Time(b)
TAL/Part 375 Metals (total) SW-846 6010D	Soil Water	8 oz wide mouth glass, teflon lined cap 250 mL plastic, teflon lined cap	Cool to 4°C Nitric acid	180 days, Hg 28 days
Mercury SW-846 7471B	Soil Water	2 oz wide mouth glass, teflon lined cap 500 mL plastic bottle	Cool to 4°C Cool to 4°C	
Hexavalent Chromium	Soil	2 oz wide mouth glass, teflon lined cap	Cool to 4°C	30 days from sample collection to extraction; 7 days following extraction
SW-846 7196A	Water	500 mL plastic bottle	Cool to 4°C	24 hours from sample collection
Total Cyanide SW-846 9010C	Soil Water	4 oz wide mouth glass, teflon lined cap 250 mL plastic bottle	Cool to 4°C NaOH	14 days from sample collection 14 days from sample collection
PFAS via EPA 1633A	Soil	One 8 oz HDPE bottle One 2 oz HDPE bottle	Cool to 4°C	14 days to extract, 40 days to analysis
PFAS via EPA 1633A	Water	Two, 250 mL HDPE bottles	Cool to 4°C	14 days from sample collection
1,4-Dioxane via 8270D 1,4-Dioxane via 8270D-SIM	Soil Water	4 oz wide mouth glass, teflon lined cap Two, 250 mL amber glass	Cool to 4°C Cool to 4°C	14 days to extract, 40 days to analysis 7 days from sample collection
TO-15	Air	6 liter 8-hour runtime Summa canister	None	14 days from sample collection
Target Compound List (TCL)/Part 375				
TCL/Part 375 Volatile Organic Compounds (VOCs)	Soil	Three 5 gram Encore samplers One 2 oz plastic bottle, teflon lined cap	Cool to 4°C	48 hours, or 14 days if extruded into sealed vial and either frozen to -7 degrees C or extruded into methanol
SW-846 8260C	Water	Three 40mL VOA vials, teflon lined cap	Hydrochloric Acid	14 days from sample collection
TCL/Part 375 Semivolatile Organic Compounds (SVOCs) SW-846 8270D	Soil Water	8 oz wide mouth glass, teflon lined cap Two 250 milliliter amber glass, teflon lined cap	Cool to 4°C	14 days to extract, 40 days to analysis 7 days to extract, 40 days to analysis
TCL/Part 375 Pesticides SW-846 8081B	Soil Water	8 oz wide mouth glass, teflon lined cap Two 120 milliliter amber glass, teflon lined cap	Cool to 4°C	14 days to extract, 40 days to analysis 7 days to extract, 40 days to analysis
TCL/Part 375 Polychlorinated biphenyls (PCBs) SW-846 8082A	Soil Water	8 oz wide mouth glass, teflon lined cap Two 120 milliliter amber glass, teflon lined cap	Cool to 4°C	14 days to extract, 40 days to analysis 7 days to extract, 40 days to analysis

(a) All soil and groundwater samples to be preserved in ice during collection and transport
(b) Days from date of sample collection.
TAL - Target Analyte List
PFAS - Per- and Polyfluoroalkyl Substances
TCL - USEPA Contract Laboratory Program Target Compound List
USEPA - United States Environmental Protection Agency

ATTACHMENTS

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4. Laboratory's Standard Operating Procedures

ATTACHMENT 1

Professional Profiles

PROFESSIONAL PROFILE



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EDUCATION
BS, Environmental Science,
Minor in Engineering,
Binghamton University, 2019
AS, Environmental Science,
Suffolk County Community
College, 2017

Brooke Hildebrand

Project Scientist

EXPERIENCE SUMMARY
Six years of professional experience: Project Scientist, Roux, Islandia, New York.

TECHNICAL SPECIALTIES
Phase I and II Environmental Site Assessments (ESAs); remedial construction and soil excavation oversight; hazardous and non-hazardous waste disposal oversight; New York State Pollutant Discharge Elimination System (SPDES) monitoring and Stormwater Pollution Prevention Plan (SWPPP) compliance; soil import oversight; waste characterization and delineation oversight; environmental site assessments focusing on soil, groundwater, and soil vapor investigations using multiple sampling techniques; drilling oversight; implementation of Community Air Monitoring Programs (CAMP); waterproofing and vapor barrier inspections; and insurance litigation support. Experience also includes management of remedial investigations and remedial actions for real estate and industrial sites in regulatory programs, including the New York State Department of Environmental Conservation (NYSDEC) Brownfields Cleanup Program (BCP), and New York City Office of Environmental Remediation (NYCOER) E-Designation and Voluntary Cleanup Programs.

REPRESENTATIVE PROJECTS

Industrial Site Assessment and Remediation

- Authored and assisted with Phase I Environmental Site Assessment portfolios for 13 parcels of land located in the southern portion of Brooklyn, New York. Responsible for maintaining report integrity and compliance with ASTM E1527-13 standards. Maintained client expectations and delivered accurate results in a timely manner to satisfy tight due diligence deadlines. Provided recommendations to the client where Phase II investigations were warranted.
- Staff on an insurance litigation case regarding the evaluation of past and future costs for the investigation and remediation of a site with past railroad, wood treating, and drum reconditioning operations. Responsibilities included the review and organization of over 20,000 documents, past cost categorization, and report writing assistance.
- Main author of various technical reports submitted to the NYSDEC for sites located throughout the boroughs, including remedial action report, construction completion report, periodic review reports, Phase II reports, daily construction reports, daily CAMP reports, final engineering report, site management plan, remedial investigation report, etc.
- Field manager for remedial excavation activities, CAMP implementation, and endpoint soil sampling at an affordable housing site located in Harlem, New York. Main author of the Site's final engineering report (FER) submitted to the NYSDEC following completion of the environmental role on-site.
- Field manager for the implementation of the On-Site Environmental Monitoring (OEM) program at a 43-acre redevelopment project in Belmont, New York. Responsibilities included ensuring environmental compliance of air, stormwater, noise, traffic, and other relevant environmental concerns during the performance of any construction-related activity across the Site.
- Field manager for a site in Sleepy Hollow, New York, performing soil importation and fill operation activities. Responsibilities include implementation of CAMP, management of soil importation, preparation of daily progress reports submitted to the NYSDEC, on-site soil tracking, and oversight of fill operations using on-site and imported fill.

- Field Staff for groundwater and soil vapor sampling monitoring programs at a former petroleum refinery and terminal in Brooklyn, New York. This work was done to monitor the largest subsurface free-product plume in North America. Field work responsibilities included the sampling of over fifty wells and monitoring points for petroleum-contaminated groundwater and soil vapor using multiple sampling methods.
- Field Manager responsible for the implementation of groundwater sampling activities at multiple locations including a high-end shopping mall site in Norwalk, Connecticut, and a Site in Brooklyn, New York.
- Field manager for NYSDEC BCP Site located in Gowanus neighborhood in Brooklyn, New York. Industrial site history and proximity to Superfund Site Gowanus Canal required a USEPA-approved remediation. Responsibilities included construction oversight, CAMP implementation, SWPPP, and groundwater sampling.
- Field manager responsible for air monitoring to collect interior air quality data during construction activities at a site in Hudson Yards, New York enrolled the NYSDEC BCP. Roux implemented monitoring by utilizing 13 stationary 5-gas meters with telemetry and two roaming 5-gas meters, capable of measuring the following air quality parameters: oxygen, carbon monoxide, hydrogen sulfide, methane (as LEL) and organic vapors (volatile organic compounds [VOCs]). Responsibilities also included construction oversight, analyzing air monitoring data, and preparing associated reporting daily.
- Field Manager responsible for environmental oversight of geotechnical work at a generating station in Astoria, New York. Responsibilities included monitoring and enforcing the subcontractor's adherence to specific procedures to prevent vertical migration of non-aqueous phase liquid (NAPL), logging and screening soil with a photo ionization detector (PID), recording lithology of soil cores, and confirming depths of clay layers and bedrock.
- Field Manager responsible for the implementation of CAMP during excavation, disposal, and import activities at multiple locations in Queens, Brooklyn, and Upstate, New York. Monitored airborne dust and VOCs potentially generated by remedial action work activities, reviewing the collected data for exceedances of the New York State Department of Health (NYSDOH) guidelines.
- Site Safety Officer for various remedial investigation sites. Responsibilities included preparation of Health and Safety Plans (HASPs), development and review of job safety analysis (JSA) documents, on-site safety meeting management, safety document preparation (Lessons Learned, Near Loss, Field Audits, etc.), and planning execution of corrective actions.
- Project Manager for multiple public sector sites located in Queens and Manhattan with petroleum contamination. Responsibilities include project management, coordination of groundwater monitoring and product recovery events, data evaluation and reporting, and client communications.
- Project Manager for NYSDEC BCP Site located in Upstate, New York. Responsibilities included project management of the remedial investigation, coordination of day and night shifts, work within an active commercial building, data evaluation, preparation of a remedial investigation report and remedial action work plan, client communications, and NYSDEC communications.
- Project Manager for multiple Phase II investigations conducted at sites located in Brooklyn, Queens, and Manhattan. Responsibilities included subcontractor coordination, project management, data evaluation, reporting, and client communications.
- Project Manager for two affordable housing redevelopment projects located in Bedford-Stuyvesant, Brooklyn with E-designations. Responsibilities included project management of Phase II ESAs, preparation of NYSDEC BCP applications, Remedial Investigation Work Plans, HASPs, CAMP, Quality Assurance Project Plans, and client management.
- Project Manager for a commercial mall site within the New York City Office of Environmental Remediation (NYCOER) Voluntary Cleanup Program (VCP) located in Staten Island. Responsibilities included project management related to soil disturbance oversight and air monitoring, tenant and OER coordination, and preparation of remediation summary documents to receive notices of no objection (NNO) from OER for tenant occupancy.
- Project Manager for the largest residential geothermal building in New York State located in Greenpoint, Brooklyn. Responsibilities included coordination of groundwater monitoring well and soil vapor point installation, preparation of NYSDEC BCP documents including FER, Site Management Plan, Soil Vapor Intrusion Evaluation Work Plan, client management, and coordination with NYSDEC and NYCOER to obtain NNOs for temporary certificates of occupancies.

PROFESSIONAL PROFILE



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EDUCATION

BCE, Civil Engineering,
University of Delaware, 1987
MBA, Management, Adelphi
University, 1992

PROFESSIONAL LICENSES

Professional Engineer:
New York, New Jersey, Pennsylvania, Rhode Island, Connecticut, Vermont, Virginia, North Carolina, Ohio, Michigan, and Montana

Charles J. McGuckin, PE

Vice President | Principal Engineer

EXPERIENCE SUMMARY

Over thirty years of experience: Principal, Senior and Project Engineer with Roux Associates, Inc.; President of Remedial Engineering, P.C.; and Design Engineer at Dvirka and Bartilucci Consulting Engineers.

TECHNICAL SPECIALTIES

Engineering design of soil and groundwater remediation systems. Brownfields clean-up plans, stormwater studies, and engineered natural treatment systems.

REPRESENTATIVE PROJECTS

- Principal Engineer for remedial investigation scoping, fate and transport analysis, and feasibility study preparation for a 1300+ acre Former Aluminum Smelter USEPA Superfund Site, Montana. Due to the size, complexity, and unique exposure areas of the Site it was broken up into multiple decision units including landfills, percolation ponds, operational areas, river area, soil, and groundwater. The contaminants of concern included metals, PAHs, cyanide, and fluoride. Remedial technologies evaluated in detail included landfill capping, groundwater extraction and treatment, pond and river dredging, soil excavation, slurry wall containment, in situ stabilization, and in situ groundwater treatment. Detailed effectiveness, implementability, and cost evaluations were prepared to support preferred remedial alternatives for each decision unit.
- Principal Engineer for remedial action implementation for a 2.5-acre former subway transit power station along the Gowanus Canal, Brooklyn, New York. The project remedial action was completed under an extremely tight deadline to obtain the certificate of completion under the NYSDEC BCP program. The project also involves close coordination with USEPA due to the adjacent canal superfund status. The investigation required in depth review of historic facility operations and sampling of subsurface tunnel structures. The remedy included excavation of PCB impacted soils, LNAPL recovery, shoreline stabilization, concrete scarification, sewer cleaning and capping. The project has required close coordination with the development team to ensure management of impacted materials in conformance with the Site Management Plan.
- Principal Engineer for the preparation of field implementation plans, construction monitoring, and Engineers Certification Report for a former manufactured gas Plant (MGP) site in West Chelsea, Manhattan, New York. The site was one of the first projects completed under the NYS Brownfields Clean-up Program. The remedy included soil excavation and offsite thermal treatment, a sheet pile barrier wall, a vapor barrier, and basement ventilation system. A comprehensive air monitoring program was conducted due to the concerns over coal tar residue emissions and odors on the surrounding community. The remedy was incorporated into the design and construction of the headquarters office building of an international media company.
- Principal engineer for the preparation of the feasibility study, IRM plans, and remedial design/remedial action plans for a 40-acre former manufacturing facility in Rensselaer, New York. IRM Soil remediation included excavation of over 10,000 cubic yards of CVOC and metals source material for disposal at multiple facilities based on waste characteristics. Basement cleaning was performed in three large buildings to remove accumulated process sludges. Lagoon closure plans included sediment removal, dewatering, soil washing, and soil capping. The final remedy for the site includes a groundwater perimeter containment trench and 40 gpm treatment system for metals and VOCs and a 9-acre vegetated cap for a former landfill.

- Principal Engineer for the feasibility studies and remedial action work plans for multiple operable units of a large railyard located in Sunnyside, Queens, New York under the NYSDEC Inactive hazardous waste program. For the former engine house and maintenance area unit, pre-design studies included product plume thickness data collection and modeling, ex situ biopiles treatment, in situ enhanced bioremediation, and in situ chemical oxidation. The final design consisted of decontamination and removal of structures, excavation of hot spot soils for PCBs and lead, UST closures, a dual phase high vacuum extraction system and in situ bioremediation.
- Project Engineer for the design and construction management of a 600 gpm groundwater extraction and treatment system to prevent offsite migration at a petroleum storage and pipeline transfer facility in Providence, Rhode Island. The treatment system was designed to remove iron, BTEX, and naphthalene from the groundwater to below surface water discharge standards for the Providence River. The system processes consisted of equalization, aeration, de-aeration, flocculation, clarification, air stripping, dual media filtration, granular activated carbon adsorption (liquid and vapor phase), and sludge thickening and dewatering. The system included an outfall diffuser designed in accordance with the CORMIX computer model.
- Senior Engineer responsible for the design, construction management, and O&M of a 60,000-gpd constructed wetlands treatment system for a former manufacturing facility in Virginia. The 16-acre treatment system was designed within an existing phragmites wetland to remove zinc and iron from landfill leachate prior to discharge to an adjacent creek. The treatment system consisted of alkalinity producing cells, toxic ponds, compost and limestone berms, anaerobic cells, and aerobic cells. The design included a 400-foot reinforced earthen dike together with hydraulic control structures and piping to maintain cell water levels and flow rates. The system also includes a pump station and force main for both effluent discharge and irrigation purposes. Joint wetlands and local permit approvals were obtained for the project.
- Senior Engineer for the performance of a feasibility study and remedial design for the closure of a concrete oil/water separator filled with refinery sludge and demolition materials impacted with lead at a former refinery in Providence, Rhode Island. Remedial alternatives were developed and evaluated including capping and containment using a perimeter slurry wall, sheet piling or concrete wall sealing; excavation and disposal; and in situ solidification. The capping and containment using a slurry wall alternative was selected for implementation of the remedial design. The design consisted of removal and replacement of existing monitoring wells, sealing of separator wall openings, a 2-acre multi-layer cap, a 1200-foot long by 30-foot deep soil-bentonite slurry wall, and a perimeter drainage swale. The multi-layer cap included a 40-mil HDPE geomembrane and a geosynthetic clay liner. The slurry wall was keyed into the existing clay confining layer beneath the separator. The design incorporated disposal of an additional 10,000 cubic yards of petroleum impacted soil under the cap.
- Principal Engineer for environmental consulting support services for a large landfill O&M contract under review by the New York City Department of Investigation. The work entailed reviewing the scope of routine vs. non-routine work performed over a one-year period for compliance with contract requirements. The O&M Work included routine cover maintenance, groundwater and gas monitoring, landfill gas extraction, major system repairs and waste handling. Memos of findings were prepared assessing acceptability of work, compliance with permit regulations and providing recommendations for improvements.
- Principal Engineer for the independent engineering review of change orders for the New York MTA Office of the Inspector General associated with electric utility substations reconstruction damaged during the 2012 Superstorm Sandy. The cost review focused on contracting procedures, waste classification of impacted structures and soils, proper waste management and disposal. Findings were compiled in a report to determine if costs were legitimate and justifiable and providing recommendations for improved specifications for bidding and of management waste handling contracts.
- Principal Engineer for remedial action plan implementation oversight and certification for the CornellTech campus development on Roosevelt Island, New York. The first phase of the campus development included lead paint and ACM abatement and demolition of the former Goldwater Hospital, construction of six main campus buildings, new utilities, roadways, and lawn/landscaped areas. Responsibilities include oversight of soil/subsurface structures excavation handling, disposal, and reuse; community air monitoring; dewatering permit compliance; and SWPPP inspections.
- Senior and Principal Engineer for the decommissioning and decontamination of a pharmaceutical facility covering seven city blocks as a part of a NYSDEC Voluntary Cleanup Agreement in Brooklyn, New York. The former office and laboratory complex would be decontaminated for reuse as a school and small business space. Multiple other buildings were demolished. Responsibilities included preparation of interior abatement plans to address mercury, lead and PCBs in building materials and review of Interim Remedial Measure (IRM) work plans for lead, benzene and mercury-contaminated soil excavation and disposal. Groundwater remediation design

included air sparge/soil vapor extraction, in situ oxidation and a reactive barrier wall using colloidal carbon and ZVI.

- Principal Engineer for the performance of a Brownfields Demonstration Pilot Program in the Hamlet of New Cassel for the Town of North Hempstead, New York. Under an EPA grant, Roux Associates created an inventory of 50 potential commercial/industrial properties within New Cassel and evaluated these properties based on perceived contamination and potential for redevelopment/reuse. Eight sites exhibiting the greatest potential for redevelopment were selected to perform Phase I Environmental Site Assessments. Of these eight sites, four sites were selected for Brownfield Site investigations to identify the nature and extent of contamination in soil and groundwater and provide potential remedial alternatives and cleanup costs to revitalize these properties. The Brownfields Demonstration Pilot Program also included community outreach activities to promote a unified approach to the redevelopment of Brownfields in new Cassel.
- Principal Engineer responsible for engineering certification of all remediation activities related to the seven-city-block Barclay's Arena and Atlantic Yards redevelopment in Brooklyn, New York. This multi-billion-dollar redevelopment includes the Arena, which will be focal point of the largest redevelopment project in Brooklyn, consisting of an urban complex of housing, commercial and retail space, as well as several acres of landscaped public open space. The existing properties being redeveloped are residential, commercial, and industrial properties, including a large railroad yard. Engineering certification included multiple RAWPs under NYSDEC Spills Program, UST removals, soil excavation, in situ groundwater treatment and remedy oversight services. The project also includes ACM abatement, building demolition, soil pre-waste-classification, coordination (with the receiving facilities), and oversight of the removal of 1,000,000 cubic yards of soil (~550,000 yards removed to date), representing one of the largest excavation and soil removal projects performed in New York City.
- Principal Engineer responsible for the preparation of the remediation completion report at Captain's Cove former municipal landfill State Superfund Site located in Glen Cove, New York. This work has been performed in accordance with Title 3 of the NYS Environmental Quality Bond Act under contract to the City of Glen Cove. Design elements included excavation plans, radiological waste monitoring, demo debris and waste separation and screening, dewatering water management, waste disposal, and site restoration. Additional work included the delisting of a six acre "clean" portion of the site to allow the development of a ferry terminal and esplanade and development of alternative cleanup standards consistent with future site uses. Site remediation will accommodate site redevelopment as a commercial waterfront and operating ferry service and seaport area.
- Principal Engineer providing program management of interior building materials surveys for 22 residential buildings along the south shore of long island under an Army Corps of Engineers contract for dune reconstruction. Building materials surveys included testing and analysis of suspect contaminants and reporting in support of building abatement and demolition planning. Testing was completed using multiple teams on a tight timeline to meet project schedule requirements.
- Principal Engineer for the remediation of a former Manufactured Gas Plant (MGP) facility in Brooklyn, NY, including oversight of the excavation of both the former gasholders, and adjacent contaminated hotspots requiring offsite thermal desorption of over 30,000 tons of coal tar impacted soil. Directed the Community Air Monitoring Program (CAMP) specific to the MGP impacted soil removal, as required by both New York State Department of Environmental Conservation (NYSDEC) and New York State Department of Health (NYSDOH). Remedial activity met all substantive requirements of the NYSDEC approved Remedial Action Work Plan for the Site. The remedy included design of a passive subsurface vapor monitoring/recovery system for a 500,000 sq. ft. retail structure in Brooklyn, NY. The system design integrated a perforated piping system complemented by a protective vapor barrier below the structural floor slab to monitor and mitigate volatile organic compound vapors. Multiple vapor barrier options were evaluated to determine the optimum design based on the site conditions.
- Principal Engineer providing expert settlement support services to a county municipality in New York State. The case involved an EPA Order for underground storage tank (UST) compliance for over 50 county operated facilities with over 125 USTs. The project involved the field inventory of the USTs at each facility and development of both Interim and final compliance plans to comply with EPA, NYSDEC and local UST regulations. Detailed cost estimates were prepared for multiple scenarios for upgrading USTs including tightness testing, manway repairs, leak detection and overfill protection monitoring systems, UST removal and replacement, and new piping. The upgrade evaluation and negotiations included incorporation of Supplemental Environmental Project (SEPs) in accordance with EPA requirements. SEPs included centralized monitoring systems for leak detection and inventory control.
- Principal Engineer for preparation of a site management plan for redevelopment of a former watch case factory in Sag Harbor, New York. The primary engineering controls for the former factory conversion to a residential building consisted of a vapor barrier and an active sub slab depressurization system

(SSDS) to address chlorinated VOCs. The SSDS system was complicated due to the existing 100-year-old structure. A unique raised floor approach was designed to allow for the SSDS installation. The system design, approved by NYSDEC and NYSDOH includes multiple legs, dual blowers, low vacuum alarms and monitoring points.

- Principal Engineer for the Remedial Action Work Plan (RAWP) for redevelopment of a shopping center in the Bronx, New York. The RAWP elements included soil and groundwater management plans, stormwater management, air monitoring and vapor mitigation systems. To address vapor intrusion, active sub slab depressurization systems were designed for two pad buildings. One system for a new retail building construction and one retrofit system for an existing building to be used as a restaurant. Closure reports were prepared and certified documenting all remediation work and approved by NYC Mayor's Office of Environmental Remediation (OER).
- Principal Engineer for the preparation of a preliminary remedial design for the remediation and restoration of a pond and surface water tributaries to Canaan Lake that have been impacted from leachate generated from an upgradient former municipal landfill located in Holtsville, New York. Completed a preliminary remedial design for the construction of a compost-based permeable reactive barrier for the removal and treatment of leachate prior to discharge to the surface water, followed by restoration of the surface water body and surrounding wetlands. The project included development of a long term remedial strategy to reduce rainfall infiltration into the landfill and minimize leachate generation. Current plans to reduce rainfall infiltration include the planting of 3,250 hybrid poplars, regrading and lining of drainage swales, and the resurfacing of low lying areas consistent with recreational facilities.
- Principal Engineer for final capping elements and wetlands restoration work and completion of the Final Engineering Report for an inactive hazardous waste site in Syracuse, New York. The project included onsite consolidation of lead impacted waste; 7-acre landfill cap with vegetated layer, cover soil, and geomembrane; stormwater runoff controls; reconstruction of wastewater ponds; and an 8-acre wetland restoration. An O &M Plan was prepared and implemented consisting of groundwater, surface water and landfill gas monitoring, and annual cap and wetland inspections.
- Principal Engineer for the preparation of the remedial action work plan for an 11-acre former Department of Defense owned Site that manufactured airplane parts along Hempstead Harbor in Manorhaven, New York. The project is regulated under the NYSDEC Voluntary Cleanup Program. The remedial design consisted of both soil vapor extraction/air sparging and in situ enhanced bioremediation systems for Site groundwater impacted by chlorinated VOCs. The final remedial design and site management plan are expected to include soil capping, vapor barriers and passive ventilation systems to be incorporated into a residential redevelopment with waterfront access.
- Senior Engineer for the performance of a stormwater runoff evaluation for a manufacturing facility in Watertown, New York. Roux Associates was retained as third party to evaluate the drainage design and construction elements for an industrial landfill cap. The evaluation was performed for the facility owner in support of potential litigation arising from onsite building flooding incidents following a severe snow and rainstorm event. The scope of work included an evaluation of the existing onsite storm sewer system capacity, calculation of runoff flow rates for the 300-acre contributing area, review of landfill cap surface drainage design, review of erosion control measures implemented during construction, and analysis of specific flooding incident causes. The runoff analyses were performed using the TR 55 Method for three conditions: pre-capped, capping under construction prior to establishment of vegetation, and final vegetated cap design. Recommendations were made to improve the site drainage including design of surface drainage swales, temporary berms and sediment traps during construction and modification of snow handling practices.
- Principal Engineer for the management of a soil and groundwater remediation system for a nationwide overnight delivery distribution center in Brooklyn, New York as part of the NYSDEC Voluntary Cleanup Program. A risk-based remedial approach that called for the remediation of "hot spot" source area soils, and mass-reduction of VOCs was successfully utilized for the Site. As a result, the focus of remediation was on reducing the mass of VOCs in on-site groundwater to a level where natural attenuation would be effective in remediation of VOCs. To address the contamination in the source area, a soil vapor extraction (SVE) and air sparge (AS) system consisting of 8 SVE wells and 17 AS wells was designed, constructed, operated and maintained for a period of approximately 3 years. Permanent shutdown of the system was approved by the NYSDEC.
- Senior Engineer for the design and construction management of a soil remediation and stormwater management project at a 16-acre former pesticide warehouse facility in Dayton, New Jersey. The Site was redeveloped for storage and trailer parking. The project consisted of consolidation of pesticide contaminated soils; asphalt capping of the 3.5-acre contaminated soils area; stormwater collection, conveyance, and detention; and site regrading. The evaluation included TR-55 runoff modeling for pre and post capping and development conditions. The storm sewer system consisted of

multiple catch basins, over 2,000 linear feet of reinforced concrete pipe ranging in size from 15 to 30 inches, and a recharge basin. A Soil Erosion and Sedimentation Control Plan and a NJPDES General Permit were prepared for the project.

- Project Principal for the performance of LNAPL remediation studies at the New Jersey Transit former Lake Street Bus Garage in Newark, New Jersey. The studies involved evaluating remedial alternatives for free product recovery, performance of an LNAPL recovery pilot test and cost estimating. A RAWP and engineering design plans were prepared for both the bus garage and the adjacent park properties. The remedy included excavation of the source area, horizontal recovery wells, a vertical recovery trench, in situ oxidation injections and product recovery using vacuum extraction.
- Senior Engineer for the performance of a stormwater management analysis for a 28-acre industrial landfill in Virginia. The principal objective of the study was to identify engineering controls to minimize stormwater runoff to a metals-contaminated sediment impoundment. The study included TR-55 runoff modeling and storage analyses for multiple detention ponds. Three engineering control alternatives were identified including landfill cap regrading, diversion using berms and swales, and diking and weir raising.
- Senior Engineer for the investigation, design, and construction management of the closure of a 2-acre fire-water supply pond and modification of the stormwater conveyance system at a former manufacturing facility in Williamsburg, Virginia. The investigation phase of the project was focused on determining the sources and loading of metals influent to the pond. Field activities included examination of the existing stormwater drainage system, sub watershed delineation, groundwater monitoring, and installation of automatic stormwater sampling devices. The final design included 400 feet of open concrete channels, 250 feet of culvert replacement, slip lining of 370 feet of 36-inch RCP culvert, reconstruction of five catch basins, placement of 10,000 cubic yards of clay fill within the pond and regrading of existing drainage ditches. Erosion control measures and slope stabilization were also included as well as the design of a special outlet structure for minimizing erosion at the outfall.
- Project Principal for the investigation and closure of five USTs at the New Jersey Transit Broad Street Station site in Summit, New Jersey. Tank sizes ranged from 20,000 to 30,000-gallon capacity. UST closure program completed in accordance with the NJDEP Technical Requirements for Site Remediation. Closure report prepared and submitted to the NJDEP and subsequent issuance of a No Further Action letter from the NJDEP.
- Project Engineer of the underground storage tank (UST) program for a major retail chain store in the New York, New Jersey, and Pennsylvania region. Responsibilities included preparation of a UST management plan based on federal, state, and local regulations and costs to prioritize UST maintenance. The tank designs included plans and specifications for the removal and replacement, or upgrading, of USTs to meet regulatory requirements. The engineering design involved fuel requirements for dual heating and back-up generator usage, mechanical pumping equipment and fire wall design.
- Project Engineer for the design and construction management of a 1,000 sq. ft. hazardous and flammable materials storage facility in Syosset, New York. The facility included concrete secondary containment dikes, access ramps, sprinkler system modifications, and lighting. The separate flammable materials area included 2-hour fire rated concrete block walls and doors, ventilation equipment and a fire alarm system. Permitting services were performed for the Nassau County Department of Health, the Nassau County Fire Marshall, and the Building Department.
- Project Engineer for the design of a 2,000 sq. ft. hazardous waste storage facility in Astoria, New York. Prior to construction, demolition of an existing building was required and included removal of asbestos and lead paint. The project included driving treated timber piles and excavation and removal of contaminated soil and groundwater. The structure consisted of a steel frame with a metal standing seam roof system, decorative masonry block walls, and a roll-up door. Temporary and permanent fencing were required along with concrete sidewalk replacement.
- Senior Engineer providing construction management services in support of the BNYCP Cogeneration Facility construction and Brooklyn Navy Yard facility decommissioning. Work included preparation of construction management plans, supervision of soil, concrete, and sediment disposal activities, asbestos surveys, and PCB sampling and analysis work. A NYCDEP wastewater discharge permit was prepared for the million gallon per day stream condensate and wastewater backwash flow rate.
- Project Principal for performing remedial alternative cost estimating for a New Jersey Transit site in Montclair, New Jersey, which is to be redeveloped as a firehouse. A cost estimate prepared by another consultant was reviewed as part of the scope of work. The proposed remedial alternative for the site consisted of excavation and disposal of PAH-impacted fill material and capping. The alternative remedy proposed by Roux Associates was a more risk-based approach, resulting in a cost savings of approximately $100,000 for New Jersey Transit.

- Project Engineer for the design and construction management of cap repair and drainage improvement measures for an industrial hazardous waste landfill in Tennessee. Components of the design included replacement of the primary clay cover material, temporary and permanent erosion and sedimentation control measures, and a lined drainage channel to minimize the generation of landfill leachate. The project included the performance of a focused feasibility study to characterize the flow, quality, and treatability of the leachate. A feasibility study was also performed in order to evaluate constructed wetlands remedial technology as a method of effective and economical treatment of leachate.
- Senior Engineer for the remedial design and construction management of a 7-acre off-spec asphalt waste pond at a former refinery in New England. The asphalt material exhibited a low load bearing capacity combined with a viscous, tacky surface. An in situ solidification mix design was developed consisting of liquification using hot water and a 2-stage lime kiln dust reagent injection and mixing step. Gravel was added to the mix when the existing subgrade material was of insufficient bearing capacity. Solidified material was tested for unconfined compressive strength, durability, and TCLP. The final cover material consisted of a 6-inch vegetated layer.
- Principal Engineer for the performance of LNAPL remediation studies for a former bus maintenance facility and a segment of a Metropolitan Subway System in Newark, New Jersey. The studies involved evaluating groundwater and soil monitoring data, performance of LNAPL recovery pilot tests, evaluation of remedial alternatives and cost estimating. Recommendations included the use of mobile high vacuum extraction methods to collect LNAPL while minimizing capital expenditures and permanent low vacuum extraction methods to minimize odors to subway cars and surrounding communities.

Litigation Support Experience

- Project Engineer for the evaluation of remedial investigations and remedial cost estimates for a 30-acre former book publishing facility in Poughkeepsie, New York. The evaluation included the review of Phase I and Phase II investigation reports, remedial investigation (RI) and feasibility study (FS) reports, and the remedial investigation work plan. The findings included the presence of chlorinated volatile organic compounds in the soil and groundwater as well as identification of underground storage tanks. Deficiencies were identified in both the RI and FS reports by comparing with the NYSDEC's required criteria and recommendations were proposed for the RI work plan to further delineate source areas. Based on the remedial investigation review, revised costing assumptions were made, and remedial cost estimates were prepared totaling $3.6 million.
- Project Engineer for the evaluation of expected remedial costs for nine hazardous waste sites, two of which are federal superfund sites. The evaluation of both single and multiple PRP sites was performed to identify costs for an insurance claim. The expected remedial costs for nine sites, which include landfills or facility surface impoundments, totalled approximately $65 million. Remedial plans evaluated for multiple site operable units included groundwater pump and treat, alternative water supply systems, soil/sludge in situ solidification and treatment, and wetlands restoration. Additional work included evaluating invoices for site work previously performed and allocating expenses into their appropriate operable unit and work type, i.e., defense or indemnity.

Water Treatment Experience

- Senior Engineer for the engineering design of a 10 gpm groundwater recovery and treatment system at a former tank farm in Rhode Island. The recovery system included a 200-foot slotted HDPE horizontal well, a 400-foot coated concrete swale and curbing, and a series of seepage collection points manifolded to a common receiving structure. The entire system was designed for passive recovery and gravity flow transmission targeting free-product seepage areas. The treatment system consisted of a collection sump retrofitted within an existing separator, a coalescing plate oil/water separator, a surge tank, a bag filter, and carbon adsorption units. The project included a permit modification for discharge to the Providence River.
- Design Engineer for the design and start-up operation of a 2 mgd packed tower aeration system for potable water in Williston Park, New York. The primary contaminants were trichloroethane and tetrachloroethene which were stripped below drinking water standards. The design process included full scale pilot testing to assure proper removal levels.
- Design Engineer for the design, construction, and start-up operation of a 5 mgd industrial cooling water treatment system utilizing mechanical surface aeration. The system consisted of two lined aeration basins operating in series with floating mechanical aerators to remove volatile organic contaminants to levels suitable for recharge into the Long Island groundwater aquifer. The primary contaminants were 1,1-dichloroethene, trichloroethane, tetrachloroethene and vinyl chloride.
- Design Engineer for the design and construction of a 4 mgd granular activated carbon system for potable water in Hempstead, New York. The primary contaminants consisted of more than 8 volatile and semi volatile organic compounds. Responsibilities included site inspection for the installation of the six vessels containing 20,000 lbs. of carbon in each. The

system was designed for 99.9% removal efficiency with two units operating in series.

Constructed Wetlands Experience

- Senior engineer for the conceptual design of a constructed wetlands stormwater treatment system for a coal handling freight railroad facility in Norfolk, Virginia. The design consists of treatment of contaminated stormwater runoff generated from maintenance and fuel handling areas onsite. The design treatment performance objective is the reduction of total suspended solids, oil and grease, and selected metals to levels below the SPDES permit discharge standards established for two of the site's outfalls discharging to the Elizabeth River. The 3 acre system consists of a passively operated 200,000 gpd subsurface-type constructed wetlands with a low visual impact and specialized structural design to meet the needs of a busy railyard facility. Additional design components include stormwater bypass structures, jacking beneath tracks, a grit chamber, a lift station, and outfall modifications. A joint wetlands permit will be prepared for the project.
- Senior Engineer for the feasibility study, conceptual design, and construction of four constructed wetlands units and sedimentation basin for a stormwater treatment system along Cedar Swamp Creek for the City of Glen Cove, New York. The project consisted of review of stormwater studies of the 12 square mile contributing watershed, compilation of USGS water quality and flow data, evaluation of stormwater treatment methods and best management practices and optimum site selection along the creek. The constructed wetlands design included a forebay, high and low marsh cells, a micropool, and stormwater bypass structures for removal of sediment, nitrogen, phosphorus, and trace metals during first flush events. Final design for the first 1.8 acre constructed wetlands unit was completed and performance of construction management is ongoing. Design activities include structural and hydraulic design tasks with specific emphasis on storm water bypass. The design has been integrated into an into an intermodal transportation project with the addition of bicycle and walking paths. NYSDEC and Army Corps permits were obtained for the project.
- Project Engineer for the design of a 7,000 gpd subsurface flow-type constructed wetlands treatment system for a refinery site in Rhode Island. The system was designed to treat a surface-water stream impacted by petroleum hydrocarbons. The system's high aesthetic, low visual impact appeal was ideal for its golf course setting. Both phragmites SPP and Typha SPP wetland species were incorporated in the design in order to assess the biodegradation/biotransformation processes effectiveness. A growth and maturation plan and a treatment evaluation plan were developed in order to evaluate the system performance.
- Lead Engineer responsible for technical review of a design for modifications to a constructed wetlands system in Nicholas County, West Virginia. The system was designed to treat the leachate from a solid waste landfill at a maximum capacity of 30 gpm. The complete water-tight treatment system consisted of a sedimentation basin, stabilization basin, a series of three wetland cells and a finishing ditch. The wetland cells consisted of a double liner system with leachate collection piping overlaid with stone fill and a matrix of plant life. The technology combines physical, geochemical, and biological removal mechanisms operating simultaneously.

Permitting/Compliance Plans

- Project Engineer for the preparation of a Spill Prevention Control and Countermeasure (SPCC) Plan and a Storm Water Pollution Prevention Plan (SWPPP) for an 850-acre petroleum storage terminal in New England. The SPCC Plan involved the inventory of 50 bulk storage tanks and miscellaneous storage vessels and an assessment of barge loading areas, truck loading racks, additive loading areas, pumping stations, and a network of aboveground pipelines. The SWPPP encompassed an inventory and surveying of the existing storm sewer system, an evaluation of oil/water separator performance and identification of storm water management controls and practices.
- Project Engineer for the design of modifications to multiple discharge facilities along the Providence and Runnins Rivers in Rhode Island. Permitting activities were performed with the following agencies: Rhode Island Department of Environmental Management (RIDEM) Pollutant Discharge Elimination System (RPDES), RIDEM Division of Freshwater Wetlands, Coastal Resources Management Council (CRMC), and the Army Corps of Engineers.

Sanitary Experience

- Design Engineer for the evaluation of a municipal sanitary sewer system consisting of approximately 70 miles of piping ranging in size from 8 inches to 16 inches, in Garden City, New York. The sewer system was evaluated for existing and proposed flow capacity, surcharging, infiltration of groundwater, inflow of storm water, root encroachment, and sewer breaks. Evaluation methods consisted of hydraulic profile analysis, television inspection of piping, field inspection of manholes, and flow measurement. Sewer upgrading methods were evaluated including direct replacement, manhole restoration and pipe slip lining, and a rehabilitation program was implemented.
- Design Engineer for the City of Glen Cove's industrial wastewater pretreatment program which was established to monitor significant industrial users discharging to the city's wastewater treatment plant to minimize upsets to the

biological treatment mechanisms. The program work included annual facility inspections, wastewater discharge sampling, review and evaluation of quarterly self-monitoring results, calculation of discharge penalty fees, preparation of annual monitoring reports for each facility and development of wastewater discharge permits to comply with City regulations.

- Design Engineer for a heavy metals study for the municipal sanitary sewer system in the City of Glen Cove, New York. The heavy metals study consisted of the development and performance of a city-wide sewer sampling program to identify the sources of heavy metals loadings on the wastewater treatment plant. The evaluation included industrial sources, scavengers, non-industrial sources, the plant operation itself, and review of existing heavy metal studies. Recommendations were provided for minimization of loadings and pretreatment to protect the plant operations.

Stormwater Experience

- Design Engineer for the evaluation and conceptual design of a water management plan for a 200-acre proposed office complex in Bethpage, New York. The design included inlets, piping and recharge basin sizing for peak storm water runoff flows as well as a system of architectural ponds and level control structures. For dry periods, the design included flow controls connected to an existing cooling water system to maintain pond levels and for utilization as a water supply for an irrigation sprinkler system during the growing season.
- Design Engineer for the design of a municipal storm drainage system for a 200-acre contributing area in Garden City, New York. The purpose of the drainage system was to alleviate severe flooding problems for eight homes located in a local low point of a residential neighborhood. The system included over 4,800 linear feet of reinforced concrete piping ranging in size from 12 to 60 inches. Design considerations included hydraulic gradient analysis, inlet capacity, utility crossings, minimization of removals of established trees, a county road crossing, utilization of existing structures and piping, and a headwall discharge to a recharge basin. Additional design items included pavement restoration, service line relocations, curbs and sidewalks, and maintenance and protection of traffic.

Site Assessment Experience

- Senior Engineer for coordination and review of Phase I environmental site assessments for five large research and development complexes located throughout the eastern United States for a major chemical company. The site assessments were performed for due diligence prior to engaging in long-term property lease agreements. The site assessments evaluated chemical storage and handling areas and previous site usage.
- Senior Engineer for coordination and review of Phase I environmental site assessments for 12 properties associated with tennis centers acquisition on Long Island, New York. The properties were either active tennis center facilities or vacant parcels available for new construction. All site assessments were conducted in accordance with ASTM standards for commercial real estate transactions. Primary concerns identified were USTs, drum storage areas, and unauthorized dumping.
- Project Manager representing a group of banks investing in a 20 acre commercial property in Westchester, New York. The onsite soil was contaminated with several volatile and semi volatile organics. Performed an evaluation of the remediation plan which included onsite biological treatment of soils and aeration and oil water separation of groundwater.

Water Main Experience

- Project Engineer for the design of over 6,000 feet of ductile iron water main in sizes from 4 to 16 inches for Town of Hempstead, New York Department of Water and the Nassau County, New York Department of Public Works. The designs included wet and dry connections to existing mains, fittings, valves, copper services and fire hydrants. Restoration work included replacement of asphalt pavement, concrete sidewalk and curbs, and grass areas.
- Design Engineer for the design and construction management of over 10,000 feet of ductile iron water main in sizes from 6 to 12 inches for the Town of Wallkill, New York. The designs included booster pump station upgrades, a stream crossing, a wetlands crossing, jacking of 36-inch casing beneath a state highway, air release chambers, copper service re-connections, fire hydrants, valves, and appurtenances. Restoration work included wetlands restoration, backfilling, and regrading within a NYSDOT right-of-way and grass and pavement replacement.
- Design Engineer for the design and construction management of upgrades to a 3.7 mgd potable water booster pump station for the Town of Wallkill, New York. The design featured the replacement of a hydropneumatic tank and pump system with three larger capacity centrifugal pumps. The upgrades were performed while maintaining the pump station service. The pump station revisions included piping, pump pads, shut-off valves, silent check valves, pressure relief valves, gauges, ventilation equipment and a motor control center.

Feasibility Study Experience

- Senior Engineer for the performance of a feasibility study and remedial design of a free product containment and recovery system at a former refinery in New England. The areal extent of the free-product plume was approximately 10 acres with a measured thickness of up to eight feet. Pilot testing activities consisted of pump tests, baildown tests, and funnel and gate

systems with and without sheeting. The selected remedial alternative consisted of re-routing and repair of active storm sewer piping, closure-in place of a former 72-inch storm drain using clay fill material to form a barrier wall, and installation of multiple recovery trenches totaling 450 linear feet. The recovery trenches were installed to a depth of 14 feet using a deep trenching machine and were completed with gravel, horizontal perforated piping, recovery wells, and monitoring wells to accommodate both passive and active product recovery pumping equipment. Product recovery enhancement pilot testing was also performed by using non-ionic surfactants, mechanical reworking of soil and vacuum extraction methods.

- Project Engineer for the performance of a feasibility study for the containment of a free-product plume beneath a refinery site in Rhode Island. The feasibility study included analysis of groundwater modeling, bench and pilot scale treatability studies, groundwater quality characterization, identification and screening of discharge alternatives, and treatment process evaluations. The work also included the evaluation of the discharge of treatment system effluent to several receptors including groundwater, wetlands, sanitary sewers, and storm sewers. Discharge requirements were evaluated for process water, off-gas air, and residual wastes. Several treatment processes were also evaluated including metals precipitation and sludge dewatering, VOC and SVOC removal, and off-gas treatment. Preferred alternatives for each process were selected for remedial design development.
- Project Engineer for the performance of a feasibility study for a hazardous waste landfill located at a Superfund site in Tennessee. The feasibility study focused on the characterization and quantification of landfill leachate consisting of chlorinated organic compounds as well as proprietary pesticide compounds. The remedial technologies which were evaluated included leachate collection alternatives, onsite treatment alternatives and offsite disposal methods. An analysis was performed for onsite treatment technologies which included constructed wetlands, biological fluidized bed reactor, and granular activated carbon adsorption. The technologies were assembled into four feasible remedial alternatives and treatability studies were recommended to confirm the suitability of selected processes.

PROFESSIONAL AFFILIATIONS

National Society of Professional Engineers

American Society of Civil Engineers

WEF Hazardous Waste Committee, 1996–1998

PUBLICATIONS

Assessment and Remediation of Off-Spec Asphalt Disposal Areas - Co-authored, Contaminated Soils, Volume 3, Amherst Scientist Publishers, 1998

Use of a Subsurface Flow Constructed Wetlands for Collection and Removal of Water Containing BTEX, Co-authored, Proceedings of the 2000 Petroleum Hydrocarbons and Organic Chemicals in Groundwater Conference, National Ground Water Association

PROFESSIONAL PROFILE



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EDUCATION
BS, Chemical Engineering,
Cornell University, 2000

Dana M. Hignell

Senior Engineer II

EXPERIENCE SUMMARY
Over twenty years of experience: Staff, Project, and Senior Engineer with Roux.

TECHNICAL SPECIALTIES
Engineering services for the investigation, design, construction, and operation and maintenance of remedial systems for the treatment of contaminated soil, sediment, and groundwater. Remedial designs have included excavation, in-situ groundwater remediation, groundwater pump and treat systems, air sparging with soil vapor extraction, vapor barriers and sub slab depressurization systems, and landfill soil cover systems.

REPRESENTATIVE PROJECTS

- Senior Engineer and Project Manager of a former wire and conduit manufacturer (former NYS Inactive Hazardous Waste Site) remediation in Syosset, NY. The Site was accepted into the NYSDEC BCP to support additional remediation and future redevelopment. Activities completed included development of RIR/RAWP and manage oversight of construction activities for approved remedy.
- Senior Engineer and Project Manager for the design and installation of a Constructed Treatment Wetlands (CTW) to treat nitrogen loading from a sanitary system in Brentwood, New York.
- Senior Engineer for the investigation of nitrogen loading to a creek in East Hampton, New York that is part of the Peconic Bay Estuary, and evaluation of the use of a permeable reactive barrier to reduce the flux of nitrogen.
- Senior Engineer for the design and start-up of a soil vapor extraction (SVE) and air sparge (AS) system at an active gasoline station in Staten Island, New York. The system was installed to address soil and groundwater contaminants and consists of 17 vertical SVE wells and 10 AS wells. The recovered soil vapor is treated by catalytic oxidation before being discharged. Over 1,200 pounds of hydrocarbon have been recovered since the system start-up.
- Senior Engineer for the design of in-situ groundwater remediation to treat chlorinated volatile organic compounds (CVOCs) at a former dry cleaner in Bronx, New York. In-situ chemical oxidation (ISCO) was selected as the groundwater remedy, with injections completed over a half an acre of property. The ISCO remedy was successful and allowed for redevelopment of the property within the required schedule.
- Senior Engineer for the design and implementation of a passive product-recovery system using horizontal recovery wells at a former bus garage in Newark, New Jersey. The remedial action also included excavation and offsite disposal of approximately 7,000 tons of petroleum contaminated soil, and in-situ chemical oxidation to treat residual soil and groundwater contamination.
- Senior Engineering managing the investigation, design, and implementation of product-recovery skimming systems for a former petroleum refinery in Queens, New York. Over 100,000 gallons of product has been recovered to date.
- Senior Engineer for the design of a sub-slab depressurization system with vapor barrier for the construction of a 19-story residential building in Brooklyn, New York.

- Senior Engineer for the investigation, design, and implementation of a soil remediation project at a Former Oil Terminal in Sag Harbor, New York. The remedy completed includes excavation and offsite disposal of approximately 2,000 tons of petroleum contaminated soil in an active roadway surrounded by underground utilities.
- Senior Engineer for the investigation, design, and implementation of a soil remediation project at a 4-acre Former Oil Terminal in Cold Spring Harbor, New York. The remedy completed includes excavation and offsite disposal of approximately 20,000 tons of petroleum contaminated and/or hazardous lead contaminated soil in accordance with the future use of the site under an Environmental Easement. Additional activities completed at the site included asbestos remediation followed by building demolition, UST removal, and cesspool remediation.
- Project Engineer for the complete design for a 9-acre landfill in Rensselaer, New York as part of the NYSDEC Voluntary Cleanup Program. The design was in accordance with the future use of the Site as a landfill with an integrated wildlife habitat vegetative cap and included hot spot soil excavation and installation of a vegetative cap.
- Project Engineer for the design of a soil and groundwater remediation system for a Former Oil Terminal in Oceanside, New York. A risk based remedial approach that called for the remediation of "hot spot" source area soils, and mass-reduction of VOCs in groundwater was successfully utilized for the Site. To address the soil contamination in the source areas, "hot spot" soil excavation was completed. A groundwater pump and treat system was designed, constructed, and continues to be operated and maintained to address the VOC contaminants in groundwater. The system consists of seven recovery wells, an oil/water separator, and a packed column air stripping tower. Over 11 million gallons of groundwater have been recovered and treated.
- Project Engineer for the design and startup of a soil vapor extraction (SVE) and air sparge (AS) system at an 11-acre active Oil Terminal in Glenwood Landing, New York. The system was installed to address soil and groundwater contaminants and consists of four horizontal SVE wells and 8 AS wells. The recovered soil vapor is treated by carbon units before being discharged. Approximately 12,000 pounds of hydrocarbon have been recovered since system start up.
- Project Engineer for the design and startup of a soil vapor extraction (SVE) and air sparge (AS) system at an 11-acre active Oil Terminal in Inwood, New York. The system was installed to address soil and groundwater contaminants and consists of 4 vertical SVE wells and six AS wells. The recovered soil vapor is treated by carbon units before being discharged. Approximately 32,000 pounds of hydrocarbon have been recovered since system start up.

Operation and Maintenance (O&M) Experience

- Project Engineer responsible for the O&M and monitoring of a SVE system and a dual-pump, product recovery system at an active Oil Terminal in Inwood, New York. O&M activities included system operation and maintenance, performance monitoring, and preparation of quarterly status reports for submission to the NYSDEC. The SVE system consists of 5 SVE wells and was designed, constructed, operated, and maintained for a period of approximately 6 years. The SVE system was permanently shut down and this area of the Site is currently in the post-remediation monitoring phase. The dual-pump system, including groundwater and product recovery and packed column air stripping towers, is currently still in operation.
- Project Engineer responsible for the operation and maintenance of soil and groundwater remediation systems for an active Oil Terminal in Glenwood Landing, New York. A groundwater pump and treat system, consisting of 1 recovery well, two packed column air stripping towers, bag filters and granular activated carbon units, has been in operation at the Site to address VOC contaminants in groundwater. In addition, an SVE/AS system, previously installed, was upgraded to further address soil and groundwater contaminants at the Site boundaries. The SVE system consisted of vertical wells in an area of shallow groundwater. The system was upgraded with seven horizontal wells and an additional five AS wells were installed. Following an on-site diesel spill of approximately 5,000 gallons in 2005, free-product recovery efforts were implemented, including removal of saturated soils and installation of a free-product recovery system.
- Staff Engineer responsible for the O&M and monitoring of an SVE/AS system for nationwide distribution center in Brooklyn, New York as part of the NYSDEC Voluntary Cleanup Program. O&M activities included system operation and maintenance, performance monitoring, soil gas monitoring, quarterly monitoring, and preparation of quarterly and annual status reports for submission to the NYSDEC. The SVE and AS system consists of 8 SVE wells and 17 AS wells and was designed, constructed, operated and maintained for a period of approximately 3 years. The SVE and AS system is permanently shut down and the Site is currently in the post-remediation monitoring phase.
- Staff Engineer responsible for the O&M of a 430 gpm, dual-pump, product recovery system in Greenpoint, Brooklyn, New York. Processes and system maintained include dual-pump groundwater and product recovery, low profile air strippers and a catalytic oxidation unit. The Site encompasses one of the nation's largest petroleum releases (18 million gallons).

UST Experience

- Project Engineer for the management of the excavation and removal of a 10,000-gallon UST and four ASTs ranging from 284,000-gallon capacity to 976,000-gallon capacity at a former Oil Terminal in Cold Spring Harbor, New York. The scope of

work entailed preparation of a UST and AST closure work plan, project management of the field work, post excavation and tank footprint sampling and preparation of a UST and AST Closure Report.

- Project Engineer for the excavation oversight of a 2,000-gallon heating oil UST at an active Oil Terminal in Glenwood Landing, New York. Field oversight included post-excavation and waste characterization soil sampling, health and safety monitoring, supervision during the removal, cleaning, and disposal of the USTs and preparation of a Closure Report.
- Project Engineer for the excavation oversight of a 2,000-gallon heating oil UST at an active Oil Terminal in Inwood, New York. Field oversight included post-excavation and waste characterization soil sampling, health and safety monitoring, supervision during the removal, cleaning, and disposal of the USTs and preparation of a Closure Report. Also included was the management and field oversight of the closure of an in-ground oil/water separator at an active Oil Terminal Inwood, New York. The scope of work entailed preparation of an oil/water separator closure work plan, project management of the field work, excavation of petroleum contaminated soils associated with the separator, post-excavation sampling, and preparation of an Oil/Water Separator Closure Report.
- Staff Engineer for the excavation oversight of three 8,000-gallon USTs, two pump islands and three hydraulic lifts and all associated piping at a former service station in Norwalk, Connecticut. Field oversight included post-excavation and waste characterization soil sampling, health and safety monitoring, supervision during the removal, cleaning, and disposal of the USTs, and preparation of a Closure Report.
- Staff Engineer for the excavation oversight of three 8,000-gallon USTs, two pump islands and all associated piping at a former service station in Hartford, Connecticut. Field oversight included post-excavation and waste characterization soil sampling, health and safety monitoring, supervision during the removal, cleaning, and disposal of the USTs and preparation of a Closure Report.

Soil and Groundwater Remediation

- Project Engineer for the design of soil remediation at an active Oil Terminal in Stamford, Connecticut to remove contaminated soils following a 30,000-gallon petroleum spill in 2006. The remediation included excavation of approximately 1,000 cubic yards of petroleum-impacted soil with post-excavation sampling.
- Staff Engineer responsible for construction management of remediation of a former major pharmaceutical plant located in Hicksville, New York as part of the State Superfund Program. The project consisted of the excavation of non-hazardous soil from five on-site drywells and a former waste disposal area, implementation of a community air monitoring plan, coordination with the Long Island Rail Road (LIRR) for work performed within the LIRR's right of way, steel sheeting installation and removal, backfilling, monitoring well abandonment and replacement, transportation and disposal of 3,300 tons of VOC, SVOC and metal contaminated soil, and restoration of approximately 9,800 square feet of asphalt. A 7-foot diameter steel caisson was used to support the deeper excavation required at the invert of two drywells. This innovative approach saved the client approximately $50,000 in costs that would have been incurred by using a traditional steel sheeting support system to protect the on-site commercial building.

Stormwater

- Project Engineer for the design and construction management of a stormwater drainage upgrade project for the parking lot of a former manufacturing facility (costume jewelry and then surgical needles) located in Queens Village, New York. The stormwater drainage system consisted of multiple catch basins, a new drain and curb, and all associated piping.
- Project Engineer for the design and construction of two stormwater management ponds and swales for a former landfill in Rensselaer, New York. The ponds and swales were constructed as part of the landfill cap project to direct stormwater to existing catch basins, allow stormwater overflow control and to enhance the wildlife habitat.

PROFESSIONAL PROFILE



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EDUCATION

MS, Geology, Wright State University, 1990
BS, Geology, Hofstra University, 1983

PROFESSIONAL LICENSES

Licensed Professional Geologist, NY (License No. 000119)

Joseph Duminuco, PG

Executive Vice President | Principal Hydrogeologist
Director of Site Investigation and Remediation Services

EXPERIENCE SUMMARY

Over thirty-five years of experience: Director of Site Investigation and Remediation Services, Executive Vice President, Brownfields Practice Area Leader, Vice President, Office Manager, Principal, Senior, and Project Hydrogeologist at Roux; Staff Hydrogeologist at Geraghty & Miller; and Geologist at Mueser Rutledge Consulting Engineers.

TECHNICAL SPECIALTIES

Mr. Duminuco provides environmental consulting services and strategic planning, focusing on the use of innovative solutions. He is Roux's Director of Site Investigation and Remediation Services, encompassing focused efforts within the Brownfields Redevelopment, Public Sector, Industrial Services, and Petroleum-Energy practice areas. He specializes in investigation and remediation of soil, groundwater, and vapor at commercial and industrial sites, including Brownfield Redevelopment projects.

REPRESENTATIVE PROJECTS

- **Principal-in-Charge of the 45-acre Development of a State-of-the-Art UBS Sports Arena And Commercial/Retail Complex at the Belmont Park Horse Racing Venue on Long Island, New York.** The Roux scope of work included Phase I and Phase II environmental site assessments (ESAs) as part of due diligence, executive information system (EIS) support, Waste Characterization sampling design, implementation, and oversight of all soil disposal/reuse activities of approximately 400,000 cubic yards of soil. In addition, Roux provided onsite environmental manager (OEM) services including daily implementation of the Community Air Monitoring Program (CAMP), compliance inspections, and reporting.

- **Principal-in-Charge of the Redevelopment of an Entire City Block into a Mix of Public and Private Open Space with Community Gardens, 655 Mixed-income Residential Units, and Community Services Containing Three Multi-use Buildings in Harlem, New York.** The buildings will be certified to Passive House standards. Responsibilities include Phase I and II ESAs, remedial action plans (RAPs), waste characterization sampling design, and reporting to the New York City Department of Environmental Protection (NYCDEP). Subsequently, the project was entered as two separate New York State Department of Environmental Conservation (NYSDEC) Brownfields Cleanup Program (BCP) sites. Activities have included BCP Application, preparation, and implementation of a remedial investigation work plan (RIWP), preparation and implementation of an IRM Work Plan, preparation of a combined remedial investigation report (RIR)/remedial action work plan (RAWP), and implementation of CAMP and soil/fill excavation oversight and management and preparation of a site management plan (SMP) and final engineering report (FER) to obtain a certificate of completion (COC).

- **Project Executive of the Design-Build Project to Prepare the Bronx Jail Property for Construction at the Location of the Former Lincoln Hospital.** The hospital and all associated structures were demolished, and the demolition debris was buried at the Site. This debris included asbestos-containing material (ACM). Roux's work consisted of a site-wide geophysical survey, a site-wide radiological gamma walkover survey, extensive waste characterization and asbestos sampling, completion of downhole gamma logging in most of the soil borings, and completion of ten test pits for asbestos and radiological delineation.

During construction Roux provided environmental oversight, CAMP, noise monitoring, and perimeter asbestos air monitoring during the construction phase of work. Due to the nature of the Site and the presence of ACM, all ground-intrusive soil work required those working within the exclusion zone to wear Level C Personal Protective Equipment (PPE).

- **Principal-in-Charge of the Redevelopment of a Waterfront Site that includes Two High-Rise Mixed-use Affordable/Senior Living Residential Towers in Queens, New York.** Additionally, the development includes ground floor retail, community facility public spaces and two piers extending into the East River. The two towers were entered as separate NYSDEC BCP sites and are immediately adjacent to the Newtown Creek Federal Superfund Site. Activities have included BCP Application, preparation, and implementation of a RIWP, preparation of a combined RIR/RAWP, and implementation of CAMP and soil/fill excavation oversight and management, and preparation of an SMP and FER to obtain a COC.
- **Principal-in-Charge of the Redevelopment of a Former Garage and Auto Repair Operation and a Manufacturing Facility on two Adjacent lots into a Multi-story Single-family Residence in lower Manhattan, New York.** The Site contains an E-Designation and as such is going through the New York City Office of Environmental Remediation (NYCOER) Voluntary Cleanup Program (VCP). Roux completed a Phase I ESA, an RI, a RAWP, a waste characterization plan, and is providing oversight of waste management, underground storage tank (UST) removals, and CAMP.
- **Principal-in-Charge of a NYSDEC BCP Redevelopment of a Property Adjacent to a Dry Cleaning Solvent Distribution Facility in Brooklyn, New York.** The Site was a former freight railyard, and offloading spillage on-site and migration from the off-site solvent facility resulted in significant soil, groundwater, and vapor contamination with chlorinated volatile organic compounds (VOCs). The Site was developed into multifamily units with first floor retail use and the remedy consisted of soil hot spot removal, a physical barrier to limit on-site migration, a permeable reactive wall to eliminate off-site migration, hot spot in situ injections, and a sub-slab depressurization system. The Site contained an E-Designation which was satisfied through the NYCOER simultaneously with the BCP process. Roux was awarded the Big Apple Brownfield Award for Innovation based on our successful cleanup approach.
- **Principal-in-Charge of a NYSDEC BCP Redevelopment Project that required a RCRA-compliant Facility Closure at a Former Paint Factory in Queens, New York.** Historical site operations adversely impacted the subsurface including a light non-aqueous phase liquid (LNAPL) plume, in addition to petroleum hydrocarbon impacts to the soil and groundwater. Roux completed a RI at the Site which characterized the nature and extent of the impacts. The remedial action included a large excavation that required support of excavation (SOE) and was completed under a tent due to odor concerns, multiple in situ chemical oxidation (ISCO) injections, UST removal/abandonment, installation of a LNAPL recovery system, and installation of a sub-slab depressurization system (SSDS). Additionally, Roux provided oversight of Resource Conservation and Recovery Act (RCRA) closure activities at the Site, which included emptying, cleaning, and scrapping 65 aboveground storage tanks (ASTs)/vessels; decontaminating the ceilings, walls, and floors of the Paint Factory Building; and collection of compliance samples.
- **Principal-in-Charge of Multiple Dry Cleaner Remediation Project Takeovers in NYC, Long Island, New Jersey, and Connecticut.** Sites included a mixed use multifamily affordable housing neighborhood retail complex, a healthcare facility, a light manufacturing facility, and several retail shopping centers. Impacts included soil, groundwater (bedrock and overburden), surface water, soil vapor, indoor air, and building material contamination from chlorinated VOCs from the former dry cleaner operations. Activities included historical research, re-delineation of contaminant source areas, negotiations with regulatory agencies, and remediation including hot spot soil removal, soil vapor extraction (SVE), in situ groundwater treatment, and negative pressure approaches (SSDS) for vapor mitigation in the existing buildings.
- **Principal-in-Charge of a NYSDEC VCP Redevelopment of a Former MGP Site into a Big Box Retail Site in Brooklyn, New York.** The project consisted of negotiations with the NYSDEC and Roux limited remediation to former gasholders filled with coal tar, soil hot spots with mobile coal tar, and perimeter containment of coal tar. All the remaining soil at the Site was impacted with manufactured gas plant (MGP) waste and most of the Site was underlain by liquid coal tar. Roux negotiated the use of institutional/engineering controls to allow significant contamination to remain in place. An SSDS and vapor barrier were installed to address the mobile coal tar left below the retail building.
- **Principal-in-Charge for a NYSDEC BCP Redevelopment Project at a Site in White Plains, New York**. This Site consists of 16 separate parcels spanning 4.5 acres and had a variety of former uses including automotive service/repair and multiple dry cleaners. The Site has both chlorinated and petroleum hydrocarbon impacts to the soil and groundwater. The remedy will consist of a site cover system, soil hot spot removals, in situ chemical oxidation for groundwater contamination, and installation of an SSDS.

- **Principal-in-Charge of a Former Wire and Conduit Manufacturer (former NYS Inactive Hazardous Waste Site) Remediation on Long Island, New York.** The Site was accepted into the NYSDEC BCP to support additional remediation and future redevelopment. Activities completed included compiling, reviewing, extracting, and summarizing numerous historical environmental reports prepared for the Site; interacting with the NYSDEC, United States Environmental Protection Agency (USEPA), and Nassau County Health Department (NCDOH); completing a supplemental soil investigation (including extensive use of XRF Technology as metals are the compound of concern); and a groundwater investigation (water is over 100 feet deep). Subsequently an RI was performed and an RIR/RAWP was submitted. The approved remedy is a site cover system.
- **Principal-in-Charge for a NYSDEC BCP Redevelopment in Staten Island, New York of a former Retail Service Station Site.** There is soil, groundwater, and vapor contamination from petroleum-related constituents in the vicinity of the former gasoline piping and pump island (the petroleum source area), as well as historic fill across the entire Site. The remedy, described in the Remedial Action Work Plan prepared by Roux, will consist of a sheet pile containment wall around the petroleum source area, a Site Cover System across the entire Site comprised of concrete building slab/walkways, asphalt parking areas and limited landscaped areas, and site-wide a sub-slab depressurization system to prevent vapor intrusion into the proposed retail building and off-site migration of impacted soil vapor.
- **Principal-in-Charge of a Mixed-use (Public School and Residential) Brownfield Redevelopment in Lower Manhattan, New York.** Project consisted of a Phase I and a Phase II ESA to satisfy New York City Department of Environmental Protection (NYCDEP) requirements. Due to the presence of contaminated historic fill, Roux performed in situ waste characterization to assist in the development of NYCDEP-required plans. In addition, Roux provided oversight of the waste removal, completion of waste manifests, and full-time CAMP during all soil moving activities. Roux provided support to the excavation contractor when two previously unknown USTs were discovered during excavation activities.
- **Principal-in-Charge of a Multi-phased NYSDEC VCP Redevelopment of a Former Defense Site to Waterfront, Upscale Housing in Long Island, New York.** This investigation included determining the nature and extent of chlorinated VOCs in soil, groundwater, and vapor-phase contamination on-site and off-site. Utilized a risk assessment to argue the level of residual contamination allowed to remain on-site with an intended residential future use. Remedial alternatives were selected in accordance with future development plans and institutional/engineering controls were proposed to limit cleanup costs. Successfully argued the technical impracticability of remediation of the heavily contaminated deeper aquifer beneath the site and off-site.
- **Principal-in-Charge of a Retail/Commercial Redevelopment in The Bronx, New York.** The Site contained a NYCDEP E-Designation due to a previous on-site service station UST release. In addition, a previous Phase I and Phase II ESA identified a former dry cleaner with a chlorinated VOC release. Roux performed a focused Phase II ESA at the dry cleaner and determined the chlorinated solvent release was not a hazard. Roux obtained closure under the NYSDEC Spills group, and the Site was redeveloped with a restaurant, a pharmacy, and reuse of a former supermarket.
- **Principal-in-Charge of the Redevelopment and Expansion of an Automobile Dealer/Service Center in New York, New York into the US Flagship Dealer for a Major European Luxury Car Manufacturer.** Supported the client and legal team during lease negotiations. Worked closely with the NYCOER to address NYCDEP E-Designation. Coordination with the NYCOER to implement remedial investigation and develop a Site Materials Management Plan as part of the expansion. Also, worked closely with the NYSDEC to address an on-site spill, as well as coordinate efforts to evaluate whether a 19,000-gallon dielectric fluid release by others impacted the Site.
- **Principal-in-Charge for the Completion of Phase I and Phase II Environmental Site Assessment Activities Associated with a Proposed Mixed-use Redevelopment Located in Westchester, New York, Waterfront.** Work included management of subsurface investigation activities to characterize soil conditions, and working closely with the client's architects and construction contractors to integrate the proposed site remediation into the project development plan (including evaluating multiple potential disposal scenarios). Site contaminants included hydrocarbons (including free-product plume from former USTs) and historic fill constituents.
- **Principal-in-Charge of an 80-acre Redevelopment in Yonkers, New York.** Work included Phase I and Phase II investigations, asbestos surveys and abatement support, and response to a free product impact from an adjacent landowner. Coordinated with the NYSDEC and responsible party to address contamination issue and not impact the client's construction schedule.
- **Principal-in-Charge for the Redevelopment of a Property in Brooklyn, New York into Supportive Housing.** Worked closely with the NYCOER to address the NYCDEP E-Designation. Coordination with the NYCOER to implement remedial investigation and develop RAP/ Construction Health and Safety Plan (CHASP) as part of the NYC VCP.

- **Principal-in-Charge for the Redevelopment of a Property in The Bronx, New York into Supportive Housing.** Worked closely with NYCDEP to address E-Designation. Coordination with NYCDEP to implement remedial investigation and develop RAP/CHASP as part of the redevelopment. Also performed an American Society for Testing and Materials (ASTM) vapor encroachment condition (VEC) to address vapor concerns.
- **Principal-in-Charge of a Brownfield Redevelopment for a Large Vacant Parcel (460 acres) on Long Island, New York.** The project involved an extensive investigation, UST, and PCB remediation; removal and proper disposal of numerous tanks, drums, abandoned vehicles and transformers; and participation in contentious public meetings. The Site was redeveloped into a golf course and a senior care facility.
- **Principal-in-Charge for a Property Transfer Support Project at a Heavily Contaminated (Chlorinated Volatile Organic Compounds from an Adjacent Dry Cleaner and On-site MGP Waste) Distribution Facility in the Bronx, New York.** The Site was a former MGP being handled under the VCP, in addition to an open petroleum spill under the regional spills group. Roux performed a Phase I for the buyer, a Phase II and remedial cost estimate for the owner, and negotiated with the buyer's consultant and the NYSDEC to limit the scope of the investigation and cleanup.
- **Principal-in-Charge of Investigation and Remediation of a Catastrophic Heating Oil Release for a Commercial Office Building in Brooklyn, New York.** All work was performed under the oversight of the NYSDEC Spills Group and time was of the essence for the initial response as the heating season was fast approaching. Roux performed free product delineation and remediation and indoor air monitoring at adjacent buildings. Site closure was obtained from the NYSDEC.
- **Principal-in-Charge of a Mixed-use (Residential, Retail, Commercial) Brownfield Redevelopment in The Bronx, New York.** Project consisted of a Phase I and Phase II ESA to satisfy NYCDEP requirements. The media investigated included soil, groundwater, soil vapor, and building materials. During the Phase II ESA, Roux performed preliminary in situ waste characterization.
- **Principal-in-Charge of an Interior Brownfield Redevelopment of a PCB, Metals, and Hydrocarbon-Contaminated Wire Manufacturing Facility in Westchester County, New York Into Use as a Movie Studio.** Activities included delineation and characterization of building surfaces, design of a remediation program, and interim cleanups to allow studio use as the project progressed.
- **Principal-in-Charge of Multiple Phases of Brownfield Project for Construction of a Cogeneration Facility in Brooklyn, New York.** The project consisted of construction oversight; environmental compliance monitoring; asbestos and lead paint abatement oversight; data evaluation and report preparation; soil, offshore sediment, and sewer effluent sampling; PCB-contaminated material remediation; preparation of a waste handling and disposal plan; and permitting.
- **Principal-in-Charge of Multiple Retail Developments in Harlem, New York.** Work included Phase I and Phase II investigations to satisfy the NYCDEP and lender requirements. Lead-based paint and asbestos surveys were performed as part of due diligence. Extensive asbestos issues were identified in building materials and soil backfill. Worked with asbestos contractors to determine the best abatement approaches for the redevelopments.
- **Project Coordinator of Multi-year Environmental Consulting Contracts with Amtrak and New Jersey Transit.** Responsibilities include contract negotiations, workload/ resource distribution, compliance with contract requirements including utilization of Minority and Women-owned Business Enterprise (M/WBE) contractors, client-staff liaison, adherence to budgets and schedules, and overall quality assurance. The remedy will likely consist of hot spot removals, a site cover system and a sub-slab depressurization system.
- **Principal-in-Charge of a Multi-phase RI/FS at a PCB and Diesel Fuel-contaminated Railroad Yard in New York City.** The Site is on the State Superfund list because PCBs were detected in soil, groundwater, hydrocarbon plume, sewer water, and sewer sediment. Responsibilities included preparation of work plans; delineation of PCB hot spots with immunoassays; sewer investigation including pump outs, monitoring, flow measurements, and video surveys prior to abandonment; investigation and remediation of numerous USTs including gasoline, solvents, and fuel oils; support of construction activities; report preparation; negotiations with the NYSDEC and New York State Department of Health (NYSDOH); participation in public meetings; and implementation of interim remedial measures to mitigate the PCB-contaminated hydrocarbon plume; interim remedial measures to mitigate PCB, PAH, and lead-contaminated soil hot spots; and agency acceptance of alternate cleanup levels for site soils that resulted in savings of over $80 million.
- **Principal-in-Charge of a Project to Support the Construction of a High-speed Rail Program.** Performed Phase I and II Environmental Site Assessments as part of due diligence at three major railyards. Prepared reports and presentations regarding environmental conditions to regulatory agencies and the design-build consortium. Performed pre-construction sampling and hot spot remediation programs. Also, prepared

environmental contingency plans for construction contractors to follow.

- **Principal-in-Charge of an Investigation at a PCB and Solvent-Contaminated Transformer Manufacturing/Repair Facility in North Carolina.** Responsibilities included preparation of a work plan and oversight of the project which consists of soil borings and sampling, immunoassay testing, monitoring well installation, groundwater sampling, report preparation, and remedial alternatives evaluation.
- **Principal-in-Charge of a Multi-year Quarterly Monitoring and Reporting Program at a Municipal Landfill Complex on Long Island, New York.** The complex consists of multiple landfills, leachate containment systems, and leachate holding tanks. The project involves the collection of water level and water quality data from dozens of monitoring wells, sampling of leachate containment systems, coordination with contract laboratory, data validation, data evaluation, and report preparation.
- **Project Director of all UST investigative and remedial work performed at service station sites in New England for a major oil company.** Responsibilities included preparation and negotiation of work orders; coordination of monitoring and sampling; communication with client, regulator, and site owner contacts; management of technical aspects of all projects; strategy evaluation with client; administration of all contracts; and operation and maintenance of remediation systems to mitigate UST releases which included groundwater pump and treat, product recovery, and soil venting systems.
- **Principal-in-Charge of an NPL Superfund Site in Delaware.** Responsibilities include ongoing performance monitoring of a groundwater extraction system. The remedial system was installed to capture a chlorinated solvent plume emanating from a former PVC manufacturing facility. In addition, prepared and implemented an RI work plan for a USEPA-required off-site investigation of adjacent chemical manufacturing facilities and a large petroleum refinery. Also included dense non-aqueous phase liquid (DNAPL) investigation and deep aquifer study.
- **Principal-in-Charge of a NJDEP-ECRA and ISRA Investigation and Cleanup Involving Groundwater and Soil Contamination at a Pesticide Formulation and Distribution Facility in New Jersey.** Responsibilities include delineating the nature and extent of the off-site contaminant plume; determining groundwater flow patterns in a two-aquifer system; using a three-dimensional computer model to determine proper location for extraction and injection wells; and preparing work plans and summary reports for New Jersey Department of Environmental Protection (NJDEP)-required additional delineation of the nature and extent of on-site soil contamination.

PROFESSIONAL TRAININGS

NYCOER Turbo Training Certified Professional

PROFESSIONAL AFFILIATIONS

Advisory Council Hofstra University NSF GEOPaths Grant

PRESENTATIONS

Incentives: Programs and Lessons. 2018 Environmental Law Forum – New Jersey State Bar Association; Cape May, NJ; June 2018.

Environmental Law in Real Estate Transactions – Working with Technical Professionals. Hofstra University Law School; January 13, 2013.

Duminuco J., Coyle F., Property Redevelopment and Brownfield Sites. Proceedings of the 11th Annual Environmental Law Conference; ISBA Conference; May 2012.

Transactions and the Environment: Contaminated Property Issues in Real Estate and Corporate Matters. New York State Bar Association; Tarrytown, NY; June 2006.

JUDY V. HARRY
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North Creek, NY 12853

Occupation: Data Validator/Environmental Technical Consultant

Years Experience: 41

Education: B.S., Chemistry, Magna cum laude, 1976, Phi Beta Kappa

Certifications: New York State Woman-Owned Business Enterprise (WBE)

Relevant Work History:

Data Validation Services: September 1989 - present

Sole proprietor of Data Validation Services, a woman-owned small business registered with SAM, providing consultation/validation services to regulatory and commercial clients.

These services include the review of analytical laboratory data for compliance with respect to specific protocols, accuracy and defensibility of data, verification of reported values, and evaluation of quality parameters for analytical usability of results. Approved by USEPA, NYSDEC, NJDEP, NYSERDA, and NYCDEP as a data validator for projects, including USEPA Superfund, Brownfield, and lead sites, and those contracted through the NYSDEC Division of Hazardous Waste Remediation, Division of Solid Waste, and Division of Water Quality.

Performed validation for compliance with laboratory analytical protocols including USEPA OLM, USEPA OLC, USEPA ILM, USEPA DFLM, USEPA SOW3/90, USEPA SOW 7/87 CLP, USEPA SOW 2/88 CLP, USEPA SW846, RCRA, AFCEE, NYS 6 NYCRR Part 360, 40 CFR, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, including TO-15, 1989/1991/1995/2000/2005 NYSDEC ASPs, and 1987 NYSDEC CLP.

Performed validation according to the USEPA National and Regional SOPs and Functional Guidelines, AFCEE requirements, NYSDEC Validation Scope of Work, NYS DUSR, and NJDEP Division of Hazardous Site Mitigation/Publicly Funded Site Remediation SOPs.

Performed validation for USEPA Superfund Sites including Salem Acres, York Oil, Port Washington L-4 Landfill, Bridgeport Rental and Oil Services, GE-MRFA, MMR/ OTIS AFB, LCP, and Peter Cooper site; and for USEPA lead sites including SJ&J Piconne, Maska, Bowe System, Jones Sanitation, and Syossett Landfill, involving CLP, RAS, and SAS protocols.

Contracted for NYSDEC Superfund Standby Contracts with LMS Engineers, HDR, CDM Smith, Malcolm-Pirnie/ARCADIS, Ecology & Environment, Shaw Environmental, CG&I, O'Brien & Gere Engineers, and EC Jordan, involving samples collected at NYS Superfund Sites and analyzed under the NYSDEC ASP.

Performed validation services for NYSDEC Phase II remedial investigations, RI/FS projects, Brownfield sites, and PRP over-site projects for hazardous waste sites.

Performed validation services for clients conducting RI/FS activities involving samples of many matrices, including waste, air, sludges, leachates, solids/sediments, aqueous, and biota.

Clients have included AECOM, ARCADIS, Barton & Loguidice, Benchmark Engineering, Bergmann Associates, Blasland, Bouck & Lee, Brown and Caldwell, CDM Smith, CB&I Shaw Environmental, C&S Consulting Engineers, Chazen Companies, Clough Harbour & Associates, Columbia Analytical Services, C.T. Male, Dames & Moore, Day Engineering, EA Engineering, EcolSciences, Ecology & Environment, Ecosystems, EC Jordan, Environmental Chemical Corporation, EHRT, ENSR Consulting, ELM, ERM-Northeast, Fagan Engineers, Fanning Phillips & Molnar, FluorDaniel GTI, Frontier, Foster Wheeler Environmental Corp, Frontier Technical, Galson Consultants, GE&R, Geomatrix Consultants, GZA Environmental, Handex of N, H2M Group, HDR, HRP, IT Corp, Jacques Whitford, JTM Associates, Labella Associates, Langan Engineers, Leader Environmental, Lockwood, Kessler & Bartlett, LMS Engineers, Malcolm-Pirnie, Metcalf & Eddy, NWEC&C, O'Brien & Gere Engineers, Pace, Parsons Engineering-Science, Plumley Engineering, Prescott Environmental, P. W. Grosser, Rizzo Associates, Roux Associates, Sear Brown Group, SECOR, Shaw Environmental, Stantec, ThermoRemediation Inc., TRC Environmental, Turnkey Environmental Restoration, TVGA Engineering, URS Consultants, Wehran Emcon, Weston, YEC, and private firms.

Provided consultation services to laboratories regarding analytical procedures and protocol interpretation, and to law firms for litigation support.

Provided services to firms involving audits of environmental analytical laboratories to determine analytical capability, particularly for compliance with NYSDEC ASP and AFCEE requirements.

Guest speaker on a panel discussing Data Review/Compliance and Usability, for an analysis workshop for the New York Association of Approved Environmental Laboratories, 1993.

Adirondack Environmental Services: June 1987 - August 1989

Senior mass spectroscopist for AES. Responsible for GC/MS analyses of environmental samples by USEPA and NYSDEC protocols, development of the GC/MS laboratory, initiating the instrumental and computer operations from the point of installation, and for implementing the procedures and methodologies for Contract Laboratory Protocol.

CompuChem Laboratories: May 1982 - January 1987

Managed a GC/MS production laboratory; developed, implemented, and supervised QA/QC criteria at three different levels of review; and was responsible for the development and production of the analysis of environmental and clinical samples. Directed a staff of 23 technical and clerical personnel, and managed the extraction and GC/MS labs and data review operations.

Research Triangle Institute: December 1979 - May 1982

Worked as an analytical research chemist responsible for development of analytical methods for the EPA Federal Register at RTI. This involved analysis of biological and environmental samples for priority pollutants, primarily relating to wastewaters and to human sampling studies. Method development included modification and interfacing of the initially developed Tekmar volatile purge apparatus to GC/MS, development and refinement of methods for entrapment and concentration of the air medium for subsequent volatile analysis, and the analysis and resolution/ identification of individual PCB congeners within Aroclor mixtures by capillary column and mass spectra.

Guardsman Chemical Company: February 1977 - November 1979

Performed all quality control functions for the manufacturing plant. Performed research and development on coatings and dyes.

Almay Cosmetics: May 1976 - December 1976

Product evaluation chemist. Responsible for analytical QC of manufactured products.

Publication

Pellizzzari, E.D., Moseley, M.A., Cooper, S.D., Harry, J.V., Demian, B., & Mullin, M. D. (1985). Recent Advances in the Analysis of Polychlorinated Biphenyls in Environmental and Biological Media. *Journal of Chromatography*, 334(3) 277-314.

ATTACHMENT 2

NYSDEC PFAS Sampling Guidance

NEW YORK STATE OF OPPORTUNITY.

Department of Environmental Conservation

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

Under NYSDEC's Part 375 Remedial Programs

April 2023



www.dec.ny.gov

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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	PFAS should be further assessed and considered as a potential contaminant of concern in groundwater or surface water (…) 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.	PFOA and PFOS should be further assessed and considered as potential contaminants of concern in groundwater or surface water (…) 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.	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Soil Sample Results, page 10	"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."	"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. " [Interim SCO Table] "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. 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. "	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Testing for Imported Soil Page 11	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. 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.	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. 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.	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Footnotes	None	[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. [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).	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	"In addition, further assessment of water may be warranted if either of the following screening levels are met: a. any other individual PFAS (not PFOA or PFOS) is detected in water at or above 100 ng/L; or b. total concentration of PFAS (including PFOA and PFOS) is detected in water at or above 500 ng/L"	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 text ….EPA 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 ± 2º 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 ± 2º 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 ± 2º 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 ± 2º 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° F (<8° C) immediately following data processing. As soon as possible, freeze at -20° C ± 5° 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

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	

richter: revised 21 April 2014; becker: 23 March 2017, 26 April, 2019

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 envelops, 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	PFTrDA	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 6ºC 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

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

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

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.

ATTACHMENT 3

Roux's Standard Operating Procedures

Date: May 5, 2000

1.0 PURPOSE

The purpose for this standard operating procedure (SOP) is to establish the guidelines for decontamination of all field equipment potentially exposed to contamination during drilling, and soil and water sampling. The objective of decontamination is to ensure that all drilling, and soil-sampling and water-sampling equipment is decontaminated (free of potential contaminants): 1) prior to being brought onsite to avoid the introduction of potential contaminants to the site; 2) between drilling and sampling events/activities onsite to eliminate the potential for cross-contamination between boreholes and/or wells; and 3) prior to the removal of equipment from the site to prevent the transportation of potentially contaminated equipment offsite.

In considering decontamination procedures, state and federal regulatory agency requirements must be considered because of potential variability between state and federal requirements and because of variability in the requirements of individual states. Decontamination procedures must be in compliance with state and/or federal protocols in order that regulatory agency(ies) scrutiny of the procedures and data collected do not result in non-acceptance (invalidation) of the work undertaken and data collected.

2.0 PROCEDURE FOR DRILLING EQUIPMENT

The following is a minimum decontamination procedure for drilling equipment. Drilling equipment decontamination procedures, especially any variation from the method itemized below, will be documented on an appropriate field form or in the field notebook.

2.1 The rig and all associated equipment should be properly decontaminated by the contractor before arriving at the test site.

2.2 The augers, drilling casings, rods, samplers, tools, rig, and any piece of equipment that can come in contact (directly or indirectly) with the soil, will be steam cleaned onsite prior to set up for drilling to ensure proper decontamination.

2.3 The same steam cleaning procedures will be followed between boreholes (at a fixed on-site location[s], if appropriate) and before leaving the site at the end of the study.

2.4 All on-site steam cleaning (decontamination) activities will be monitored and documented by a member(s) of the staff of Roux Associates, Inc.

2.5 If drilling activities are conducted in the presence of thick, sticky oils (e.g., PCBs) which coat drilling equipment, then special decontamination procedures may have to be utilized before steam cleaning (e.g., hexane scrub and wash).

2.6 Containment of decontamination fluids may be necessary (e.g., rinseate from steam cleaning) or will be required (e.g., hexane), and disposal must be in accordance with state and/or federal procedures.

3.0 PROCEDURE FOR SOIL-SAMPLING EQUIPMENT

The following is a minimum decontamination procedure for soil-sampling equipment (e.g., split spoons, stainless-steel spatulas). Soil-sampling equipment decontamination procedures, especially any variation from the method itemized below, will be documented on an appropriate field form or in the field notebook.

3.1 Wear disposable gloves while cleaning equipment to avoid cross-contamination and change gloves as needed.

3.2 Steam clean the sampler or rinse with potable water. If soil-sampling activities are conducted in the presence of thick, sticky oils (e.g., PCBs) which coat sampling equipment, then special decontamination procedures may have to be utilized before steam cleaning and washing in detergent solution (e.g., hexane scrub and wash).

3.3 Prepare a non-phosphate, laboratory-grade detergent solution and distilled or potable water in a clean bucket.

3.4 Disassemble the sampler, as necessary and immerse all parts and other sampling equipment in the solution.

3.5 Scrub all equipment in the bucket with a brush to remove any adhering particles.

3.6 Rinse all equipment with copious amounts of potable water followed by distilled or deionized water.

3.7 Place clean equipment on a clean plastic sheet (e.g., polyethylene)

3.8 Reassemble the cleaned sampler, as necessary.

3.9 Transfer the sampler to the driller (or helper) making sure that this individual is also wearing clean gloves or wrap the equipment with a suitable material (e.g., plastic bag, aluminum foil.

As part of the decontamination procedure for soil-sampling equipment, state and/or federal protocols must be considered. These may require procedures above those specified as minimum for Roux Associates, Inc., such as the use of nitric acid, acetone, etc. Furthermore, the containment and proper disposal of decontamination fluids must be considered with respect to regulatory agency(ies) requirements.

4.0 PROCEDURE FOR WATER-SAMPLING EQUIPMENT

The following is a decontamination procedure for water-sampling equipment (e.g., bailers, pumps). Water-sampling equipment decontamination procedures, especially any variation from the method itemized below, will be documented on an appropriate field form or in the field notebook.

4.1 Decontamination procedures for bailers follow:

a. Wear disposable gloves while cleaning bailer to avoid cross-contamination and change gloves as needed.

b. Prepare a non-phosphate, laboratory-grade detergent solution and potable water in a bucket.

c. Disassemble bailer (if applicable) and discard cord in an appropriate manner and scrub each part of the bailer with a brush and solution.

d. Rinse with potable water and reassemble bailer.

e. Rinse with copious amounts of distilled or deionized water.

f. Air dry.

g. Wrap equipment with a suitable material (e.g., clean plastic bag, aluminum foil).

h. Rinse bailer at least three times with distilled or deionized water before use.

4.2 Decontamination procedures for pumps follow:

a. Wear disposable gloves while cleaning pump to avoid cross-contamination and change gloves as needed.

b. Prepare a non-phosphate, laboratory-grade detergent solution and potable water in a clean bucket, clean garbage can, or clean 55-gallon drum.

c. Flush the pump and discharge hose (if not disposable) with the detergent solution and discard disposable tubing and/or cord in an appropriate manner.

d. Flush the pump and discharge hose (if not disposable) with potable water.

e. Place the pump on clear plastic sheeting.

f. Wipe any pump-related equipment (e.g., electrical lines, cables, discharge hose) that entered the well with a clean cloth and detergent solution, and rinse or wipe with a clean cloth and potable water.

g. Air dry.

h. Wrap equipment with a suitable material (e.g., clean plastic bag).

As part of the decontamination procedure for water-sampling equipment, state and/or federal protocols must be considered. These may require procedures above those specified as minimum for Roux Associates, Inc., such as the use of nitric acid, acetone, etc. Furthermore, the containment and proper disposal of decontamination fluids must be considered with respect to regulatory agency(ies) requirements.

Date: May 5, 2000

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish guidelines for the collection of soil samples for laboratory analysis. This SOP is applicable to soil samples collected from split-spoon samplers during drilling, hand auger samples, grab samples from stockpiled soils, surface samples, test pit samples, etc.

2.0 CONSIDERATIONS

Soil samples may be collected in either a random or biased manner. Random samples can be based on a grid system or statistical methodology. Biased samples can be collected in areas of visible impact or suspected source areas. Soil samples can be collected at the surface, shallow subsurface, or at depth. When samples are collected at depth the water content should be noted, since generally "soil sampling" is restricted to the unsaturated zone. Equipment selection will be determined by the depth of the sample to be collected. A thorough description of the sampling locations and proposed methods of sample collection should be included in the work plan.

Commonly, surface sampling refers to the collection of samples at a 0 to 6-inch depth interval. Certain regulatory agencies may define the depth interval of a surface sample differently, and this must be defined in the work plan. Collection of surface soil samples is most efficiently accomplished with the use of a stainless-steel trowel or scoop. For samples at greater depths a decontaminated bucket auger or power auger may be needed to advance the hole to the point of sample collection. Another clean bucket auger should then be used to collect the sample. To collect samples at depths of greater than approximately six feet the use of a drill rig and split spoon samples will usually be necessary. In some situations, sample locations are accessed with the use of a backhoe.

3.0 MATERIALS/EQUIPMENT

a. A work plan which outlines soil sampling requirements.

b. Field notebook, field form(s), maps, chain-of-custody forms, and custody seals.

c. Decontamination supplies (including: non-phosphate, laboratory grade detergent, buckets, brushes, potable water, distilled water, regulatory-required reagents, aluminum foil, plastic sheeting, etc.).

d. Sampling device (split-spoon sampler, stainless steel hand auger, stainless steel trowel, etc.).

e. Stainless steel spoons or spatulas.

f. Disposable sampling gloves.

g. Laboratory-supplied sample containers with labels.

h. Cooler with blue or wet ice.

i. Plastic sheeting.

j. Black pen and indelible marker.

k. Zip-lock bags and packing material.

l. Tape measure.

m. Paper towels or clean rags.

n. Masking and packing tape.

o. Overnight (express) mail forms.

4.0 DECONTAMINATION

All reusable sampling equipment will be thoroughly cleaned according to the decontamination SOP. Where possible, thoroughly pre-cleaned and wrapped sampling equipment should be used and dedicated to individual sampling locations. Disposable items such as sampling gloves, aluminum foil, and plastic sheeting will be changed after each use and discarded in an appropriate manner.

5.0 PROCEDURE

5.1 Prior to collecting soil samples, ensure that all sampling equipment has been thoroughly cleaned according to the decontamination SOP. If samples are to be collected at depth, then the boring must be advanced with thoroughly cleaned equipment to the desired sampling horizon and a different thoroughly cleaned sampler must be used to collect the sample.

5.2 Using disposable gloves and a pre-cleaned, stainless steel spatula or spoon, extract the soil sample from the sampler, measure the recovery, and separate the wash from the true sample. Where allowed by regulatory agency(ies), disposable plastic spoons may be used.

5.3 Place the sample in a laboratory-supplied, pre-cleaned sample container. This should be done as quickly as possible and this is especially important when sampling for volatile organic compounds (VOCs). Samples to be analyzed for VOCs must be collected prior to other constituents.

5.4 The sample container will be labeled with appropriate information such as, client name, site location, sample identification (location, depth, etc.), date and time of collection, and sampler's initials.

5.5 Using the remaining portion of soil from the sampler, log the sample in detail and record sediment characteristics (color, odor, moisture, texture, density, consistency, organic content, layering, grain size, etc.).

5.6 If soil samples are to be composited in the field, then equal portions from selected locations will be placed on a clean plastic sheet and homogenized. Alternately, several samples may be submitted to the laboratory for compositing by weight. The method used is dependent upon regulatory requirements. Specific compositing procedures shall be approved by the appropriate regulatory agency and described in the work plan. Samples to be analyzed for VOCs will not be composited unless required by a regulatory agency.

5.7 After the sample has been collected, labeled, and logged in detail, it is placed in a zip-lock bag and stored in a cooler at 4°C.

5.8 A chain-of-custody form is completed for all samples collected. One copy is retained and two are sent with the samples in a zip-lock bag to the laboratory. A custody seal is placed on the cooler prior to shipment.

5.9 Samples collected from Monday to Friday are to be delivered to the laboratory within 24 hours of collection. If Saturday delivery is unavailable, samples collected on Friday must be delivered by Monday morning. Check the work plan to determine if any analytes require a shorter delivery time.

5.10 The field notebook and appropriate forms should include, but not be limited to the following: client name, site location, sample location, sample depth, sample identification, date and time collected, sampler's name, method of sample collection, number and type of containers, geologic description of material, description of decontamination procedures, etc. A site map should be prepared with exact measurements to each sample location in case follow-up sampling is necessary.

5.11 All reusable sampling equipment must be thoroughly cleaned in accordance with the decontamination SOP. Following the final decontamination (after all samples are collected) the sampling equipment is wrapped in aluminum foil. Discard any gloves, foil, plastic, etc. in an appropriate manner that is consistent with site conditions.

END OF PROCEDURE

Date: May 5, 2000

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to establish guidelines for sample handling which will allow consistent and accurate results. Valid chemistry data are integral to investigations that characterize media-quality conditions. Thus, this SOP is designed to ensure that once samples are collected, they are preserved, packed and delivered in a manner which will maintain sample integrity to as great an extent as possible. The procedures outlined are applicable to most sampling events and any required modifications must be clearly described in the work plan.

2.0 CONSIDERATIONS

Sample containers, sampling equipment decontamination, quality assurance/quality control (QA/QC), sample preservation, and sample handling are all components of this SOP.

2.1 Sample Containers

Prior to collection of a sample, considerations must be given to the type of container that will be used to store and transport the sample. The type and number of containers selected is usually based on factors such as sample matrix, potential contaminants to be encountered, analytical methods requested, and the laboratory's internal quality assurance requirements. In most cases, the overriding considerations will be the analytical methodology, or the state or federal regulatory requirements because these regulations generally encompass the other factors. The sample container selected is usually based on some combination of the following criteria:

a. Reactivity of Container Material with Sample

Choosing the proper composition of sample containers will help to ensure that the chemical and physical integrity of the sample is maintained. For sampling potentially hazardous material, glass is the recommended container type because it is chemically inert to most substances. Plastic containers are not recommended for most hazardous wastes because the potential exists for contaminants to adsorb to the surface of the plastic or for the plasticizer to leach into the sample.

In some instances, however, the sample characteristics or analytes of interest may dictate that plastic containers be used instead of glass. Because some metals species will adhere to the sides of the glass containers in an aqueous matrix, plastic bottles (e.g., nalgene) must be used for samples collected for metals analysis. A separate, plastic

container should accompany glass containers if metals analysis is to be performed along with other analyses. Likewise, other sample characteristics may dictate that glass cannot be used. For example, in the case of a strong alkali waste or hydrofluoric solution, plastic containers may be more suitable because glass containers may be etched by these compounds and create adsorptive sites on the container's surface.

b. Volume of the Container

The volume of sample to be collected will be dictated by the analysis being performed and the sample matrix. The laboratory must supply bottles of sufficient volume to perform the required analysis. In most cases, the methodology dictates the volume of sample material required to complete the analysis. However, individual laboratories may provide larger volume containers for various analytes to ensure sufficient quantities for duplicates or other QC checks.

To facilitate transfer of the sample from the sampler into the container and to minimize spillage and sample disturbance, wide-mouth containers are recommended. Aqueous volatile organic samples must be placed into 40-milliliter (ml) glass vials with polytetrafluoroethylene (PTFE) (e.g., TeflonTM) septums. Non-aqueous volatile organic samples should be collected in the same type of vials or in 4-ounce (oz) wide-mouth jars provided by the laboratory. These jars should have PTFE-lined screw caps.

c. Color of Container

Whenever possible, amber glass containers should be used to prevent photodegradation of the sample, except when samples are being collected for metals analysis. If amber containers are not available, then containers holding samples should be protected from light (i.e., place in cooler with ice immediately after filling).

d. Container Closures

Container closures must screw on and off the containers and form a leak-proof seal. Container caps must not be removed until the container is ready to be filled with the sample, and the container cap must be replaced (securely) immediately after filling it. Closures should be constructed of a material which is inert with respect to the sampled material, such as PTFE (e.g., TeflonTM). Alternately, the closure may be separated from the sample by a closure liner that is inert to the sample material such as PTFE sheeting. If soil or sediment samples are being collected, the threads of the container must be wiped clean with a dedicated paper towel or cloth, so the cap can be threaded properly.

e. Decontamination of Sample Containers

Sample containers must be laboratory cleaned by the laboratory performing the analysis. The cleaning procedure is dictated by the specific analysis to be performed on the sample. Sample containers must be carefully examined to ensure that all containers appear clean. Do not mistake the preservative as unwanted residue. The bottles should not be field cleaned. If there is any question regarding the integrity of the bottle, then the laboratory must be contacted immediately and the bottle(s) replaced.

f. Sample Bottle Storage and Transport

No matter where the sample bottles are, whether at the laboratory waiting to be packed for shipment or in the field waiting to be filled with sample, care must be taken to avoid contamination. Sample shuttles or coolers, and sample bottles must be stored and transported in clean environments. Sample bottles and clean sampling equipment must never be stored near solvents, gasoline, or other equipment that is a potential source of cross-contamination. When under chain of custody, sample bottles must be secured in locked vehicles, and custody sealed in shuttles or in the presence of authorized personnel. Information which documents that proper storage and transport procedures have been followed must be included in the field notebook and on appropriate field forms.

2.2 Decontamination of Sampling Equipment

Proper decontamination of all re-usable sampling equipment is critical for all sampling episodes. The SOP for Decontamination of Field Equipment and SOPs for method-specific or instrument-specific tasks must also be referred to for guidance for decontamination of various types of equipment.

2.3 Quality Assurance/Quality Control Samples

QA/QC samples are intended to provide control over the proper collection and tracking of environmental measurements, and subsequent review, interpretation and validation of generated analytical data. The SOPs for Collection of Quality Control Samples, for Evaluation and Validation of Data, and for Field Record Keeping and Quality Assurance/Quality Control must be referred to for detailed guidance regarding these respective procedures. SOPs for method-specific or instrument-specific tasks must also be referred to for guidance for QA/QC procedures.

2.4 Sample Preservation Requirements

Certain analytical methodologies for specific analytes require chemical additives in order to stabilize and maintain sample integrity. Generally, this is accomplished under the following two scenarios:

a. Sample bottles are preserved at the laboratory prior to shipment into the field.

b. Preservatives are added in the field immediately after the samples are collected.

Many laboratories provide pre-preserved bottles as a matter of convenience and to help ensure that samples will be preserved immediately upon collection. A problem associated with this method arises if not enough sample could be collected, resulting in too much preservative in the sample. More commonly encountered problems with this method include the possibility of insufficient preservative provided to achieve the desired pH level or the need for additional preservation due to chemical reactions caused by the addition of sample liquids to pre-preserved bottles. The use of pre-preserved bottles is acceptable; however, field sampling teams must always be prepared to add additional preservatives to samples if the aforementioned situations occur. Furthermore, care must be exercised not to overfill sample bottles containing preservatives to prevent the sample and preservative from spilling and therefore diluting the preservative (i.e., not having enough preservative for the volume of sample).

When samples are preserved after collection, special care must be taken. The transportation and handling of concentrated acids in the field requires additional preparation and adherence to appropriate preservation procedures. All preservation acids used in the field should be trace-metal or higher-grade.

2.5 Sample Handling

After the proper sample bottles have been received under chain-of-custody, properly decontaminated equipment has been used to collect the sample, and appropriate preservatives have been added to maintain sample integrity, the final step for the field personnel is checking the sample bottles prior to proper packing and delivery of the samples to the laboratory.

All samples should be organized and the labels checked for accuracy. The caps should be checked for tightness and any 40-ml volatile organic compound (VOC) bottles must be checked for bubbles. Each sample bottle must be placed in an individual Ziploc® bag to protect the label, and placed on ice. The bottles must be carefully packed to prevent breakage during transport. When several bottles have been collected for an individual sample, they should not be placed adjacent to each other in the cooler to prevent possible breakage of all bottles for a given sample. If there are any samples which are known or suspected to be highly

contaminated, these should be placed in an individual cooler under separate chain-of-custody to prevent possible cross contamination. Sufficient ice (wet or blue packs) should be placed in the cooler to maintain the temperature at 4 degrees Celsius (°C) until delivery at the laboratory. Consult the work plan to determine if a particular ice is specified as the preservation for transportation (e.g., the United States Environmental Protection Agency does not like the use of blue packs because they claim that the samples will not hold at 4°C). If additional coolers are required, then they should be purchased. The chain-of-custody form should be properly completed, placed in a "zip-lock" bag, and placed in the cooler. One copy must be maintained for the project files. The cooler should be sealed with packing tape and a custody seal. The custody seal number should be noted in the field book. Samples collected from Monday through Friday will be delivered to the laboratory within 24 hours of collection. If Saturday delivery is not available, samples collected on Friday must be delivered by Monday morning. Check the work plan to determine if certain analytes require a shorter delivery time. If overnight mail is utilized, then the shipping bill must be maintained for the files and the laboratory must be called the following day to confirm receipt.

3.0 EQUIPMENT AND MATERIALS

3.1 General equipment and materials may include, but not necessarily be limited to, the following:

a. Sample bottles of proper size and type with labels.

b. Cooler with ice (wet or blue pack).

c. Field notebook, appropriate field form(s), chain-of-custody form(s), custody seals.

d. Black pen and indelible marker.

e. Packing tape, "bubble wrap," and "zip-lock" bags.

f. Overnight (express) mail forms and laboratory address.

g. Health and safety plan (HASP).

h. Work plan/scope of work.

i. Pertinent SOPs for specified tasks and their respective equipment and materials.

3.2 Preservatives for specific samples/analytes as specified by the laboratory. Preservatives must be stored in secure, spillproof glass containers with their content, concentration, and date of preparation and expiration clearly labeled.

3.3 Miscellaneous equipment and materials including, but not necessarily limited to, the following:

a. Graduated pipettes.

b. Pipette bulbs.

c. Litmus paper.

d. Glass stirring rods.

e. Protective goggles.

f. Disposable gloves.

g. Lab apron.

h. First aid kit.

i. Portable eye wash station.

j. Water supply for immediate flushing of spillage, if appropriate.

k. Shovel and container for immediate containerization of spillage-impacted soils, if appropriate.

4.0 PROCEDURE

4.1 Examine all bottles and verify that they are clean and of the proper type, number, and volume for the sampling to be conducted.

4.2 Label bottles carefully and clearly with project name and number, site location, sample identification, date, time, and the sampler's initials using an indelible marker.

4.3 Collect samples in the proper manner (refer to specific sampling SOPs).

4.4 Conduct preservation activities as required after each sample has been collected. Field preservation must be done immediately and must not be done later than 30 minutes after sample collection.

4.5 Conduct QC sampling, as required.

4.6 Seal each container carefully and place in an individual "zip lock" bag.

4.7 Organize and carefully pack all samples in the cooler immediately after collection (e.g., bubble wrap). Insulate samples so that breakage will not occur.

4.8 Complete and place the chain-of-custody form in the cooler after all samples have been collected. Maintain one copy for the project file. If the cooler is to be transferred several times prior to shipment or delivery to the laboratory, it may be easier to tape the chain-of-custody to the exterior of the sealed cooler. When exceptionally hazardous samples are known or suspected to be present, this should be identified on the chain-of-custody as a courtesy to the laboratory personnel.

4.9 Add additional ice as necessary to ensure that it will last until receipt by the laboratory.

4.10 Seal the cooler with packing tape and a custody seal. Record the number of the custody seal in the field notebook and on the field form. If there are any exceptionally hazardous samples, then shipping regulations should be examined to ensure the sample containers and coolers are in compliance and properly labeled.

4.11 Samples collected from Monday through Friday will be delivered to the laboratory within 24 hours of collection. If Saturday delivery is not available, samples collected on Friday must be delivered by Monday morning. Check the work plan to determine if certain analytes require a shorter delivery time.

4.12 Maintain the shipping bill for the project files if overnight mail is utilized and call the laboratory the following day to confirm receipt.

END OF PROCEDURE

Date: May 5, 2000

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to establish guidelines for screening soil samples for volatile organic vapors using a portable photoionization detector (PID). This SOP is applicable to soil samples collected from split-spoon samplers during drilling, hand auger samples, and grab samples from stockpiled soils.

2.0 CONSIDERATIONS

The primary objective of photoionization screening of soil samples is to obtain a qualitative understanding of the distribution of volatile organic compounds (VOCs) in soil. The proper design of an organic vapor screening program requires an understanding of site hydrogeology, potential source areas, and potential constituents of concern. Sample locations and frequency must be fully defined in the work plan. The work plan should outline the type of lamp to be utilized in the PID based on the ionization potentials and response factors of the constituents of concern. The work plan must also clearly describe the heating or equilibration procedures to be employed if they differ from those described in this SOP. Regardless of the specific equilibration procedure employed, it is imperative that each sample be treated identically to allow the photoionization results from different locations to be compared. Observations such as water, clay, and organic content should be noted to facilitate interpretation of the data. Every effort should be made to collect a representative portion of soil from the sampling device.

3.0 MATERIALS/EQUIPMENT

a. A work plan which outlines photoionization screening requirements.

b. Decontamination supplies (including: non-phosphate, laboratory grade detergent, buckets, brushes, potable water, distilled water, regulatory-required reagents [e.g., acetone, nitric acid, hexane, etc.], aluminum foil, plastic sheeting, etc.).

c. Field notebook, field form(s), maps, chain-of-custody forms.

d. Sampling device (split-spoon sampler, stainless steel hand auger, stainless steel trowel, etc.).

e. Stainless steel spoons or spatulas.

f. Disposable plastic spoons.

g. Plastic sheeting.

h. Aluminum foil.

i. Mason jars or driller's jars.

j. Water bath (hot plate, extension cord, water tray, thermometer).

k. Photoionization detector with charging unit.

l. Calibration gases with regulator.

m. Indelible marker.

n. Masking tape.

o. Disposable sampling gloves.

4.0 DECONTAMINATION

Where possible, thoroughly pre-cleaned and wrapped sampling equipment must be used and dedicated to individual sampling locations. Disposable items such as sampling gloves, aluminum foil, and sample jars will be changed after each use and discarded in an appropriate manner. If only photoionization results are to be obtained, then split-spoon samples and hand augers may be cleaned with a soap and water wash and potable water rinse or steam cleaning, and a final distilled water rinse. However, if samples are to be collected concurrently for laboratory analytical results, then all reusable sampling equipment must be thoroughly decontaminated according to the SOP for decontamination of field equipment.

5.0 CALIBRATION

The PID must be calibrated according to the manufacturer's specifications at a minimum frequency of once per day prior to collecting photoionization readings. In addition, periodic checks (e.g., every 2 hours or every ten samples) with the standard gas will be conducted to confirm that the calibration has not drifted. The time, date, and calibration procedure must be clearly documented in the field notebook and the calibration log. If at any time the photoionization results appear erratic or inconsistent with field observations, then the unit must be recalibrated. If calibration is difficult to achieve, then the unit's lamp should be checked for dirt or moisture and cleaned, as necessary. During humid or wet conditions, the unit should be calibrated on a more frequent basis as determined by field personnel.

6.0 PROCEDURE

6.1 Extract the soil sample from the sampler, quickly measure the recovery, and separate the wash from the true sample by using a dedicated, stainless steel spatula. Where allowed by regulatory agency(ies), disposable plastic spoons may be used.

6.2 Place the sample in a pre-cleaned glass jar (as quickly as possible to avoid loss of VOCs) filling the jar half full. Place an aluminum foil seal between the glass and metal cap and screw tight.

6.3 Label jars with the boring number, depth of sample, date of collection and blow counts. In addition, the field personnel will ensure the following: samples are taken at appropriate depths; unrepresentative portions of the sample are discarded properly; that the sampler is decontaminated properly between use; and the driller uses proper methods during sample collection and does not use oil or grease on tools entering the borehole.

6.4 Log the sample in detail and record sediment characteristics (color, odor, moisture, texture, density, consistency, organic content, and layering).

6.5 After the sample has been collected, heat the sample under controlled conditions in a water bath for a 2 minute period.

6.6 Ensure that the PID has been calibrated and that the calibration information is documented in the field book. Pierce the aluminum foil seal with the probe from the PID and measure the relative concentration of VOCs in the headspace of the soil sample. The initial (peak) reading must be recorded.

6.7 Record the PID reading in the field notebook, on an appropriate field form, and on the base map, if appropriate.

6.8 Place any material not representative of the interval sampled in a pile with the other cuttings from the borehole.

6.9 If only photoionization results are to be obtained, then reusable sampling devices may be cleaned with a soap and water wash and a potable water rinse. The sampler will then be rinsed with distilled water, assembled and placed on plastic sheeting for reuse. A more rigorous decontamination procedure is required when samples are also being collected for laboratory analysis. Refer to the SOP for collection of soil samples for laboratory analysis for additional information.

END OF PROCEDURE

ATTACHMENT 4

Laboratory's Standard Operating Procedures

The information contained herein is of a highly confidential and proprietary nature. Eurofins Environment Testing North Central, LLC specifically prohibits the dissemination, copy, disclosure, transfer, or modification of this information without the express written approval of Eurofins Environment Testing North Central, LLC.

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Revision Log

Revision: 02	**Effective date:**	This version
Section	**Justification**	**Changes**
		Update procedure.

Reference

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

Cross Reference

Document	Document Title

Document	Document Title
NC-WC-092	Determination of Solids in Waters and Wastes (Total Suspended Solids)
NDSC-QA-SOP42091	Detection and Quantitation Limits
NDSC-QA-SOP43862	Manual Integrations
NC-QA-015	Support Equipment Calibration and Verification
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
QA-003	Quality Control Program
NC-QA-017	Standards and Reagents
NDSC-QA-QP44940	Calibration Curves and the Selection of Calibration Points
NDSC-US-EHS QP46060	NDSC EH&S Manual
NC-QAM-001	QA Manual

Scope

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

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

Analyte	Acronym	CAS#
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonic acid	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1

Analyte	Acronym	CAS#
Perfluoro-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA, PES	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

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

Interferences

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

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.

3. All part of the SPE manifold must be cleaned every 2 weeks per manufacturer recommendations.

Safety Precautions and Waste Handling

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

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

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

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

Personnel Training and Qualifications

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

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

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

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

Sample Collection, Preservation, and Handling

A. Sample Collection

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

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

B. Sample Storage and Shipment

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

2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, and protected from light until extraction. Alternatively, to meet project requirements, samples may be stored at ≤ -20°C and protected from light until extraction.

3. Water samples must be extracted within 28 days when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Water samples must be extracted within 90 days when stored at a temperature < -20°C and protected from light. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light.

Apparatus and Equipment

A. Apparatus

1. 125 mL HDPE bottles: Environmental Sampling Supply; #0125-1902-QC, or equivalent.

2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 14955237 or equivalent

3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288 or equivalent.

4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC, or equivalent.

5. Analytical Balance – Capable of weighing to 0.0001 g

6. Top-Loading Balance – Capable of weighing to 0.01 g

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

8. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product # 54258-U.

9. SPE Tube Adapter - Millipore-Sigma; Product # 57020-U.

10. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.

11. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.

12. Vacuum manifold for Solid Phase Extraction (SPE).

13. Centrifuge – "Thermo Sorvall ST4" with TX-750 rotor or equivalent, capable of a maximum rotational force of 3800 rcf.

14. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. FisherScientific cat # 14-287-150, or equivalent.

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

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

17. Pipettes – Disposable transfer. Fisher Scientific, Cat. No. 13-711-7M

18. Vortex mixer, variable speed, Fisher Scientific or equivalent.

19. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control. Organomation, Inc. Cat. #11250-NT, or equivalent.

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

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

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

23. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent

24. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125

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

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

27. Syringe filter - Acrodisc, Syringe Filter, 25 mm, 0.2 µm, 50/pkg, Fisher part # 50786068.

28. Silanized glass wool, Supelco # 20411 or equivalent

29. Wheaton Bottle, 15ml, narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Cat. No. 209044SP, or equivalent

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

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

2. PromoChrom automated extractor, model, SPE-03, or equivalent
   a. Promochrom Filters, Catalog Number F-HC-30

3. HPLC columns

   a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-Y0 or equivalent

   b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

Reagents and Standards

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

A. Reagents:

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

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

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

B. Standards:

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

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

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

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

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

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

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

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

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

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

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 04*.
If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

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

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

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

Attachment 05 - Native PFAS Intermediate A
Attachment 06 - Native PFAS Intermediate B
Attachment 07 - Native PFAS Intermediate M
Attachment 08 - Extraction Standard Mix
Attachment 09 - Internal Standard Mix
Attachment 10 - Initial Calibration Standards Preparation
Attachment 11 - Initial Calibration Standards Concentrations
Attachment 12 - Bile Salts Stock Solutions
Attachment 13 - Bile Salts Working Solution A
Attachment 14 - Bile Salts Working Solution B
Attachment 15 - PFAS Linear/Branched Mix A
Attachment 16 - PFAS Linear/Branched Mix B
Attachment 17 - Linear/Branched Bile Salts Solution
Attachment 18 - ICV Working Standard

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order(quadratic) curve fit. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.

2. Analyze a Cal4 level standard that contains Bile Salt retention time markers and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the

calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

Example Initial Calibration Sequence:

1. Instrument Blank
2. Instrument Blank
3. Instrument Blank
4. CAL 1
5. CAL 2
6. CAL 3
7. CAL 4
8. CAL 5
9. CAL 6
10. CAL 7
11. ICB (Instrument Blank)
12. ICV
13. WDM (Linear Branched/Bile Salts standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 02* for additional information about compound relationships.

4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic acid (TCDCA), and Tauroursodeoxycholic acid (TUDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

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

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA

PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

5. Fit the curve

a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.

c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

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

6. Initial Calibration Verification (ICV)

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

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

2. Acceptance criteria

a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

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

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

A. Sample Preparation

1. Weigh sample container with contents on a calibrated top loading balance, and record the first reading in the automated prep entry system.

   a. For all samples, the full bottle must be extracted. The sample must remain in the original sample container until after spiking solutions have been added.

   b. If limited sample is submitted, spike sample in original container, then add reagent water to bring to final volume of 125 mL prior to SPE extraction (see B.6 for spiking details).
   c. If the sample has dissolved and/or settleable solids content (i.e; is cloudy or has a layer of sediment/solids at the bottom of the bottle), the sample must be centrifuged in order to minimize the difficulty of passing throug the SPE sorbent bed. In order to preserve the integrity of the sample and ensure the full voume of the container is used, see Procedure C.

2. Use a 125 mL HDPE bottle for the method blank, the laboratory control sample (LCS) and the low level laboratory control sample (LLCS). Fill each bottle with 125 mL of reagent water. Record 125 mL as the volume for the batch QC samples on the batchlog.

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

B. Solid Phase Extraction (SPE) - Manual Sample Extraction

1. Pack clean silanized glass wool to a quarter of the height of the SPE cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batch log.

3. Condition each SPE cartridge with the following reagents in the following order without allowing the cartridges to go dry:

   a. 15 mL 1% methanolic ammonium hydroxide

   b. 5 mL 0.3M formic acid

   c. Discard conditioning eluent(s)

4. Label each sample bottle, cap and reservoir with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Vortex all spike solutions prior to use.

6. Spike QC and all samples with 25 µl of Working labeled extraction standard spike solution (PFAS EIS Mix). Spike LCS/MS/MSD with 200 µl of native 1633 mid-level spike solution (PFAS Native M). Spike LLCS with 200 µl of native 1633 low-level spike solution (PFAS Native B). Vortex/Shake containers to mix thoroughly.

7. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges.

8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5 mL 1:1 0.1M formic acid/methanol and pass the rinses through the cartridge using vacuum. Apply full

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

NOTE: If a given sample is too problematic to extract using one cartridge with full vacuum, a second cartridge is created following Procedure B.1-3 above.

9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge ensuring the delivery needles to do not touch the sides of the tubes.

10. Rinse the inside of each empty sample/QC bottle with 5mL of 1% methanolic ammonium hydroxide.

11. Using a glass pipette, transfer the rinse from the bottles to the SPE reservoirs, washing the walls of the reservoirs. Set empty bottles aside to air dry.

12. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

13. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

Note: If a second cartridge was required, the elutant from the second cartridge is added to the first cartridge elutant with a methanol rinse and then blown down to 5 mL on an N-evap prior to step 14 below.

14. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix.

15. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

16. Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to a clean 15-mL polypropylene centrifuge collection tube.

17. Place a syringe filter (25-mm filter, 0.2-µm nylon membrane) on a 5 mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the plunger and filter the entire extract into the new collection tube containing the internal standard.

18. Bring each sample extract to final volume 5ml using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

19. Cap and vortex to mix.

20. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

21. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for dilution or reinjection if needed.

C. Samples containing Dissolved and/or Settleable Solids

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

2. Spike sample with appropriate spikes as in Procedure B.6.

3. Centrifuge the full bottle.

4. DO NOT SHAKE BOTTLE FOLLOWING THE CENTRIFUGE STEP.

5. Follow steps in Procedure B, 1 through 4.

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

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

8. Continue extraction process with Procedure B. 8.

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

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

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

1. Pack clean silanized glass wool to a quarter of the height of the SPE cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

3. Check to make sure all reagent bottles are filled and that reagents are not expired.

4. Label each sample bottle and cap with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Vortex all spike solutions prior to use.

6. Spike QC and all samples with 25 µl of Working labeled extraction standard spike solution (PFAS EIS Mix). Spike LCS/MS/MSD with 200 µl of native 1633 mid-level spike solution (PFAS Native M). Spike LLCS with 200 µl of native 1633 low-level spike solution (PFAS Native B). Vortex/Shake containers to mix thoroughly.

7. Select "Clean sys-004" from the drop down menu to start the cleaning cycle prior to SPE. Make sure the "Clean" cartridges and bottles are attached for this cycle. After the "Clean sys-004" has finished, attach Promochrom filters to the bottles, select "Clean Filter" from the drop down menu to clean filters prior to SPE.

8. Remove "Clean" cartridges and attach the labeled cartridges containing a graphitized carbon that will be used when running the samples when the system cleaning is complete. Disconnect the "Clean" bottles. Attach the labeled sample bottles by twisting the bottles and not the cap. Place sample bottles upside down in corresponding numbered location on shaker. Poke two holes into the lip of each bottle. Load labeled centrifuge tubes in the moving tray beneath the cartridges.

9. Select "EPA 1633-004" from the drop down menu and press the green check mark to start the procedure. Note: N2 dry down should be 15 minutes long.

10. Once the cycle is complete, remove centrifuge tubes and discard the used cartridges and bottles. Reattach "Clean" bottles and cartridges, select "clean sys-004" from menu, select "start". This must be done between each batch and at the beginning and the end of the day.

11. Add 25ul of Acetic acid to sample extracts and vortex to mix thoroughly. Extracts must be stored in a cooler.

12. Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to a clean 15-mL polypropylene centrifuge collection tube. Using a syringe, push ~ 3mL of MeOH through each syringe filter and allow to dry..

13. Place a syringe filter (25-mm filter, 0.2-µm nylon membrane) on a 5 mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the plunger and filter the entire extract into the new collection tube containing the internal standard.

14. Bring each sample extract to final volume 5ml using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

15. Cap and vortex to mix.

16. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

17. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for reinjection if needed.

E. LC/MS/MS Analysis

1. Mass Calibration and Tuning

   a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

   b. When masses fall outside of the +/- 0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Example acquisition method: See *Attachment 03*. Mass Transitions: See *Attachment 01*.

4. Instrument Sensitivity Check (ISC) and Instrument Blanks
   a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the

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

b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salt RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

   Example Sample Sequence:
   1. Instrument blank
   2. Instrument blank
   3. Instrument blank
   4. Instrument Sensitivity Check (CCV 0_CAL1)
   5. CCV 2_CAL4
   6. Linear Branched/Bile Salts marker (WDM)
   7. Instrument Blank (ICB)
   8. Method Blank (MB)
   9. Low Level LCS (LLCS)
   10. LCS
   11. Sample (10 or less)
   12. CCV 3_CAL4
   13. Instrument Blank

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.

b. If LLCS and LCSD recoveries are acceptable, proceed to sample quantitation.

c. If the LCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS must be reextracted.

d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Calculate the relative percent difference(RPD) between the sample and the matrix duplicate(DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data.

11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. (See Statistical Infromation/Method Performance section of this SOP.) If the extraction standard recovery(ies) is(are) outside the QC limit(s), revial a fresh aliquot and reanalyze. If the extraction standard recovery(ies) are still outside the QC limit(s), reanalyze the sample at a dilution. If the extraction standard recovery(ies) are still outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) are still outside the QC limit(s), narrate with an NCM and report the data.

12. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

13. Compare the retention times of all of the analytes, surrogates, and internals standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than

    a. 0.4 minutes for isotopically-labeled compounds

    b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound

    c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

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

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

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

15. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.

16. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of internal standard spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring dilutions greater than 10X should be reextracted using a reduced aliquot.

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

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

    On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

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

4. Sample Concentration

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

5. Ion Ratio

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

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

Statistical Information/Method Performance

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

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

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

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

Quality Assurance/Quality Control

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

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

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

Attachment 08 - Extraction Standard Mix (.pdf)
Attachment 09 - Internal Standard Mix (.pdf)
Attachment 10 - Initial Calibration Standards Preparation (.pdf)
Attachment 11 - Initial Calibration Standard Concentrations (.pdf)
Attachment 12 - Bile Salt Stock Solutions (.pdf)
Attachment 13 - Bile Salts Working Solution A (.pdf)
Attachment 14 - Bile Salts Working Solution B (.pdf)
Attachment 15 - PFAS Linear Branched Mix A (.pdf)
Attachment 16 - PFAS Linear Branched Mix B (.pdf)
Attachment 17 - Linear Branched Bile Salts Solution (.pdf)
Attachment 18 - ICV Working Standard (.pdf)
Attachment 19 - Table 5 (1633 Draft V3) (.docx)
Attachment 20 - Table 8 (1633 Draft V3) (.docx)

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Attachment: Attachment 11 - Initial Calibration Standard Concentrations (pdf)
Attachment: Attachment 12 - Bile Salt Stock Solutions (pdf)
Attachment: Attachment 13 - Bile Salts Working Solution A (pdf)
Attachment: Attachment 14 - Bile Salts Working Solution B (pdf)
Attachment: Attachment 15 - PFAS Linear Branched Mix A (pdf)
Attachment: Attachment 16 - PFAS Linear Branched Mix B (pdf)
Attachment: Attachment 17 - Linear Branched Bile Salts Solution (pdf)
Attachment: Attachment 18 - ICV Working Standard (pdf)
Attachment: Attachment 19 - Table 5 (1633 Draft V3) (docx)
Attachment: Attachment 20 - Table 8 (1633 Draft V3) (docx)

End of document

Version history

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

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

Attachment 1

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

Attachment 1

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

Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

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

Extraction Standards

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

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

Native PFAS Compounds

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

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

Acquisition Method

1633

Mass Spec 12.500 min
Period 12.500 mi
-MRM
Integrated Valve
LC System
Equilibrate
Injection

Mass Spectrometer Method Properties

Period 1:

Scans in Period:	1250
Min. Dwell Time:	3 ms
Max. Dwell Time:	250 ms
Relative Start Time:	0.00 msec
Scheduled Ionization:	Off
Experiments in Period:	1
Use target Cycle Time:	No
Target Cycle Time:	N/A

Period 1 Experiment 1:

Scan Type:	MRM (MRM)
Scheduled MRM:	Yes
Polarity:	Negative
Scan Mode:	N/A
Ion Source:	Turbo Spray
sMRM Q1Q3 Resolution:	No
MRM detection window:	60 sec
Target Scan Time:	0.6000 sec
Resolution Q1:	Unit
Resolution Q3:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Step Size:	0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
212.800	168.900	2.71	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	84.900	2.94	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	2.98	DP	-50.00	-50.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	3.21	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	79.900	3.67	DP	-90.00	-90.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.100	3.32	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	134.900	3.84	DP	-70.00	-70.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	3.52	DP	-30.00	-30.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.41	DP	-110.00	-110.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.55	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

349.100	79.900	4.00	DP	-90.00	-90.00	PFPeS	
			CE	-70.00	-70.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.68	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.85	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.30	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	3.96	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	3.98	DP	-90.00	-90.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.68	DP	-110.00	-110.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.12	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	5.22	DP	-90.00	-90.00	PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.43	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.40	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.61	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.72	DP	-100.00	-100.00	PFNS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.85	DP	-60.00	-60.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.58	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	6.14	DP	-110.00	-110.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.79	DP	-80.00	-80.00	NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	6.16	DP	-90.00	-90.00	PFDS
			CE	-130.00	-130.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.39	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	5.04	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.49	DP	-90.00	-90.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.87	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.94	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	7.08	DP	-90.00	-90.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.94	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	7.24	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.36	DP	-90.00	-90.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	619.000	6.30	DP	-60.00	-60.00	PFTrDA
			CE	-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.71	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.66	DP	-110.00	-110.00	PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.52	DP	-10.00	-10.00	PFECA B_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.41	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.55	DP	-40.00	-40.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.00	DP	-110.00	-110.00	PFPeS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.68	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.85	DP	-50.00	-50.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.30	DP	-90.00	-90.00	PFHxS_2

			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	3.98	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.68	DP	-100.00	-100.00	PFHpS_2
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.12	DP	-50.00	-50.00	PFOA_2
			CE	-26.00	-26.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	5.21	DP	-110.00	-110.00	PFOS_2
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	4.43	DP	-50.00	-50.00	PFNA_2
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	98.800	5.72	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	219.000	4.85	DP	-60.00	-60.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	80.800	4.58	DP	-100.00	-100.00	8:2 FTS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	483.000	4.79	DP	-90.00	-90.00	NMeFOSAA_2
			CE	-24.00	-24.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	98.800	6.16	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	269.100	5.39	DP	-70.00	-70.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	526.000	5.04	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	319.000	5.87	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

663.000	168.900	6.30	DP		-60.00	-60.00	PFTrDA_2
			CE		-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.71	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.21	DP	-40.00	-40.00	PFPeA_2
			CE	-55.00	-55.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	6.13	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	7.08	DP	-110.00	-110.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.35	DP	-90.00	-90.00	NEtPFOSA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	3.95	DP	-40.00	-40.00	DONA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.60	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.48	DP	-90.00	-90.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	2.98	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.40	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.83	DP	-70.00	-70.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.94	DP	-90.00	-90.00	PFDoS_2
			CE	-140.00	-140.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.71	DP	-40.00	-40.00	13C3-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.71	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.21	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.66	DP	-90.00	-90.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.41	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.54	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.54	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.68	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.85	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.30	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.30	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	3.97	DP	-90.00	-90.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.12	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.12	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	5.21	DP	-90.00	-90.00	13C4-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	79.900	5.21	DP	-90.00	-90.00	13C8-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.42	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
519.100	474.100	4.85	DP	-60.00	-60.00	13C6-PFDA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.100	470.100	4.85	DP	-60.00	-60.00	13C2-PFDA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	80.900	4.58	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
506.100	77.800	6.13	DP	-110.00	-110.00	13C8-PFOSA
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
573.200	419.000	4.78	DP	-90.00	-90.00	d3-NMeFOSAA
			CE	-27.00	-27.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.100	5.38	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.200	419.000	5.02	DP	-90.00	-90.00	d5-NEtFOSAA

			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.86	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.92	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	7.07	DP	-90.00	-90.00	d3-NMePFOSA
			CE	-37.00	-37.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	7.21	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.34	DP	-90.00	-90.00	d5-NEtPFOSA
			CE	-38.00	-38.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.70	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.54	DP	-40.00	-40.00	13C5-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.66	DP	-110.00	-110.00	13C3-PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.29	DP	-90.00	-90.00	13C3-PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	5.20	DP	-110.00	-110.00	13C8-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.41	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	3.97	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.58	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.68	DP	-75.00	-75.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

315.100	119.400	3.54	DP	-40.00	-40.00	13C2-PFHxA_2	
			CE	-31.00	-31.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	5.21	DP	-110.00	-110.00	13C4-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.78	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.70	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.39	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
CAD:	10.00
EP	-10.00
CXP	-14.00

Acquisition Method

1633

Mass Spec 12.500 min
   Period 12.500 min
      -MRM
Integrated Valve
LC System
   Equilibrate
   Injection

Acquisition Information:

Acquisition Method:	1633.dam
Created:	Wednesday May 24 2023 14:48:39 PM
Last Modified:	Wednesday June 14 2023 11:54:50 AM
Comment:	
Synchronization Mode:	LC Sync
Auto-Equilibration:	Off
Acquisition Duration:	12min30sec
Number Of Scans:	1250
Periods In File:	1
Acquisition Module:	Acquisition Method
Software version	Analyst 1.7.3

Period 1:

Scans in Period:	1250
Min. Dwell Time:	3 ms
Max. Dwell Time:	250 ms
Relative Start Time:	0.00 msec
Scheduled Ionization:	Off
Experiments in Period:	1
Use target Cycle Time:	No
Target Cycle Time:	N/A

Period 1 Experiment 1:

Scan Type:	MRM (MRM)
Scheduled MRM:	Yes
Polarity:	Negative
Scan Mode:	N/A
Ion Source:	Turbo Spray
sMRM Q1Q3 Resolution:	No
MRM detection window:	60 sec
Target Scan Time:	0.6000 sec
Resolution Q1:	Unit
Resolution Q3:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Step Size:	0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
212.800	168.900	2.71	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	84.900	2.94	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	2.98	DP	-50.00	-50.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	3.21	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	79.900	3.67	DP	-90.00	-90.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.100	3.32	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	134.900	3.84	DP	-70.00	-70.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	3.52	DP	-30.00	-30.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.41	DP	-110.00	-110.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.55	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	79.900	4.00	DP	-90.00	-90.00	PFPeS
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.68	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.85	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.30	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	3.96	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	3.98	DP	-90.00	-90.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.68	DP	-110.00	-110.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.12	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	5.22	DP	-90.00	-90.00	PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.43	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.40	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.61	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.72	DP	-100.00	-100.00	PFNS

			CE	-110.00	-110.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.85	DP	-60.00	-60.00	PFDA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.58	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	6.14	DP	-110.00	-110.00	PFOSA
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.79	DP	-80.00	-80.00	NMeFOSAA
			CE	-27.00	-27.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	6.16	DP	-90.00	-90.00	PFDS
			CE	-130.00	-130.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.39	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	5.04	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.49	DP	-90.00	-90.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.87	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.94	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	7.08	DP	-90.00	-90.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.94	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	7.24	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.36	DP	-90.00	-90.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

663.000	619.000	6.30	DP		-60.00	-60.00	PFTrDA
			CE		-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.71	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.66	DP	-110.00	-110.00	PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.52	DP	-10.00	-10.00	PFECA B_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.41	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.55	DP	-40.00	-40.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.00	DP	-110.00	-110.00	PFPeS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.68	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.85	DP	-50.00	-50.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.30	DP	-90.00	-90.00	PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	3.98	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.68	DP	-100.00	-100.00	PFHpS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.12	DP	-50.00	-50.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	5.21	DP	-110.00	-110.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	4.43	DP	-50.00	-50.00	PFNA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	98.800	5.72	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	219.000	4.85	DP	-60.00	-60.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	80.800	4.58	DP	-100.00	-100.00	8:2 FTS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	483.000	4.79	DP	-90.00	-90.00	NMeFOSAA_2
			CE	-24.00	-24.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	98.800	6.16	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	269.100	5.39	DP	-70.00	-70.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	526.000	5.04	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	319.000	5.87	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	168.900	6.30	DP	-60.00	-60.00	PFTrDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.71	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.21	DP	-40.00	-40.00	PFPeA_2
			CE	-55.00	-55.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	6.13	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	7.08	DP	-110.00	-110.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.35	DP	-90.00	-90.00	NEtPFOSA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	3.95	DP	-40.00	-40.00	DONA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.60	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.48	DP	-90.00	-90.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	2.98	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.40	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.83	DP	-70.00	-70.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.94	DP	-90.00	-90.00	PFDoS_2
			CE	-140.00	-140.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.71	DP	-40.00	-40.00	13C3-PFBA

			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.71	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.21	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.66	DP	-90.00	-90.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.41	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.54	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.54	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.68	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.85	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.30	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.30	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	3.97	DP	-90.00	-90.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.12	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.12	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	5.21	DP	-90.00	-90.00	13C4-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

507.100	79.900	5.21	DP		-90.00	-90.00	13C8-PFOS
				CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.42	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
519.100	474.100	4.85	DP	-60.00	-60.00	13C6-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.100	470.100	4.85	DP	-60.00	-60.00	13C2-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	80.900	4.58	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
506.100	77.800	6.13	DP	-110.00	-110.00	13C8-PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
573.200	419.000	4.78	DP	-90.00	-90.00	d3-NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.100	5.38	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.200	419.000	5.02	DP	-90.00	-90.00	d5-NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.86	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.92	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	7.07	DP	-90.00	-90.00	d3-NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	7.21	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.34	DP	-90.00	-90.00	d5-NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.70	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.54	DP	-40.00	-40.00	13C5-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.66	DP	-110.00	-110.00	13C3-PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.29	DP	-90.00	-90.00	13C3-PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	5.20	DP	-110.00	-110.00	13C8-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.41	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	3.97	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.58	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.68	DP	-75.00	-75.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	119.400	3.54	DP	-40.00	-40.00	13C2-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	5.21	DP	-110.00	-110.00	13C4-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.78	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.70	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.39	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
CAD:	10.00
EP	-10.00

CXF -14.00

Integrated Valve Method Properties
Valve Diverter

	Total Time (min)	Position
1	1.0	B
2	8.0	A

LC Method Properties

LC system Equlibration time = 0.00 min
LC system Injection Volume = 10.00 ul

Pump

Model:	A: LC-40D XF B: LC-40D XR
Pump mode:	Binary gradient
Stop time:	12.50 min
<Gradient start adjustment>	
Mode:	None
<B.GE1>	
<General>	
Flow/Pressure:	Flow
Flow:	0.5000 mL/min
Time to reach the flow:	0.00 min
B.Conc:	5.0 %
B.Curve:	0
Pressure limits Minimum:	0 psi
Pressure limits Maximum:	6500 psi

<Gradient>

Time min	Flow mL/min	B.Conc %	B.Curve
0.00	0.5000	5.0	0
0.20	0.5000	5.0	0
3.00	0.5000	60.0	0
3.80	0.5000	60.0	0
6.70	0.5000	98.0	0
8.50	0.5000	98.0	0
8.70	0.6500	5.0	0
12.50	0.5000	5.0	0

<PumpA>	
<Mobile phase switching valve>	
Selected mobile phase port:	A
<Mobile phase settings>	
Set compressibility:	No

Port	Mobile phase name
A	Mobile phase A

<Advanced>	
LPGE cycle:	Standard
<PumpB>	

```
      <Mobile phase switching valve>
         Selected mobile phase port:                    A
      <Mobile phase settings>
         Set compressibility:                          No
      Port  Mobile phase name
      ----  -----------------
         A     Mobile phase A
      <Advanced>
         LPGE cycle:                             Standard
<Autopurge settings>
   Execute autopurge before analysis:                  No
   <Autosampler purge settings>
      Execute sampler purge:                           No
   <Init conc-replacement>
      Use Init conc-replacement:                       No
   <Warm up>
      Wait time:                                  0 min
      B.GE1 flow:                          0.0000 mL/min
   <Execute after autopurge>
      Turn oven on after autopurge:                    No
      Turn pump on after autopurge:                    No
<Time program>
Autosampler
===========
Model:                                        SIL-40C XR
<General>
   Use Autosampler:                                  Yes
<Sample plate settings>
   Specify plate:                                    No
   Specify needle stroke:                            No
<Injection settings>
   Sampling speed:                             1.0 µL/s
   Use cooler temperature:                           Yes
   Cooler temperature:                            15 °C
   Use air gap volume:                               No
   Use low dispersion injection:                     No
<Acquisition cycle time optimization>
   Pretreatment start timing:                        Off
<Cut off loop settings>
   Cut off loop:                                     No
<Rinse settings>
   Rinse type:                           External only
   Rinse mode:                 Before and after aspiration
   Rinse dip time:                                0 s
   Rinse method:                        Rinse port only
   Rinse time:                                  2.0 s
   Rinsing speed:                              35 µL/s
   Rinsing volume:                             500 µL
   Measuring line purge volume:                100 µL
<Details>
   Wait for temperature equilibration before run (sample cooler):
                                                     No
```

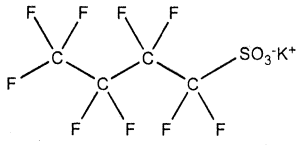
```
<Pretreatment>
   Mode:                                          Standard
<Time program>
Oven
====
Model:                                         A: CTO-40C
<Column Oven A>
   <General>
      Use Column Oven:                               Yes
      Oven temperature:                           40 °C
      Temperature limit(Maximum):                 90 °C
      Wait for temperature equilibration before run:  Off
   <Advanced>
      Fan speed:                                  Auto
      Cooler mode:                                Auto
<Time program>
System Controller
==================
Model:                                           CBM-40
<External output settings>
   Event 1:                                          No
   Event 2:                                          No
<Shutdown settings>
   Power off after shutdown:                         No
<Time program>
Time  Command  Value
 min
---- -------- ------
0.01    Event      1
```

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

PRODUCT CODE: L-PFBS
LOT NUMBER: LPFBS1022
COMPOUND: Potassium perfluoro-1-butanesulfonate

STRUCTURE:
CAS #: 29420-49-3



MOLECULAR FORMULA: $C_4F_9SO_3K$
MOLECULAR WEIGHT: 338.19
CONCENTRATION: 50.0 ± 2.5 µg/mL (K salt)
44.4 ± 2.2 µg/mL (PFBS acid)
44.2 ± 2.2 µg/mL (PFBS anion)
SOLVENT(S): Methanol
CHEMICAL PURITY: >98%
LAST TESTED: (mm/dd/yyyy) 10/04/2022
EXPIRY DATE: (mm/dd/yyyy) 10/04/2027
RECOMMENDED STORAGE: Store ampoule in a cool, dark place

DOCUMENTATION/ DATA ATTACHED:

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

ADDITIONAL INFORMATION:

- See page 2 for further details.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Certified By: _____ **Date:** 10/14/2022 (mm/dd/yyyy)
B.G. Chittim, General Manager

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

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

INTENDED USE:

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

HANDLING:

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

SYNTHESIS / CHARACTERIZATION:

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

HOMOGENEITY:

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

UNCERTAINTY:

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

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters $x_1, x_2, \ldots x_n$ on which it depends is:

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

QUALITY MANAGEMENT:

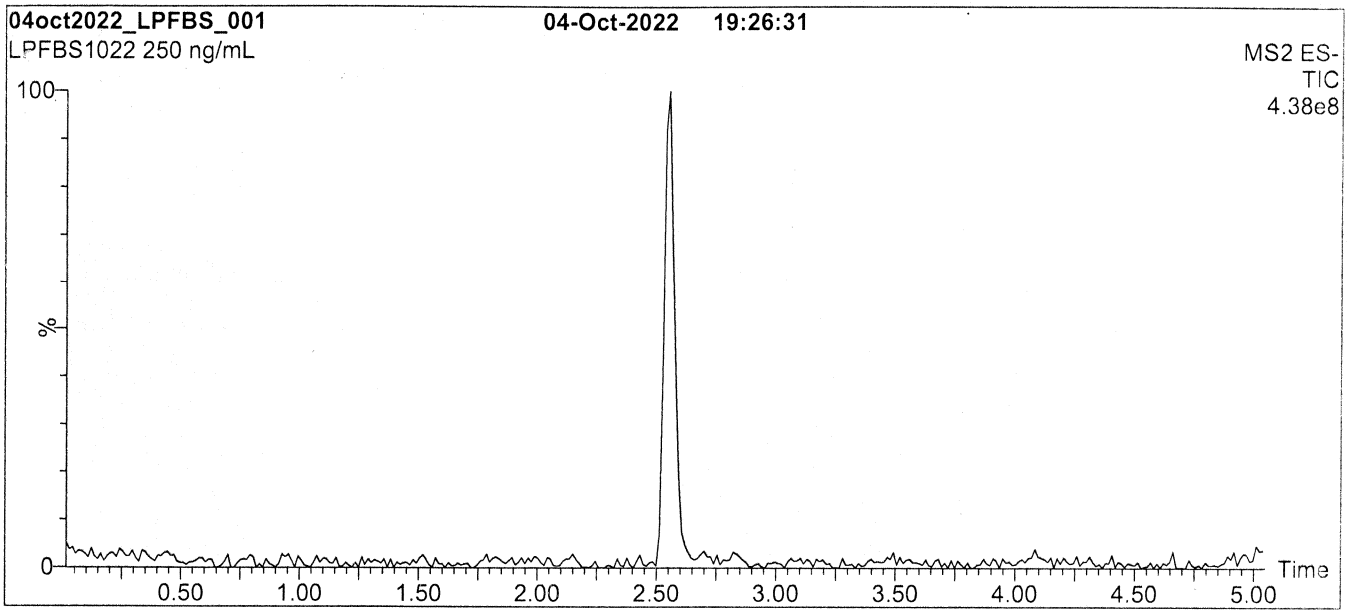
This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A1226), and ISO 17034 by ANSI National Accreditation Board (ANAB; AR-1523).

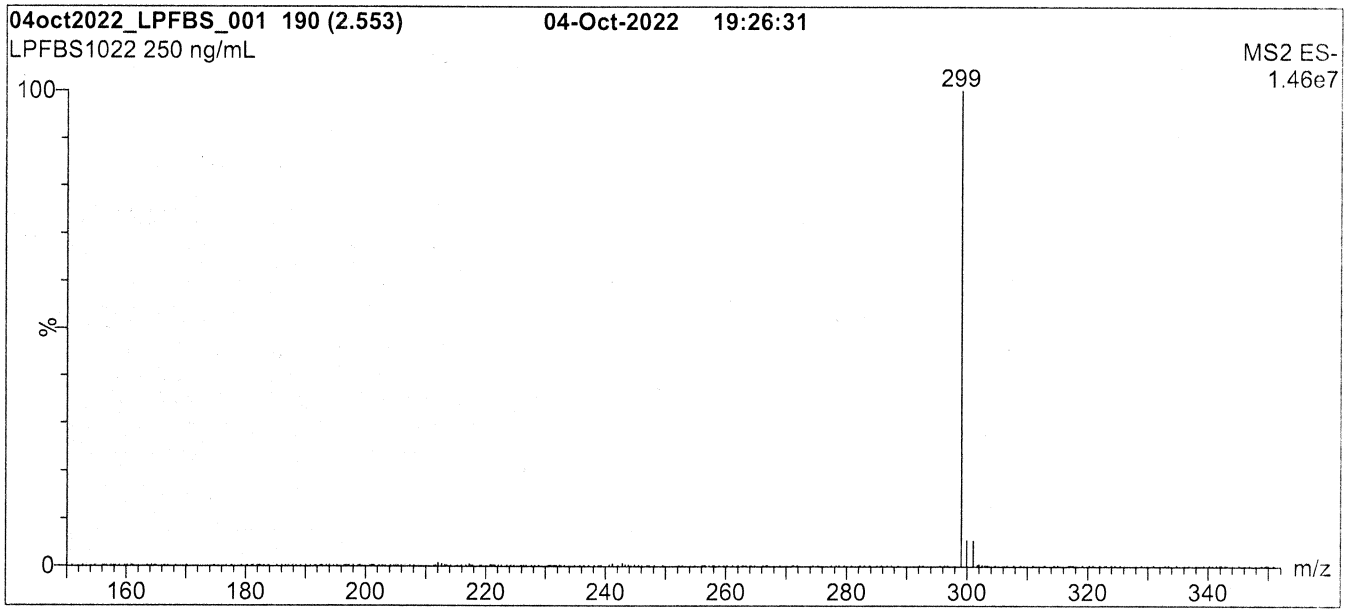
CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

Figure 1: **L-PFBS; LC/MS Data (Full Scan and Mass Spectrum)**





Conditions for Figure 1:

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

Chromatographic Conditions:

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

Mobile phase: Gradient
Start: 60% H_2O / 40% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions over 0.75 min.
Time: 12 min

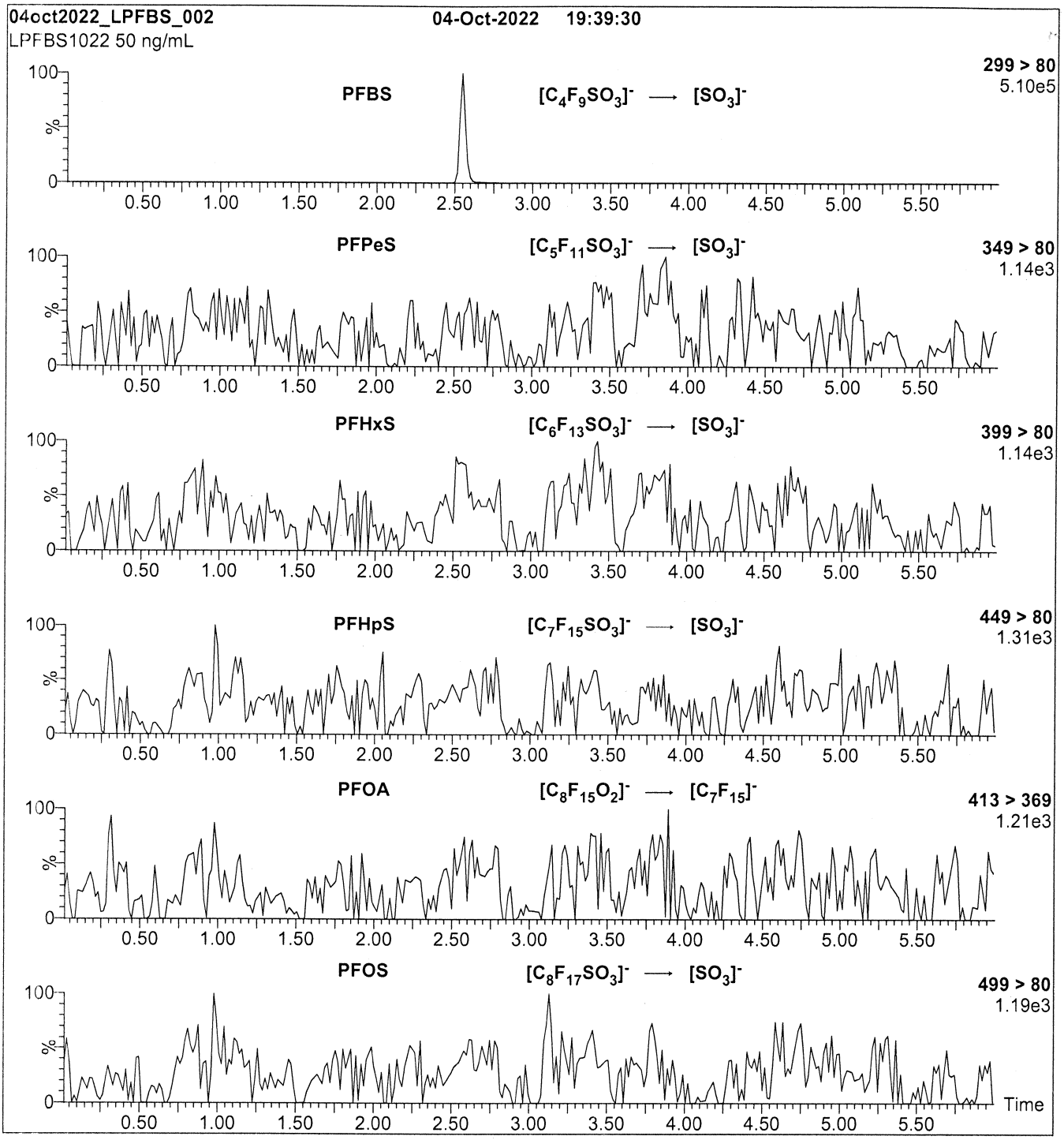
Flow: 300 µL/min

MS Parameters:

Experiment: Full Scan (150 - 850 amu)

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = 10.00
Desolvation Temperature (°C) = 500
Desolvation Gas Flow (L/hr) = 1000

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



Conditions for Figure 2:

Injection:	On-column (L-PFBS)
Mobile phase:	Same as Figure 1
Flow:	300 µL/min

MS Parameters:

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

Attachment 5

Native PFAS Intermediate A								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Native PFAS Intermediate A (ng/ml)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.1	10	500
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.05		250
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.025		125
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.025		125
Wellington	PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.025		125
Wellington	PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.025		125
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.025		125
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000	0.025		125
Wellington	PFDoA	Perfluorododecanoic acid	307-55-1	PFDoA	50000	0.025		125
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.025		125
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.025		125
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.025		111
Wellington	L-PFPeS	Perfluoropentansulfonic acid	2706-91-4	PFPeS	46900	0.025		117
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.025		114
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.025		119
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.025		117
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.025		120
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.025		121
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400	0.025		121
Wellington	4;2 FTS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	46700	0.1		467
Wellington	6;2 FTS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	47400	0.1		474
Wellington	8;2 FTS	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	47900	0.1		479
Wellington	FOSA-I	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.025		125
Wellington	N-MeFOSA-M	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	50000	0.025		125
Wellington	N-EtFOSA-M	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	50000	0.025		125
Wellington	br-NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.025		125
Wellington	br-NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.025		125
Wellington	N-MeFOSE-M	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	50000	0.25		1250
Wellington	N-EtFOSE-M	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	50000	0.25		1250
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	50000	0.1		500
Wellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	47100	0.1		471
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.05		250
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50000	0.05		250
Wellington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	50000	0.05		250
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.1		466
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.1		471
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.05		223
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000	0.625		3125
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000	0.625		3125

Attachment 6

Native PFAS Intermediate B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. Native PFAS Intermediate B (ng/ml)**
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.1	10	5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			2.5
	Perfluorohexanoic acid	307-24-4	PFHxA	125			1.25
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			1.25
	Perfluorooctanoic acid	335-67-1	PFOA	125			1.25
	Perfluorononanoic acid	375-95-1	PFNA	125			1.25
	Perfluorodecanoic acid	335-76-2	PFDA	125			1.25
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			1.25
	Perfluorododecanoic acid	307-55-1	PFDoA	125			1.25
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			1.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			1.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			1.11
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117.25			1.17
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75			1.14
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			1.19
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5			1.17
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			1.20
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5			1.21
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			1.21
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			4.67
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			4.74
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			4.79
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			1.25
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			1.25
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			1.25
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			1.25
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			12.5
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			12.5
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			5
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			4.71
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			2.5
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			2.5
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			2.5
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466			4.66
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471			4.71
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			2.23
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			6.25
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			31.3
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			31.3

Attachment 7

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

Attachment 8

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

Attachment 9

Internal Standard Mix								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Concentration (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. Internal Standard Mix (ng/ml)**
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	50000	50	10	250
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	50000	25		125
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	50000	12.5		62.5
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	47800	25		120
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	50000	12.5		62.5
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	50000	25		125
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	47300	25		118

Attachment 10

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

Attachment 11

1633 Initial Calibration Standard Concentrations							
	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Compound Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
PFpeA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFHxA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFHpA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFOA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFNA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFUnA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDoA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTrDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTeDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFBS	0.044	0.111	0.276	0.553	1.11	2.76	13.8
PFPeS	0.047	0.117	0.293	0.586	1.17	2.93	14.7
PFHxS	0.046	0.114	0.284	0.569	1.14	2.84	14.2
PFHpS	0.048	0.119	0.298	0.595	1.19	2.98	14.9
PFOS	0.047	0.117	0.291	0.583	1.17	2.91	14.6
PFNS	0.048	0.120	0.300	0.600	1.20	3.00	15.0
PFDS	0.048	0.121	0.301	0.603	1.21	3.01	15.1
PFDoS	0.048	0.121	0.303	0.605	1.21	3.03	15.1
4:2 FTS	0.187	0.467	1.17	2.34	4.67	11.7	58.4
6:2 FTS	0.190	0.474	1.19	2.37	4.74	11.9	59.3
8:2 FTS	0.192	0.479	1.20	2.40	4.79	12.0	59.9
PFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
NEtFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
HFPO-DA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
DONA	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFMPA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFMBA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
NFDHA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
9Cl-PF3ONS	0.186	0.466	1.17	2.33	4.66	11.7	58.3
11Cl-PF3OUdS	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFEESA	0.089	0.223	0.56	1.11	2.23	5.56	27.8
3:3 FTCA	0.250	0.625	1.56	3.13	6.25	15.6	78.1
5:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
7:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
13C4-PFBA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
13C5-PFPeA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C5-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C4-PFHpA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C8-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C9-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C6-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C7-PFUnA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFDoA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFTeDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C3-PFBS	0.581	0.581	0.581	0.581	0.581	0.581	0.581
13C3-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591
13C8-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C8-PFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
D5-NEtFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-4:2FTS	1.17	1.17	1.17	1.17	1.17	1.17	1.17
13C2-6:2FTS	1.19	1.19	1.19	1.19	1.19	1.19	1.19
13C2-8:2FTS	1.20	1.20	1.20	1.20	1.20	1.20	1.20
13C3-HFPO-DA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
D7-NMeFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
D9-NEtFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
D5-NEtFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C3-PFBA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C4-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C2-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C4-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C5-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
18O2-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591

Attachment 12

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

Attachment 13

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

Attachment 14

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

Attachment 15

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

Attachment 16

PFAS Linear/Branched Mix B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. PFAS Linear/Branched Mix B (ng/ml)**
PFAS Linear/Branched Mix A	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	500	0.5	2	125
	Perfluorooctanoic acid	335-67-1	PFOA	500			125
	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500			125
	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500			125
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500			125
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			125
	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			125
	Perfluoro-7-methyloctanoic acid	15899-31-7	PF7MOA	500			125
	Perfluorononanoic acid	375-95-1	PFNA	500			125

Attachment 17

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

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

Attachment 18

1633 ICV Working Standard							
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume* (ml)	Final Conc. 1633 ICV Working Standard (ng/ml)
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.01	2	2.50
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			1.25
	Perfluorohexanoic acid	307-24-4	PFHxA	125			0.625
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			0.625
	Perfluorooctanoic acid	335-67-1	PFOA	125			0.625
	Perfluorononanoic acid	375-95-1	PFNA	125			0.625
	Perfluorodecanoic acid	335-76-2	PFDA	125			0.625
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			0.625
	Perfluorododecanoic acid	307-55-1	PFDoA	125			0.625
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			0.625
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			0.625
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			0.553
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117			0.586
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	114			0.569
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			0.595
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	117			0.583
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			0.600
	Perfluorodecanesulfonic acid	335-77-3	PFDS	121			0.603
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			0.605
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			2.34
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			2.37
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			2.40
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			0.625
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			0.625
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			0.625
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			0.625
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			0.625
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			6.25
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			6.25
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			2.50
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			2.36
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			1.25
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			1.25
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			1.25
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466			2.33
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471			2.36
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			1.11
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			3.13
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			15.6
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			15.6
Extraction Standard Mix	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500	0.01		2.5
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250			1.25
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125			0.625
	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	116			0.581
	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	118			0.591
	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	120			0.598
	Perfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250			1.25
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	250			1.25
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	234			1.17
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	238			1.19
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	240			1.20
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	500			2.5
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	1250			6.25
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250			6.25
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
Internal Standard Mix	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250	0.01		1.25
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	120			0.598
	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	118			0.591

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

ATTACHMENT 21

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

	Aqueous Matrix [1, 2]			
	IPR			
Compounds	Recovery (%)	RSD (%)	OPR Recovery (%)	LLOPR Recovery (%)
PFBA	60 – 147	20	58 – 148	44 - 157
PFPeA	56 – 150	20	54 – 152	57 - 148
PFHxA	59 – 148	25	55 – 152	62 - 149
PFHpA	60 – 149	25	54 – 154	56 - 150
PFOA	55 – 158	25	52 – 161	57 - 161
PFNA	64 – 144	25	59 – 149	53 - 157
PFDA	57 – 142	25	52 – 147	43 - 158
PFUnA	54 – 153	30	48 – 159	50 - 155
PFDoA	73 – 133	25	64 – 142	60 - 141
PFTrDA	52 – 145	25	49 – 148	52 - 140
PFTeDA	49 - 158	25	47 – 161	52 - 156
PFBS	66 – 141	20	62 – 144	63 - 145
PFPeS	66 – 144	25	59 – 151	58 - 144
PFHxS	62 – 141	25	57 – 146	44 - 158
PFHpS	59 – 148	25	55 – 152	51 - 150
PFOS	61 – 145	20	58 – 149	43 - 162
PFNS	57 – 143	25	52 – 148	46 - 151
PFDS	56 – 142	25	51 – 147	50 - 144
PFDoS	41 – 140	30	36 – 145	30 - 138
4:2FTS	77 – 135	25	67 – 146	52 - 158
6:2FTS	75 – 137	30	61 – 151	48 - 158
8:2FTS	79 – 136	30	63 – 152	46 - 165
PFOSA	65 – 144	20	61 – 148	47 - 163
NMeFOSA	76 – 132	25	63 – 145	54 - 155
NEtFOSA	75 – 129	25	65 – 139	49 - 156
NMeFOSAA	69 – 134	25	58 – 144	32 - 160
NEtFOSAA	65 – 140	25	59 – 146	51 - 154
NMeFOSE	79 – 129	20	71 – 136	56 - 151
NEtFOSE	79 – 126	25	69 – 137	60 - 147
HFPO-DA	72 – 135	25	63 – 144	58 - 154
ADONA	75 – 138	20	68 – 146	61 - 148
PFMPA	55 – 141	25	51 – 145	48 - 150
PFMBA	59 – 145	20	55 – 148	49 - 154
NFDHA	63 – 146	35	48 – 161	47 - 160
9Cl-PF3ONS	72 – 140	30	56 – 156	44 - 167
11Cl-PF3OUdS	61 – 140	35	46 – 156	36 - 158
PFEESA	57 – 149	20	56 – 151	56 - 144
3:3FTCA	66 – 126	20	62 – 129	32 - 161
5:3FTCA	68 – 130	20	63 – 134	39 - 156
7:3FTCA	55 – 133	25	50 – 138	36 - 149
$^{13}C_4$-PFBA	10 – 130	30	10-130	10-130
$^{13}C_5$-PFPeA	35 – 150	30	40 -150	40 -150
$^{13}C_5$-PFHxA	55 – 150	30	40 -150	40 -150
$^{13}C_4$-PFHpA	55 – 150	30	40 -150	40 -150
$^{13}C_8$-PFOA	60 – 140	30	30-140	30-140
$^{13}C_9$-PFNA	55 – 140	30	30-140	30-140
$^{13}C_6$-PFDA	50 – 140	30	20-140	20-140

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

Compounds	Aqueous Matrix [1, 2]			
	IPR			
	Recovery (%)	RSD (%)	OPR Recovery (%)	LLOPR Recovery (%)
$^{13}C_7$-PFUnA	30 – 140	30	20-140	20-140
$^{13}C_2$-PFDoA	10 – 150	30	10-150	10-150
$^{13}C_2$-PFTeDA	10 – 130	30	10-130	10-130
$^{13}C_3$-PFBS	55 – 150	30	25-150	25-150
$^{13}C_3$-PFHxS	55 – 150	30	25-150	25-150
$^{13}C_8$-PFOS	45 – 140	30	20-140	20-140
$^{13}C_2$-4:2FTS	60 – 200	30	25-200	25-200
$^{13}C_2$-6:2FTS	60 – 200	30	25-200	25-200
$^{13}C_2$-8:2FTS	50 – 200	30	25-200	25-200
$^{13}C_8$-PFOSA	30 – 130	30	10-130	10-130
D_3-NMeFOSA	15 – 130	30	10-130	10-130
D_5-NEtFOSA	10 – 130	30	10-130	10-130
D_3-NMeFOSAA	45 – 200	30	10-200	10-200
D_5-NEtFOSAA	10 – 200	30	10-200	10-200
D_7-NMeFOSE	10 – 150	30	10-150	10-150
D_9-NEtFOSE	10 – 150	30	10-150	10-150
$^{13}C_3$-HFPO-DA	25 – 160	30	25-160	25-160

[1] The recovery limits apply to the target analyte results for IPR, OPR, and LLOPR samples for wastewater matrices. Data for this matrix type are derived from the multi-laboratory validation study and are therefore the limits required for this method.

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

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

EIS Compound	Recovery Range (%)
$^{13}C_4$-PFBA	10 – 130 *
$^{13}C_5$-PFPeA	35 - 150
^{13}C5-PFHxA	55 - 150
$^{13}C_4$-PFHpA	55 - 150
$^{13}C_8$-PFOA	60 - 140
$^{13}C_9$-PFNA	55 - 140
$^{13}C_6$-PFDA	50 - 140
$^{13}C_7$-PFUnA	30 - 140
$^{13}C_2$-PFDoA	10 - 150
$^{13}C_2$-PFTeDA	10 – 130 *
$^{13}C_3$-PFBS	55 - 150
$^{13}C_3$-PFHxS	55 - 150
$^{13}C_8$-PFOS	45 - 140
$^{13}C_2$-4:2FTS	60 – 200 *
$^{13}C_2$-6:2FTS	60 - 200 *
$^{13}C_2$-8:2FTS	50 – 200 *
$^{13}C_8$-PFOSA	30 – 130
D_3-NMeFOSA	15 – 130
D_5-NEtFOSA	10 – 130
D_3-NMeFOSAA	45 – 200 *
D_5-NEtFOSAA	10 – 200
D_7-NMeFOSE	10 – 150 *
D_9-NEtFOSE	10 – 150 *
$^{13}C_3$-HFPO-DA	25 - 160

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

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Revision Log
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Quality Assurance/Quality Control

Revision Log

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

Reference

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

Cross Reference

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

Document	Document Title
NC-QA-015	Support Equipment Calibration and Verification
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
QA-003	Quality Control Program
NC-QA-017	Standards and Reagents
NDSC-QA-QP44940	Calibration Curves and the Selection of Calibration Points
NDSC-US-EHSQP46060	NDSC EH&S Manual
NC-QAM-001	QA Manual

Scope

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

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

Analyte	Acronym	CAS#
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA, PES	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

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

Interferences

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

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.

3. All parts of the SPE manifold must be cleaned every 2 weeks per manufacturer recommendations.

Safety Precautions and Waste Handling

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

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

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

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

Personnel Training and Qualifications

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

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

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

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

Sample Collection, Preservation, and Handling

A. Sample Collection

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

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

B. Sample Storage and Shipment

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

2. Solid and Biosolid samples stored in the lab must be protected from light and held at a temperature of 0° to 6°C, or ≤ -20°C, until extraction.

3. Solid and Biosolid samples must be extracted within 90 days. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light.

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

Apparatus and Equipment

A. Apparatus

1. Specimen container; PP; natural; graduated; non-sterile; separate 1/4-turn red screwcap; 4 oz; 600 EA, Greenwood Products, Inc. Part No. HP30011

2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 14955237 or equivalent

3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288 or equivalent.

4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 0125-1902-PC.

5. Analytical Balance – Capable of weighing to 0.0001 g

6. Top-Loading Balance – Capable of weighing to 0.01 g

7. Waters Oasis WAX 150 mg/6cc (part # 186002493) with 20 mg loose carbon added.

8. Phenomenex Strata PFAS WAX/GCB, 500mg/50mg/6cc (part # CS0-9208) or equivalent. This cartridge incorporates graphitized carbon.

9. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product # 54258-U.

10. SPE Tube Adapter - Millipore-Sigma; Product # 57020-U.

11. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.

12. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.

13. Centrifuge – "Thermo Sorvall ST4" with TX-750 rotor or equivalent, capable of a minimum rotational force of 3800 rcf.

14. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent.

15. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. FisherScientific cat # 14-287-150, or equivalent.

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

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

18. Pipettes – Disposable transfer. Fisher Scientific, Cat. No. 13-711-7M

19. Vortex mixer, variable speed, Fisher Scientific or equivalent.

20. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control. Organomation, Inc. Cat. #11250-NT, or equivalent.

21. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTPF08831.

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

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

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

25. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent

26. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125

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

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

29. Syringe filter - Acrodisc, Syringe Filter, GHP,13 mm, 0.2 µm, Aqueous, 100/pkg, Part # WAT097962.

30. Silanized glass wool (Sigma-Alrich, Cat #20411 or equivalent)

31. Syringe filter - Acrodisc, Syringe Filter, 25 mm, 0.2 µm, 50/pkg, Fisher part # 50786068.

32. Variable speed mixing table (Fisherbrand™ Nutating mixer or equivalent)

33. Evaporation/concentrator tubes: 60 mL clear glass vial, 30x125 mm, without caps (Wheaton Cat # W226060 or equivalent).

34. Wooden Tongue Depressors - Fisher; Cat. # 11-700-555, or equivalent.

35. Wheaton Bottle, 15ml, Narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Cat. No. 209044SP, or equivalent

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

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

2. Promochrom automated extraction unit, model SPE-03 or equivalent

a. Promochrom filters, catalog # F-HC-30

3. HPLC columns

   a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-YO or equivalent

   b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

1. Methanol (MeOH) – J.T. Baker Ultra Resi-Analyzed, Cat. No. UN1230 or equivalent
2. Acetonitrile (ACN) – J.T. Baker Ultra Resi-Analyzed, Cat. No. UN1648 or equivalent
3. Water – Fisher Scientific, Optima, Cat. No. W6-4 or equivalent
4. Ammonium acetate – Fisher Scientific, Cat. No. A637-500 or equivalent
5. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
6. Methanolic ammonium hydroxide (0.3%) – add ammonium hydroxide (10 mL, 30%) to methanol (990 mL), store at room temperature, replace after 1 month
7. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
8. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
9. Acetic Acid – ACS grade or equivalent, store at room temperature
10. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
11. Formic acid -(greater than 96% purity or equivalent), store at room temperature.

    a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years

    b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years

c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years

d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years

e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years

12. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.

13. Solids reference matrix – Ottawa or reagent-grade sand

14. 20 mM ammonium acetate solution in 95:5(v/v) Milli-Q water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL Milli-Q water and mix well to dissolve the ammonium acetate. Add 100 mL acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

15. 20 mM ammonium acetate solution in 90:10 acetonitrile/Milli-Q water – Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 200 mL of Milli-Q water and mix well to dissolve the Ammonium Acetate. Add 1800 mL of acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

B. Standards:

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

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

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

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

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

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

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS

extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

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

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

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

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 04*.
If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

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

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

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

Attachment 05 - Native PFAS Intermediate A
Attachment 06 - Native PFAS Intermediate B
Attachment 07 - Native PFAS Intermediate M
Attachment 08 - Extraction Standard Mix
Attachment 09 - Internal Standard Mix
Attachment 10 - Initial Calibration Standards Preparation
Attachment 11 - Initial Calibration Standards Concentrations
Attachment 12 - Bile Salts Stock Solutions
Attachment 13 - Bile Salts Working Solution A
Attachment 14 - Bile Salts Working Solution B
Attachment 15 - PFAS Linear/Branched Mix A
Attachment 16 - PFAS Linear/Branched Mix B
Attachment 17 - Linear/Branched Bile Salts Solution
Attachment 18 - ICV Working Standard

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit(quadratic). In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.

2. Analyze a Cal4 level standard that contains Bile Salts retention time marker and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

   Example Initial Calibration Sequence:
   1. Instrument Blank
   2. Instrument Blank
   3. Instrument Blank
   4. CAL 1
   5. CAL 2
   6. CAL 3
   7. CAL 4
   8. CAL 5
   9. CAL 6
   10. CAL 7
   11. ICB (Instrument Blank)
   12. ICV
   13. MDL
   14. WDM (Linear Branched/Bile Salts standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 02* for additional information about compound relationships.

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	

PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	

11Cl-PF3OUdS	

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

4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic Acid (TCDCA), and Tauroursodeoxycholic Acid (TUDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

5. Fit the curve

   a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

   b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for the compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.

   c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

   d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

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

6. Initial Calibration Verification (ICV)

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

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

2. Acceptance criteria

   a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV

recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

A. Sample Preparation

NOTE: Prior to weighing out samples, thoroughly mix each sample using a wooden tongue depressor or stainless steel spoon to ensure a homogeneous sample matrix. Stir from the bottom to the top in a circular motion along the sides of the jar, breaking particles to less than 1 mm by pressing against the side of the container. Remove rocks, invertebrates, and foreign objects. Vegetation can either be removed or cut into smaller pieces based on project requirements.

1. On a calibrated, top-loading balance, accurately weigh 1.25 ± 0.10g (dry weight; note: maximum 10g sample aliquot) (0.5 g for biosolids(dry weight; note: maximum 1g sample aliquot)) of solid sample into a tared, labeled 50-mL centrifuge tube using a disposable polypropylene spatula. Record sample weight in the prep entry system.

2. For each batch - maximum 20 samples - include the following quality control samples:
   a. Method Blank: Weigh 1.25 ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 g for biosolids) of reagent water
   b. LCS: Fortify 1.25g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 mL for biosolids) reagent water and spiked with 200 µL of Native 1633 Mid-Level Spike Solution (PFAS Native M).
   c. LLCS: Fortify 1.25g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 mL for biosolids) reagent water and spiked with 200 µL of Native 1633 Low-Level Spike Solution (PFAS Native B).
   d. Matrix Spike/Matrix Spike Duplicate (MS/MSD): Fortify 1.25g ± 0.10 g(dry weight; note: maximum 10g sample aliquot) (0.5 g for biosolids(dry weight; note: maximum 1g sample aliquot)) of sample as specified in sample preparation log with 200 µL of Native 1633 Mid-Level Spike Solution (PFAS Native M).

3. Add 25 µl working labeled extraction standard spike solution (PFAS EIS Mix) to each sample/QC tube.

4. Cap and vortex for approximately 30 seconds.

5. Allow samples/QC to equilibrate for at least 30 minutes.

6. Add 10 mL of 0.3% methanolic ammonium hydroxide to each centrifuge tube.

7. Cap and vortex

8. Shake for 30 minutes on a variable speed mixing table

9. Centrifuge @ 3000 rcf for 10 minutes and transfer supernatant to a clean 50 mL polypropylene centrifuge tube.

10. Add 15 mL of 0.3% methanolic ammonium hydroxide to the remaining solid sample in each centrifuge tube. Cap and vortex.

11. Shake for 30 minutes on a variable speed mixing table

12. Centrifuge @ 3000 rcf for 10 minutes and decant the supernatant from the second extraction into the centrifuge tube with the supernatant from the first extraction.

13. Add another 5 mL of 0.3% methanolic ammonium hydroxide to the remaining sample in each centrifuge tube.

14. Shake by hand to disperse.

15. Immediately decant the supernatant from the third extraction into the centrifuge tube with the supernatant from the first and second extraction.

16. Centrifuge for 10 minutes @ 3000 rcf.

17. Decant the extract from each tube into a clean, labeled, 125 ml HDPE bottle. Bring the 125-mL HDPE container up to 125 mL with Milli-Q water.

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

19. Invert container to thoroughly mix.

B. Solid Phase Extraction (SPE) - Manual Sample Extraction

1. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

2. Condition each SPE cartridge with the following reagents in the following order:

    a. 15 mL 1% methanolic ammonium hydroxide

    b. 5 mL 0.3M formic acid

    c. Discard conditioning eluent(s)

3. Label each sample bottle, cap and reservoir with the same number to insure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

4. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges. Allow full volume to pass through each cartridge by gravity, if possible. Apply light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.

5. After full volume has passed through the cartridges, dry the cartridges with vacuum - no more than 15" of Hg - for approximately five minutes. After five minutes, visually inspect the cartridge to determine if the sorbent is dry. This done by comparing the cartridge to a visual standard (an unused SPE cartridge). If the sorbent is not dry, continue to check at one minute intervals until the cartridge is dry.

6. Discard the waste and rinse the waste reservoir with DI water. Wipe each needle with a Kim-wipe/methanol.

7. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5ml of **1:1 0.1M formic acid/methanol** and pass the rinses through the cartridge using vacuum. Dry the cartridge by pulling air through for 15 seconds. Discard the rinse solution.

8. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge.

9. Rinse the inside of the evaporation/concentrator tube using 5mL of 1% methanolic ammonium hydroxide.

10. Using a glass pipette, transfer the rinse to the SPE reservoirs, washing the walls of the reservoirs.

11. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

12. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

13. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix thoroughly.

14. Add 25 uL of Intenal Standard Spike Solution (PFAS NIS Mix) to each sample extract.

15. Bring each sample extract to final volume 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

16. Cap and vortex to mix. Using a syringe, push ~ 3mL of MeOH through each filter and allow to dry

17. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 6 mL polypropylene syringe. Take the plunger out and carefully decant ~1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter ~1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

18. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for dilution or reinjection if needed.

C. Solid Phase Extraction (SPE) – Automated Sample Extraction using Promochrom

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

1. Verify pH of all samples/QC(needs to be between pH 4.0 ± 0.5). Modify with 50% Formic acid or 30% NH4OH if necessary. (Record the lot number of the acid/base used). **NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.**

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

3. Check to make sure all reagent bottles are filled and that reagents are not expired.

4. Select "Clean sys-004" from the drop down menu to start the cleaning cycle prior to SPE. Make sure the "Clean" cartridges and bottles are attached for this cycle.

5. Remove "Clean" cartridges and attach the labeled cartridges containing graphitized carbon that will be used when running the samples. Disconnect the "Clean" bottles. Attach the sample bottle by twisting the bottle and not the cap. Place sample bottle upside down in corresponding numbered location on shaker. Poke two holes into the lip of each bottle. Load labeled centrifuge tubes in the moving tray beneath the cartridges.

6. Select "EPA 1633-004" from the drop down menu and press the green check mark to start the procedure. NOTE: N2 drydown should be 15 minutes long.

7. Once the cycle is complete, remove centrifuge tubes and discard the used cartridges and bottles. Reattach "Clean" bottles and cartridges, select "clean sys-004" from menu, select "start". This must be done between each batch and at the beginning and the end of the day.

8. Add 25ul of Acetic acid to sample extracts and vortex to mix thoroughly before capping tubes.

9. Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to each sample extract.

10. Bring each sample extract to final volume 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

11. Cap and vortex to mix. Using a syringe, push ~ 3mL of MeOH through each filter and allow to dry.

12. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 3 mL polypropylene syringe. Take the plunger out and carefully decant approximately 1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter approximately 1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

13. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for reinjection if needed.

D. LC/MS/MS Analysis

1. Mass Calibration and Tuning

   a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

   b. When masses fall outside of the ±0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Example acquisition method: See *Attachment 03*. Mass Transitions: See *Attachment 01*.

4. Instrument Sensitivity Check (ISC) and Instrument Blanks

   a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within ±30% of their true value.The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
   b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salts RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

   Example Sample Sequence:
   1. Instrument blank
   2. Instrument blank
   3. Instrument blank
   4. Instrument Sensitivity Check (CCV0 _CAL1)
   5. CCV 2_CAL4
   6. Linear Branched/ Bile Salt marker (WDM)
   7. Instrument Blank (ICB)
   8. Method Blank (MB)
   9. Low Level LCS (LLCS)
   10. LCS
   11. Sample (10 or less)
   12. CCV 3_CAL4
   13. Instrument Blank

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

    a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.

    b. If LLCS and LCS recoveries are acceptable, proceed to sample quantitation.

    c. If the LCS and LLCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS/LLCS must be reextracted.

    d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Calculate the relative percent difference(RPD) between the sample and the matrix duplicate(DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data

11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), revial a fresh aliquot and reanalyze. If the extraction standard recovery(ies) are still outside the QC limit(s), reanalyze the sample at a dilution. If the extraction standard recovery(ies) are still outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) are still outside the QC limit(s), narrate with an NCM and report the data.

12. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

13. Compare the retention times of all of the analytes, surrogates, and internals standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than

    a. 0.4 minutes for isotopically-labeled compounds

    b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound

    c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

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

14. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of 13C4-PFBA, 13C5-PFPeA, 13C4-PFHpA, 13C8-PFOA, 13C9-PFNA, 13C6-PFDA, 13C7-PFUnA, 13C2-PFDA,13C2-PFDoDA 13C2-PFTeDA, 13C8-PFOSA, D3-NMePFOSA, D5-NEtFOSAA, D3-NMeFOSAA, D5-NEtPFOSA, D7-NMePFOSAE, D9-NEtPFOSAE, 13C3-PFBA, 13C4-PFOA, 13C5-PFNA, 13C2-PFOA, 18O2-PFHxS, PFBA, PFECA F(PFMPA), PFECA A(PFMBA), NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required in prepped samples. Ion ratios must be in control in calibration solutions. If they are outside of limits, stop the analysis and correct the issues.

15. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.

16. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of Internal Standard Spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring greater than a 10x dilution should be reextracted using a reduced aliquot.

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

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

    On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

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

4. Sample Concentration

Sample concentration (ng/g) = (On-column concentration x Final Sample Volume x DF) ÷ Initial Sample Volume

5. Ion Ratio

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

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

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are method defined.

QC parameter	Lower acceptance limit	High acceptance limit
Extracted Internal standard (EIS)	20%	150%
Non-extracted Internal Standard (NIS)	>30% of the average NIS from the initial calibration	200%
Analyte recoveries LCS/LLCS/MS/MSD	40%	150%
Sample/Duplicate	</= 30%	NA

Note: lower acceptance limit for EIS cannot not be <20%, lower acceptance limit for analyte recovery cannot be <40%.

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

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

Quality Assurance/Quality Control

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

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

Attachment:
Attachment 01 - Mass Transitions (.doc)
Attachment 02 - Standard Relationships (.docx)
Attachment 03 - Acquisition Parameters (.pdf)
Attachment 04 - Example Certificate of Analysis (.pdf)
Attachment 05 - Native PFAS Intermediate A (.pdf)
Attachment 06 - Native PFAS Intermediate B (.pdf)
Attachment 07 - Native PFAS Intermediate M (.pdf)
Attachment 08 - Extraction Standard Mix (.pdf)
Attachment 09 - Internal Standard Mix (.pdf)
Attachment 10 - Initial Calibration Standards Preparation (.pdf)
Attachment 11 - Initial Calibration Standard Concentrations (.pdf)
Attachment 12 - Bile Salt Stock Solutions (.pdf)
Attachment 13 - Bile Salts Working Solution A (.pdf)
Attachment 14 - Bile Salts Working Solution B (.pdf)
Attachment 15 - PFAS Linear Branched Mix A (.pdf)
Attachment 16 - PFAS Linear Branched Mix B (.pdf)
Attachment 17 - Linear Branched Bile Salts Solution (.pdf)
Attachment 18 - ICV Working Standard (.pdf)

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Attachment: Attachment 12 - Bile Salt Stock Solutions (pdf)
Attachment: Attachment 13 - Bile Salts Working Solution A (pdf)
Attachment: Attachment 14 - Bile Salts Working Solution B (pdf)
Attachment: Attachment 15 - PFAS Linear Branched Mix A (pdf)
Attachment: Attachment 16 - PFAS Linear Branched Mix B (pdf)
Attachment: Attachment 17 - Linear Branched Bile Salts Solution (pdf)
Attachment: Attachment 18 - ICV Working Standard (pdf)

End of document

Version history

Version	Approval	Revision information	
0	25.SEP.2023		
1	03.NOV.2023		
2	20.NOV.2023		

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

Attachment 1

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

Attachment 1

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

Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

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

Extraction Standards

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

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

Native PFAS Compounds

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

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

Acquisition Method

1633

Mass Spec 12.500 min
Period 12.500 mi
-MRM
Integrated Valve
LC System
Equilibrate
Injection

Mass Spectrometer Method Properties

Period 1:

Scans in Period:	1250
Min. Dwell Time:	3 ms
Max. Dwell Time:	250 ms
Relative Start Time:	0.00 msec
Scheduled Ionization:	Off
Experiments in Period:	1
Use target Cycle Time:	No
Target Cycle Time:	N/A

Period 1 Experiment 1:

Scan Type:	MRM (MRM)
Scheduled MRM:	Yes
Polarity:	Negative
Scan Mode:	N/A
Ion Source:	Turbo Spray
sMRM Q1Q3 Resolution:	No
MRM detection window:	60 sec
Target Scan Time:	0.6000 sec
Resolution Q1:	Unit
Resolution Q3:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Step Size:	0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
212.800	168.900	2.71	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	84.900	2.94	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	2.98	DP	-50.00	-50.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	3.21	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	79.900	3.67	DP	-90.00	-90.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.100	3.32	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	134.900	3.84	DP	-70.00	-70.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	3.52	DP	-30.00	-30.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.41	DP	-110.00	-110.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.55	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

349.100	79.900	4.00	DP		-90.00	-90.00	PFPeS
				CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.68	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.85	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.30	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	3.96	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	3.98	DP	-90.00	-90.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.68	DP	-110.00	-110.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.12	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	5.22	DP	-90.00	-90.00	PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.43	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.40	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.61	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.72	DP	-100.00	-100.00	PFNS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.85	DP	-60.00	-60.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.58	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	6.14	DP	-110.00	-110.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.79	DP	-80.00	-80.00	NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	6.16	DP	-90.00	-90.00	PFDS
			CE	-130.00	-130.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.39	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	5.04	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.49	DP	-90.00	-90.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.87	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.94	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	7.08	DP	-90.00	-90.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.94	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	7.24	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.36	DP	-90.00	-90.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	619.000	6.30	DP	-60.00	-60.00	PFTrDA
			CE	-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.71	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.66	DP	-110.00	-110.00	PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.52	DP	-10.00	-10.00	PFECA B_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.41	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.55	DP	-40.00	-40.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.00	DP	-110.00	-110.00	PFPeS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.68	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.85	DP	-50.00	-50.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.30	DP	-90.00	-90.00	PFHxS_2

			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	3.98	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.68	DP	-100.00	-100.00	PFHpS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.12	DP	-50.00	-50.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	5.21	DP	-110.00	-110.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	4.43	DP	-50.00	-50.00	PFNA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	98.800	5.72	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	219.000	4.85	DP	-60.00	-60.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	80.800	4.58	DP	-100.00	-100.00	8:2 FTS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	483.000	4.79	DP	-90.00	-90.00	NMeFOSAA_2
			CE	-24.00	-24.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	98.800	6.16	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	269.100	5.39	DP	-70.00	-70.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	526.000	5.04	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	319.000	5.87	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

663.000	168.900	6.30	DP		-60.00	-60.00	PFTrDA_2
			CE		-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.71	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.21	DP	-40.00	-40.00	PFPeA_2
			CE	-55.00	-55.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	6.13	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	7.08	DP	-110.00	-110.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.35	DP	-90.00	-90.00	NEtPFOSA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	3.95	DP	-40.00	-40.00	DONA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.60	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.48	DP	-90.00	-90.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	2.98	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.40	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.83	DP	-70.00	-70.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.94	DP	-90.00	-90.00	PFDoS_2
			CE	-140.00	-140.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.71	DP	-40.00	-40.00	13C3-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.71	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.21	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.66	DP	-90.00	-90.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.41	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.54	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.54	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.68	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.85	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.30	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.30	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	3.97	DP	-90.00	-90.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.12	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.12	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	5.21	DP	-90.00	-90.00	13C4-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	79.900	5.21	DP	-90.00	-90.00	13C8-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.42	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	
519.100	474.100	4.85	DP	-60.00	-60.00	13C6-PFDA
			CE	-18.00	-18.00	
515.100	470.100	4.85	DP	-60.00	-60.00	13C2-PFDA
			CE	-18.00	-18.00	
529.100	80.900	4.58	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	
506.100	77.800	6.13	DP	-110.00	-110.00	13C8-PFOSA
			CE	-80.00	-80.00	
573.200	419.000	4.78	DP	-90.00	-90.00	d3-NMeFOSAA
			CE	-27.00	-27.00	
570.000	525.100	5.38	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	
589.200	419.000	5.02	DP	-90.00	-90.00	d5-NEtFOSAA

			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.86	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.92	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	7.07	DP	-90.00	-90.00	d3-NMePFOSA
			CE	-37.00	-37.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	7.21	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.34	DP	-90.00	-90.00	d5-NEtPFOSA
			CE	-38.00	-38.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.70	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.54	DP	-40.00	-40.00	13C5-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.66	DP	-110.00	-110.00	13C3-PFBS_2
			CE	-35.00	-35.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.29	DP	-90.00	-90.00	13C3-PFHxS_2
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	5.20	DP	-110.00	-110.00	13C8-PFOS_2
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.41	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	3.97	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.58	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.68	DP	-75.00	-75.00	13C3-HFPODA_2
			CE	-10.00	-10.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

315.100	119.400	3.54	DP	-40.00	-40.00	13C2-PFHxA_2	
			CE	-31.00	-31.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	5.21	DP	-110.00	-110.00	13C4-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.78	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.70	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.39	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
CAD:	10.00
EP	-10.00
CXP	-14.00

Acquisition Method

```
1633

Mass Spec  12.500 min
   Period  12.500 min
      -MRM
Integrated Valve
LC System
   Equilibrate
   Injection
```

Acquisition Information:

Acquisition Method:	1633.dam
Created:	Wednesday May 24 2023 14:48:39 PM
Last Modified:	Wednesday June 14 2023 11:54:50 AM
Comment:	
Synchronization Mode:	LC Sync
Auto-Equilibration:	Off
Acquisition Duration:	12min30sec
Number Of Scans:	1250
Periods In File:	1
Acquisition Module:	Acquisition Method
Software version	Analyst 1.7.3

Period 1:

Scans in Period:	1250
Min. Dwell Time:	3 ms
Max. Dwell Time:	250 ms
Relative Start Time:	0.00 msec
Scheduled Ionization:	Off
Experiments in Period:	1
Use target Cycle Time:	No
Target Cycle Time:	N/A

Period 1 Experiment 1:

Scan Type:	MRM (MRM)
Scheduled MRM:	Yes
Polarity:	Negative
Scan Mode:	N/A
Ion Source:	Turbo Spray
sMRM Q1Q3 Resolution:	No
MRM detection window:	60 sec
Target Scan Time:	0.6000 sec
Resolution Q1:	Unit
Resolution Q3:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Step Size:	0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
212.800	168.900	2.71	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	84.900	2.94	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	2.98	DP	-50.00	-50.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	3.21	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	79.900	3.67	DP	-90.00	-90.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.100	3.32	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	134.900	3.84	DP	-70.00	-70.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	3.52	DP	-30.00	-30.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.41	DP	-110.00	-110.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.55	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	79.900	4.00	DP	-90.00	-90.00	PFPeS
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.68	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.85	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.30	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	3.96	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	3.98	DP	-90.00	-90.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.68	DP	-110.00	-110.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.12	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	5.22	DP	-90.00	-90.00	PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.43	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.40	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.61	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.72	DP	-100.00	-100.00	PFNS

			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.85	DP	-60.00	-60.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.58	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	6.14	DP	-110.00	-110.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.79	DP	-80.00	-80.00	NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	6.16	DP	-90.00	-90.00	PFDS
			CE	-130.00	-130.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.39	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	5.04	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.49	DP	-90.00	-90.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.87	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.94	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	7.08	DP	-90.00	-90.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.94	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	7.24	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.36	DP	-90.00	-90.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

663.000	619.000	6.30	DP		-60.00	-60.00	PFTrDA
			CE		-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.71	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.66	DP	-110.00	-110.00	PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.52	DP	-10.00	-10.00	PFECA B_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.41	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.55	DP	-40.00	-40.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.00	DP	-110.00	-110.00	PFPeS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.68	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.85	DP	-50.00	-50.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.30	DP	-90.00	-90.00	PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	3.98	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.68	DP	-100.00	-100.00	PFHpS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.12	DP	-50.00	-50.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	5.21	DP	-110.00	-110.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	4.43	DP	-50.00	-50.00	PFNA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	98.800	5.72	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	219.000	4.85	DP	-60.00	-60.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	80.800	4.58	DP	-100.00	-100.00	8:2 FTS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	483.000	4.79	DP	-90.00	-90.00	NMeFOSAA_2
			CE	-24.00	-24.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	98.800	6.16	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	269.100	5.39	DP	-70.00	-70.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	526.000	5.04	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	319.000	5.87	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	168.900	6.30	DP	-60.00	-60.00	PFTrDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.71	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.21	DP	-40.00	-40.00	PFPeA_2
			CE	-55.00	-55.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	6.13	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	7.08	DP	-110.00	-110.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.35	DP	-90.00	-90.00	NEtPFOSA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	3.95	DP	-40.00	-40.00	DONA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.60	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.48	DP	-90.00	-90.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	2.98	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.40	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.83	DP	-70.00	-70.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.94	DP	-90.00	-90.00	PFDoS_2
			CE	-140.00	-140.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.71	DP	-40.00	-40.00	13C3-PFBA

			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.71	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.21	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.66	DP	-90.00	-90.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.41	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.54	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.54	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.68	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.85	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.30	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.30	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	3.97	DP	-90.00	-90.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.12	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.12	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	5.21	DP	-90.00	-90.00	13C4-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

507.100	79.900	5.21	DP		-90.00	-90.00	13C8-PFOS
				CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.42	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
519.100	474.100	4.85	DP	-60.00	-60.00	13C6-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.100	470.100	4.85	DP	-60.00	-60.00	13C2-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	80.900	4.58	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
506.100	77.800	6.13	DP	-110.00	-110.00	13C8-PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
573.200	419.000	4.78	DP	-90.00	-90.00	d3-NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.100	5.38	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.200	419.000	5.02	DP	-90.00	-90.00	d5-NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.86	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.92	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	7.07	DP	-90.00	-90.00	d3-NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	7.21	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.34	DP	-90.00	-90.00	d5-NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.70	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.54	DP	-40.00	-40.00	13C5-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.66	DP	-110.00	-110.00	13C3-PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.29	DP	-90.00	-90.00	13C3-PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	5.20	DP	-110.00	-110.00	13C8-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.41	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	3.97	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.58	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.68	DP	-75.00	-75.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	119.400	3.54	DP	-40.00	-40.00	13C2-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	5.21	DP	-110.00	-110.00	13C4-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.78	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.70	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.39	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
CAD:	10.00
EP	-10.00

CXF -14.00

Integrated Valve Method Properties
Valve Diverter

	Total Time (min)	Position
1	1.0	B
2	8.0	A

LC Method Properties

LC system Equlibration time = 0.00 min
LC system Injection Volume = 10.00 ul

Pump

Model:	A: LC-40D XF B: LC-40D XR
Pump mode:	Binary gradient
Stop time:	12.50 min
<Gradient start adjustment>	
Mode:	None
<B.GE1>	
<General>	
Flow/Pressure:	Flow
Flow:	0.5000 mL/min
Time to reach the flow:	0.00 min
B.Conc:	5.0 %
B.Curve:	0
Pressure limits Minimum:	0 psi
Pressure limits Maximum:	6500 psi

<Gradient>

Time min	Flow mL/min	B.Conc %	B.Curve
0.00	0.5000	5.0	0
0.20	0.5000	5.0	0
3.00	0.5000	60.0	0
3.80	0.5000	60.0	0
6.70	0.5000	98.0	0
8.50	0.5000	98.0	0
8.70	0.6500	5.0	0
12.50	0.5000	5.0	0

<PumpA>	
<Mobile phase switching valve>	
Selected mobile phase port:	A
<Mobile phase settings>	
Set compressibility:	No

Port	Mobile phase name
A	Mobile phase A

<Advanced>	
LPGE cycle:	Standard
<PumpB>	

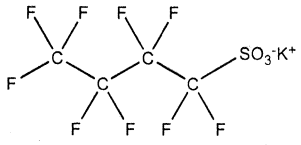
```
      <Mobile phase switching valve>
         Selected mobile phase port:                        A
      <Mobile phase settings>
         Set compressibility:                              No
      Port  Mobile phase name
      ----  -----------------
         A     Mobile phase A
      <Advanced>
         LPGE cycle:                                 Standard
<Autopurge settings>
   Execute autopurge before analysis:                      No
   <Autosampler purge settings>
      Execute sampler purge:                               No
   <Init conc-replacement>
      Use Init conc-replacement:                           No
   <Warm up>
      Wait time:                                       0 min
      B.GE1 flow:                                0.0000 mL/min
   <Execute after autopurge>
      Turn oven on after autopurge:                        No
      Turn pump on after autopurge:                        No
<Time program>
Autosampler
===========
Model:                                             SIL-40C XR
<General>
   Use Autosampler:                                       Yes
<Sample plate settings>
   Specify plate:                                       No
   Specify needle stroke:                               No
<Injection settings>
   Sampling speed:                                  1.0 µL/s
   Use cooler temperature:                               Yes
   Cooler temperature:                                15 °C
   Use air gap volume:                                  No
   Use low dispersion injection:                        No
<Acquisition cycle time optimization>
   Pretreatment start timing:                           Off
<Cut off loop settings>
   Cut off loop:                                        No
<Rinse settings>
   Rinse type:                                 External only
   Rinse mode:                  Before and after aspiration
   Rinse dip time:                                    0 s
   Rinse method:                              Rinse port only
   Rinse time:                                      2.0 s
   Rinsing speed:                                  35 µL/s
   Rinsing volume:                                500 µL
   Measuring line purge volume:                   100 µL
<Details>
   Wait for temperature equilibration before run (sample cooler):
                                                        No
```

<Pretreatment>
   Mode: Standard
<Time program>
Oven
====
Model: A: CTO-40C
<Column Oven A>
   <General>
      Use Column Oven: Yes
      Oven temperature: 40 °C
      Temperature limit(Maximum): 90 °C
      Wait for temperature equilibration before run: Off
   <Advanced>
      Fan speed: Auto
      Cooler mode: Auto
<Time program>
System Controller
==========
Model: CBM-40
<External output settings>
   Event 1: No
   Event 2: No
<Shutdown settings>
   Power off after shutdown: No
<Time program>
Time Command Value
 min
---- -------- ------
0.01   Event   1

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

PRODUCT CODE:	L-PFBS	**LOT NUMBER:**	LPFBS1022
COMPOUND:	Potassium perfluoro-1-butanesulfonate		
STRUCTURE:		**CAS #:**	29420-49-3



MOLECULAR FORMULA:	$C_4F_9SO_3K$	**MOLECULAR WEIGHT:**	338.19
CONCENTRATION:	50.0 ± 2.5 µg/mL (K salt)	**SOLVENT(S):**	Methanol
	44.4 ± 2.2 µg/mL (PFBS acid)		
	44.2 ± 2.2 µg/mL (PFBS anion)		
CHEMICAL PURITY:	>98%		
LAST TESTED: (mm/dd/yyyy)	10/04/2022		
EXPIRY DATE: (mm/dd/yyyy)	10/04/2027		
RECOMMENDED STORAGE:	Store ampoule in a cool, dark place		

DOCUMENTATION/ DATA ATTACHED:

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

ADDITIONAL INFORMATION:

- See page 2 for further details.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Certified By: _____ **Date:** 10/14/2022 (mm/dd/yyyy)
B.G. Chittim, General Manager

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

Attachment "" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 Page 67 of 84 "
Printed by Dorothy Love, d. Mon 20 Nov 2023 15:26 EST

INTENDED USE:

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

HANDLING:

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

SYNTHESIS / CHARACTERIZATION:

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

HOMOGENEITY:

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

UNCERTAINTY:

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

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters $x_1, x_2, \ldots x_n$ on which it depends is:

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

QUALITY MANAGEMENT:

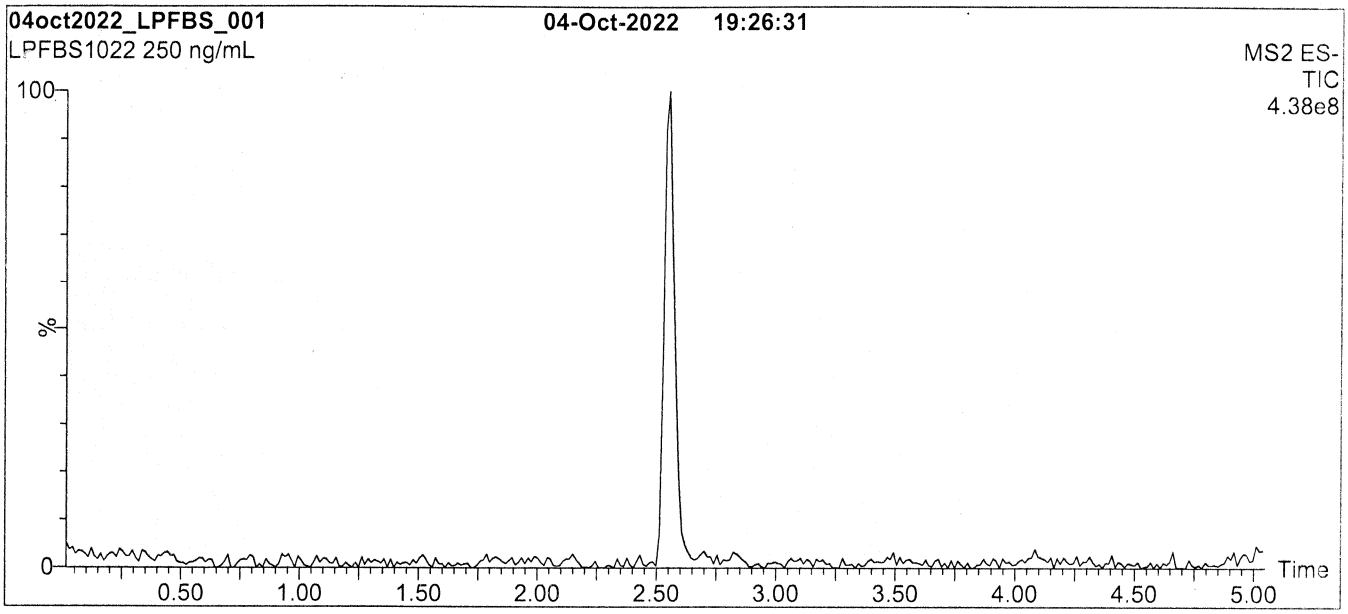
This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A1226), and ISO 17034 by ANSI National Accreditation Board (ANAB; AR-1523).

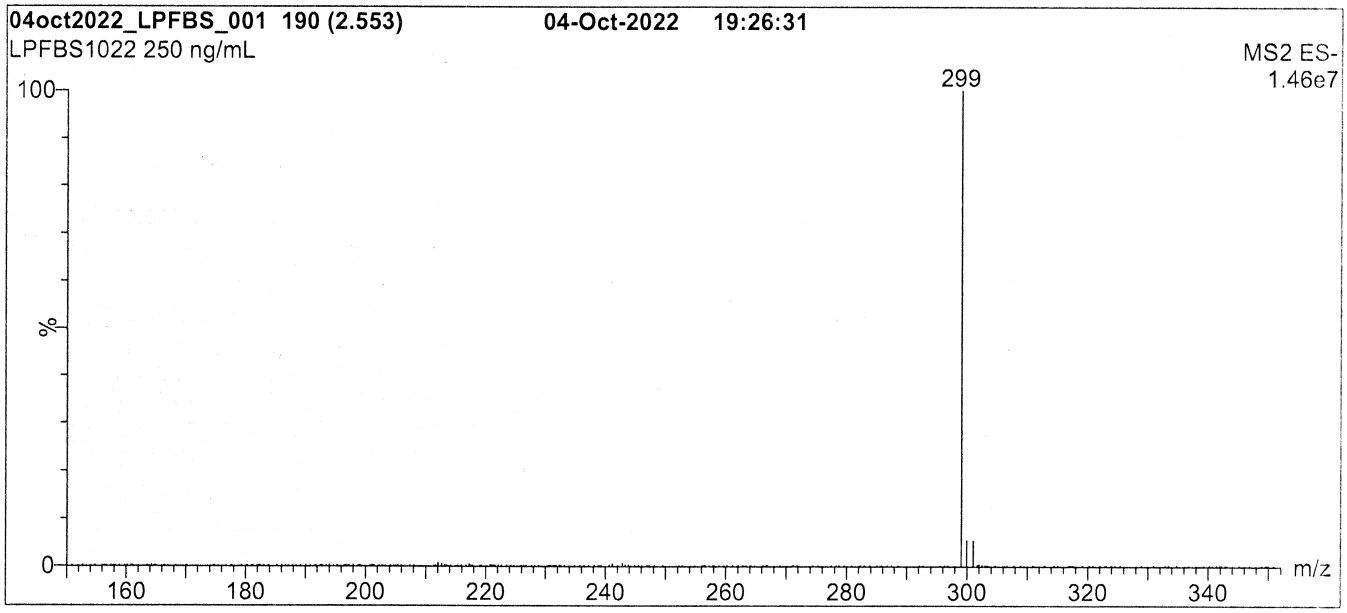
CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

Figure 1: **L-PFBS; LC/MS Data (Full Scan and Mass Spectrum)**





Conditions for Figure 1:

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

Chromatographic Conditions:

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

Mobile phase: Gradient
Start: 60% H_2O / 40% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions over 0.75 min.
Time: 12 min

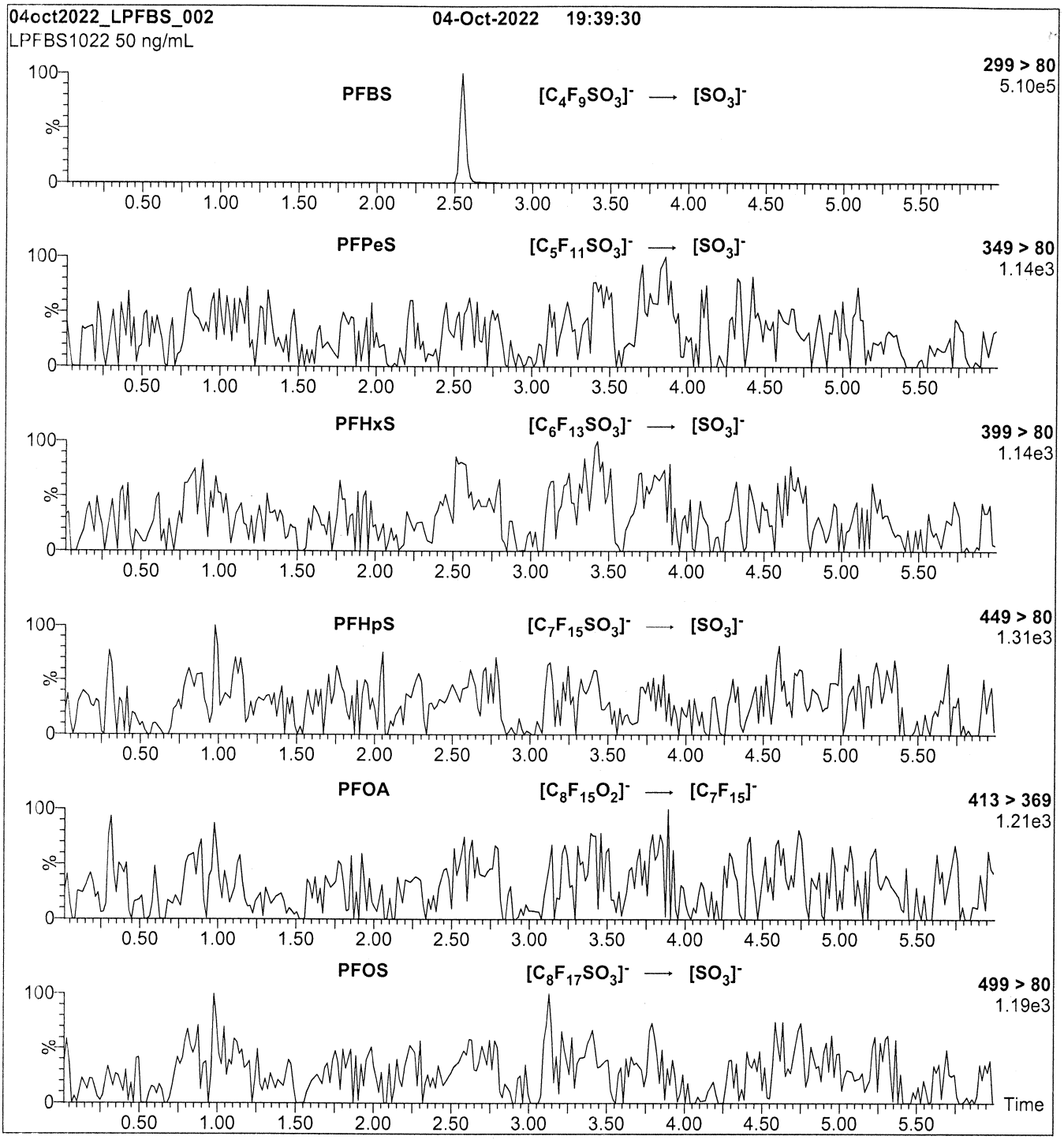
Flow: 300 µL/min

MS Parameters:

Experiment: Full Scan (150 - 850 amu)

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = 10.00
Desolvation Temperature (°C) = 500
Desolvation Gas Flow (L/hr) = 1000

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



Conditions for Figure 2:

Injection:	On-column (L-PFBS)
Mobile phase:	Same as Figure 1
Flow:	300 µL/min

MS Parameters:

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

Attachment 5

Native PFAS Intermediate A								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Native PFAS Intermediate A (ng/ml)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.1	10	500
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.05		250
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.025		125
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.025		125
Wellington	PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.025		125
Wellington	PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.025		125
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.025		125
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000	0.025		125
Wellington	PFDoA	Perfluorododecanoic acid	307-55-1	PFDoA	50000	0.025		125
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.025		125
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.025		125
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.025		111
Wellington	L-PFPeS	Perfluoropentansulfonic acid	2706-91-4	PFPeS	46900	0.025		117
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.025		114
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.025		119
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.025		117
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.025		120
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.025		121
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400	0.025		121
Wellington	4;2 FTS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	46700	0.1		467
Wellington	6;2 FTS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	47400	0.1		474
Wellington	8;2 FTS	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	47900	0.1		479
Wellington	FOSA-I	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.025		125
Wellington	N-MeFOSA-M	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	50000	0.025		125
Wellington	N-EtFOSA-M	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	50000	0.025		125
Wellington	br-NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.025		125
Wellington	br-NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.025		125
Wellington	N-MeFOSE-M	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	50000	0.25		1250
Wellington	N-EtFOSE-M	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	50000	0.25		1250
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	50000	0.1		500
Wellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	47100	0.1		471
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.05		250
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50000	0.05		250
Wellington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	50000	0.05		250
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.1		466
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.1		471
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.05		223
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000	0.625		3125
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000	0.625		3125

Attachment 6

Native PFAS Intermediate B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. Native PFAS Intermediate B (ng/ml)**
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.1	10	5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			2.5
	Perfluorohexanoic acid	307-24-4	PFHxA	125			1.25
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			1.25
	Perfluorooctanoic acid	335-67-1	PFOA	125			1.25
	Perfluorononanoic acid	375-95-1	PFNA	125			1.25
	Perfluorodecanoic acid	335-76-2	PFDA	125			1.25
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			1.25
	Perfluorododecanoic acid	307-55-1	PFDoA	125			1.25
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			1.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			1.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			1.11
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117.25			1.17
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75			1.14
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			1.19
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5			1.17
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			1.20
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5			1.21
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			1.21
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			4.67
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			4.74
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			4.79
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			1.25
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			1.25
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			1.25
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			1.25
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			12.5
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			12.5
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			5
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			4.71
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			2.5
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			2.5
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			2.5
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466			4.66
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471			4.71
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			2.23
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			6.25
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			31.3
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			31.3

Attachment 7

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

Attachment 8

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

Attachment 9

Internal Standard Mix								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Concentration (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. Internal Standard Mix (ng/ml)**
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	50000	50	10	250
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	50000	25		125
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	50000	12.5		62.5
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	47800	25		120
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	50000	12.5		62.5
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	50000	25		125
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	47300	25		118

Attachment 10

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

Attachment 11

1633 Initial Calibration Standard Concentrations							
	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Compound Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
PFpeA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFHxA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFHpA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFOA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFNA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFUnA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDoA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTrDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTeDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFBS	0.044	0.111	0.276	0.553	1.11	2.76	13.8
PFPeS	0.047	0.117	0.293	0.586	1.17	2.93	14.7
PFHxS	0.046	0.114	0.284	0.569	1.14	2.84	14.2
PFHpS	0.048	0.119	0.298	0.595	1.19	2.98	14.9
PFOS	0.047	0.117	0.291	0.583	1.17	2.91	14.6
PFNS	0.048	0.120	0.300	0.600	1.20	3.00	15.0
PFDS	0.048	0.121	0.301	0.603	1.21	3.01	15.1
PFDoS	0.048	0.121	0.303	0.605	1.21	3.03	15.1
4:2 FTS	0.187	0.467	1.17	2.34	4.67	11.7	58.4
6:2 FTS	0.190	0.474	1.19	2.37	4.74	11.9	59.3
8:2 FTS	0.192	0.479	1.20	2.40	4.79	12.0	59.9
PFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
NEtFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
HFPO-DA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
DONA	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFMPA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFMBA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
NFDHA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
9Cl-PF3ONS	0.186	0.466	1.17	2.33	4.66	11.7	58.3
11Cl-PF3OUdS	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFEESA	0.089	0.223	0.56	1.11	2.23	5.56	27.8
3:3 FTCA	0.250	0.625	1.56	3.13	6.25	15.6	78.1
5:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
7:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
13C4-PFBA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
13C5-PFPeA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C5-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C4-PFHpA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C8-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C9-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C6-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C7-PFUnA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFDoA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFTeDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C3-PFBS	0.581	0.581	0.581	0.581	0.581	0.581	0.581
13C3-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591
13C8-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C8-PFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
D5-NEtFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-4:2FTS	1.17	1.17	1.17	1.17	1.17	1.17	1.17
13C2-6:2FTS	1.19	1.19	1.19	1.19	1.19	1.19	1.19
13C2-8:2FTS	1.20	1.20	1.20	1.20	1.20	1.20	1.20
13C3-HFPO-DA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
D7-NMeFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
D9-NEtFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
D5-NEtFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C3-PFBA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C4-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C2-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C4-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C5-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
18O2-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591

Attachment 12

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

Attachment 13

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

Attachment 14

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

Attachment 15

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

Attachment 16

PFAS Linear/Branched Mix B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. PFAS Linear/Branched Mix B (ng/ml)**
PFAS Linear/Branched Mix A	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	500	0.5	2	125
	Perfluorooctanoic acid	335-67-1	PFOA	500			125
	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500			125
	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500			125
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500			125
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			125
	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			125
	Perfluoro-7-methyloctanoic acid	15899-31-7	PF7MOA	500			125
	Perfluorononanoic acid	375-95-1	PFNA	500			125

Attachment 17

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

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

Attachment 18

1633 ICV Working Standard							
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume* (ml)	Final Conc. 1633 ICV Working Standard (ng/ml)
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.01	2	2.50
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			1.25
	Perfluorohexanoic acid	307-24-4	PFHxA	125			0.625
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			0.625
	Perfluorooctanoic acid	335-67-1	PFOA	125			0.625
	Perfluorononanoic acid	375-95-1	PFNA	125			0.625
	Perfluorodecanoic acid	335-76-2	PFDA	125			0.625
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			0.625
	Perfluorododecanoic acid	307-55-1	PFDoA	125			0.625
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			0.625
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			0.625
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			0.553
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117			0.586
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	114			0.569
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			0.595
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	117			0.583
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			0.600
	Perfluorodecanesulfonic acid	335-77-3	PFDS	121			0.603
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			0.605
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			2.34
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			2.37
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			2.40
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			0.625
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			0.625
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			0.625
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			0.625
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			0.625
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			6.25
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			6.25
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			2.50
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			2.36
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			1.25
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			1.25
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			1.25
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466			2.33
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471			2.36
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			1.11
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			3.13
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			15.6
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			15.6
Extraction Standard Mix	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500	0.01		2.5
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250			1.25
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125			0.625
	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	116			0.581
	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	118			0.591
	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	120			0.598
	Perfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250			1.25
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	250			1.25
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	234			1.17
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	238			1.19
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	240			1.20
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	500			2.5
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	1250			6.25
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250			6.25
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
Internal Standard Mix	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250	0.01		1.25
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	120			0.598
	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	118			0.591

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

Title: Mercury Analysis for Solid and Semisolid Waste Samples using the Leeman Mercury Analyzer (Cold Vapor Technique) by SW846 Method 7471B

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

SW846 Method 7471B is applicable to the determination of total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be digested prior to analysis. If this digestion procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

The typical detection limit using a 0.6 gram sample size is 0.017 mg/Kg Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

The sample is digested as described in this SOP and is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance at 253.7-nm is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Potassium permanganate is added to eliminate possible interferences from sulfide.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/Kg Copper.

4.3 Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 254 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mL).

4.4 Certain volatile organic materials that absorb at 253.7 nm may also cause interference. The analysis of the undigested sample should determine if this type of interference is present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra IIAA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software.

6.1.3. Top loader balance, 300gm capacity, and minimum sensitivity of ±1.0 mg

6.1.4. Hotblock digestor: Adjustable and capable of maintaining a temperature between 95 ±3°C.

6.2. Supplies

6.2.1 Pipettes and tips in various sizes

6.2.2 100 ml volumetric flasks

6.2.3 15 ml sample cups

6.2.4 Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab

- Rinse, Black tab

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6.2.5 Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.6 Nitrogen or Argon supply - capable of producing 80 PSI.

7.0 Reagents and Standards

Storage requirements: store at room temperature

Life of Reagent: Concentrated acids: refer to manufacturer's instructions
Laboratory prepared reagents and diluted acids: one year

Record reagent preparations in the TALS Reagent Module.

7.1 Reagents

7.1.1 Nitric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions

7.1.2 Hydrochloric acid-Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3 Potassium Permanganate (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.4 Sodium Chloride (analytical reagent grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.5 Hydroxylamine Hydrochloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.6 Stannous Chloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.7 Deionized water - 18 megohm minimum

7.1.8 10% Hydrochloric Acid- Cautiously add 200 ml of concentrated HCl to a container and bring to final volume of 2 liters with deionized water. Store at room temperature; stable for one year.

7.1.9 Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution. Store at room temperature; stable for one year.

7.1.10 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water. Store at room temperature; stable for one year.

7.1.11 Potassium permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water. Store at room temperature; stable for one year.

7.1.12 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature

Shelf-life:	Stock standards – refer to manufacturer's instructions Intermediate standards – made fresh daily Working standards – made fresh daily (Note: expiration date must not go beyond the expiration date of the source stock).
Concentration:	see Attachment 1 for example certificates of analysis (COA) of Hg standards listed below. The COA lists the manufacturer's Lot number, certified concentration and shelf life.

Document standard preparation in TALS, see Sec 11.5.3.

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2 Secondary Stock Mercury Calibration Standard (10 mg/L Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.3 Intermediate Calibration Standard (DCAL-Int), 100 ug/L Hg: Dilute 1 ml of Hg calibration stock standard solution (Sec 7.2.1) to 100 ml with 0.15% HNO_3.

7.2.4 Intermediate Initial Calibration Verification Standard (DQCS-Int), 100 ug/L Hg: Dilute 1 ml of Hg stock Calibration Verification standard, 10ppm Hg (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3.

7.2.5 Calibration Standards: Use six 50 ml hotblock cups to prepare the standards. Spike the calibrations standards cups as follows:

Calibration Standard	DCAL-Int Spike Volume (mL)	0.15% HNO_3 (mL)	Final Conc (ug/L)
Std1 (Cal Blk)	0	5	0
Std 2	0.1	4.9	0.2
Std 3	0.5	4.5	1
Std 4	1	4	2
Std 5	2.5	2.5	5
Std 6	5	0	10

Digest the standards following Sample preparation in Section 10.1.

7.2.6 Initial Verification Standard (ICV) 5.0 ppb: Using a hotblock cup, add 2.5 mL of 0.15 HNO_3 and 2.5 ml of DQCS-INT, (Sec 7.2.4). Digest the standard following Section 10.1. After digestion, the ICV will contain 5.0 ppb of Hg.

7.2.7 Continuing Calibration Verification Standard (CCV) 5.0 ppb: Follow the preparation instructions for Std 5 (see Sec 7.2.5)

8.0 Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Plastic Glass	5 grams	Cool 4 ± 2ºC	28 Days	SW846 Method 7471B

[1] Inclusive of digestion and analysis.

Samples are to be analyzed without drying. A separate procedure is used to determine the percent solids in the sample.

For sample homogenization procedures refer to TestAmerica Edison SOP ED-GEN-007 (Subsampling).

9.0 Quality Control

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

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< RL; 5% of the regulatory limit; 5% of the measured concentration in the sample
Laboratory Control Soil Sample Reference Material (LCSSRM) in soil samples	1 in 20 or fewer samples	Vendor's certified limit
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Matrix Spike (MS)[1]	1 in 20 or fewer samples	80-120%
Matrix Spike Duplicate (MSD)[2]	1 in 20 or fewer samples	80-120%; If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Serial Dilution (SD)	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client; Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.
[2] A MSD is prepared and analyzed when requested by the client.

9.1.1. Method Blank: One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion. Results must be less than the RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample. If the analyte concentration in the method blank is above this control limit, the batch must be prepared again and the samples reanalyzed.

9.1.2. Laboratory Control Sample Soil Reference Material (LCSSRM: A laboratory control sample must be analyzed with each group of samples digested. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the 'QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.1.3 Matrix Duplicate (DUP): A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL.

9.1.4 Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required.

9.1.5 Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required. If matrix spike sample and matrix spike duplicate sample are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL

9.1.6 Serial Dilution (SD): A five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. The results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2. Instrument QC

9.2.1 Initial Calibration Verification (ICV): Initial calibration is verified after calibration using an independent check standard at a concentration near

the mid-point of the calibration (5.0ppb); see Sec 7.2.6 for preparation instructions. The results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2 **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. Use a concentration of mercury at the midpoint of the calibration range (5.0 ppb). See Sec. 7.2.7 for preparation instructions. The value obtained must be within 10% of the true value. If not, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification. .

9.2.3 **Initial and Continuing Calibration Blank (ICB/CCB):** ICB/ CCB must be analyzed after the calibration curve, every 10 samples, and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

10 Procedure

10.1. Sample Preparation (includes all samples, standards, and blanks)

10.1.1 Mix the sample well and weigh 0.5 - 0.6 grams of sample (including the LCSSRM) and place in the bottom of an appropriately identified 50 mL hotblock cup. For QA samples, weigh three portions of 0.5 - 0.6 grams of sample and place in the bottoms of three hotblock cups labeled as SAMPLE, DUP, and MS. Before adding any reagents, spike the MS sample with 0.5 mL of DCAL-Int standard.

10.1.2 Except the calibration standards, add 5 mL of deionized water to all hotblock cups (i.e., all field samples, MB, LCSSRM, Dup, and MS).

10.1.3 Add 1.5 ml concentrated HNO_3 and 4.5 ml concentrated HCl. Heat 2 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.4 Add 15 mL deionized water and 15 mL potassium permanganate solution (Sec 7.1.11) to each hotblock cup. Mix well. The same amount of $KMnO_4$ must be added to the standards and samples.

10.1.5 Cap the hotblock cups loosely enough so that pressure does not build up but also tight enough so that the caps stay on and that volume loss due to heating is minimized. Heat 30 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.6 To each hotblock cup, add 6 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce excess permanganate.

10.1.7 Using deionized water, bring to a 50 mL final volume. Cap and mix well.

10.2. Calibration

10.2.1 The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in the in TALS reagent module.

10.2.2 Prepare the calibration standards and Calibration Verification Standards as stated in Sections 7.2.5, 7.2.6., & 7.2.7.

10.2.3. Calibration Curve Read-Back:

10.2.3.1. Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% of the true value (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate.

10.2.3.2. Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% of the true value (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate

10.3. Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used for samples analyzed under Method 7471B:

Instrument Calibration (Blank and five standards)
ICV
ICB
CCV
CCB

10 Samples
CCV
CCB
10 Samples
CCV
CCB
Repeat until run is complete
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1 Turn on instrument, computer and monitor.

10.3.4 Plumbing the Reagent Lines:

10.3.4.1 One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.4.2 Reductant (Red); Connect tab end of tube to the reductant bottle and the other end to the bottom of the mixing tee.

10.3.4.3 Sample (Blue); Connect tab end of tube to the autosampler probe and the other end to the top of the mixing tee.

10.3.4.4 Drain (Blue) Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.4.5 Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.5 Preparation of Reagents:

10.3.5.1 Pour the $SnCl_2$ solution into the reductant bottle and connect to the red reductant tube connector.

10.3.5.2 Pour the ten percent HCl solution into the Rinse reservoir bottle.

10.3.6 Starting Program:

10.3.6.1 Click the Envoy icon on the computer desktop

10.3.6.2 Click Method, select 7471_7471B, and select OK
10.3.6.3 On the main screen, click the StartUp icon. Wait 15 minutes before analyzing calibrating.
10.3.6.4 Click Sequence Tab on bottom of screen
10.3.6.5 Click Sequence on top of screen
10.3.6.6 Select Open, then select New
10.3.6.7 Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions
10.3.6.8 Pour out the digested calibration standards and samples into the proper locations on the autosampler.
10.3.6.9 Click the Run Sequence icon to begin calibration and to run samples.

10.3.7 Creating the Raw Data PDF:

10.3.7.1 Click the Analysis Tab, select Detailed

10.3.7.2 Click Load, select Use This To Make PDF

10.3.7.3 Click Report, Click Printer

10.3.7.4 Include the calibration curve graph:

10.3.7.4.1 Go to Methods Tab and click Calibration, click Print

10.3.7.5 Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the "Doc's" location in the analytical batch.

10.3.8 Shutting Down the Instrument:

10.3.8.1 Click the Stop icon on the main screen

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.3. Concentration: (mg/Kg) = $\frac{C \times V1 \times D}{W}$

Where: C= Element concentration from instrument (ppb)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample
W= Initial weight of sample digested (in gram)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Import Data to TALS

11.5.2.1. Click the Analysis tab
11.5.2.2. Select the analytical run that needs to be imported
11.5.2.3. Select Statistics
11.5.2.4. Click Load and select TALS Import, click OK
11.5.2.5. Click Report, click CSV File
11.5.2.6. Name the import file (e.g., batch name, today's date)
11.5.2.7. The newly created import file is in the Import Folder on the desktop. Send to TALS Import Folder

11.5.3. Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.4. All reagents must be recorded in TALS Reagent Module.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.5.5.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.5.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.5.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

11.5.6. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying the average of the standard deviations obtained from the analysis of seven reagent blanks by 3.14.

12.3 Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.4 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.5. Lower Limit of Quantitation (LLOQ)

12.5.1. LLOQ verification: prior to analyzing samples under method 7470A, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ (0.2 ppb). The seven replicates must be digested prior to analysis. The LLOQ is verified when the mean recovery of the 7 replicates is +/-30% of the true value and the RSD < 20%.

12.5.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water spiked at the LLOQ (0.2 ppb). The MB and LLOQ samples must be digested prior to analysis. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-30% of the .true value.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 7471B, Revision 2, January 1998.

15.2. Leeman Hydra IIAA Operating Manual.

15.3. TestAmerica Edison Document ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.6. TestAmerica Edison Subsampling SOP, *ED-GEN-007,* most current revision.

15.7. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

16.0. Method Modifications:

Item	Method #	Modification
1 (Sec 10.3.5.1)	7471B	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's

		improvement that will reduce error due to loss of Mercury.
Sample preparation	7471B	The amount of DI water added to the sample at digestion is decreased to 15 ml from 50 ml (water bath procedure), while keeping the volume of reagents added during sample digestion the same. Sample final volume for hotblock procedure is modified to 50 ml (100 ml – water bath procedure) which allows for lesser sample volume hence less sample waste produced; hotblock digestion cup has maximum volume of 50 ml.

17.0. Attachments

Attachment 1: Certificate of Analysis of stock standards

18.0. Revision History

- Revision 5, dated 11 August 2022
  - Sec 12.5: Added the lower limit of quantitation (LLOQ), associated instructions and recovery limits.
- Revision 4, dated 03 August 2020
  - Updated SOP header with Eurofin logo.
  - Sec 6.1.1 & 15.2: Replaced Hydra AA with Hydra IIAA.
  - Sec 9.1 Table and 9.1.1: Revised MB control limits to <RL.
  - Sec 9.1, 9.1.4 and 9.1.5: Revised MS and MSD acceptance limits to 80-120%.
  - Sec 9.2.2: Revised CCV acceptance limits to +/-10%.
  - Sec 10.2.3: Added calibration curve read-back criteria.
  - Sec 11.4: Added Relative % error calculation.
  - Sec 11.5: Added Relative % error calculation.
  - Sec 11.5.6.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.
- Revision 3, dated 12 October 2018
  - Sec 6.1.2: Removed WinHg instrument software reference and replaced with current instrument software, Envoy.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix
  - Sec 10.3.6: Updated the Starting Program instructions to reflect the new instrument software, Envoy.
  - Sec 10.3.7 & 10.3.8: Updated instructions for creating raw data pdf and shutting down instrument.
  - Sec 11.4.2: Updated instructions for importing data to TALS.
  - Sec 11.4.5: Replaced Metals Data Review Checklist with Data Review Checker (DRC) in TALS.

- Sec 15.8 & 15.9: Removed reference to EDS-WI-007 & EDS-WI-125; WI not applicable.

- Revision 2, dated 29 Nov 2016
  - Sec 6.1.4, Sec 10.1.3, & 10.1.5: updated the hotblock temperature range per method 7471B requirements
  - Sec 6.2.7: added Argon gas
  - Sec 7.2.4: the name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.6: removed the CCV preparation instructions using the second source standard. The name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.7: added preparation instructions for the CCV using the primary source standard
  - Sec 9.1: added the quality control sample MSD to the table
  - Sec 9.1.5: added quality control information for the MSD
  - Sec 11.4.5: Metals Data Review Checklist control # has been updated from ED-WI-007 to CA-Q-WI-042

- Revision 1, dated 12 May 2014
  - Sec 1.1: Revised the typical detection limit from 0.033 mg/kg to 0.017 mg/kg to reflect the 50mL final volume – hotblock procedure.
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 6.1.4: Added hotblock digestor to the list of equipment; removed autoclave from the list.
  - Sec 6.2: Added 50 mL hotblock digestion cups to replace the BOD bottles. Removed supplies which are no longer applicable (BOD bottles, graduated cylinders).
  - Sec 7.0 & 11.4.4: Removed Hg Reagent Logbook and replaced with TALS Reagent Module
  - Sec 7.1. Deleted Sulfuric Acid (previously referenced in Sec 7.1.1). Added 0.15% HNO_3 to list of reagents.
  - Sec 7.2.5: Updated Calibration standards' spiking instructions to reflect the 50 ml final volume.
  - Sec 7.2.6: Updated ICV/CCV spiking instructions to reflect the 50 ml final volume.
  - Sec 8.1: Added plastic container to the list of acceptable sample containers.
  - Sec 9.1 & 9.1.3: Clarified the acceptance limits for Matrix duplicate to reflect actual laboratory practices.
  - Sec 9.1.4: Revised the spiking amount added to the MS sample.
  - Replaced LCSS reference with LCSSRM in various sections of the SOP to reflect TALS QC type reference.
  - Sec 10.1: Revised sample preparation procedure to include hotblock digestor procedure; deleted all reference to autoclave digestion procedure.

- Sec 11.4.5 & 15.9: Added Work instructions EDS-WI-125 (TestAmerica Edison Metals Initial Calibration Data Review for Mercury).
- Sec 16: Added Sample prep method modification (i.e. final volume).

- Revision 0, dated 04 February 2011
  New

Attachment 1

Certificate of Analysis

Catalogue Number :	141-110-111/141-110-112/141-110-115
Description :	*Plasma*CAL - **ICP-MS Verification Standard 1 Solution B**
Lot Number :	**SC8298812**
Expiration Date :	**January 2010**

Analysis of Solution Standard by Inductively Coupled Plasma Spectroscopy (ICP-AES) traceable to NIST Standard Reference Materials : 3133

Actual Concentrations

Hg : 9.98 µg/ml

Matrix : **10% HNO_3**
Density : **1.040 g/ml @ 22.0°C**

MS 0882
Rec'd
1/14/09

Certified by : _____
Thomas Znoj, Chemistry Manager

Certification Date : **October 28, 2008**

This ICP-AES & ICP-MS Standard is guaranteed to be stable and accurate to within plus or minus 1.0% of the actual concentration up to the expiry date, provided the solution is kept tightly capped and stored under normal laboratory conditions. For these solutions, 18 megohm/cm double deionized water, high-purity acids, Class A glassware and acid-cleaned bottles are used. The Material Safety Data Sheet and this Certificate of Analysis are available on our web site. (Également disponible en Français)

Manufactured according to an ISO 9001:2000 Quality System and ISO 17025 (in-process)

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CERTIFICATE OF ANALYSIS

MS0879
Rec'd 1-12-09

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001:2000 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

Catalog Number:	MSHG-10PPM
Lot Number:	**B2-HG02061**
Starting Material:	Hg metal
Starting Material Purity (%):	99.999549
Starting Material Lot No:	05214TX
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 10.027 ± 0.020 µg/mL

Certified Density: 1.019 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons to the following NIST SRMs:

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

Revision Log

Revision: 02	**Effective date:**	This version
Section	**Justification**	**Changes**
		Update procedure.

Reference

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

Cross Reference

Document	Document Title

Document	Document Title
NC-WC-092	Determination of Solids in Waters and Wastes (Total Suspended Solids)
NDSC-QA-SOP42091	Detection and Quantitation Limits
NDSC-QA-SOP43862	Manual Integrations
NC-QA-015	Support Equipment Calibration and Verification
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
QA-003	Quality Control Program
NC-QA-017	Standards and Reagents
NDSC-QA-QP44940	Calibration Curves and the Selection of Calibration Points
NDSC-US-EHS QP46060	NDSC EH&S Manual
NC-QAM-001	QA Manual

Scope

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

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

Analyte	Acronym	CAS#
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonic acid	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1

Analyte	Acronym	CAS#
Perfluoro-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA, PES	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

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

Interferences

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

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.

3. All part of the SPE manifold must be cleaned every 2 weeks per manufacturer recommendations.

Safety Precautions and Waste Handling

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

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

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

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

Personnel Training and Qualifications

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

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

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

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

Sample Collection, Preservation, and Handling

A. Sample Collection

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

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

B. Sample Storage and Shipment

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

2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, and protected from light until extraction. Alternatively, to meet project requirements, samples may be stored at ≤ -20°C and protected from light until extraction.

3. Water samples must be extracted within 28 days when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Water samples must be extracted within 90 days when stored at a temperature < -20°C and protected from light. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light.

Apparatus and Equipment

A. Apparatus

1. 125 mL HDPE bottles: Environmental Sampling Supply; #0125-1902-QC, or equivalent.

2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 14955237 or equivalent

3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288 or equivalent.

4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC, or equivalent.

5. Analytical Balance – Capable of weighing to 0.0001 g

6. Top-Loading Balance – Capable of weighing to 0.01 g

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

8. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product # 54258-U.

9. SPE Tube Adapter - Millipore-Sigma; Product # 57020-U.

10. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.

11. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.

12. Vacuum manifold for Solid Phase Extraction (SPE).

13. Centrifuge – "Thermo Sorvall ST4" with TX-750 rotor or equivalent, capable of a maximum rotational force of 3800 rcf.

14. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. FisherScientific cat # 14-287-150, or equivalent.

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

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

17. Pipettes – Disposable transfer. Fisher Scientific, Cat. No. 13-711-7M

18. Vortex mixer, variable speed, Fisher Scientific or equivalent.

19. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control. Organomation, Inc. Cat. #11250-NT, or equivalent.

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

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

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

23. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent

24. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125

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

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

27. Syringe filter - Acrodisc, Syringe Filter, 25 mm, 0.2 µm, 50/pkg, Fisher part # 50786068.

28. Silanized glass wool, Supelco # 20411 or equivalent

29. Wheaton Bottle, 15ml, narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Cat. No. 209044SP, or equivalent

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

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

2. PromoChrom automated extractor, model, SPE-03, or equivalent
   a. Promochrom Filters, Catalog Number F-HC-30

3. HPLC columns

   a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-Y0 or equivalent

   b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

Reagents and Standards

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

A. Reagents:

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

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

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

B. Standards:

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

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

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

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

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

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

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

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

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

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

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 04*.
If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

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

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

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

Attachment 05 - Native PFAS Intermediate A
Attachment 06 - Native PFAS Intermediate B
Attachment 07 - Native PFAS Intermediate M
Attachment 08 - Extraction Standard Mix
Attachment 09 - Internal Standard Mix
Attachment 10 - Initial Calibration Standards Preparation
Attachment 11 - Initial Calibration Standards Concentrations
Attachment 12 - Bile Salts Stock Solutions
Attachment 13 - Bile Salts Working Solution A
Attachment 14 - Bile Salts Working Solution B
Attachment 15 - PFAS Linear/Branched Mix A
Attachment 16 - PFAS Linear/Branched Mix B
Attachment 17 - Linear/Branched Bile Salts Solution
Attachment 18 - ICV Working Standard

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order(quadratic) curve fit. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.

2. Analyze a Cal4 level standard that contains Bile Salt retention time markers and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the

calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

Example Initial Calibration Sequence:

1. Instrument Blank
2. Instrument Blank
3. Instrument Blank
4. CAL 1
5. CAL 2
6. CAL 3
7. CAL 4
8. CAL 5
9. CAL 6
10. CAL 7
11. ICB (Instrument Blank)
12. ICV
13. WDM (Linear Branched/Bile Salts standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 02* for additional information about compound relationships.

4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic acid (TCDCA), and Tauroursodeoxycholic acid (TUDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

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

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA

PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

5. Fit the curve

a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.

c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

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

6. Initial Calibration Verification (ICV)

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

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

2. Acceptance criteria

a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

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

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

A. Sample Preparation

1. Weigh sample container with contents on a calibrated top loading balance, and record the first reading in the automated prep entry system.

   a. For all samples, the full bottle must be extracted. The sample must remain in the original sample container until after spiking solutions have been added.

   b. If limited sample is submitted, spike sample in original container, then add reagent water to bring to final volume of 125 mL prior to SPE extraction (see B.6 for spiking details).
   c. If the sample has dissolved and/or settleable solids content (i.e; is cloudy or has a layer of sediment/solids at the bottom of the bottle), the sample must be centrifuged in order to minimize the difficulty of passing throug the SPE sorbent bed. In order to preserve the integrity of the sample and ensure the full voume of the container is used, see Procedure C.

2. Use a 125 mL HDPE bottle for the method blank, the laboratory control sample (LCS) and the low level laboratory control sample (LLCS). Fill each bottle with 125 mL of reagent water. Record 125 mL as the volume for the batch QC samples on the batchlog.

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

B. Solid Phase Extraction (SPE) - Manual Sample Extraction

1. Pack clean silanized glass wool to a quarter of the height of the SPE cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batch log.

3. Condition each SPE cartridge with the following reagents in the following order without allowing the cartridges to go dry:

   a. 15 mL 1% methanolic ammonium hydroxide

   b. 5 mL 0.3M formic acid

   c. Discard conditioning eluent(s)

4. Label each sample bottle, cap and reservoir with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Vortex all spike solutions prior to use.

6. Spike QC and all samples with 25 µl of Working labeled extraction standard spike solution (PFAS EIS Mix). Spike LCS/MS/MSD with 200 µl of native 1633 mid-level spike solution (PFAS Native M). Spike LLCS with 200 µl of native 1633 low-level spike solution (PFAS Native B). Vortex/Shake containers to mix thoroughly.

7. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges.

8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5 mL 1:1 0.1M formic acid/methanol and pass the rinses through the cartridge using vacuum. Apply full

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

NOTE: If a given sample is too problematic to extract using one cartridge with full vacuum, a second cartridge is created following Procedure B.1-3 above.

9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge ensuring the delivery needles to do not touch the sides of the tubes.

10. Rinse the inside of each empty sample/QC bottle with 5mL of 1% methanolic ammonium hydroxide.

11. Using a glass pipette, transfer the rinse from the bottles to the SPE reservoirs, washing the walls of the reservoirs. Set empty bottles aside to air dry.

12. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

13. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

Note: If a second cartridge was required, the elutant from the second cartridge is added to the first cartridge elutant with a methanol rinse and then blown down to 5 mL on an N-evap prior to step 14 below.

14. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix.

15. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

16. Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to a clean 15-mL polypropylene centrifuge collection tube.

17. Place a syringe filter (25-mm filter, 0.2-µm nylon membrane) on a 5 mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the plunger and filter the entire extract into the new collection tube containing the internal standard.

18. Bring each sample extract to final volume 5ml using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

19. Cap and vortex to mix.

20. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

21. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for dilution or reinjection if needed.

C. Samples containing Dissolved and/or Settleable Solids

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

2. Spike sample with appropriate spikes as in Procedure B.6.

3. Centrifuge the full bottle.

4. DO NOT SHAKE BOTTLE FOLLOWING THE CENTRIFUGE STEP.

5. Follow steps in Procedure B, 1 through 4.

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

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

8. Continue extraction process with Procedure B. 8.

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

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

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

1. Pack clean silanized glass wool to a quarter of the height of the SPE cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

3. Check to make sure all reagent bottles are filled and that reagents are not expired.

4. Label each sample bottle and cap with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Vortex all spike solutions prior to use.

6. Spike QC and all samples with 25 µl of Working labeled extraction standard spike solution (PFAS EIS Mix). Spike LCS/MS/MSD with 200 µl of native 1633 mid-level spike solution (PFAS Native M). Spike LLCS with 200 µl of native 1633 low-level spike solution (PFAS Native B). Vortex/Shake containers to mix thoroughly.

7. Select "Clean sys-004" from the drop down menu to start the cleaning cycle prior to SPE. Make sure the "Clean" cartridges and bottles are attached for this cycle. After the "Clean sys-004" has finished, attach Promochrom filters to the bottles, select "Clean Filter" from the drop down menu to clean filters prior to SPE.

8. Remove "Clean" cartridges and attach the labeled cartridges containing a graphitized carbon that will be used when running the samples when the system cleaning is complete. Disconnect the "Clean" bottles. Attach the labeled sample bottles by twisting the bottles and not the cap. Place sample bottles upside down in corresponding numbered location on shaker. Poke two holes into the lip of each bottle. Load labeled centrifuge tubes in the moving tray beneath the cartridges.

9. Select "EPA 1633-004" from the drop down menu and press the green check mark to start the procedure. Note: N2 dry down should be 15 minutes long.

10. Once the cycle is complete, remove centrifuge tubes and discard the used cartridges and bottles. Reattach "Clean" bottles and cartridges, select "clean sys-004" from menu, select "start". This must be done between each batch and at the beginning and the end of the day.

11. Add 25ul of Acetic acid to sample extracts and vortex to mix thoroughly. Extracts must be stored in a cooler.

12. Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to a clean 15-mL polypropylene centrifuge collection tube. Using a syringe, push ~ 3mL of MeOH through each syringe filter and allow to dry..

13. Place a syringe filter (25-mm filter, 0.2-µm nylon membrane) on a 5 mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the plunger and filter the entire extract into the new collection tube containing the internal standard.

14. Bring each sample extract to final volume 5ml using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

15. Cap and vortex to mix.

16. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

17. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for reinjection if needed.

E. LC/MS/MS Analysis

1. Mass Calibration and Tuning

   a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

   b. When masses fall outside of the +/- 0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Example acquisition method: See *Attachment 03*. Mass Transitions: See *Attachment 01*.

4. Instrument Sensitivity Check (ISC) and Instrument Blanks
   a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the

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

b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salt RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

   Example Sample Sequence:
   1. Instrument blank
   2. Instrument blank
   3. Instrument blank
   4. Instrument Sensitivity Check (CCV 0_CAL1)
   5. CCV 2_CAL4
   6. Linear Branched/Bile Salts marker (WDM)
   7. Instrument Blank (ICB)
   8. Method Blank (MB)
   9. Low Level LCS (LLCS)
   10. LCS
   11. Sample (10 or less)
   12. CCV 3_CAL4
   13. Instrument Blank

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.

b. If LLCS and LCSD recoveries are acceptable, proceed to sample quantitation.

c. If the LCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS must be reextracted.

d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Calculate the relative percent difference(RPD) between the sample and the matrix duplicate(DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data.

11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. (See Statistical Infromation/Method Performance section of this SOP.) If the extraction standard recovery(ies) is(are) outside the QC limit(s), revial a fresh aliquot and reanalyze. If the extraction standard recovery(ies) are still outside the QC limit(s), reanalyze the sample at a dilution. If the extraction standard recovery(ies) are still outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) are still outside the QC limit(s), narrate with an NCM and report the data.

12. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

13. Compare the retention times of all of the analytes, surrogates, and internals standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than

    a. 0.4 minutes for isotopically-labeled compounds

    b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound

    c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

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

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

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

15. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.

16. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of internal standard spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring dilutions greater than 10X should be reextracted using a reduced aliquot.

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

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

    On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

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

4. Sample Concentration

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

5. Ion Ratio

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

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

Statistical Information/Method Performance

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

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

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

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

Quality Assurance/Quality Control

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

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

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

Attachment 08 - Extraction Standard Mix (.pdf)
Attachment 09 - Internal Standard Mix (.pdf)
Attachment 10 - Initial Calibration Standards Preparation (.pdf)
Attachment 11 - Initial Calibration Standard Concentrations (.pdf)
Attachment 12 - Bile Salt Stock Solutions (.pdf)
Attachment 13 - Bile Salts Working Solution A (.pdf)
Attachment 14 - Bile Salts Working Solution B (.pdf)
Attachment 15 - PFAS Linear Branched Mix A (.pdf)
Attachment 16 - PFAS Linear Branched Mix B (.pdf)
Attachment 17 - Linear Branched Bile Salts Solution (.pdf)
Attachment 18 - ICV Working Standard (.pdf)
Attachment 19 - Table 5 (1633 Draft V3) (.docx)
Attachment 20 - Table 8 (1633 Draft V3) (.docx)

Attachment: Attachment 01 - Mass Transitions (doc)
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Attachment: Attachment 05 - Native PFAS Intermediate A (pdf)
Attachment: Attachment 06 - Native PFAS Intermediate B (pdf)
Attachment: Attachment 07 - Native PFAS Intermediate M (pdf)
Attachment: Attachment 08 - Extraction Standard Mix (pdf)
Attachment: Attachment 09 - Internal Standard Mix (pdf)
Attachment: Attachment 10 - Initial Calibration Standards Preparation (pdf)
Attachment: Attachment 11 - Initial Calibration Standard Concentrations (pdf)
Attachment: Attachment 12 - Bile Salt Stock Solutions (pdf)
Attachment: Attachment 13 - Bile Salts Working Solution A (pdf)
Attachment: Attachment 14 - Bile Salts Working Solution B (pdf)
Attachment: Attachment 15 - PFAS Linear Branched Mix A (pdf)
Attachment: Attachment 16 - PFAS Linear Branched Mix B (pdf)
Attachment: Attachment 17 - Linear Branched Bile Salts Solution (pdf)
Attachment: Attachment 18 - ICV Working Standard (pdf)
Attachment: Attachment 19 - Table 5 (1633 Draft V3) (docx)
Attachment: Attachment 20 - Table 8 (1633 Draft V3) (docx)

End of document

Version history

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

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

Attachment 1

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

Attachment 1

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

Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

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

Extraction Standards

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

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

Native PFAS Compounds

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

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

Acquisition Method

1633

Mass Spec 12.500 min
Period 12.500 mi
-MRM
Integrated Valve
LC System
Equilibrate
Injection

Mass Spectrometer Method Properties

Period 1:

Scans in Period:	1250
Min. Dwell Time:	3 ms
Max. Dwell Time:	250 ms
Relative Start Time:	0.00 msec
Scheduled Ionization:	Off
Experiments in Period:	1
Use target Cycle Time:	No
Target Cycle Time:	N/A

Period 1 Experiment 1:

Scan Type:	MRM (MRM)
Scheduled MRM:	Yes
Polarity:	Negative
Scan Mode:	N/A
Ion Source:	Turbo Spray
sMRM Q1Q3 Resolution:	No
MRM detection window:	60 sec
Target Scan Time:	0.6000 sec
Resolution Q1:	Unit
Resolution Q3:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Step Size:	0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
212.800	168.900	2.71	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	84.900	2.94	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	2.98	DP	-50.00	-50.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	3.21	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	79.900	3.67	DP	-90.00	-90.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.100	3.32	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	134.900	3.84	DP	-70.00	-70.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	3.52	DP	-30.00	-30.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.41	DP	-110.00	-110.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.55	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

349.100	79.900	4.00	DP	-90.00	-90.00	PFPeS	
			CE	-70.00	-70.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.68	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.85	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.30	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	3.96	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	3.98	DP	-90.00	-90.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.68	DP	-110.00	-110.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.12	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	5.22	DP	-90.00	-90.00	PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.43	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.40	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.61	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.72	DP	-100.00	-100.00	PFNS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.85	DP	-60.00	-60.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.58	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	6.14	DP	-110.00	-110.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.79	DP	-80.00	-80.00	NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	6.16	DP	-90.00	-90.00	PFDS
			CE	-130.00	-130.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.39	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	5.04	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.49	DP	-90.00	-90.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.87	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.94	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	7.08	DP	-90.00	-90.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.94	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	7.24	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.36	DP	-90.00	-90.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	619.000	6.30	DP	-60.00	-60.00	PFTrDA
			CE	-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.71	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.66	DP	-110.00	-110.00	PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.52	DP	-10.00	-10.00	PFECA B_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.41	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.55	DP	-40.00	-40.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.00	DP	-110.00	-110.00	PFPeS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.68	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.85	DP	-50.00	-50.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.30	DP	-90.00	-90.00	PFHxS_2

			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	3.98	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.68	DP	-100.00	-100.00	PFHpS_2
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.12	DP	-50.00	-50.00	PFOA_2
			CE	-26.00	-26.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	5.21	DP	-110.00	-110.00	PFOS_2
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	4.43	DP	-50.00	-50.00	PFNA_2
			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	98.800	5.72	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	219.000	4.85	DP	-60.00	-60.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	80.800	4.58	DP	-100.00	-100.00	8:2 FTS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	483.000	4.79	DP	-90.00	-90.00	NMeFOSAA_2
			CE	-24.00	-24.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	98.800	6.16	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	269.100	5.39	DP	-70.00	-70.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	526.000	5.04	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	319.000	5.87	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

663.000	168.900	6.30	DP		-60.00	-60.00	PFTrDA_2
			CE		-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.71	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.21	DP	-40.00	-40.00	PFPeA_2
			CE	-55.00	-55.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	6.13	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	7.08	DP	-110.00	-110.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.35	DP	-90.00	-90.00	NEtPFOSA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	3.95	DP	-40.00	-40.00	DONA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.60	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.48	DP	-90.00	-90.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	2.98	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.40	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.83	DP	-70.00	-70.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.94	DP	-90.00	-90.00	PFDoS_2
			CE	-140.00	-140.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.71	DP	-40.00	-40.00	13C3-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.71	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.21	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.66	DP	-90.00	-90.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.41	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.54	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.54	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.68	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.85	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.30	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.30	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	3.97	DP	-90.00	-90.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.12	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.12	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	5.21	DP	-90.00	-90.00	13C4-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	79.900	5.21	DP	-90.00	-90.00	13C8-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.42	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
519.100	474.100	4.85	DP	-60.00	-60.00	13C6-PFDA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.100	470.100	4.85	DP	-60.00	-60.00	13C2-PFDA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	80.900	4.58	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
506.100	77.800	6.13	DP	-110.00	-110.00	13C8-PFOSA
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
573.200	419.000	4.78	DP	-90.00	-90.00	d3-NMeFOSAA
			CE	-27.00	-27.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.100	5.38	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.200	419.000	5.02	DP	-90.00	-90.00	d5-NEtFOSAA

			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.86	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.92	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	7.07	DP	-90.00	-90.00	d3-NMePFOSA
			CE	-37.00	-37.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	7.21	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.34	DP	-90.00	-90.00	d5-NEtPFOSA
			CE	-38.00	-38.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.70	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.54	DP	-40.00	-40.00	13C5-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.66	DP	-110.00	-110.00	13C3-PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.29	DP	-90.00	-90.00	13C3-PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	5.20	DP	-110.00	-110.00	13C8-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.41	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	3.97	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.58	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.68	DP	-75.00	-75.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

315.100	119.400	3.54	DP	-40.00	-40.00	13C2-PFHxA_2	
			CE	-31.00	-31.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	5.21	DP	-110.00	-110.00	13C4-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.78	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.70	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.39	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
CAD:	10.00
EP	-10.00
CXP	-14.00

Acquisition Method

1633

Mass Spec 12.500 min
   Period 12.500 min
      -MRM
Integrated Valve
LC System
   Equilibrate
   Injection

Acquisition Information:

Acquisition Method:	1633.dam
Created:	Wednesday May 24 2023 14:48:39 PM
Last Modified:	Wednesday June 14 2023 11:54:50 AM
Comment:	
Synchronization Mode:	LC Sync
Auto-Equilibration:	Off
Acquisition Duration:	12min30sec
Number Of Scans:	1250
Periods In File:	1
Acquisition Module:	Acquisition Method
Software version	Analyst 1.7.3

Period 1:

Scans in Period:	1250
Min. Dwell Time:	3 ms
Max. Dwell Time:	250 ms
Relative Start Time:	0.00 msec
Scheduled Ionization:	Off
Experiments in Period:	1
Use target Cycle Time:	No
Target Cycle Time:	N/A

Period 1 Experiment 1:

Scan Type:	MRM (MRM)
Scheduled MRM:	Yes
Polarity:	Negative
Scan Mode:	N/A
Ion Source:	Turbo Spray
sMRM Q1Q3 Resolution:	No
MRM detection window:	60 sec
Target Scan Time:	0.6000 sec
Resolution Q1:	Unit
Resolution Q3:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Step Size:	0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
212.800	168.900	2.71	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	84.900	2.94	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	2.98	DP	-50.00	-50.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	3.21	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	79.900	3.67	DP	-90.00	-90.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.100	3.32	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	134.900	3.84	DP	-70.00	-70.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	3.52	DP	-30.00	-30.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.41	DP	-110.00	-110.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.55	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	79.900	4.00	DP	-90.00	-90.00	PFPeS
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.68	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.85	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.30	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	3.96	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	3.98	DP	-90.00	-90.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.68	DP	-110.00	-110.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.12	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	5.22	DP	-90.00	-90.00	PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.43	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.40	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.61	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.72	DP	-100.00	-100.00	PFNS

			CE	-110.00	-110.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.85	DP	-60.00	-60.00	PFDA
			CE	-18.00	-18.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.58	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	6.14	DP	-110.00	-110.00	PFOSA
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.79	DP	-80.00	-80.00	NMeFOSAA
			CE	-27.00	-27.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	6.16	DP	-90.00	-90.00	PFDS
			CE	-130.00	-130.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.39	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	5.04	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.49	DP	-90.00	-90.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.87	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.94	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	7.08	DP	-90.00	-90.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.94	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	7.24	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.36	DP	-90.00	-90.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

663.000	619.000	6.30	DP		-60.00	-60.00	PFTrDA
			CE		-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.71	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.66	DP	-110.00	-110.00	PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.52	DP	-10.00	-10.00	PFECA B_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.41	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.55	DP	-40.00	-40.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.00	DP	-110.00	-110.00	PFPeS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.68	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.85	DP	-50.00	-50.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.30	DP	-90.00	-90.00	PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	3.98	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.68	DP	-100.00	-100.00	PFHpS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.12	DP	-50.00	-50.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	5.21	DP	-110.00	-110.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	4.43	DP	-50.00	-50.00	PFNA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	98.800	5.72	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	219.000	4.85	DP	-60.00	-60.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	80.800	4.58	DP	-100.00	-100.00	8:2 FTS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	483.000	4.79	DP	-90.00	-90.00	NMeFOSAA_2
			CE	-24.00	-24.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	98.800	6.16	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	269.100	5.39	DP	-70.00	-70.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	526.000	5.04	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	319.000	5.87	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	168.900	6.30	DP	-60.00	-60.00	PFTrDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.71	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.21	DP	-40.00	-40.00	PFPeA_2
			CE	-55.00	-55.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	6.13	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	7.08	DP	-110.00	-110.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.35	DP	-90.00	-90.00	NEtPFOSA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	3.95	DP	-40.00	-40.00	DONA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.60	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.48	DP	-90.00	-90.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	2.98	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.40	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.83	DP	-70.00	-70.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.94	DP	-90.00	-90.00	PFDoS_2
			CE	-140.00	-140.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.71	DP	-40.00	-40.00	13C3-PFBA

			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.71	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.21	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.66	DP	-90.00	-90.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.41	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.54	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.54	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.68	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.85	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.30	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.30	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	3.97	DP	-90.00	-90.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.12	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.12	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	5.21	DP	-90.00	-90.00	13C4-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

507.100	79.900	5.21	DP		-90.00	-90.00	13C8-PFOS
				CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.42	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
519.100	474.100	4.85	DP	-60.00	-60.00	13C6-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.100	470.100	4.85	DP	-60.00	-60.00	13C2-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	80.900	4.58	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
506.100	77.800	6.13	DP	-110.00	-110.00	13C8-PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
573.200	419.000	4.78	DP	-90.00	-90.00	d3-NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.100	5.38	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.200	419.000	5.02	DP	-90.00	-90.00	d5-NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.86	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.92	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	7.07	DP	-90.00	-90.00	d3-NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	7.21	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.34	DP	-90.00	-90.00	d5-NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.70	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.54	DP	-40.00	-40.00	13C5-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.66	DP	-110.00	-110.00	13C3-PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.29	DP	-90.00	-90.00	13C3-PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	5.20	DP	-110.00	-110.00	13C8-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.41	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	3.97	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.58	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.68	DP	-75.00	-75.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	119.400	3.54	DP	-40.00	-40.00	13C2-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	5.21	DP	-110.00	-110.00	13C4-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.78	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.70	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.39	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
CAD:	10.00
EP	-10.00

CXF -14.00

Integrated Valve Method Properties
Valve Diverter

	Total Time (min)	Position
1	1.0	B
2	8.0	A

LC Method Properties

LC system Equlibration time = 0.00 min
LC system Injection Volume = 10.00 ul

Pump

Model:	A: LC-40D XF B: LC-40D XR
Pump mode:	Binary gradient
Stop time:	12.50 min
<Gradient start adjustment>	
Mode:	None
<B.GE1>	
<General>	
Flow/Pressure:	Flow
Flow:	0.5000 mL/min
Time to reach the flow:	0.00 min
B.Conc:	5.0 %
B.Curve:	0
Pressure limits Minimum:	0 psi
Pressure limits Maximum:	6500 psi

<Gradient>

Time min	Flow mL/min	B.Conc %	B.Curve
0.00	0.5000	5.0	0
0.20	0.5000	5.0	0
3.00	0.5000	60.0	0
3.80	0.5000	60.0	0
6.70	0.5000	98.0	0
8.50	0.5000	98.0	0
8.70	0.6500	5.0	0
12.50	0.5000	5.0	0

<PumpA>	
<Mobile phase switching valve>	
Selected mobile phase port:	A
<Mobile phase settings>	
Set compressibility:	No

Port	Mobile phase name
A	Mobile phase A

<Advanced>	
LPGE cycle:	Standard
<PumpB>	

```
      <Mobile phase switching valve>
         Selected mobile phase port:                    A
      <Mobile phase settings>
         Set compressibility:                          No
      Port  Mobile phase name
      ----  -----------------
         A     Mobile phase A
      <Advanced>
         LPGE cycle:                             Standard
<Autopurge settings>
   Execute autopurge before analysis:                  No
   <Autosampler purge settings>
      Execute sampler purge:                           No
   <Init conc-replacement>
      Use Init conc-replacement:                       No
   <Warm up>
      Wait time:                                  0 min
      B.GE1 flow:                          0.0000 mL/min
   <Execute after autopurge>
      Turn oven on after autopurge:                    No
      Turn pump on after autopurge:                    No
<Time program>
Autosampler
===========
Model:                                        SIL-40C XR
<General>
   Use Autosampler:                                  Yes
<Sample plate settings>
   Specify plate:                                    No
   Specify needle stroke:                            No
<Injection settings>
   Sampling speed:                             1.0 µL/s
   Use cooler temperature:                           Yes
   Cooler temperature:                            15 °C
   Use air gap volume:                               No
   Use low dispersion injection:                     No
<Acquisition cycle time optimization>
   Pretreatment start timing:                        Off
<Cut off loop settings>
   Cut off loop:                                     No
<Rinse settings>
   Rinse type:                           External only
   Rinse mode:                 Before and after aspiration
   Rinse dip time:                                0 s
   Rinse method:                        Rinse port only
   Rinse time:                                  2.0 s
   Rinsing speed:                              35 µL/s
   Rinsing volume:                             500 µL
   Measuring line purge volume:                100 µL
<Details>
   Wait for temperature equilibration before run (sample cooler):
                                                     No
```

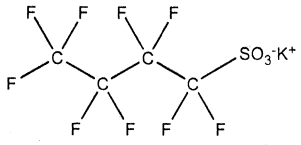
```
<Pretreatment>
   Mode:                                          Standard
<Time program>
Oven
====
Model:                                         A: CTO-40C
<Column Oven A>
   <General>
      Use Column Oven:                               Yes
      Oven temperature:                           40 °C
      Temperature limit(Maximum):                 90 °C
      Wait for temperature equilibration before run:  Off
   <Advanced>
      Fan speed:                                  Auto
      Cooler mode:                                Auto
<Time program>
System Controller
==================
Model:                                           CBM-40
<External output settings>
   Event 1:                                          No
   Event 2:                                          No
<Shutdown settings>
   Power off after shutdown:                         No
<Time program>
Time  Command  Value
 min
---- -------- ------
0.01    Event      1
```

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

PRODUCT CODE: L-PFBS
LOT NUMBER: LPFBS1022
COMPOUND: Potassium perfluoro-1-butanesulfonate

STRUCTURE:
CAS #: 29420-49-3



MOLECULAR FORMULA: $C_4F_9SO_3K$
MOLECULAR WEIGHT: 338.19
CONCENTRATION: 50.0 ± 2.5 µg/mL (K salt)
44.4 ± 2.2 µg/mL (PFBS acid)
44.2 ± 2.2 µg/mL (PFBS anion)
SOLVENT(S): Methanol
CHEMICAL PURITY: >98%
LAST TESTED: (mm/dd/yyyy) 10/04/2022
EXPIRY DATE: (mm/dd/yyyy) 10/04/2027
RECOMMENDED STORAGE: Store ampoule in a cool, dark place

DOCUMENTATION/ DATA ATTACHED:

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

ADDITIONAL INFORMATION:

- See page 2 for further details.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Certified By: _____ **Date:** 10/14/2022 (mm/dd/yyyy)
B.G. Chittim, General Manager

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

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

INTENDED USE:

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

HANDLING:

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

SYNTHESIS / CHARACTERIZATION:

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

HOMOGENEITY:

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

UNCERTAINTY:

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

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters $x_1, x_2, \ldots x_n$ on which it depends is:

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

QUALITY MANAGEMENT:

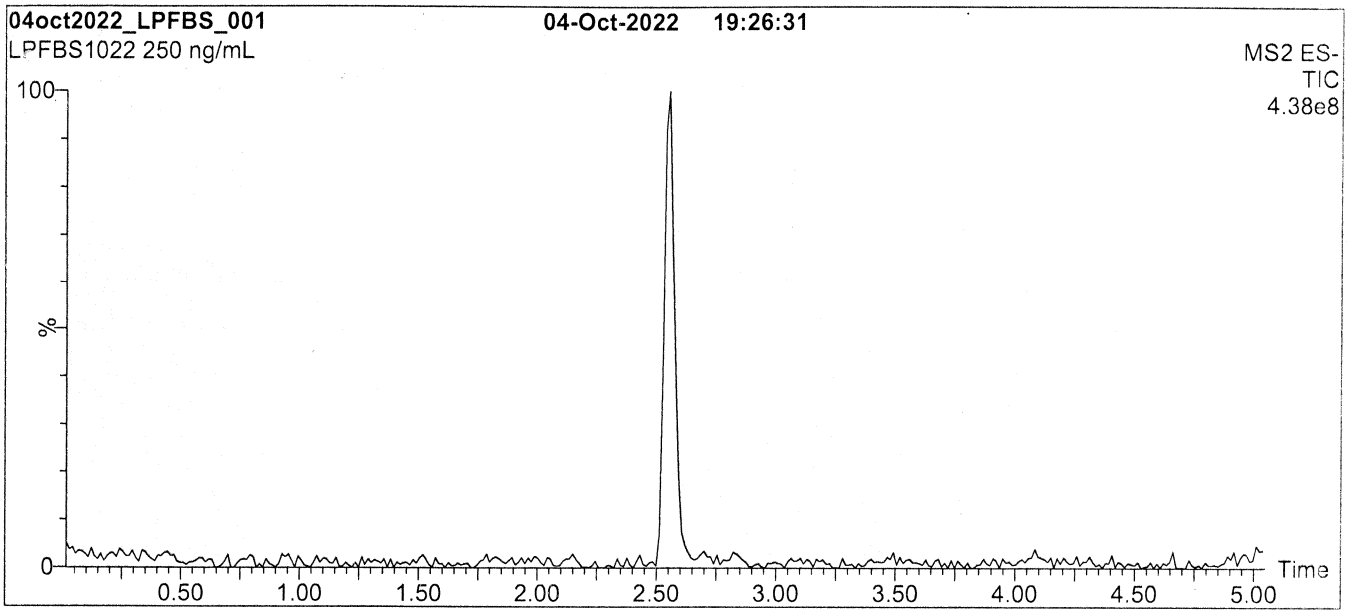
This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A1226), and ISO 17034 by ANSI National Accreditation Board (ANAB; AR-1523).

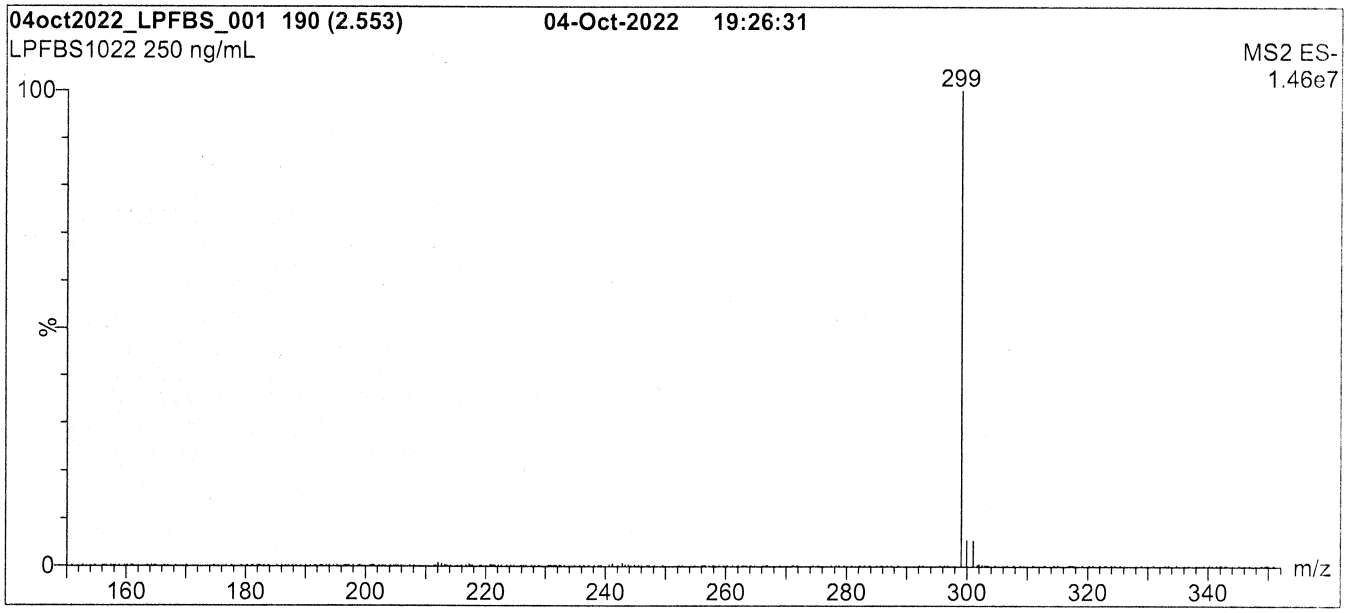
CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

Figure 1: **L-PFBS; LC/MS Data (Full Scan and Mass Spectrum)**





Conditions for Figure 1:

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

Chromatographic Conditions:

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

Mobile phase: Gradient
Start: 60% H_2O / 40% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions over 0.75 min.
Time: 12 min

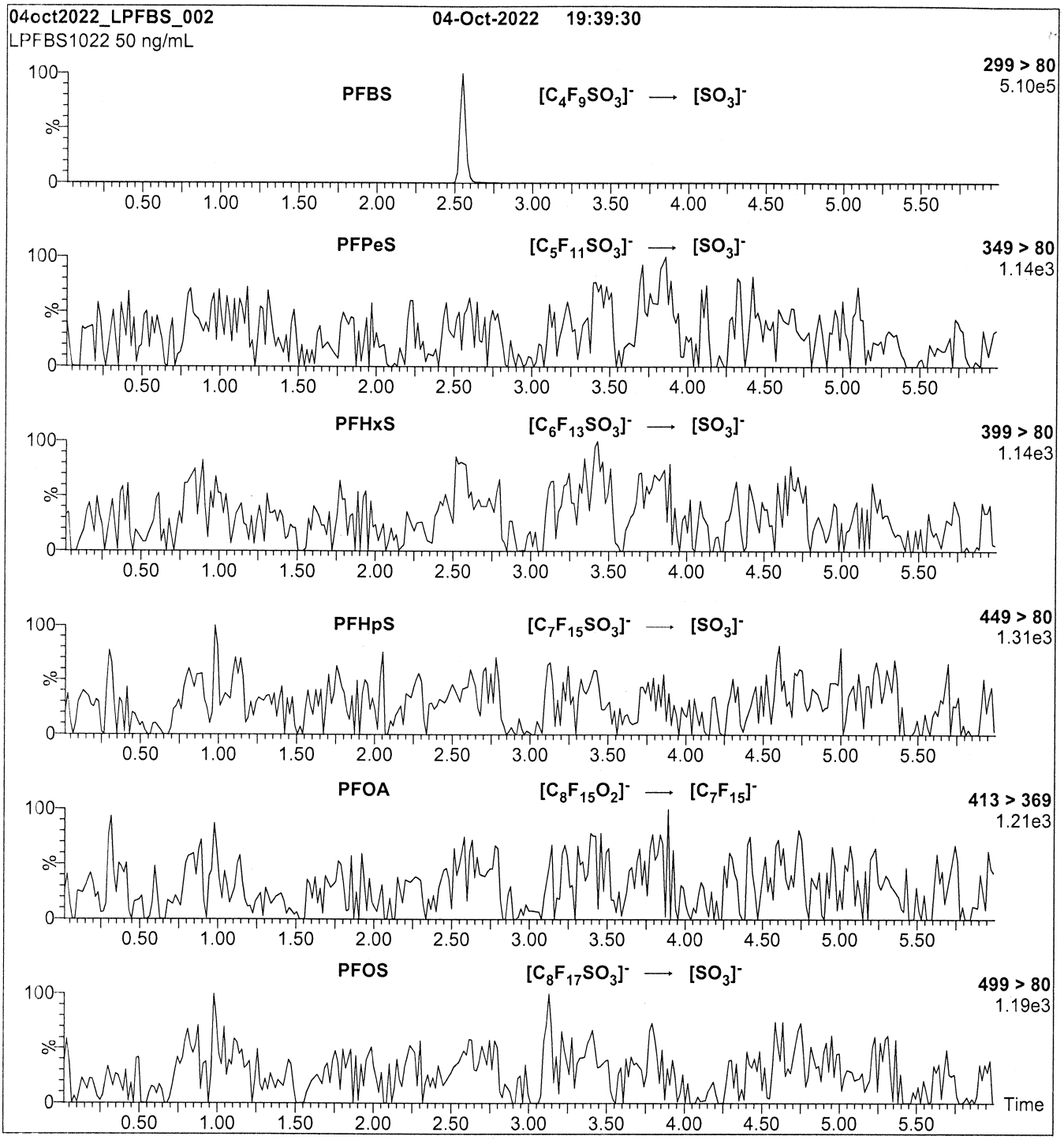
Flow: 300 µL/min

MS Parameters:

Experiment: Full Scan (150 - 850 amu)

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = 10.00
Desolvation Temperature (°C) = 500
Desolvation Gas Flow (L/hr) = 1000

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



Conditions for Figure 2:

Injection:	On-column (L-PFBS)
Mobile phase:	Same as Figure 1
Flow:	300 µL/min

MS Parameters:

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

Attachment 5

Native PFAS Intermediate A								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Native PFAS Intermediate A (ng/ml)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.1	10	500
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.05		250
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.025		125
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.025		125
Wellington	PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.025		125
Wellington	PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.025		125
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.025		125
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000	0.025		125
Wellington	PFDoA	Perfluorododecanoic acid	307-55-1	PFDoA	50000	0.025		125
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.025		125
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.025		125
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.025		111
Wellington	L-PFPeS	Perfluoropentansulfonic acid	2706-91-4	PFPeS	46900	0.025		117
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.025		114
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.025		119
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.025		117
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.025		120
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.025		121
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400	0.025		121
Wellington	4;2 FTS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	46700	0.1		467
Wellington	6;2 FTS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	47400	0.1		474
Wellington	8;2 FTS	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	47900	0.1		479
Wellington	FOSA-I	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.025		125
Wellington	N-MeFOSA-M	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	50000	0.025		125
Wellington	N-EtFOSA-M	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	50000	0.025		125
Wellington	br-NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.025		125
Wellington	br-NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.025		125
Wellington	N-MeFOSE-M	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	50000	0.25		1250
Wellington	N-EtFOSE-M	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	50000	0.25		1250
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	50000	0.1		500
Wellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	47100	0.1		471
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.05		250
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50000	0.05		250
Wellington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	50000	0.05		250
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.1		466
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.1		471
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.05		223
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000	0.625		3125
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000	0.625		3125

Attachment 6

Native PFAS Intermediate B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. Native PFAS Intermediate B (ng/ml)**
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.1	10	5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			2.5
	Perfluorohexanoic acid	307-24-4	PFHxA	125			1.25
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			1.25
	Perfluorooctanoic acid	335-67-1	PFOA	125			1.25
	Perfluorononanoic acid	375-95-1	PFNA	125			1.25
	Perfluorodecanoic acid	335-76-2	PFDA	125			1.25
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			1.25
	Perfluorododecanoic acid	307-55-1	PFDoA	125			1.25
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			1.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			1.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			1.11
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117.25			1.17
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75			1.14
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			1.19
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5			1.17
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			1.20
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5			1.21
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			1.21
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			4.67
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			4.74
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			4.79
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			1.25
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			1.25
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			1.25
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			1.25
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			12.5
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			12.5
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			5
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			4.71
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			2.5
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			2.5
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			2.5
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466			4.66
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471			4.71
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			2.23
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			6.25
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			31.3
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			31.3

Attachment 7

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

Attachment 8

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

Attachment 9

Internal Standard Mix								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Concentration (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. Internal Standard Mix (ng/ml)**
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	50000	50	10	250
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	50000	25		125
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	50000	12.5		62.5
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	47800	25		120
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	50000	12.5		62.5
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	50000	25		125
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	47300	25		118

Attachment 10

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

Attachment 11

1633 Initial Calibration Standard Concentrations							
	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Compound Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
PFpeA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFHxA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFHpA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFOA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFNA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFUnA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDoA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTrDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTeDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFBS	0.044	0.111	0.276	0.553	1.11	2.76	13.8
PFPeS	0.047	0.117	0.293	0.586	1.17	2.93	14.7
PFHxS	0.046	0.114	0.284	0.569	1.14	2.84	14.2
PFHpS	0.048	0.119	0.298	0.595	1.19	2.98	14.9
PFOS	0.047	0.117	0.291	0.583	1.17	2.91	14.6
PFNS	0.048	0.120	0.300	0.600	1.20	3.00	15.0
PFDS	0.048	0.121	0.301	0.603	1.21	3.01	15.1
PFDoS	0.048	0.121	0.303	0.605	1.21	3.03	15.1
4:2 FTS	0.187	0.467	1.17	2.34	4.67	11.7	58.4
6:2 FTS	0.190	0.474	1.19	2.37	4.74	11.9	59.3
8:2 FTS	0.192	0.479	1.20	2.40	4.79	12.0	59.9
PFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
NEtFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
HFPO-DA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
DONA	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFMPA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFMBA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
NFDHA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
9Cl-PF3ONS	0.186	0.466	1.17	2.33	4.66	11.7	58.3
11Cl-PF3OUdS	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFEESA	0.089	0.223	0.56	1.11	2.23	5.56	27.8
3:3 FTCA	0.250	0.625	1.56	3.13	6.25	15.6	78.1
5:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
7:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
13C4-PFBA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
13C5-PFPeA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C5-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C4-PFHpA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C8-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C9-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C6-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C7-PFUnA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFDoA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFTeDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C3-PFBS	0.581	0.581	0.581	0.581	0.581	0.581	0.581
13C3-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591
13C8-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C8-PFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
D5-NEtFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-4:2FTS	1.17	1.17	1.17	1.17	1.17	1.17	1.17
13C2-6:2FTS	1.19	1.19	1.19	1.19	1.19	1.19	1.19
13C2-8:2FTS	1.20	1.20	1.20	1.20	1.20	1.20	1.20
13C3-HFPO-DA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
D7-NMeFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
D9-NEtFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
D5-NEtFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C3-PFBA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C4-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C2-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C4-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C5-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
18O2-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591

Attachment 12

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

Attachment 13

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

Attachment 14

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

Attachment 15

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

Attachment 16

PFAS Linear/Branched Mix B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. PFAS Linear/Branched Mix B (ng/ml)**
PFAS Linear/Branched Mix A	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	500	0.5	2	125
	Perfluorooctanoic acid	335-67-1	PFOA	500			125
	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500			125
	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500			125
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500			125
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			125
	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			125
	Perfluoro-7-methyloctanoic acid	15899-31-7	PF7MOA	500			125
	Perfluorononanoic acid	375-95-1	PFNA	500			125

Attachment 17

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

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

Attachment 18

1633 ICV Working Standard							
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume* (ml)	Final Conc. 1633 ICV Working Standard (ng/ml)
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.01	2	2.50
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			1.25
	Perfluorohexanoic acid	307-24-4	PFHxA	125			0.625
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			0.625
	Perfluorooctanoic acid	335-67-1	PFOA	125			0.625
	Perfluorononanoic acid	375-95-1	PFNA	125			0.625
	Perfluorodecanoic acid	335-76-2	PFDA	125			0.625
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			0.625
	Perfluorododecanoic acid	307-55-1	PFDoA	125			0.625
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			0.625
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			0.625
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			0.553
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117			0.586
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	114			0.569
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			0.595
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	117			0.583
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			0.600
	Perfluorodecanesulfonic acid	335-77-3	PFDS	121			0.603
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			0.605
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			2.34
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			2.37
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			2.40
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			0.625
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			0.625
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			0.625
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			0.625
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			0.625
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			6.25
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			6.25
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			2.50
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			2.36
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			1.25
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			1.25
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			1.25
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466			2.33
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471			2.36
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			1.11
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			3.13
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			15.6
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			15.6
Extraction Standard Mix	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500	0.01		2.5
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250			1.25
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125			0.625
	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	116			0.581
	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	118			0.591
	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	120			0.598
	Perfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250			1.25
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	250			1.25
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	234			1.17
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	238			1.19
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	240			1.20
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	500			2.5
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	1250			6.25
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250			6.25
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
Internal Standard Mix	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250	0.01		1.25
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	120			0.598
	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	118			0.591

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

ATTACHMENT 21

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

	Aqueous Matrix [1, 2]			
	IPR			
Compounds	Recovery (%)	RSD (%)	OPR Recovery (%)	LLOPR Recovery (%)
PFBA	60 – 147	20	58 – 148	44 - 157
PFPeA	56 – 150	20	54 – 152	57 - 148
PFHxA	59 – 148	25	55 – 152	62 - 149
PFHpA	60 – 149	25	54 – 154	56 - 150
PFOA	55 – 158	25	52 – 161	57 - 161
PFNA	64 – 144	25	59 – 149	53 - 157
PFDA	57 – 142	25	52 – 147	43 - 158
PFUnA	54 – 153	30	48 – 159	50 - 155
PFDoA	73 – 133	25	64 – 142	60 - 141
PFTrDA	52 – 145	25	49 – 148	52 - 140
PFTeDA	49 - 158	25	47 – 161	52 - 156
PFBS	66 – 141	20	62 – 144	63 - 145
PFPeS	66 – 144	25	59 – 151	58 - 144
PFHxS	62 – 141	25	57 – 146	44 - 158
PFHpS	59 – 148	25	55 – 152	51 - 150
PFOS	61 – 145	20	58 – 149	43 - 162
PFNS	57 – 143	25	52 – 148	46 - 151
PFDS	56 – 142	25	51 – 147	50 - 144
PFDoS	41 – 140	30	36 – 145	30 - 138
4:2FTS	77 – 135	25	67 – 146	52 - 158
6:2FTS	75 – 137	30	61 – 151	48 - 158
8:2FTS	79 – 136	30	63 – 152	46 - 165
PFOSA	65 – 144	20	61 – 148	47 - 163
NMeFOSA	76 – 132	25	63 – 145	54 - 155
NEtFOSA	75 – 129	25	65 – 139	49 - 156
NMeFOSAA	69 – 134	25	58 – 144	32 - 160
NEtFOSAA	65 – 140	25	59 – 146	51 - 154
NMeFOSE	79 – 129	20	71 – 136	56 - 151
NEtFOSE	79 – 126	25	69 – 137	60 - 147
HFPO-DA	72 – 135	25	63 – 144	58 - 154
ADONA	75 – 138	20	68 – 146	61 - 148
PFMPA	55 – 141	25	51 – 145	48 - 150
PFMBA	59 – 145	20	55 – 148	49 - 154
NFDHA	63 – 146	35	48 – 161	47 - 160
9Cl-PF3ONS	72 – 140	30	56 – 156	44 - 167
11Cl-PF3OUdS	61 – 140	35	46 – 156	36 - 158
PFEESA	57 – 149	20	56 – 151	56 - 144
3:3FTCA	66 – 126	20	62 – 129	32 - 161
5:3FTCA	68 – 130	20	63 – 134	39 - 156
7:3FTCA	55 – 133	25	50 – 138	36 - 149
$^{13}C_4$-PFBA	10 – 130	30	10-130	10-130
$^{13}C_5$-PFPeA	35 – 150	30	40 -150	40 -150
$^{13}C_5$-PFHxA	55 – 150	30	40 -150	40 -150
$^{13}C_4$-PFHpA	55 – 150	30	40 -150	40 -150
$^{13}C_8$-PFOA	60 – 140	30	30-140	30-140
$^{13}C_9$-PFNA	55 – 140	30	30-140	30-140
$^{13}C_6$-PFDA	50 – 140	30	20-140	20-140

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

Compounds	Aqueous Matrix [1, 2]			
	IPR		OPR Recovery (%)	LLOPR Recovery (%)
	Recovery (%)	RSD (%)		
$^{13}C_7$-PFUnA	30 – 140	30	20-140	20-140
$^{13}C_2$-PFDoA	10 – 150	30	10-150	10-150
$^{13}C_2$-PFTeDA	10 – 130	30	10-130	10-130
$^{13}C_3$-PFBS	55 – 150	30	25-150	25-150
$^{13}C_3$-PFHxS	55 – 150	30	25-150	25-150
$^{13}C_8$-PFOS	45 – 140	30	20-140	20-140
$^{13}C_2$-4:2FTS	60 – 200	30	25-200	25-200
$^{13}C_2$-6:2FTS	60 – 200	30	25-200	25-200
$^{13}C_2$-8:2FTS	50 – 200	30	25-200	25-200
$^{13}C_8$-PFOSA	30 – 130	30	10-130	10-130
D_3-NMeFOSA	15 – 130	30	10-130	10-130
D_5-NEtFOSA	10 – 130	30	10-130	10-130
D_3-NMeFOSAA	45 – 200	30	10-200	10-200
D_5-NEtFOSAA	10 – 200	30	10-200	10-200
D_7-NMeFOSE	10 – 150	30	10-150	10-150
D_9-NEtFOSE	10 – 150	30	10-150	10-150
$^{13}C_3$-HFPO-DA	25 – 160	30	25-160	25-160

[1] The recovery limits apply to the target analyte results for IPR, OPR, and LLOPR samples for wastewater matrices. Data for this matrix type are derived from the multi-laboratory validation study and are therefore the limits required for this method.

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

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

EIS Compound	Recovery Range (%)
$^{13}C_4$-PFBA	10 – 130 *
$^{13}C_5$-PFPeA	35 - 150
^{13}C5-PFHxA	55 - 150
$^{13}C_4$-PFHpA	55 - 150
$^{13}C_8$-PFOA	60 - 140
$^{13}C_9$-PFNA	55 - 140
$^{13}C_6$-PFDA	50 - 140
$^{13}C_7$-PFUnA	30 - 140
$^{13}C_2$-PFDoA	10 - 150
$^{13}C_2$-PFTeDA	10 – 130 *
$^{13}C_3$-PFBS	55 - 150
$^{13}C_3$-PFHxS	55 - 150
$^{13}C_8$-PFOS	45 - 140
$^{13}C_2$-4:2FTS	60 – 200 *
$^{13}C_2$-6:2FTS	60 - 200 *
$^{13}C_2$-8:2FTS	50 – 200 *
$^{13}C_8$-PFOSA	30 – 130
D_3-NMeFOSA	15 – 130
D_5-NEtFOSA	10 – 130
D_3-NMeFOSAA	45 – 200 *
D_5-NEtFOSAA	10 – 200
D_7-NMeFOSE	10 – 150 *
D_9-NEtFOSE	10 – 150 *
$^{13}C_3$-HFPO-DA	25 - 160

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

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

Revision Log

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

Reference

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

Cross Reference

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

Document	Document Title
NC-QA-015	Support Equipment Calibration and Verification
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
QA-003	Quality Control Program
NC-QA-017	Standards and Reagents
NDSC-QA-QP44940	Calibration Curves and the Selection of Calibration Points
NDSC-US-EHSQP46060	NDSC EH&S Manual
NC-QAM-001	QA Manual

Scope

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

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

Analyte	Acronym	CAS#
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA, PES	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

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

Interferences

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

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.

3. All parts of the SPE manifold must be cleaned every 2 weeks per manufacturer recommendations.

Safety Precautions and Waste Handling

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

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

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

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

Personnel Training and Qualifications

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

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

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

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

Sample Collection, Preservation, and Handling

A. Sample Collection

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

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

B. Sample Storage and Shipment

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

2. Solid and Biosolid samples stored in the lab must be protected from light and held at a temperature of 0° to 6°C, or ≤ -20°C, until extraction.

3. Solid and Biosolid samples must be extracted within 90 days. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light.

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

Apparatus and Equipment

A. Apparatus

1. Specimen container; PP; natural; graduated; non-sterile; separate 1/4-turn red screwcap; 4 oz; 600 EA, Greenwood Products, Inc. Part No. HP30011

2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 14955237 or equivalent

3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288 or equivalent.

4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 0125-1902-PC.

5. Analytical Balance – Capable of weighing to 0.0001 g

6. Top-Loading Balance – Capable of weighing to 0.01 g

7. Waters Oasis WAX 150 mg/6cc (part # 186002493) with 20 mg loose carbon added.

8. Phenomenex Strata PFAS WAX/GCB, 500mg/50mg/6cc (part # CS0-9208) or equivalent. This cartridge incorporates graphitized carbon.

9. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product # 54258-U.

10. SPE Tube Adapter - Millipore-Sigma; Product # 57020-U.

11. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.

12. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.

13. Centrifuge – "Thermo Sorvall ST4" with TX-750 rotor or equivalent, capable of a minimum rotational force of 3800 rcf.

14. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent.

15. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. FisherScientific cat # 14-287-150, or equivalent.

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

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

18. Pipettes – Disposable transfer. Fisher Scientific, Cat. No. 13-711-7M

19. Vortex mixer, variable speed, Fisher Scientific or equivalent.

20. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control. Organomation, Inc. Cat. #11250-NT, or equivalent.

21. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTPF08831.

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

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

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

25. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent

26. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125

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

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

29. Syringe filter - Acrodisc, Syringe Filter, GHP,13 mm, 0.2 µm, Aqueous, 100/pkg, Part # WAT097962.

30. Silanized glass wool (Sigma-Alrich, Cat #20411 or equivalent)

31. Syringe filter - Acrodisc, Syringe Filter, 25 mm, 0.2 µm, 50/pkg, Fisher part # 50786068.

32. Variable speed mixing table (Fisherbrand™ Nutating mixer or equivalent)

33. Evaporation/concentrator tubes: 60 mL clear glass vial, 30x125 mm, without caps (Wheaton Cat # W226060 or equivalent).

34. Wooden Tongue Depressors - Fisher; Cat. # 11-700-555, or equivalent.

35. Wheaton Bottle, 15ml, Narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Cat. No. 209044SP, or equivalent

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

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

2. Promochrom automated extraction unit, model SPE-03 or equivalent

a. Promochrom filters, catalog # F-HC-30

3. HPLC columns

   a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-YO or equivalent

   b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

1. Methanol (MeOH) – J.T. Baker Ultra Resi-Analyzed, Cat. No. UN1230 or equivalent
2. Acetonitrile (ACN) – J.T. Baker Ultra Resi-Analyzed, Cat. No. UN1648 or equivalent
3. Water – Fisher Scientific, Optima, Cat. No. W6-4 or equivalent
4. Ammonium acetate – Fisher Scientific, Cat. No. A637-500 or equivalent
5. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
6. Methanolic ammonium hydroxide (0.3%) – add ammonium hydroxide (10 mL, 30%) to methanol (990 mL), store at room temperature, replace after 1 month
7. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
8. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
9. Acetic Acid – ACS grade or equivalent, store at room temperature
10. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
11. Formic acid -(greater than 96% purity or equivalent), store at room temperature.

    a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years

    b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years

c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years

d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years

e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years

12. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.

13. Solids reference matrix – Ottawa or reagent-grade sand

14. 20 mM ammonium acetate solution in 95:5(v/v) Milli-Q water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL Milli-Q water and mix well to dissolve the ammonium acetate. Add 100 mL acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

15. 20 mM ammonium acetate solution in 90:10 acetonitrile/Milli-Q water – Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 200 mL of Milli-Q water and mix well to dissolve the Ammonium Acetate. Add 1800 mL of acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

B. Standards:

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

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

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

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

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

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

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS

extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

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

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

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

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 04*.
If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

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

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

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

Attachment 05 - Native PFAS Intermediate A
Attachment 06 - Native PFAS Intermediate B
Attachment 07 - Native PFAS Intermediate M
Attachment 08 - Extraction Standard Mix
Attachment 09 - Internal Standard Mix
Attachment 10 - Initial Calibration Standards Preparation
Attachment 11 - Initial Calibration Standards Concentrations
Attachment 12 - Bile Salts Stock Solutions
Attachment 13 - Bile Salts Working Solution A
Attachment 14 - Bile Salts Working Solution B
Attachment 15 - PFAS Linear/Branched Mix A
Attachment 16 - PFAS Linear/Branched Mix B
Attachment 17 - Linear/Branched Bile Salts Solution
Attachment 18 - ICV Working Standard

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit(quadratic). In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.

2. Analyze a Cal4 level standard that contains Bile Salts retention time marker and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

   Example Initial Calibration Sequence:
   1. Instrument Blank
   2. Instrument Blank
   3. Instrument Blank
   4. CAL 1
   5. CAL 2
   6. CAL 3
   7. CAL 4
   8. CAL 5
   9. CAL 6
   10. CAL 7
   11. ICB (Instrument Blank)
   12. ICV
   13. MDL
   14. WDM (Linear Branched/Bile Salts standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 02* for additional information about compound relationships.

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	

PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	

11Cl-PF3OUdS	

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

4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic Acid (TCDCA), and Tauroursodeoxycholic Acid (TUDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

5. Fit the curve

   a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

   b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for the compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.

   c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

   d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

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

6. Initial Calibration Verification (ICV)

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

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

2. Acceptance criteria

   a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV

recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

A. Sample Preparation

NOTE: Prior to weighing out samples, thoroughly mix each sample using a wooden tongue depressor or stainless steel spoon to ensure a homogeneous sample matrix. Stir from the bottom to the top in a circular motion along the sides of the jar, breaking particles to less than 1 mm by pressing against the side of the container. Remove rocks, invertebrates, and foreign objects. Vegetation can either be removed or cut into smaller pieces based on project requirements.

1. On a calibrated, top-loading balance, accurately weigh 1.25 ± 0.10g (dry weight; note: maximum 10g sample aliquot) (0.5 g for biosolids(dry weight; note: maximum 1g sample aliquot)) of solid sample into a tared, labeled 50-mL centrifuge tube using a disposable polypropylene spatula. Record sample weight in the prep entry system.

2. For each batch - maximum 20 samples - include the following quality control samples:
   a. Method Blank: Weigh 1.25 ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 g for biosolids) of reagent water
   b. LCS: Fortify 1.25g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 mL for biosolids) reagent water and spiked with 200 µL of Native 1633 Mid-Level Spike Solution (PFAS Native M).
   c. LLCS: Fortify 1.25g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 mL (0.25 mL for biosolids) reagent water and spiked with 200 µL of Native 1633 Low-Level Spike Solution (PFAS Native B).
   d. Matrix Spike/Matrix Spike Duplicate (MS/MSD): Fortify 1.25g ± 0.10 g(dry weight; note: maximum 10g sample aliquot) (0.5 g for biosolids(dry weight; note: maximum 1g sample aliquot)) of sample as specified in sample preparation log with 200 µL of Native 1633 Mid-Level Spike Solution (PFAS Native M).

3. Add 25 µl working labeled extraction standard spike solution (PFAS EIS Mix) to each sample/QC tube.

4. Cap and vortex for approximately 30 seconds.

5. Allow samples/QC to equilibrate for at least 30 minutes.

6. Add 10 mL of 0.3% methanolic ammonium hydroxide to each centrifuge tube.

7. Cap and vortex

8. Shake for 30 minutes on a variable speed mixing table

9. Centrifuge @ 3000 rcf for 10 minutes and transfer supernatant to a clean 50 mL polypropylene centrifuge tube.

10. Add 15 mL of 0.3% methanolic ammonium hydroxide to the remaining solid sample in each centrifuge tube. Cap and vortex.

11. Shake for 30 minutes on a variable speed mixing table

12. Centrifuge @ 3000 rcf for 10 minutes and decant the supernatant from the second extraction into the centrifuge tube with the supernatant from the first extraction.

13. Add another 5 mL of 0.3% methanolic ammonium hydroxide to the remaining sample in each centrifuge tube.

14. Shake by hand to disperse.

15. Immediately decant the supernatant from the third extraction into the centrifuge tube with the supernatant from the first and second extraction.

16. Centrifuge for 10 minutes @ 3000 rcf.

17. Decant the extract from each tube into a clean, labeled, 125 ml HDPE bottle. Bring the 125-mL HDPE container up to 125 mL with Milli-Q water.

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

19. Invert container to thoroughly mix.

B. Solid Phase Extraction (SPE) - Manual Sample Extraction

1. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

2. Condition each SPE cartridge with the following reagents in the following order:

    a. 15 mL 1% methanolic ammonium hydroxide

    b. 5 mL 0.3M formic acid

    c. Discard conditioning eluent(s)

3. Label each sample bottle, cap and reservoir with the same number to insure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

4. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges. Allow full volume to pass through each cartridge by gravity, if possible. Apply light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.

5. After full volume has passed through the cartridges, dry the cartridges with vacuum - no more than 15" of Hg - for approximately five minutes. After five minutes, visually inspect the cartridge to determine if the sorbent is dry. This done by comparing the cartridge to a visual standard (an unused SPE cartridge). If the sorbent is not dry, continue to check at one minute intervals until the cartridge is dry.

6. Discard the waste and rinse the waste reservoir with DI water. Wipe each needle with a Kim-wipe/methanol.

7. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5ml of **1:1 0.1M formic acid/methanol** and pass the rinses through the cartridge using vacuum. Dry the cartridge by pulling air through for 15 seconds. Discard the rinse solution.

8. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge.

9. Rinse the inside of the evaporation/concentrator tube using 5mL of 1% methanolic ammonium hydroxide.

10. Using a glass pipette, transfer the rinse to the SPE reservoirs, washing the walls of the reservoirs.

11. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

12. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

13. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix thoroughly.

14. Add 25 uL of Intenal Standard Spike Solution (PFAS NIS Mix) to each sample extract.

15. Bring each sample extract to final volume 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

16. Cap and vortex to mix. Using a syringe, push ~ 3mL of MeOH through each filter and allow to dry

17. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 6 mL polypropylene syringe. Take the plunger out and carefully decant ~1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter ~1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

18. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for dilution or reinjection if needed.

C. Solid Phase Extraction (SPE) – Automated Sample Extraction using Promochrom

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

1. Verify pH of all samples/QC(needs to be between pH 4.0 ± 0.5). Modify with 50% Formic acid or 30% NH4OH if necessary. (Record the lot number of the acid/base used). **NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.**

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

3. Check to make sure all reagent bottles are filled and that reagents are not expired.

4. Select "Clean sys-004" from the drop down menu to start the cleaning cycle prior to SPE. Make sure the "Clean" cartridges and bottles are attached for this cycle.

5. Remove "Clean" cartridges and attach the labeled cartridges containing graphitized carbon that will be used when running the samples. Disconnect the "Clean" bottles. Attach the sample bottle by twisting the bottle and not the cap. Place sample bottle upside down in corresponding numbered location on shaker. Poke two holes into the lip of each bottle. Load labeled centrifuge tubes in the moving tray beneath the cartridges.

6. Select "EPA 1633-004" from the drop down menu and press the green check mark to start the procedure. NOTE: N2 drydown should be 15 minutes long.

7. Once the cycle is complete, remove centrifuge tubes and discard the used cartridges and bottles. Reattach "Clean" bottles and cartridges, select "clean sys-004" from menu, select "start". This must be done between each batch and at the beginning and the end of the day.

8. Add 25ul of Acetic acid to sample extracts and vortex to mix thoroughly before capping tubes.

9. Add 25 uL of Internal Standard Spike Solution (PFAS NIS Mix) to each sample extract.

10. Bring each sample extract to final volume 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

11. Cap and vortex to mix. Using a syringe, push ~ 3mL of MeOH through each filter and allow to dry.

12. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 3 mL polypropylene syringe. Take the plunger out and carefully decant approximately 1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter approximately 1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

13. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for reinjection if needed.

D. LC/MS/MS Analysis

1. Mass Calibration and Tuning

   a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

   b. When masses fall outside of the ±0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Example acquisition method: See *Attachment 03*. Mass Transitions: See *Attachment 01*.

4. Instrument Sensitivity Check (ISC) and Instrument Blanks

   a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within ±30% of their true value.The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
   b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salts RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

   Example Sample Sequence:
   1. Instrument blank
   2. Instrument blank
   3. Instrument blank
   4. Instrument Sensitivity Check (CCV0 _CAL1)
   5. CCV 2_CAL4
   6. Linear Branched/ Bile Salt marker (WDM)
   7. Instrument Blank (ICB)
   8. Method Blank (MB)
   9. Low Level LCS (LLCS)
   10. LCS
   11. Sample (10 or less)
   12. CCV 3_CAL4
   13. Instrument Blank

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

    a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.

    b. If LLCS and LCS recoveries are acceptable, proceed to sample quantitation.

    c. If the LCS and LLCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS/LLCS must be reextracted.

    d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Calculate the relative percent difference(RPD) between the sample and the matrix duplicate(DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data

11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), revial a fresh aliquot and reanalyze. If the extraction standard recovery(ies) are still outside the QC limit(s), reanalyze the sample at a dilution. If the extraction standard recovery(ies) are still outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) are still outside the QC limit(s), narrate with an NCM and report the data.

12. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

13. Compare the retention times of all of the analytes, surrogates, and internals standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than

    a. 0.4 minutes for isotopically-labeled compounds

    b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound

    c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

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

14. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of 13C4-PFBA, 13C5-PFPeA, 13C4-PFHpA, 13C8-PFOA, 13C9-PFNA, 13C6-PFDA, 13C7-PFUnA, 13C2-PFDA,13C2-PFDoDA 13C2-PFTeDA, 13C8-PFOSA, D3-NMePFOSA, D5-NEtFOSAA, D3-NMeFOSAA, D5-NEtPFOSA, D7-NMePFOSAE, D9-NEtPFOSAE, 13C3-PFBA, 13C4-PFOA, 13C5-PFNA, 13C2-PFOA, 18O2-PFHxS, PFBA, PFECA F(PFMPA), PFECA A(PFMBA), NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required in prepped samples. Ion ratios must be in control in calibration solutions. If they are outside of limits, stop the analysis and correct the issues.

15. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.

16. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of Internal Standard Spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring greater than a 10x dilution should be reextracted using a reduced aliquot.

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

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

    On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

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

4. Sample Concentration

Sample concentration (ng/g) = (On-column concentration x Final Sample Volume x DF) ÷ Initial Sample Volume

5. Ion Ratio

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

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

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are method defined.

QC parameter	Lower acceptance limit	High acceptance limit
Extracted Internal standard (EIS)	20%	150%
Non-extracted Internal Standard (NIS)	>30% of the average NIS from the initial calibration	200%
Analyte recoveries LCS/LLCS/MS/MSD	40%	150%
Sample/Duplicate	</= 30%	NA

Note: lower acceptance limit for EIS cannot not be <20%, lower acceptance limit for analyte recovery cannot be <40%.

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

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

Quality Assurance/Quality Control

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

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

Attachment:
Attachment 01 - Mass Transitions (.doc)
Attachment 02 - Standard Relationships (.docx)
Attachment 03 - Acquisition Parameters (.pdf)
Attachment 04 - Example Certificate of Analysis (.pdf)
Attachment 05 - Native PFAS Intermediate A (.pdf)
Attachment 06 - Native PFAS Intermediate B (.pdf)
Attachment 07 - Native PFAS Intermediate M (.pdf)
Attachment 08 - Extraction Standard Mix (.pdf)
Attachment 09 - Internal Standard Mix (.pdf)
Attachment 10 - Initial Calibration Standards Preparation (.pdf)
Attachment 11 - Initial Calibration Standard Concentrations (.pdf)
Attachment 12 - Bile Salt Stock Solutions (.pdf)
Attachment 13 - Bile Salts Working Solution A (.pdf)
Attachment 14 - Bile Salts Working Solution B (.pdf)
Attachment 15 - PFAS Linear Branched Mix A (.pdf)
Attachment 16 - PFAS Linear Branched Mix B (.pdf)
Attachment 17 - Linear Branched Bile Salts Solution (.pdf)
Attachment 18 - ICV Working Standard (.pdf)

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Attachment: Attachment 08 - Extraction Standard Mix (pdf)
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Attachment: Attachment 12 - Bile Salt Stock Solutions (pdf)
Attachment: Attachment 13 - Bile Salts Working Solution A (pdf)
Attachment: Attachment 14 - Bile Salts Working Solution B (pdf)
Attachment: Attachment 15 - PFAS Linear Branched Mix A (pdf)
Attachment: Attachment 16 - PFAS Linear Branched Mix B (pdf)
Attachment: Attachment 17 - Linear Branched Bile Salts Solution (pdf)
Attachment: Attachment 18 - ICV Working Standard (pdf)

End of document

Version history

Version	Approval	Revision information	
0	25.SEP.2023		
1	03.NOV.2023		
2	20.NOV.2023		

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

Attachment 1

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

Attachment 1

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

Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

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

Extraction Standards

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

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

Native PFAS Compounds

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

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

Acquisition Method

1633

Mass Spec 12.500 min
Period 12.500 mi
-MRM
Integrated Valve
LC System
Equilibrate
Injection

Mass Spectrometer Method Properties

Period 1:

Scans in Period:	1250
Min. Dwell Time:	3 ms
Max. Dwell Time:	250 ms
Relative Start Time:	0.00 msec
Scheduled Ionization:	Off
Experiments in Period:	1
Use target Cycle Time:	No
Target Cycle Time:	N/A

Period 1 Experiment 1:

Scan Type:	MRM (MRM)
Scheduled MRM:	Yes
Polarity:	Negative
Scan Mode:	N/A
Ion Source:	Turbo Spray
sMRM Q1Q3 Resolution:	No
MRM detection window:	60 sec
Target Scan Time:	0.6000 sec
Resolution Q1:	Unit
Resolution Q3:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Step Size:	0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
212.800	168.900	2.71	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	84.900	2.94	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	2.98	DP	-50.00	-50.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	3.21	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	79.900	3.67	DP	-90.00	-90.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.100	3.32	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	134.900	3.84	DP	-70.00	-70.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	3.52	DP	-30.00	-30.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.41	DP	-110.00	-110.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.55	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

349.100	79.900	4.00	DP		-90.00	-90.00	PFPeS
				CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.68	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.85	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.30	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	3.96	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	3.98	DP	-90.00	-90.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.68	DP	-110.00	-110.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.12	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	5.22	DP	-90.00	-90.00	PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.43	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.40	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.61	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.72	DP	-100.00	-100.00	PFNS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.85	DP	-60.00	-60.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.58	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	6.14	DP	-110.00	-110.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.79	DP	-80.00	-80.00	NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	6.16	DP	-90.00	-90.00	PFDS
			CE	-130.00	-130.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.39	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	5.04	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.49	DP	-90.00	-90.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.87	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.94	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	7.08	DP	-90.00	-90.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.94	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	7.24	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.36	DP	-90.00	-90.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	619.000	6.30	DP	-60.00	-60.00	PFTrDA
			CE	-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.71	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.66	DP	-110.00	-110.00	PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.52	DP	-10.00	-10.00	PFECA B_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.41	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.55	DP	-40.00	-40.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.00	DP	-110.00	-110.00	PFPeS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.68	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.85	DP	-50.00	-50.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.30	DP	-90.00	-90.00	PFHxS_2

			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	3.98	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.68	DP	-100.00	-100.00	PFHpS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.12	DP	-50.00	-50.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	5.21	DP	-110.00	-110.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	4.43	DP	-50.00	-50.00	PFNA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	98.800	5.72	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	219.000	4.85	DP	-60.00	-60.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	80.800	4.58	DP	-100.00	-100.00	8:2 FTS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	483.000	4.79	DP	-90.00	-90.00	NMeFOSAA_2
			CE	-24.00	-24.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	98.800	6.16	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	269.100	5.39	DP	-70.00	-70.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	526.000	5.04	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	319.000	5.87	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

663.000	168.900	6.30	DP		-60.00	-60.00	PFTrDA_2
			CE		-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.71	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.21	DP	-40.00	-40.00	PFPeA_2
			CE	-55.00	-55.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	6.13	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	7.08	DP	-110.00	-110.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.35	DP	-90.00	-90.00	NEtPFOSA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	3.95	DP	-40.00	-40.00	DONA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.60	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.48	DP	-90.00	-90.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	2.98	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.40	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.83	DP	-70.00	-70.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.94	DP	-90.00	-90.00	PFDoS_2
			CE	-140.00	-140.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.71	DP	-40.00	-40.00	13C3-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.71	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.21	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.66	DP	-90.00	-90.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.41	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.54	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.54	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.68	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.85	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.30	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.30	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	3.97	DP	-90.00	-90.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.12	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.12	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	5.21	DP	-90.00	-90.00	13C4-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	79.900	5.21	DP	-90.00	-90.00	13C8-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.42	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	
519.100	474.100	4.85	DP	-60.00	-60.00	13C6-PFDA
			CE	-18.00	-18.00	
515.100	470.100	4.85	DP	-60.00	-60.00	13C2-PFDA
			CE	-18.00	-18.00	
529.100	80.900	4.58	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	
506.100	77.800	6.13	DP	-110.00	-110.00	13C8-PFOSA
			CE	-80.00	-80.00	
573.200	419.000	4.78	DP	-90.00	-90.00	d3-NMeFOSAA
			CE	-27.00	-27.00	
570.000	525.100	5.38	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	
589.200	419.000	5.02	DP	-90.00	-90.00	d5-NEtFOSAA

			CE	-30.00	-30.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.86	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.92	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	7.07	DP	-90.00	-90.00	d3-NMePFOSA
			CE	-37.00	-37.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	7.21	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.34	DP	-90.00	-90.00	d5-NEtPFOSA
			CE	-38.00	-38.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.70	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.54	DP	-40.00	-40.00	13C5-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.66	DP	-110.00	-110.00	13C3-PFBS_2
			CE	-35.00	-35.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.29	DP	-90.00	-90.00	13C3-PFHxS_2
			CE	-70.00	-70.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	5.20	DP	-110.00	-110.00	13C8-PFOS_2
			CE	-80.00	-80.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.41	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	3.97	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.58	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.68	DP	-75.00	-75.00	13C3-HFPODA_2
			CE	-10.00	-10.00	
Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

315.100	119.400	3.54	DP	-40.00	-40.00	13C2-PFHxA_2	
			CE	-31.00	-31.00		

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	5.21	DP	-110.00	-110.00	13C4-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.78	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.70	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.39	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
CAD:	10.00
EP	-10.00
CXP	-14.00

Acquisition Method

```
1633

Mass Spec  12.500 min
   Period  12.500 min
      -MRM
Integrated Valve
LC System
   Equilibrate
   Injection
```

Acquisition Information:

Acquisition Method:	1633.dam
Created:	Wednesday May 24 2023 14:48:39 PM
Last Modified:	Wednesday June 14 2023 11:54:50 AM
Comment:	
Synchronization Mode:	LC Sync
Auto-Equilibration:	Off
Acquisition Duration:	12min30sec
Number Of Scans:	1250
Periods In File:	1
Acquisition Module:	Acquisition Method
Software version	Analyst 1.7.3

Period 1:

Scans in Period:	1250
Min. Dwell Time:	3 ms
Max. Dwell Time:	250 ms
Relative Start Time:	0.00 msec
Scheduled Ionization:	Off
Experiments in Period:	1
Use target Cycle Time:	No
Target Cycle Time:	N/A

Period 1 Experiment 1:

Scan Type:	MRM (MRM)
Scheduled MRM:	Yes
Polarity:	Negative
Scan Mode:	N/A
Ion Source:	Turbo Spray
sMRM Q1Q3 Resolution:	No
MRM detection window:	60 sec
Target Scan Time:	0.6000 sec
Resolution Q1:	Unit
Resolution Q3:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Step Size:	0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
212.800	168.900	2.71	DP	-40.00	-40.00	PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
229.000	84.900	2.94	DP	-40.00	-40.00	PFECA F
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	177.000	2.98	DP	-50.00	-50.00	3:3 FTCA
			CE	-12.00	-12.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	219.000	3.21	DP	-40.00	-40.00	PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	79.900	3.67	DP	-90.00	-90.00	PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
279.000	85.100	3.32	DP	-40.00	-40.00	PFECA A
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	134.900	3.84	DP	-70.00	-70.00	PFEESA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	201.000	3.52	DP	-30.00	-30.00	PFECA B
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.41	DP	-110.00	-110.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.55	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	79.900	4.00	DP	-90.00	-90.00	PFPeS
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.68	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.85	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.30	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	3.96	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	3.98	DP	-90.00	-90.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.68	DP	-110.00	-110.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.12	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	5.22	DP	-90.00	-90.00	PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.43	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.40	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.61	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.72	DP	-100.00	-100.00	PFNS

			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.85	DP	-60.00	-60.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.58	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	6.14	DP	-110.00	-110.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.79	DP	-80.00	-80.00	NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	6.16	DP	-90.00	-90.00	PFDS
			CE	-130.00	-130.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.39	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	5.04	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.49	DP	-90.00	-90.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.87	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.94	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	7.08	DP	-90.00	-90.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.94	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	7.24	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.36	DP	-90.00	-90.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

663.000	619.000	6.30	DP		-60.00	-60.00	PFTrDA
			CE		-21.00	-21.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.71	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.66	DP	-110.00	-110.00	PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.52	DP	-10.00	-10.00	PFECA B_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.41	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.55	DP	-40.00	-40.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.00	DP	-110.00	-110.00	PFPeS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.68	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.85	DP	-50.00	-50.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.30	DP	-90.00	-90.00	PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	3.98	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.68	DP	-100.00	-100.00	PFHpS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.12	DP	-50.00	-50.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	5.21	DP	-110.00	-110.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	219.000	4.43	DP	-50.00	-50.00	PFNA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	98.800	5.72	DP	-100.00	-100.00	PFNS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	219.000	4.85	DP	-60.00	-60.00	PFDA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	80.800	4.58	DP	-100.00	-100.00	8:2 FTS_2
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	483.000	4.79	DP	-90.00	-90.00	NMeFOSAA_2
			CE	-24.00	-24.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	98.800	6.16	DP	-100.00	-100.00	PFDS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	269.100	5.39	DP	-70.00	-70.00	PFUnDA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	526.000	5.04	DP	-100.00	-100.00	NEtFOSAA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	319.000	5.87	DP	-60.00	-60.00	PFDoDA_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	168.900	6.30	DP	-60.00	-60.00	PFTrDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.71	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.21	DP	-40.00	-40.00	PFPeA_2
			CE	-55.00	-55.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	6.13	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	7.08	DP	-110.00	-110.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.35	DP	-90.00	-90.00	NEtPFOSA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	3.95	DP	-40.00	-40.00	DONA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.60	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.48	DP	-90.00	-90.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	2.98	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.40	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.83	DP	-70.00	-70.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.94	DP	-90.00	-90.00	PFDoS_2
			CE	-140.00	-140.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.71	DP	-40.00	-40.00	13C3-PFBA

			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.71	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.21	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.66	DP	-90.00	-90.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.41	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.54	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.54	DP	-30.00	-30.00	13C5-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.68	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.85	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.30	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.30	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	3.97	DP	-90.00	-90.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.12	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.12	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	5.21	DP	-90.00	-90.00	13C4-PFOS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

507.100	79.900	5.21	DP		-90.00	-90.00	13C8-PFOS
				CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.42	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
519.100	474.100	4.85	DP	-60.00	-60.00	13C6-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.100	470.100	4.85	DP	-60.00	-60.00	13C2-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	80.900	4.58	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
506.100	77.800	6.13	DP	-110.00	-110.00	13C8-PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
573.200	419.000	4.78	DP	-90.00	-90.00	d3-NMeFOSAA
			CE	-27.00	-27.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.100	5.38	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.200	419.000	5.02	DP	-90.00	-90.00	d5-NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.86	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.92	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	7.07	DP	-90.00	-90.00	d3-NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	7.21	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.34	DP	-90.00	-90.00	d5-NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.70	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.54	DP	-40.00	-40.00	13C5-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.66	DP	-110.00	-110.00	13C3-PFBS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.29	DP	-90.00	-90.00	13C3-PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	5.20	DP	-110.00	-110.00	13C8-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.41	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	3.97	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.58	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.68	DP	-75.00	-75.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	119.400	3.54	DP	-40.00	-40.00	13C2-PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	5.21	DP	-110.00	-110.00	13C4-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.78	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.70	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.39	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
CAD:	10.00
EP	-10.00

CXF -14.00

Integrated Valve Method Properties
Valve Diverter

	Total Time (min)	Position
1	1.0	B
2	8.0	A

LC Method Properties

LC system Equlibration time = 0.00 min
LC system Injection Volume = 10.00 ul

Pump

Model:	A: LC-40D XF B: LC-40D XR
Pump mode:	Binary gradient
Stop time:	12.50 min
<Gradient start adjustment>	
Mode:	None
<B.GE1>	
<General>	
Flow/Pressure:	Flow
Flow:	0.5000 mL/min
Time to reach the flow:	0.00 min
B.Conc:	5.0 %
B.Curve:	0
Pressure limits Minimum:	0 psi
Pressure limits Maximum:	6500 psi

<Gradient>

Time min	Flow mL/min	B.Conc %	B.Curve
0.00	0.5000	5.0	0
0.20	0.5000	5.0	0
3.00	0.5000	60.0	0
3.80	0.5000	60.0	0
6.70	0.5000	98.0	0
8.50	0.5000	98.0	0
8.70	0.6500	5.0	0
12.50	0.5000	5.0	0

<PumpA>	
<Mobile phase switching valve>	
Selected mobile phase port:	A
<Mobile phase settings>	
Set compressibility:	No

Port	Mobile phase name
A	Mobile phase A

<Advanced>	
LPGE cycle:	Standard
<PumpB>	

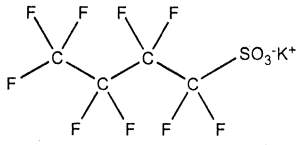
```
      <Mobile phase switching valve>
         Selected mobile phase port:                        A
      <Mobile phase settings>
         Set compressibility:                              No
      Port  Mobile phase name
      ----  -----------------
         A     Mobile phase A
      <Advanced>
         LPGE cycle:                                 Standard
<Autopurge settings>
   Execute autopurge before analysis:                      No
   <Autosampler purge settings>
      Execute sampler purge:                               No
   <Init conc-replacement>
      Use Init conc-replacement:                           No
   <Warm up>
      Wait time:                                       0 min
      B.GE1 flow:                                0.0000 mL/min
   <Execute after autopurge>
      Turn oven on after autopurge:                        No
      Turn pump on after autopurge:                        No
<Time program>
Autosampler
===========
Model:                                             SIL-40C XR
<General>
   Use Autosampler:                                       Yes
<Sample plate settings>
   Specify plate:                                       No
   Specify needle stroke:                               No
<Injection settings>
   Sampling speed:                                  1.0 µL/s
   Use cooler temperature:                               Yes
   Cooler temperature:                                15 °C
   Use air gap volume:                                  No
   Use low dispersion injection:                        No
<Acquisition cycle time optimization>
   Pretreatment start timing:                           Off
<Cut off loop settings>
   Cut off loop:                                        No
<Rinse settings>
   Rinse type:                                 External only
   Rinse mode:                  Before and after aspiration
   Rinse dip time:                                    0 s
   Rinse method:                              Rinse port only
   Rinse time:                                      2.0 s
   Rinsing speed:                                  35 µL/s
   Rinsing volume:                                500 µL
   Measuring line purge volume:                   100 µL
<Details>
   Wait for temperature equilibration before run (sample cooler):
                                                        No
```

<Pretreatment>
   Mode: Standard
<Time program>
Oven
====
Model: A: CTO-40C
<Column Oven A>
   <General>
      Use Column Oven: Yes
      Oven temperature: 40 °C
      Temperature limit(Maximum): 90 °C
      Wait for temperature equilibration before run: Off
   <Advanced>
      Fan speed: Auto
      Cooler mode: Auto
<Time program>
System Controller
==========
Model: CBM-40
<External output settings>
   Event 1: No
   Event 2: No
<Shutdown settings>
   Power off after shutdown: No
<Time program>
Time Command Value
 min
---- -------- ------
0.01   Event   1

WELLINGTON LABORATORIES

CERTIFICATE OF ANALYSIS
DOCUMENTATION

PRODUCT CODE:	L-PFBS	**LOT NUMBER:**	LPFBS1022
COMPOUND:	Potassium perfluoro-1-butanesulfonate		
STRUCTURE:		**CAS #:**	29420-49-3



MOLECULAR FORMULA:	$C_4F_9SO_3K$	**MOLECULAR WEIGHT:**	338.19
CONCENTRATION:	50.0 ± 2.5 µg/mL (K salt)	**SOLVENT(S):**	Methanol
	44.4 ± 2.2 µg/mL (PFBS acid)		
	44.2 ± 2.2 µg/mL (PFBS anion)		
CHEMICAL PURITY:	>98%		
LAST TESTED: (mm/dd/yyyy)	10/04/2022		
EXPIRY DATE: (mm/dd/yyyy)	10/04/2027		
RECOMMENDED STORAGE:	Store ampoule in a cool, dark place		

DOCUMENTATION/ DATA ATTACHED:

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

ADDITIONAL INFORMATION:

- See page 2 for further details.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Certified By: _____ **Date:** 10/14/2022 (mm/dd/yyyy)
B.G. Chittim, General Manager

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

Attachment "" to "US Eurofins Cleveland - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633 Page 67 of 84 "
Printed by Dorothy Love, d. Mon 20 Nov 2023 15:26 EST

INTENDED USE:

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

HANDLING:

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

SYNTHESIS / CHARACTERIZATION:

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

HOMOGENEITY:

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

UNCERTAINTY:

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

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters $x_1, x_2, \ldots x_n$ on which it depends is:

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

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

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

TRACEABILITY:

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

EXPIRY DATE / PERIOD OF VALIDITY:

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

LIMITED WARRANTY:

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

QUALITY MANAGEMENT:

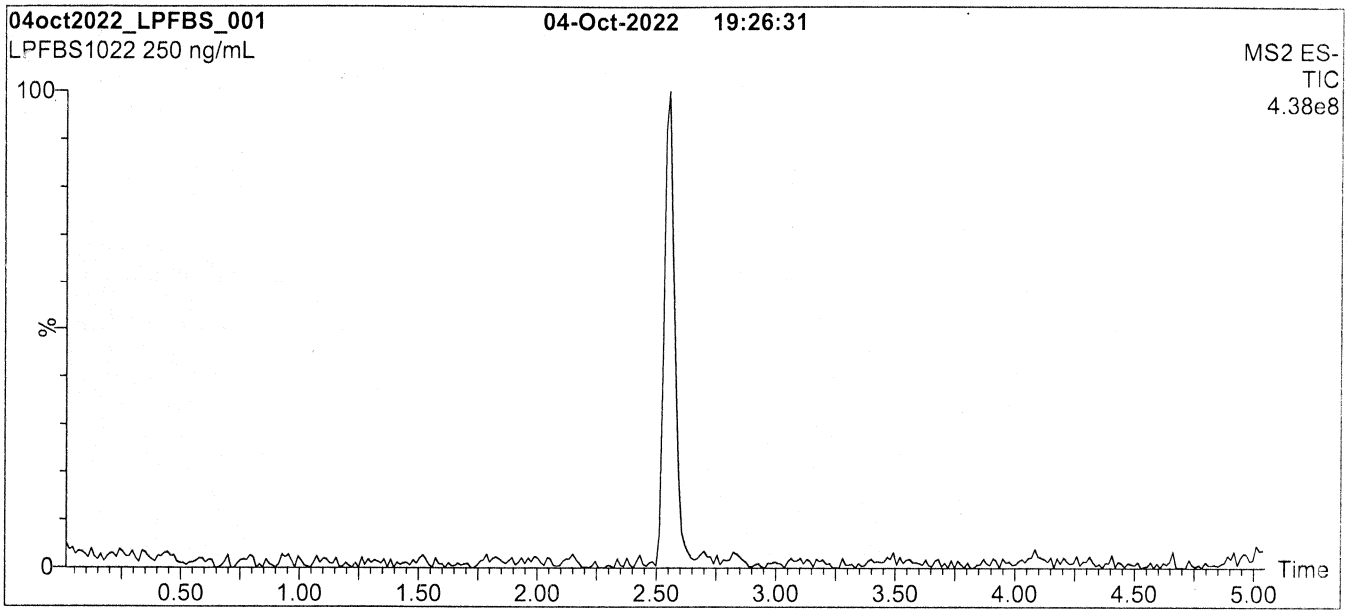
This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A1226), and ISO 17034 by ANSI National Accreditation Board (ANAB; AR-1523).

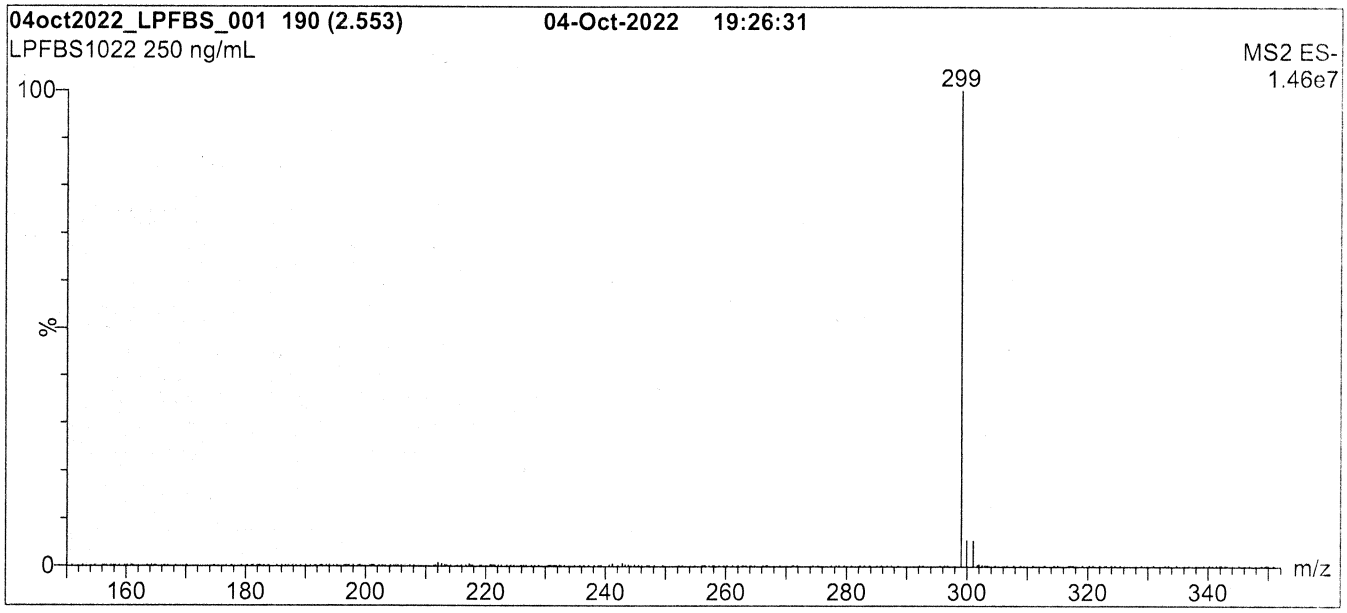
CALA Testing Accreditation No. A1226

ANAB ANSI National Accreditation Board ACCREDITED ISO 17034 REFERENCE MATERIAL PRODUCER

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

Figure 1: **L-PFBS; LC/MS Data (Full Scan and Mass Spectrum)**





Conditions for Figure 1:

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

Chromatographic Conditions:

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

Mobile phase: Gradient
Start: 60% H_2O / 40% (80:20 MeOH:ACN)
(both with 10 mM NH_4OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions over 0.75 min.
Time: 12 min

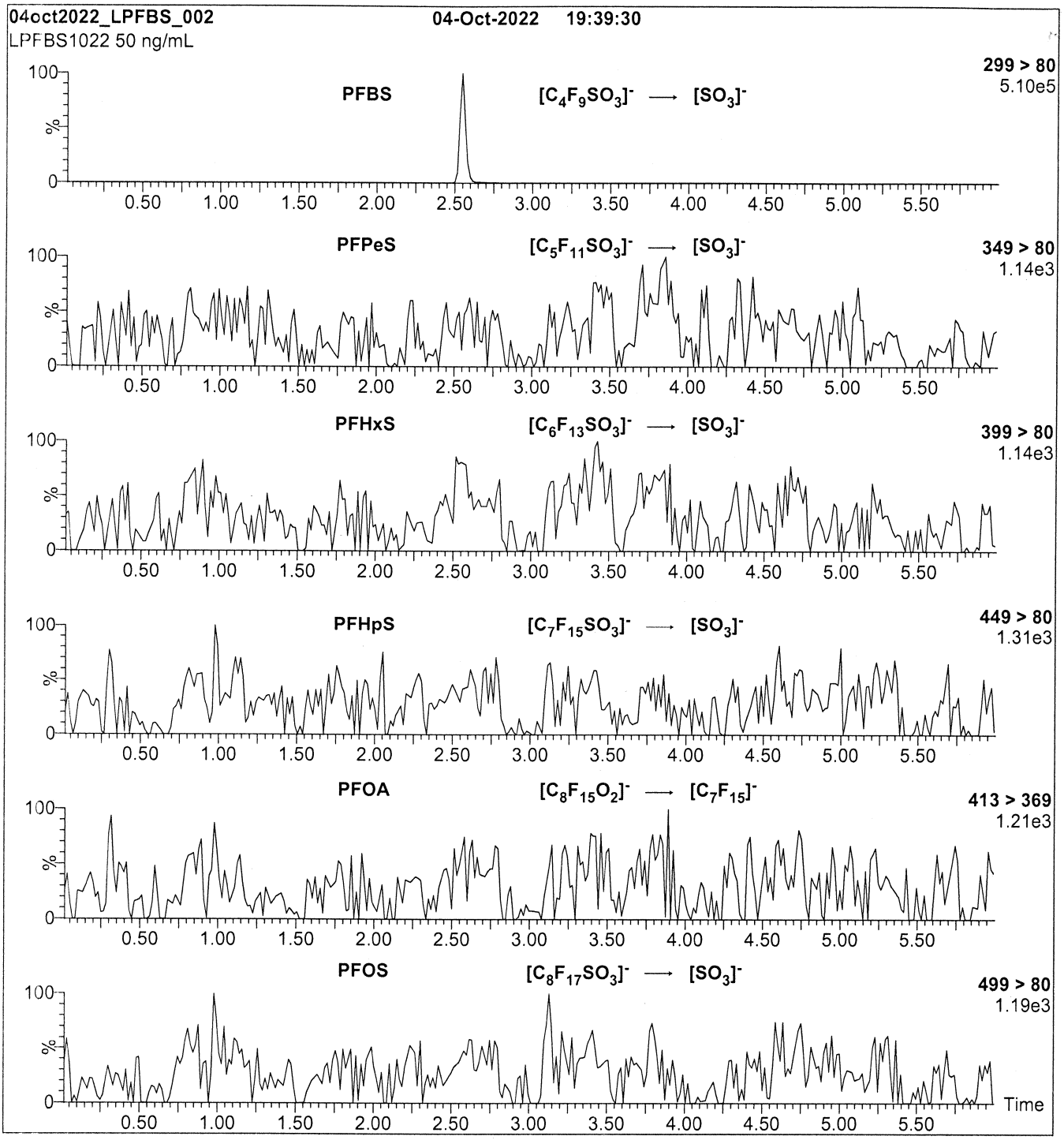
Flow: 300 µL/min

MS Parameters:

Experiment: Full Scan (150 - 850 amu)

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = 10.00
Desolvation Temperature (°C) = 500
Desolvation Gas Flow (L/hr) = 1000

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



Conditions for Figure 2:

Injection:	On-column (L-PFBS)
Mobile phase:	Same as Figure 1
Flow:	300 µL/min

MS Parameters:

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

Attachment 5

Native PFAS Intermediate A								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume (ml) Methanol	Final Conc. Native PFAS Intermediate A (ng/ml)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.1	10	500
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.05		250
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.025		125
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.025		125
Wellington	PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.025		125
Wellington	PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.025		125
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.025		125
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000	0.025		125
Wellington	PFDoA	Perfluorododecanoic acid	307-55-1	PFDoA	50000	0.025		125
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.025		125
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.025		125
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.025		111
Wellington	L-PFPeS	Perfluoropentansulfonic acid	2706-91-4	PFPeS	46900	0.025		117
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.025		114
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.025		119
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.025		117
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.025		120
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.025		121
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400	0.025		121
Wellington	4;2 FTS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	46700	0.1		467
Wellington	6;2 FTS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	47400	0.1		474
Wellington	8;2 FTS	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	47900	0.1		479
Wellington	FOSA-I	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.025		125
Wellington	N-MeFOSA-M	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	50000	0.025		125
Wellington	N-EtFOSA-M	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	50000	0.025		125
Wellington	br-NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.025		125
Wellington	br-NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.025		125
Wellington	N-MeFOSE-M	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	50000	0.25		1250
Wellington	N-EtFOSE-M	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	50000	0.25		1250
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	50000	0.1		500
Wellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	47100	0.1		471
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.05		250
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50000	0.05		250
Wellington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	50000	0.05		250
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.1		466
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.1		471
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.05		223
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000	0.625		3125
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000	0.625		3125

Attachment 6

Native PFAS Intermediate B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. Native PFAS Intermediate B (ng/ml)**
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.1	10	5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			2.5
	Perfluorohexanoic acid	307-24-4	PFHxA	125			1.25
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			1.25
	Perfluorooctanoic acid	335-67-1	PFOA	125			1.25
	Perfluorononanoic acid	375-95-1	PFNA	125			1.25
	Perfluorodecanoic acid	335-76-2	PFDA	125			1.25
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			1.25
	Perfluorododecanoic acid	307-55-1	PFDoA	125			1.25
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			1.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			1.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			1.11
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117.25			1.17
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75			1.14
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			1.19
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5			1.17
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			1.20
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5			1.21
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			1.21
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			4.67
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			4.74
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			4.79
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			1.25
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			1.25
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			1.25
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			1.25
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			12.5
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			12.5
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			5
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			4.71
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			2.5
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			2.5
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			2.5
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466			4.66
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471			4.71
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			2.23
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			6.25
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			31.3
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			31.3

Attachment 7

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

Attachment 8

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

Attachment 9

Internal Standard Mix								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Concentration (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. Internal Standard Mix (ng/ml)**
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	50000	50	10	250
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	50000	25		125
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	50000	12.5		62.5
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	47800	25		120
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	50000	12.5		62.5
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	50000	25		125
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	47300	25		118

Attachment 10

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

Attachment 11

1633 Initial Calibration Standard Concentrations							
	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Compound Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
PFpeA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFHxA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFHpA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFOA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFNA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFUnA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDoA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTrDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTeDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFBS	0.044	0.111	0.276	0.553	1.11	2.76	13.8
PFPeS	0.047	0.117	0.293	0.586	1.17	2.93	14.7
PFHxS	0.046	0.114	0.284	0.569	1.14	2.84	14.2
PFHpS	0.048	0.119	0.298	0.595	1.19	2.98	14.9
PFOS	0.047	0.117	0.291	0.583	1.17	2.91	14.6
PFNS	0.048	0.120	0.300	0.600	1.20	3.00	15.0
PFDS	0.048	0.121	0.301	0.603	1.21	3.01	15.1
PFDoS	0.048	0.121	0.303	0.605	1.21	3.03	15.1
4:2 FTS	0.187	0.467	1.17	2.34	4.67	11.7	58.4
6:2 FTS	0.190	0.474	1.19	2.37	4.74	11.9	59.3
8:2 FTS	0.192	0.479	1.20	2.40	4.79	12.0	59.9
PFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NEtFOSAA	0.050	0.125	0.313	0.625	1.3	3.13	15.6
NMeFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
NEtFOSE	0.500	1.25	3.13	6.25	12.5	31.3	156
HFPO-DA	0.200	0.500	1.25	2.50	5.00	12.5	62.5
DONA	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFMPA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
PFMBA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
NFDHA	0.100	0.250	0.625	1.25	2.50	6.25	31.3
9Cl-PF3ONS	0.186	0.466	1.17	2.33	4.66	11.7	58.3
11Cl-PF3OUdS	0.188	0.471	1.18	2.36	4.71	11.8	58.9
PFEESA	0.089	0.223	0.56	1.11	2.23	5.56	27.8
3:3 FTCA	0.250	0.625	1.56	3.13	6.25	15.6	78.1
5:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
7:3 FTCA	1.25	3.13	7.81	15.6	31.3	78.1	391
13C4-PFBA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
13C5-PFPeA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C5-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C4-PFHpA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C8-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C9-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C6-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C7-PFUnA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFDoA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFTeDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C3-PFBS	0.581	0.581	0.581	0.581	0.581	0.581	0.581
13C3-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591
13C8-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C8-PFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
D5-NEtFOSAA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-4:2FTS	1.17	1.17	1.17	1.17	1.17	1.17	1.17
13C2-6:2FTS	1.19	1.19	1.19	1.19	1.19	1.19	1.19
13C2-8:2FTS	1.20	1.20	1.20	1.20	1.20	1.20	1.20
13C3-HFPO-DA	2.50	2.50	2.50	2.50	2.50	2.50	2.50
D7-NMeFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
D9-NEtFOSE	6.25	6.25	6.25	6.25	6.25	6.25	6.25
D5-NEtFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C3-PFBA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C4-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C2-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C4-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C5-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
18O2-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591

Attachment 12

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

Attachment 13

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

Attachment 14

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

Attachment 15

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

Attachment 16

PFAS Linear/Branched Mix B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume (ml) Methanol**	**Final Conc. PFAS Linear/Branched Mix B (ng/ml)**
PFAS Linear/Branched Mix A	Technical Ammonium, Perfluorooctanoate (Technical Grade)	95328-99-7	T-PFOA	500	0.5	2	125
	Perfluorooctanoic acid	335-67-1	PFOA	500			125
	2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	1691-99-2	NEtPFOSAE	500			125
	2-(N-methylperfluoro-1-octanesulfonamido) ethanol	24448-09-7	NMePFOSAE	500			125
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500			125
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			125
	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			125
	Perfluoro-7-methyloctanoic acid	15899-31-7	PF7MOA	500			125
	Perfluorononanoic acid	375-95-1	PFNA	500			125

Attachment 17

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

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

Attachment 18

1633 ICV Working Standard							
Solution Name	Analyte	CAS#	Acronym	Conc. (ng/ml)	Aliquot (ml)	Final Volume* (ml)	Final Conc. 1633 ICV Working Standard (ng/ml)
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.01	2	2.50
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			1.25
	Perfluorohexanoic acid	307-24-4	PFHxA	125			0.625
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			0.625
	Perfluorooctanoic acid	335-67-1	PFOA	125			0.625
	Perfluorononanoic acid	375-95-1	PFNA	125			0.625
	Perfluorodecanoic acid	335-76-2	PFDA	125			0.625
	Perfluoroundecanoic acid	2058-94-8	PFUnA	125			0.625
	Perfluorododecanoic acid	307-55-1	PFDoA	125			0.625
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			0.625
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			0.625
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			0.553
	Perfluoropentansulfonic acid	2706-91-4	PFPeS	117			0.586
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	114			0.569
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			0.595
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	117			0.583
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			0.600
	Perfluorodecanesulfonic acid	335-77-3	PFDS	121			0.603
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	121			0.605
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			2.34
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			2.37
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			2.40
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			0.625
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	125			0.625
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	125			0.625
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			0.625
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			0.625
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	1250			6.25
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	1250			6.25
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	500			2.50
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	471			2.36
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			1.25
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			1.25
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			1.25
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466			2.33
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471			2.36
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			1.11
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			3.13
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	3125			15.6
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	3125			15.6
Extraction Standard Mix	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500	0.01		2.5
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250			1.25
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125			0.625
	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	116			0.581
	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	118			0.591
	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	120			0.598
	Perfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250			1.25
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	250			1.25
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	234			1.17
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	238			1.19
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	240			1.20
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	500			2.5
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	1250			6.25
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250			6.25
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	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
Internal Standard Mix	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250	0.01		1.25
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	120			0.598
	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	118			0.591

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

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Revision Log

Revision: 16.0	**Effective Date**	This version
Section	**Justification**	**Changes**
Throughout	Added	Cross-reference links to related documents in D4
Section 5	Updated	Current standard Safety language
Sections 6 & 7	Added	Catalog # disclaimer and storage conditions
Section 10.2.2.1	Added	Percent Error acceptance criteria for individual points in the calibration

Revision: 15.0	**Effective Date**	21 April 2023
Section	**Justification**	**Changes**
Entire Document	Format	Update to D4 format
Section 10.2.4	Updated	Removed reference to DoD specific QA requirements.
Section 10.3.4	Added	Client specific QA requirements

Previous revisions maintained by QA Department

References

- EPA Compendium Method TO-15, "Determination of Volatile Organic Compounds in Ambient Air using Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry", US EPA, January, 1999.
- USEPA Compendium Method TO-3 “Method for the Determination of Volatile Organic Compounds In Ambient Air Using Cryogenic Pre Concentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection”, USEPA, April 1984
- New Jersey Department of Environmental Protection Site Remediation Program Vapor Intrusion Technical Guidance, Version 3.1, March 2013.

Cross References

- *SOP52862 Quality Assurance Manual*
- *SOP48255 Canister Cleaning, Certification and Sampling Kit Preparation*
- *QP46060 NDSC HSE Manual*
- *SOP49961 Burlington Corporate Safety Manual Addendum*
- *SOP52272 Traceability, Handling and Storage of Standards and Reagents*
- *SOP49959 Sample Management*
- *SOP49967 Nonconformance and Corrective Action Procedures*
- *SOP43862: Manual Integrations*
- *SOP49966 Management, Retention Storage and Archive of Data Records*
- *SOP52264 Employee Training and Analyst Demonstration of Proficiency*
- *SOP47863 Tracking and Collecting of Hazardous Waste*
- *SOP48269 Procedure for Acceptance and Handling of USDA Regulated Domestic and Foreign Soil*

1) Scope and Application

This SOP describes the laboratory procedure for the analysis of polar and non-polar volatile organic compounds (VOCs) in ambient and non-ambient air. The procedure is applicable to those VOCs that have been evaluated by the laboratory for their consistent performance in meeting the control criteria put forth in Compendium Method TO-15.

While the compendium method is specifically written for the analysis of ambient air samples collected in leak-free passivated stainless-steel canisters, it may be applied to the analysis of samples that have employed the use of other collection devices such as Tedlar bags and are from sources other than ambient air such as indoor air, vapor intrusion impacted air, soil gas, landfill gas, gas cleaning apparatuses and stack emissions.

This procedure may also be used to report a variety of carbon ranges, constituent groups like TVOC (total volatile organic compounds), or as unresolved complex mixtures (e.g. Total Hydrocarbons).

1.1) Analytes, Matrix(s), and Reporting Limits

The target compound list and reporting limits for each compound are provided in the Appendix.

2) Summary of Method

An aliquot of sample is pulled from the canister through a solid multi sorbent bed trap which reduces the water content of the sample. The sample is thermally desorbed, and the VOCs are carried onto a GC column coupled to a mass spectrometer. Compounds are identified by comparison of the mass spectra for individual peaks in the total ion chromatogram to the fragmentation patterns of ions corresponding to VOCs including the intensity of primary and secondary ions as well as the patterns of stored spectra acquired under similar conditions.

The concentration of the target compound is calculated by internal standard technique using the average response factor or slope and intercept of that compound as determined by the initial calibration.

If the laboratory has modified the method, a list of these adjustments may be found in the Method Modifications section of this SOP.

3) Definitions

A list of terms and definitions are provided in the Appendix. Refer to the Laboratory *SOP52862 Quality Assurance Manual* (QAM) for additional Glossary of Terms, Definitions, and Acronyms.

4) Interferences

Contamination may occur if canisters or other equipment is not properly cleaned before use. The laboratory procedures for canister and flow controller cleaning are provided in *SOP48255 Canister Cleaning, Certification and Sampling Kit Preparation*.

5) Safety

Employees must abide by the policies and procedures in the Eurofins NDSC-US EHS-*QP46060 NDSC HSE Manual*, the *SOP49961 Burlington Corporate Safety Manual Addendum* and this document. This procedure may involve hazardous material, operations and equipment.

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

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.

5.1) Specific Safety Concerns or Requirements

The analytical system contains zones with elevated or depressed temperatures that are capable of causing injury upon direct contact. The analyst needs to be aware of the locations of those zones and allow them to return to room temperature prior to maintenance activities or take measures to avoid contact with hot and/or cold surfaces. There are areas of high voltage in the analytical system. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Liquid nitrogen (LN2) is used for cryogenic purposes. In addition to avoiding contact with LN2 cooled surfaces, analysts must be aware of the potential for oxygen depletion in a confined space in the event of an unexpected large release of the product. Users should evacuate a confined space in which large amounts of LN2 have been released.

Sample canisters are occasionally pressurized for cleaning or sample dilution purposes. Lab systems are designed to ensure that the cans are not pressurized above 40 psi. Eye protection must be worn when cans are pressurized in the event of a canister failure.

5.2) Primary Materials Used

There are no materials used in this method which have a serious or significant hazard rating.
NOTE: This section does not include all materials used in the method. 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.

6) Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment meets the specification of this SOP.

6.1) Sampling Equipment

- 6L and 1L SUMMA® Canisters: Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 Valves, or equivalent. Maximum rated pressure 40 psig.
- 6L SUMMA® Canisters: Silicon Lined-Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 valves or equivalent. Maximum rated pressure 40 psig.
- Flow Controllers: Restek Catalog #24239 or equivalent.
- Flow Controller Orifice: Various sizes ranging from 0.008” to 0.060”, Restek or equivalent.
- Flow Controller Vacuum Gauges: Capable of measuring vacuum to an absolute vacuum of -30” Hg, and pressure up to 30 psi, Grainger Catalog #5WZ37 or equivalent
- Rain Guard: Stainless Steel Tubing ¼”, 10ft. Grainger or Equivalent. Cut 8” and bend into a J shape using a pipe bender.
- Stainless Steel Pre-Filter (7 um): Swagelok Catalog# SS-4F-T7-7 or equivalent
- Teflon Tape: Home Depot Brand or equivalent.

6.2) Analytical System

- Mass Spectrometer: Agilent 5973, 5975, 5975B, 5977C MSD or equivalent.
- Gas Chromatograph: Agilent 6890, 7890, 8890 or equivalent.
- VOC Autosampler: Entech 7016CA or equivalent.
- Cryogenic Concentrator: Equipped with an electronic mass flow controller that maintains a constant flow for carrier gas and sample over a range of 0-200 cc/min. Entech 7100A, 7200, 7200A or equivalent.
- Low Pressure Liquid Nitrogen: Air Gas or equivalent.
- Glass Bead Cryotrap: Capable of effectively removing water while trapping polar and non-polar compounds. Entech catalog# 01-04-11320.
- TENAX Sorbent Trap: Capable of removing CO2 and trapping the polar and non-polar compounds. Entech catalog # 01-04-11330.Primary Column: Fused silica capillary column (60 m x 0.32 mm x 1.8 µm), Restek RTX-624 or equivalent.
- Data System: PC software for Entech instrumentation. Hewlett-Packard ChemStation data acquisition software, Environment Testing Chrom and Environment Testing LIMS (TALS).
- NIST Mass Spectral Library and Search Program, 2020 release or newer

6.3) Cleaning System

- Canister Cleaner Module and Software: Capable of filling canisters with humidified air and evacuating canisters to 50 mtorr, Entech Model 3100A or equivalent.
- Vacuum Pump: Capable of evacuating sample canisters to full vacuum. Vacuubrand or equivalent.
- Cleaning Manifold: Equipped with stainless steel and Teflon transfer lines and connections for cleaning up to twelve canisters simultaneously.
- Heating Belts: Individual thermal-stated heating belts used to heat canisters to 100ºC during the manifolds cleaning cycles. Entech or equivalent.
- Cleaning oven: Capable of cleaning 6 Summa Cans simultaneously at a temperature of 100ºC. Entech or equivalent.
- Flow Controller Cleaning Manifold: Capable of flushing hot Nitrogen through 24 flow controllers simultaneously for cleaning.

6.4) Miscellaneous Supplies

- Mass Flow Controller, NIST Traceable: Capable of flow rate of 70 mL/min, Alicat MC5005CCM or equivalent. Use for the preparation of calibration and working standards.
- Syringes: Gas tight with a Luer-Lok tip, assorted sizes ranging from 1.0 mL to 1.0 L, SGE or equivalent.
- Digital Pressure Gauges, NIST Traceable: Capable of measuring pressure in the range of -30" Hg to 100 psi, Dwyer Models DPGA-12 and 67100 or equivalent
- Digital Flow Meter, NIST Traceable: Alltech or equivalent.

- Nitrogen Gas
- Digital Stop watch, NIST traceable, Thomas Scientific or equivalent

7) Reagents and Standards

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts may use equivalent-quality materials based on factors including availability. Permanent changes will be noted in future SOP revisions. All reagent and standards must be labeled with their Name and/or ID #; the Expiration Date; and the Date Prepared.

All reagents and standards are stored at room temperature in the Air Lab work area, unless noted otherwise.

For additional information refer to the *SOP52272 Traceability, Handling and Storage of Standards and Reagents*

7.1) Reagents

- Argon - Gas off bulk Liquid Argon tank. UHP Air Gas or equivalent vendor
- Nitrogen- Gas off bulk Liquid Nitrogen tank. UHP Air Gas or equivalent vendor
- Helium- UHP from Airgas or equivalent
- VOA free water

7.2) Standards

Certified stock standard solutions are purchased from approved commercial vendors. From these, intermediate and working standard solutions are prepared by diluting a known volume of standard in an appropriate solvent to the desired final concentration.

Formulations for the preparation of intermediate and working standard solutions are provided in the Appendix. Unless otherwise specified, assign an expiration date of 12 months from date of preparation, unless the parent component expires earlier in which case use the earliest expiration date.

There are small variations in analyte concentrations in the preparation of air standards. For simplicity, the nominal concentrations are referenced in this SOP.

Note: *When entering the concentrations of source standards in TALS, use the nominal concentration and not the actual concentration. Unless the actual has a > 15% difference from the nominal value. In these cases, use the actual value.*

Purchase the following stock standard mixtures from commercial vendors:

- Mixed Gas Stock Standard: - Commercially prepared standard that includes internal standard and tune standard compounds: Bromofluorobenzene, Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5, at a concentration of 100 ppbv each. Linde or equivalent.
- Calibration Stock Standard: - Commercially prepared custom gaseous stock standard that includes all target analytes except ethanol at a concentration of 1.0 ppmv. Linde or equivalent.
- Calibration Ethanol Neat Material. >99.5%
- ICV / LCS Stock Standard: - Custom made gaseous stock standard prepared from a different lot(s) of the source material(s) used to manufacture the calibration stock standard. The ICV/LCS stock standard includes all target analytes at a concentration of 1.0 ppmv. Linde.
- ICV/LCS Ethanol Neat material. >99.5% from a source other than the calibration source.

Prepare calibration and working standards mixtures by diluting a known volume of the stock standard with nitrogen to a specified volume into a humidified summa canister. The summa canister is humidified by adding 100ul of VOA free reagent water. The volume of the standard added to the canister is calculated using the set flow rate of 70mL/min using a mass flow meter multiplied by the time of the standard addition, plus the inclusion of the 25ml volume for the tubing connecting the mass flow meter to the summa canister. The formulations for standard preparation are provided in the Appendix along with recommended expiration dates and storage conditions.

Each stock standard is assigned a 1-year expiration date from manufacture and recertified annually. See *SOP52272 Traceability, Handling, and Storage of Standards and Reagents* for details on the recertification process. The ethanol neat material is assigned the expiration date given by the manufacturer.

If a new standard cannot be received prior to the expiration of the standard presently in use, the lab will notify the QA manager. The QA manager will perform a control chart review (see *SOP49965 Control Charts*). If the standard is determined to be acceptable, the lab may assign a new expiration date of not more than 30 days from the date of the last data point in the control chart review. When the new standard ID is created in TALS, the COA and the Control Chart verifying the standards stability will be attached to the reagent ID.

8) Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection, so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Air	1L or 6L Passivated Summa Canister	1L	NA	30 days from collection	EPA TO-15

1-liter Tedlar bags are only provided upon client request; however, clients are discouraged from using Tedlar bags due to the shortened holding time of 72 hours from collection.

All samples should be collected in passivated stainless-steel canisters that have been certified clean prior to sampling. The laboratory will provide certified clean canisters to the client upon request. The procedures for clean canister certification are provided in Laboratory SOP 48255: Canister Cleaning, Certification and Sampling Kit Preparation.

The laboratory can also provide flow controllers set to the appropriate flow rate for the sampling time required by the client.

The laboratory ships air canisters in custom made boxes. The boxes are equipped with custom-made foam inserts to hold the pre-set flow-controllers. The shipping materials are designed to prevent damage of equipment to and from the sampling site. The laboratory checks the equipment to ensure it is in proper working order before shipment to the client and additional checks are performed on return of the equipment to the laboratory. Sampling instructions are provided with each sampling kit. The sampling crew is advised to handle the sampling equipment using the instructions provided by the laboratory to ensure optimum performance.

The laboratory's sample acceptance policy for air samples in canisters requires that the sampling crew record the ID of the flow controller used for sample collection on the tag attached to each canister, but the association may also be recorded on the Field Test Data Sheet or a COC. With this information the laboratory can review the history of use of the FC as needed to troubleshoot potential equipment problems. Without the association, the history of use of the FC is unknown. The laboratory strongly recommends that field samplers be instructed to provide this information for each sampling event.

The return pressure of each canister should be between -10" Hg to -1"Hg, except for "grab" samples or samples with a collection rate of 100 or 200 mL/min, which must have a pressure greater than or equal to -10" Hg. The residual vacuum criteria ensure that the sample was collected over the time period the flow controller was calibrated for.

9) Quality Control

9.1) Sample QC

The following quality control samples are prepared with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See QC Summary Table
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See QC Summary Table
Internal Standard (ISTD)	Every Sample	See QC Summary Table
Laboratory Control Sample Duplicate (LCSD)	Client request or if insufficient sample for Sample Duplicate	See QC Summary Table
Sample Duplicate (SD)	1 in 20 or fewer samples if sample volume allows	See QC Summary Table
Trip Blank (TB)	Client Request	See QC Summary Table

NOTE: *The compendium reference method does not require the analysis of a laboratory control sample (LCS) or provide criteria for the evaluation of an LCS. The laboratory performs an LCS at the above-mentioned frequency as an evaluation of percent recovery in a blank matrix. The laboratory uses statistically derived control limits for the LCS.*

Unless otherwise specified by the client during project initiation, the LCSD will be used to measure precision only. The LCS will be used for evaluations for percent recovery and to determine if corrective action is necessary.

9.2) Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Tune Standard (BFB)	Each Analytical Window	See Appendices
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Appendices
Initial Calibration Verification (ICV)	Once, after each ICAL	See Appendices
RT Window Establishment	Once per ICAL	See Appendices

Relative Retention Time (RRT)	With each sample	See Appendices
Continuing Calibration Verification (CCV)	Daily, after each BFB	See Appendices

10) Procedure

10.1) Support Equipment Calibration

Verify the calibration is valid for the following;

- Mass flow controller used to prepare standards.
- Digital flow meter used to set and check the flow rates of the FC(s) used for sample collection.
- Digital pressure gauges used to check return canister pressure.
- Stopwatch used to measure standard addition time is current to the year.

Immediately notify the QA department if the calibration is not current and wait for further instruction. Equipment whose calibration has expired may not be used without documented approval from the QA department.

NOTE: *The QA department schedules the annual calibrations of the support equipment and maintains all Certificates of Calibration. The flow controllers are checked against a NIST traceable standard. This check is performed by the manufacturer of the equipment, when possible, or by an approved vendor that provides certification service.*

10.2) Instrument Calibration

All Calibration Standards and Quality Control samples have the same concentration of Internal Standards and Surrogates that are added for field sample analysis. Refer to the Analytical Sequence section for additional information.

10.2.1) Tune Standard

Analyze a tune standard (BFB) prior at the beginning of each analytical window. The tune standard is a commercially prepared mixed gas stock standard that includes Bromofluorobenzene (BFB) at a concentration of 100 ppbv.

To analyze the tune standard:

1. Establish the instrument operating conditions specified in the Sample Analysis section of this SOP. Once established these settings must be maintained for the analysis of all quality control and environmental samples.
2. Attach the mixed gas stock standard to the Entech concentrator by attaching the cylinder to the line dedicated for introduction of the internal standard (ISTD). The concentrator directly injects 20 mL of the 100 ppbv stock standard onto the instrument to yield an on-column concentration of 10 ppbv.
3. Acquire the data and evaluate the results against the acceptance criteria in the Tune Standard Criteria table in the appendix.
4. Criteria must be met prior to further analysis.
5. The official start time of the 24-hour analytical window is the injection time of a passing tune standard. All samples must be injected within 24 hours of that time.

NOTE: *The data processing system averages three scans (apex scan, scan prior, and scan following) and performs background subtraction of the single scan prior to the elution of BFB.*

10.2.2) Initial Calibration (ICAL)

The instrument must be calibrated with a minimum of five calibration standards for each target analyte at concentrations that span the working range of the method.

The laboratory routinely analyzes 8 standards at the recommended concentrations of 0.035, 0.20, 0.50, 5.0, 10.0, 15.0, 20 and 40 ppbv, except for Ethanol. For Ethanol, a 6-point curve is analyzed at the following concentrations: 5, 10, 15, 20, 40, and 100 ppbv.

Note: *These are nominal concentrations, dependent on parent can stock concentrations. The nominal 0.035 ppbv concentration is prepared to ensure the actual low calibration standard is below the reporting limit (RL).*

Even though eight calibration standards are routinely analyzed not every calibration standard is used for each analyte. Each analyte has been assigned to an analyte group that includes a calibration range of at least five standards. The analyte group associations for each target analyte are provided in Tables in the appendix. The calibration range for each analyte group is as follows:

- Group A: This analyte group is associated with a seven-point calibration curve. The calibration range is 0.20 to 40 ppbv with the 0.035 ppbv standard excluded. The limit of quantitation (LOQ) for this group of analytes is 0.20 ppbv

- Group B: This analyte group is associated with a six-point calibration curve. The calibration range is 0.50 to 40 ppbv with the 0.04 and 0.20ppbv standards excluded. The limit of quantitation (LOQ) for this group of analytes is 0.50 ppbv.

- Group C: This analyte group is associated with a five-point calibration curve. The calibration range is 5.0 to 40 ppbv with the 0.04, 0.20, and 0.50ppbv standards excluded. The limit of quantitation (LOQ) for this group of analytes is 5.0 ppbv.

- Group D: This analyte group is an eight-point calibration curve. The calibration range is 0.035 to 40 ppbv. The limit of quantitation (LOQ) for this group of analytes is 0.035 ppbv.

- Group E: (Ethanol): This analyte has a six-point calibration curve. The calibration range is 5 to 100 ppbv. The limit of quantitation (LOQ) for this analyte is 5 ppbv.

Prepare the calibration standards using the formulations provided in Appendix B.

Analyze the standards in a sequence from lowest to highest concentration using the instructions provided in the Sample Analysis section.

10.2.2.1) Initial Calibration Acceptance Criteria

The data processing system calculates a relative response factor (RRF), for each analyte and isomer pair using the assigned internal standard. The internal standard associations for each target analyte are provided in tables in the Appendix. The data processing system also calculates a mean relative response factor, relative standard deviation (RSD), relative retention time (RRT) and the mean RRT.

The following criteria must be met for a calibration to be considered acceptable:

- The RSD for each target analyte must be <30% with at most 2 exceptions up to a limit of 40%. If linear regression is used the R2 must be greater than 0.996

- The area response for the primary quantitation ion for the internal standard for each ICAL standard must be within 40% of the mean area response over the calibration range for each internal standard.

    - The RRT for each target compound at each calibration level must be within 0.06 RRT units of the mean RRT for the compound. The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.

If these criteria are not met inspect the system for problems and perform corrective action. See the corrective action section and the QC Summary Table in the appendix for guidance.

Whichever calibration model is selected, it must be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

Calculate the concentration of each calibration standard from the final calibration curve. Calculate the %Error for each point. All points must have an absolute %Error of < 30%, except the low point % Error must be < 50%.

If these conditions are not met, perform corrective action and recalibrate the instrument.

The laboratory CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.
%Error is the absolute value of the difference between the calculated concentration and the true concentration divided by the true concentration x100%.

- For example, if the 0.20mg/L standard reads back as 0.17mg/L:
  $[(0.20 - 0.17)/0.20] \times 100\% = 15\%$

Repeat initial calibration whenever instrument operating conditions are changed, a new column is installed, when significant instrument maintenance has been performed, and when the result of the CCV indicate the calibration is no longer valid.

10.2.3) Second Source Calibration Verification (ICV)

Immediately following an acceptable initial calibration verify the accuracy of the calibration by the analysis of the second source calibration verification standard (ICV).

Prepare the ICV following the formulation provided in the Appendix.

Analyze the ICV following the instructions provided in Sample Analysis section.

The percent recovery (%R) for each target analyte must be within 70-130%. If criteria are not met, perform corrective action. Recommended corrective actions are provided in QC Summary Table. If corrective action is not successful, remake your standards and recalibrate.

If after successful analysis of the ICV, time remains in the 24-hour analytical window, QC and field samples may be analyzed without analysis of a continuing calibration verification check standard. If time does not remain in the analytical window, a new analytical sequence must be initiated with a Tune Standard followed by daily calibration (CCV).

10.2.4) Continuing Calibration Verification (CCV)

Analyze the CCV immediately after the tune standard unless the analytical window includes ICAL, in which case, a CCV is not required.

Prepare the CCV standard using the formulation given in the Appendix. The recommended concentration of the CCV for each target analyte is 10.0 ppbv.

Analyze the CCV following the instructions provided in Sample Analysis section. The data system calculates a response factor for each analyte and calculates the percent difference (%D) of the RRF relative to the mean RRF in the most recent initial calibration.

- The %D for each target analyte must be within ±30%. If the above criteria are not met, repeat the analysis of the CCV once. If the second CCV meets criteria, continue with the analytical sequence. If it fails, evaluate the data to determine if one of the following conditions is met. If these conditions are not met corrective action must be taken. Guidance for troubleshooting is provided in the Troubleshooting and Corrective Action sections. After corrective action the analytical sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If these two CCVs do not meet the criteria, recalibration is required prior to further analysis.

- If the CCV criteria are exceeded high, indicating a high bias, and the associated samples have non-detects for those analytes, the analytical data may be considered usable. In the absence of instructions otherwise, proceed with analysis.

- If the CCV criteria are exceeded low, indicating a low bias, analytical results may be reported if those results exceed the project's regulatory decision level. In other words, if the analytical results are sufficiently high to counter the low bias, results may be reported. Consult with the project manager to determine if the exception is allowable for each project.

10.2.5) Troubleshooting

Check the following items in case of calibration failures:

- Loss of sensitivity or unstable ISTD recoveries are usually the result of a leak. Check the union between the GC column and Entech transfer line.
- Loss of sensitivity for individual compounds may be a result of either an active site in a transfer line or a bad trap. Troubleshoot and perform maintenance as necessary.
- Poor chromatography usually requires GC column maintenance, perform as necessary.
- Carryover is usually caused by excessive amounts of analyte introduced to the system. Analyze blanks until the system is cleaned or replace the traps and transfer lines if necessary.

10.3) Sample Preparation

10.3.1) Post Sampling Canister Pressure Check Procedure

The post-sampling canister pressure check is performed at the time of sample login by sample management staff so that any problems found are quickly identified and communicated to the client.

Refer to the current version of *SOP49959 Sample Management* for the procedure to take and record the post sampling canister pressure check.

10.3.2) Sample Screening

At the laboratory's discretion unknown samples may be screened prior to initial analysis to determine if the sample requires dilution. Unless otherwise requested by the client the laboratory does not provide screen data with the data package report even when primary dilutions are performed based on the results of the screen analysis.

To prepare a sample for screen analysis, connect the sample canister to the autosampler connected to screening instrument and analyze 20 mL of sample. Acquire and evaluate the results. If the results of screen analysis indicate that a target compound is above its upper range of calibration, calculate a recommended dilution factor (DF) by dividing the concentration of analyte found by 30. Record the recommended DF on the screen worksheet.

NOTE: *Samples are screened on a GC/MS instrument that is programmed with the operating conditions referenced in this SOP. The calibration is checked weekly with a single point calibration standard at a concentration of 10 ppbv for all target analytes. The calibration is checked more frequently when the results of instrument analysis do not correlate well with the results of the screen analysis.*

10.3.3) Sample Dilutions & Pressure Adjustment

Field samples should be diluted prior to initial analysis when the screen results indicate that the concentrations are above calibration range or when the laboratory has sufficient knowledge of the sample (history) to know that the sample will require dilution. Field samples must be reanalyzed at a dilution when the concentration of target compounds in initial analysis exceed of the upper range of calibration.

When the return negative pressure of a canister is greater than -10” Hg, make-up gas may be added to provide sufficient volume of make-up gas in order to have an adequate sample volume for analysis. The addition of make-up gas is considered a canister dilution. Some concentrators are able to pull full sample volume even if the residual vacuum is lower than -10” Hg so make-up air may not be necessary.

For samples analyzed for constituent groups (e.g., TVOC as Toluene), ranges, and or unresolved complex mixtures (Total Hydrocarbons), samples must be diluted such that the maximum peak height of any sample constituent under consideration does not exceed the equivalent peak height generated by highest calibration level standard. For evaluation it may be useful to graphically overlay sample chromatograms with that of the high point calibration standard.
To dilute the sample:

1) Attach the sample canister to the nitrogen gas line equipped with a pressure gauge that reads negative pressure in (“Hg) and positive pressure in (psig).

2) Ensure the valve of the nitrogen gas line is closed then open the valve of the sample canister. Record the negative pressure reading in the Canister Dilution Worksheet or on the canister’s tag.

3) Slowly open the valve of the nitrogen gas line and fill the canister until canister pressure gauge reads -10” Hg. Do not open the valve to such an extent that the nitrogen gas line pressure drops below 15 psig and do not allow the nitrogen gas line to reach equilibrium otherwise you will contaminate the nitrogen gas line.

4) When the desired pressure is achieved, close the canister valve and the valve on the nitrogen gas line; wait 15 seconds. The pressure should not exceed 40 psig.

5) Open the canister valve and record the final pressure reading in psig.

6) Close the canister valve and remove the valve from the zero air-line.

7) Record the initial and final pressure readings in the TALS canister dilution tracking module. If the final pressure is below ambient, the pressure will be in “Hg. Be sure to change the units in the TALS dilution tracking module.

When the return pressure of a canister is positive, the pressure must be adjusted to near ambient (0” Hg) prior to analysis. To adjust the pressure to ambient, vent the canister to ambient in a fume hood by opening the canister value for ~4-5 seconds, close the valve. For higher pressure canisters, open the valve and listen for a release of air then close the valve when the sound recedes.

If a trip blank is provided under vacuum, pressurize the trip blank canister to 10 psig. The pressurization of the trip blank is not considered a dilution.

10.3.4) QC Sample Preparation

To prepare the method blank (MB): Fill a clean canister that has never been used to collect environmental samples and has never left the laboratory to 20 psig with nitrogen gas. Continue to use this canister as the MB until the pressure of the canister reaches 0 psig, at which time, recharge with nitrogen gas to 20 psig and reuse.

To prepare the LCS: Follow the instructions provided in the Appendix for preparation of the working ICV/LCS standard. If an LCSD is requested, analyze the LCSD from the same canister as the LCS, unless client makes a specific request for an independent LCSD can.

10.4) Sample Analysis

10.4.1) Instrument Operating Conditions

Optimize the GC and MS conditions for compound separation and sensitivity.

The recommended operating conditions are as follows:

Thermal Desorb:	Initial Trap #1 Temperature: -110ºC. Desorb Temperature from Trap #1 to #2: 0 ºC. Total Volume Transfer by Mass Flow Controller: 40 mL Initial Trap #2 Temperature: -15 ºC Desorb Temperature from Trap #2 to #3: 200ºC. Transfer time 3.5 minutes Initial Trap #3 Temperature: -165 ºC Injection Trap #3 Temperature: 70ºC Injection Time: 1.5 minutes Trap #3 Temperature after Injection: -165 ºC
Carrier Gas:	Helium, Ultra High Purity
Cryogenic Focusing Gas:	Liquid Nitrogen
Flow Rate:	~1.5 mL/min
Temperature Program:	Initial Temperature: 40ºC Initial Hold Time: 4 minutes Ramp1 Rate: 20ºC/min. to 200ºC. Ramp 2 Rate: 40ºC/min. to 220ºC Final Temperature: 220ºC Final Hold Time: 6.5 minutes
Electron Energy:	70 electron volts
Mass Range:	35-265 amu
Scan Time:	≥1 scan per second.

These operating conditions may be changed but once the operating conditions are established for initial calibration the same conditions must be used until a new calibration is performed.

The mass spec tune profile may be changed to optimize the BFB tune analysis but once set, it cannot be changed until another BFB tune is analyzed.

10.4.2) Daily Instrument Maintenance

Prior to analysis initiate the flushing sequence using the Entech software. Then initiate the bake program using the Entech software.

10.4.3) Bi-Weekly Instrument Maintenance

At a minimum frequency of once every two weeks, perform an autosampler leak check. Cap all autosampler ports and initiate the leak check program using the Entech software. Record this check in the instrument maintenance log.

10.4.4) Analytical Sequence

All Calibration Standards, batch QA and field samples, except for the BFB analysis, have 20mls of the Mixed Gas Stock Standard added in conjunction with any other additions. This addition adds the internal standards and surrogates.

An example analytical sequence that includes initial calibration (ICAL) is provided below. When ICAL is not performed, the sequence begins with the tune standard and is followed by the CCV, LCS, LCSD, and method blank. If sufficient time remains in the 24 hours analytical window after initial calibration, QC and field samples may be analyzed without the CCV and the ICV will serve as the LCS for the sequence. The MB, LCS and LSCD must be analyzed at a frequency of every 20 samples or with each analytical sequence whichever is more frequent.

1. Tune Standard (BFB)
2. ICAL
3. ICV
4. CCV
5. LCS (repeat every 20 samples)
6. LCSD (when requested)
7. MB (repeat every 20 samples)
8. Field Samples (including trip blanks)

Attach the canisters to the autosampler inlet in the order of the analytical sequence then initiate the analytical sequence. The autosampler introduces 200 mL of sample volume from each canister to the instrument system and adds 20 mL of the mixed gas

standard to each sample.

Acquire the data and evaluate the results to confirm qualitative identification and quantification.

10.5) Corrective Action

An out-of-control situation may occur that is not adequately addressed in the corrective action table or troubleshooting section. The analyst must use their best analytical judgment and available resources to determine the corrective action to be taken. The analyst should seek the assistance of their immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation.

The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established.

If authorization is received from the Supervisor or QA Manager for release with data qualification, a Non-Conformance Memo (NCM) must be created to notify the project manager, who then notifies the client of the out-of-control situation. Also, the situation may be significant enough that the Supervisor or QA Manager would create a Corrective Action Report (CAR). Refer to the *SOP49967 Nonconformance and Corrective Action Procedures* for further guidance.

11) Calculations/Data Reduction

11.1) Qualitative Identification

The data processing system tentatively identifies target analytes by comparing the retention time of the peaks to the window set around the continuing calibration standard, and searches in that area for the primary ion and up to two secondary ions characteristic of the target analyte.

All tentative identifications made by the computer are reviewed and either accepted or rejected by the primary analyst. The identification made by the system is accepted when the following criteria are met:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the NIST14 database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance if a tentative identification is to be made and should be evaluated even if they are below 10% relative abundance.
- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components.

When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with corporate *SOP43862 Manual Integrations*.

11.2) Quantification of Target Analytes

After a compound has been identified, the data system quantifies the on-column concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

Samples analyzed for constituent groups, ranges, and or unresolved complex mixtures are quantified as follows;

11.2.1) TVOC as Toluene

An internal standard type of quantification response factor is generated using the RIC peak response measured for Toluene and its associated internal standard (as detailed in the calculation section). That response factor is used to quantify all RIC chromatographic sample response with the exception of those peak responses associated with internal standards. The results are reported as 'TVOC (as Toluene)'.

Retention Time Range: Propene to Naphthalene

11.2.2) GRO as Octane

An internal standard type of quantification response factor is generated using the RIC peak response measured for Octane and its associated internal standard (as detailed in the calculation section). That response factor is used to quantify all RIC chromatographic sample response with the exception of those peak responses associated with internal standards that eluted within the retention time range as defined by the elution of 2-Methyl-Butane through Decane (C5-C10). The results are reported as 'GRO (as Octane).

Retention Time Range: 2-Methyl-Butane to n-Decane

11.2.3) Unresolved Complex mixtures

An internal standard type of quantification response factor is generated using the RIC peak response measured for the un-resolved complex mixture (i.e. the gasoline envelope) and its associated internal standard (as detailed in the calculation section). That response factor is used to quantify all similarly chromatographing RIC sample response with the exception of those peak responses associated with internal standards. The results are reported using the name of the unresolved complex mixture used in the calibration (e.g. Total Hydrocarbons).

Retention Time Range: Propene to Naphthalene

Final results are calculated in TALS

11.3) Calculations

Analytical results are calculated as follows:

Dilution Factor

DF = (V2/V1) x (V4/V3)

Where:
V1 = Pre-Dilution Canister Volume
V2 = Post-Dilution Canister Volume
V4 = Base Sample Amount (200 mL)

Relative Response Factor (RRF)

RRF = (Ax)(Cis)/(Ais)(Cx)

Where:
Ax = Area of the quantitation ion of the analyte
Ais = Area of the quantitation ion of the internal standard
Cx = Concentration of analyte in concentration units (ppbv)
Cis = Concentration of internal standard in concentration units (ppbv)

Sample Concentration

Cx = [(Ax)(Cis)/(Ais)(Mean RRF)] x (DF)

Where:
Cx = Compound concentration (ppbv)
Cis = Concentration of associated internal standard (ppbv)
Ais = Area of quantitation ion for associated internal standard
Ax = Area of quantitation ion for compound
DF = Dilution Factor
Mean RRF = Average Relative Response Factor from initial calibration.

Unit Conversion from ppbv to ugm3

Analytical Result(ug/m3) = Result(ppbv) x (MW/24.45)

Where:
MW = molecular Weight

Example:
Benzene Result = 56 ppbv
Benzene mw = 78.108

Analytical Result(ug/m3) = 56ppbv x (78.108/24.45)

Result(ug/m3) = 178.9 ug/m3 reported as 180 ug/m3

Percent Recovery (%R)

%R = (Cs/Cn) x 100

Where:
Cs = Concentration of the spiked sample (ppbv)
Cn = Nominal concentration of spike added (ppbv)

Precision (%RPD)

%RPD = [(|C1-C2|)/((C1+C2)/2) x 100

Where:
C1 = Measured concentration of the first sample aliquot
C2 = Measured concentration of the second sample aliquot

Formula of Standard Addition:

Volume (mL) = (FR x T) + 25 mL

Where:
FR = flow rate in mL/min
T= time in minutes

Example:
FR = 70ml/min
Time = 10.69 minutes
Volume = 25+ (70 x 10.69) = 773 mL

Total Volume of diluted standard

Volume (mL) = CV x P

Where:
CV = canister volume in mL
P = pressure in Bar (14.7psi per Bar)

Example:
CV = 6185 mL
P= 2.5 Bar
Volume = 15462 mL

11.4) Data Review

11.4.1) Primary Review (Performed by Primary Analyst)

Upload the data files to TALS. Enter batch editor information and add the standards and reagents to the TALS batch. Review the results against acceptance criteria. If acceptance criteria are not met, make arrangements to perform corrective action.

Check the results of samples analyzed immediately after high concentration samples for signs of carry-over. Reanalyze the sample if carry over is suspected.

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

If samples are run at dilution, there should be a target peak that is at least 20% of the calibrated range but preferably in the upper half of the range or there should be an abundance of TICs which preclude a more concentrated analysis, or the sample should show matrix effects which preclude a more concentrated analysis. If a sample is diluted due to TICs or matrix, an NCM should be written explaining the reason for dilution.

Set results to primary, secondary, acceptable or rejected as appropriate.

Verify corrective action was taken for all results not within acceptance criteria. If corrective action is not taken or was unsuccessful, record all instances where criteria are not met with a nonconformance memo (NCM). Be sure to provide

explanation of your decision making in the internal comment section of the NCM. The internal comment section should list the reason the NCM is suspected, which action (if any) was taken and why, and the outcome of the action taken. Refer to the *SOP49967 Nonconformance and Corrective Action Procedures* for further guidance.

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Review that QC links have been properly made and run the data review checker (DRC) and address any anomalies.

Set the batch to 1st level review and record your review on the data review checklist.

11.4.2) Secondary Review (Performed by Peer Reviewer)

Review the project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Review records (these records include but are not limited to the Pre-Shipment Clean Canister Certification Report, Flow Controller Set Flow Rate and Leak Check Record, Field Test Data Sheets, and the Air Canister Return Pressure Check Record) associated with release and return of air sampling equipment to ensure all anomalies are properly recorded. Compare any problems noted in these documents with the analytical results and record any findings in the narrative note program or otherwise communicate your findings to the PM for inclusion in the project narrative.

Review the TALS batch editor to verify information is complete. Review the batch to verify that the procedures in this SOP were followed. If discrepancy is found, resolve the discrepancy and verify any modifications to the SOP were approved and properly documented.

Spot-check 15% of samples in the batch to verify quantitative and qualitative identification. If the samples are being analyzed under DoD methods 100% of data must be checked during secondary review.

If manual integrations were performed:

- Review all integrations to verify that the integration meets the requirements for manual integration as specified in corporate SOP CA-Q-S-002. If an error is suspected or found, consult with the analyst that performed the integration and request correction or notify the Department Manager or QA Manager. Do not "fix" the integration. Reintegration by a secondary data reviewer must not be performed except in limited circumstances as approved by the department supervisor or other laboratory management. If those instances where the secondary reviewer performs the integration, this person is now considered the primary analyst and each integration performed by the secondary reviewer must be subsequently reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in corporate SOP CA-Q-S-002.
- Check to ensure an appropriate technical reason code is provided for all manual integrations.

Verify that the performance criteria for the QC items listed in TO15 QC Summary & Recommended Corrective Action table in Appendix were met. If the results do not fall within the established limits, verify that corrective actions were performed. If corrective action was not performed; verify the reason is provided and that the situation is properly documented with an NCM. Set samples to 2nd level review. Record second level review on the data review checklist.

Run and review the deliverable. Fix any problems found. When complete set the method chain to lab complete and forward any paperwork to report/project management.

11.5) TO15 Poor Performing Analytes

NOTE: *Statistically derived limits will be no wider than 50-150%R for poor performing compounds.*

Compounds with a high boiling point which is beyond the range of typical gas compounds.

- Dodecane
- 1,2,4-Trichlorobenzene
- Hexachlorobutadiene
- Naphthalene
- 1,2,3-Trichlorobenzene

The following compounds change how they act on the traps depending on the humidity of the standard.

- 3-chloro-1-propene
- Bromoform

The following compounds are very polar and are linked to the water in the standard. Since we remove most of the water during the trapping process, a little change can produce a big result. For example removing 95% of the water instead of 94% of the water will have a negligible affect on non-polar compounds, but a 20% difference for polar compounds.

- Ethanol (made from neat material and is not part of the standard TO-15 calibration gas mix due to high reactivity
- Ethyl ether
- Acrolein
- Acetone
- Isopropyl alcohol
- 2-butanone
- Tetrahydrofuran
- 2-hexanone

11.6) Data Reporting

Report analytical results above the reporting limit (RL) as the value found. Report analytical results less than the RL, to the adjusted RL with a "U" data qualifier. If the data is to be reported to the method detection limit (MDL) the analytical results between the RL and the MDL are reported with an "estimated" footnote. Adjust the RL and MDL for sample dilution/concentration. The unadjusted RL for each target analyte is provided in Routine Compound List and TO-3 Analyte List tables. For Method TO3 the laboratory does not report values below the reporting limit.

Data reporting and creation of the data deliverable is performed by TALS using the formatters set by the project manager during project initiation.

Electronic and hardcopy data are maintained as described in laboratory *SOP49966 Management, Retention Storage and Archive of Data Records*.

12) Method Performance

12.1) Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005,

12.2) Training Requirements and Demonstration of Capabilities

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. The Group Leader is also responsible for the documentation of this training.

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the portion of the method they will be performing prior to working independently. Annually thereafter, the analyst must document continuing demonstration of capability (CDOC). See requirements for demonstration of analyst proficiency in *SOP52264 Employee Training and Analyst Demonstration of Proficiency*.

13) Pollution Control

It is Eurofins Burlington's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in NDSC-USEHS-*QP46060 NDSC HSE Manual*.

14) Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to *SOP47863 Tracking and Collecting of Hazardous Waste* and *SOP48269 Procedure for Acceptance and Handling of USDA Regulated Domestic and Foreign Soil*.

The following waste streams are produced when this method is carried out:

- None

15) Method Modifications

- This SOP utilizes and alternative detector (mass spectrometer) vs that of a FID and/or ECD as listed in the published method. (TO3)

- This SOP describes a method where an aliquot of a whole air sample collected in a passivated stainless-steel canister is cryogenically trapped and concentrated prior to injection into the MS detector equipped GC. (TO3)
- This Method SOP utilizes an RTX-624 capillary column (60M x 0.32 mm ID x 0.18um). (TO3, TO15)
- Additional quality control aspects are employed in the use of this method based on the alternative detector type, specifically, the detector tune is verified at the beginning of each period of analysis prior to the acquisition on any standard, blank, QC sample or field sample. (TO3)
- This Method SOP does not utilize sub ambient column oven temperature programming. (TO3, TO15)
- Sample traps consist of a multi bed system employing Tenax and glass beads (see manufactures system description: Entech Model 7100 Sample Pre-Concentrator). (TO15)
- Sampling apparatus consists of passivated stainless-steel canister specifically design for the collection of ambient air samples for volatile analysis (TO3)
- Nafion dryers are not used. The analytical pre-concentration system employs a moisture control system consisting of multi sorbent bed traps. (TO15)
- System calibration is verified every 24 hours within which samples are analyzed. System linearity is not verified every 4-6 hours as described. (TO3)
- Published Method requires weekly multi-point calibration be performed. Laboratory performs multi-point calibrations as necessary see section 10.2 (TO3)
- System linearity is determined through multi point calibration utilizing average response factors and internal standard technique quantification (see Section 10).(TO3)
- This SOP is applied to more than just the ambient air listed as a matrix by the method (TO15)

16) Appendices

16.1) Routine Compound List, Reporting Limit, Internal Standard and Ion Assignments

Analyte	CAS #	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Dichlorodifluoromethane	75-71-8	0.5	5	85	87		1	B
Chlorodifluoromethane	75-45-6	0.5	5	51	67	69	1	B
1,2-Dichlorotetrafluoroethane	76-14-2	0.2	2	85	135	87	1	A
Chloromethane	74-87-3	0.5	5	50	52		1	B
n-Butane	106-97-8	0.5	5	43	41	58	1	B
Vinyl Chloride	75-01-4	0.035	0.35	62	64		1	D
1,3-Butadiene	106-99-0	0.035	0.35	54			1	D
Bromomethane	74-83-9	0.2	2	94	96		1	A
Chloroethane	75-00-3	0.5	5	64	66		1	B
Isopentane	78-78-4	0.2	2	43	57	56	1	A
Bromoethene (Vinyl Bromide)	593-60-2	0.2	2	106	108	81	1	A
Trichlorofluoromethane	75-69-4	0.2	2	101	103		1	A
Pentane	109-66-0	0.5	5	43	57	56	1	B
EthylEther	60-29-7	0.2	2	59	45	74	1	A
Acrolein	107-02-8	5	50	56	55	37	1	C
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.2	2	101	151	103	1	A
1,1-Dichloroethene	75-35-4	0.035	0.35	96	161	63	1	A
Acetone	67-64-1	5	50	43	58		1	C
Isopropyl Alcohol	67-63-0	5	50	45	43		1	C
Carbon Disulfide	75-15-0	0.5	5	76			1	B
3-Chloropropene (Allyl Chloride)	107-05-1	0.5	5	41	76		1	B
Acetonitrile	75-05-8	5	50	41	40	39	1	C
Methylene Chloride	75-09-2	0.5	5	49	84	86	1	B
tert-Butyl Alcohol	75-65-0	5	50	59	41	43	1	C
Methyl tert-Butyl Ether	1634-04-4	0.5	5	73	43		1	B
trans-1,2-Dichloroethene	156-60-5	0.2	2	61	96		1	A
n-Hexane	110-54-3	0.2	2	57	86		1	B
1,1-Dichloroethane	75-34-3	0.035	0.35	63	65	83	1	A
Methyl Ethyl Ketone	78-93-3	0.5	5	72	43		1	B
cis-1,2-Dichloroethene	156-59-2	0.035	0.35	96	98	83	1	A
Tetrahydrofuran	109-99-9	5	50	42	72		2	C
Chloroform	67-66-3	0.2	2	83	85		1	A

1,1,1-Trichloroethane	71-55-6	0.2	2	97	99	61	2	A
Cyclohexane	110-82-7	0.2	2	84	56		2	A
Carbon Tetrachloride	56-23-5	0.035	0.35	117	119		2	D
2,2,4-Trimethylpentane	540-84-1	0.2	2	57	41	43	2	A
1,2-Dichloroethene (total)	540-59-0	0.2	2	61	96		1	A
Benzene	71-43-2	0.2	2	78	77		2	A
1,2-Dichloroethane	107-06-2	0.2	2	62	98		2	A
n-Heptane	142-82-5	0.2	2	43	71		2	A
Trichloroethene	79-01-6	0.035	0.35	95	130	132	2	D
Methyl Methacrylate	80-62-6	0.5	56	69	41	39	2	B
1,2-Dichloropropane	78-87-5	0.2	2	63	41		2	A
1,4-Dioxane	123-91-1	5	50	88	58		2	C
Dibromomethane	74-95-3	0.2	2	174	93	172	2	A
Bromodichloromethane	75-27-4	0.2	2	83	85		2	A
cis-1,3-Dichloropropene	10061-01-5	0.2	2	75	110		2	A
Methyl Isobutyl Ketone	108-10-1	0.5	5	43	58		2	B
n-Octane	111-65-9	0.2	2	43	57	114	2	A
Toluene	108-88-3	0.2	2	92	91		3	A
trans-1,3-Dichloropropene	10061-02-6	0.2	2	75	110		2	A
1,1,2-Trichloroethane	79-00-5	0.2	2	83	97	85	3	A
Tetrachloroethene	127-18-4	0.035	0.35	166	168	129	3	D
Methyl Butyl Ketone	591-78-6	0.5	5	43	58		3	B
Dibromochloromethane	124-48-1	0.2	2	129	127		3	A
1,2-Dibromoethane	106-93-4	0.2	2	107	109		3	A
Nonane	111-84-2	0.2	2	57	71	128	3	A
Chlorobenzene	108-90-7	0.2	2	112	77	114	3	A
Ethylbenzene	100-41-4	0.2	2	91	106		3	A
Xylene (m,p)	1330-20-7	0.5	5	106	91		3	A
Xylene (o)	95-47-6	0.2	2	106	91		3	A
Styrene	100-42-5	0.2	2	104	78		3	A
Bromoform	75-25-2	0.2	2	173	175	171	3	A
Cumene	98-82-8	0.2	2	105	120	77	3	A
1,1,2,2-Tetrachloroethane	79-34-5	0.2	2	83	131	85	3	A
Xylene (total)	1330-20-7	0.2	2	106	91		3	A
n-Decane	124-18-5	0.5	5	57	71	142	3	B
n-Propylbenzene	103-65-1	0.2	2	91	120	92	3	A
1,2,3-Trichoropropane	96-18-4	0.5	5	75	110	112	3	B
4-Ethyltoluene	622-96-8	0.2	2	105	120		3	A
1,3,5-Trimethylbenzene	108-67-8	0.2	2	105	120		3	A
2-Chlorotoluene	95-49-8	0.2	2	91	63		3	A
tert-Butylbenzene	98-06-6	0.2	2	119	91	134	3	A
1,2,4-Trimethylbenzene	95-63-6	0.2	2	105	120		3	A
sec-Butylbenzene	135-98-8	0.2	2	105	134	91	3	A
4-Isopropyltoluene	99-87-6	0.2	2	119	134	91	3	A
1,3-Dichlorobenzene	541-73-1	0.2	2	146	111	148	3	A
1,4-Dichlorobenzene	106-46-7	0.2	2	146	111	148	3	A
n-Undecane	1120-21-4	5	50	57	71	156	3	C
Benzyl Chloride	100-44-7	0.2	2	91	126	65	3	A
n-Butylbenzene	104-51-8	0.2	2	91	134	92	3	A
1,2-Dichlorobenzene	95-50-1	0.2	2	146	111	148	3	A
n-Dodecane	112-40-3	5	50	57	71	170	3	C
1,2,4-Trichlorobenzene	120-82-1	0.5	5	180	182		3	B
1,3-Hexachlorobutadiene	87-68-3	0.2	2	225	223		3	A
Naphthalene	91-20-3	0.5	5	128			3	B
1,2,3-Trichlorobenzene	87-61-6	0.2	2	180	182	145	3	A
Propylene	115-07-1	5	50	41	42	39	1	C

Vinyl Acetate	108-05-4	5	50	43	86		1	C
Ethyl Acetate	141-78-6	5	50	43	74		1	C
Ethanol	64-17-5	5	50	46	45		1	E
Bromochloromethane	74-97-5	NA	NA	128	49	130	1	NA
1,4-Difluorobenzene	540-36-3	NA	NA	114			2	NA
Chlorobenzene-d5	3114-55-4	NA	NA	117			3	NA

16.2) TO-3 Analyte List, Reporting Limit, Internal Standard and Ion Assignments

Analyte	CAS No.	RL (ppbv)	Qualifier Ion	Qualifier Ion	Qualifier Ion	ISTD Group
n-Octane	111-65-9	0.2	43	57	114	2
Toluene	108-88-3	0.2	92	91		3
TVOC as Toluene	NA	11				NA
GRO as Octane	NA	14				NA
Total Hydrocarbons	NA	76 ug/m3				NA
Internal Standards						
Bromochloromethane	74-97-5	NA	128	49	130	1
1,4-Difluorobenzene	540-36-3	NA	114			2
Chlorobenzene-d5	3114-55-4	NA	117			3

16.3) Tune Standard Criteria

Mass	Ion Abundance Criteria
50	8.0 to 40.0 percent of mass 95
75	30.0 to 66.0 percent of mass 95
95	Base Peak, 100 percent relative abundance
96	5.0 to 9.0 percent of mass 95
173	Less than 2.0 percent of mass 174
174	50.0 to 120.0 percent of mass 95
175	4.0 to 9.0 percent of mass 174
176	93.0 to 101.0 percent of mass 174
177	5.0 to 9.0 percent of mass 176

16.4) QC Summary & Recommended Corrective Action (Routine and DoD)

QC Check	Frequency	Acceptance Criteria	Recommended Corrective Action
Tune Standard	Prior to calibration and every 24 hours	See Tune Standard Criteria table	Correct Problem. Reanalyze. No samples may be analyzed without a valid tune.
Initial Calibration	Prior to sample analysis and when CCV fails	RSD for each analyte ≤ 30% with 2 exceptions up to 40% (if allowed). Individual Calibration points: < 30% Error, except < 50% for the lowest calibration point	Correct problem and repeat calibration

RRT	With each calibration	RRT of each target analyte in each calibration standard within ± 0.006 RRT units	Correct Problem. Repeat ICAL
ICV	Once after each ICAL	%R 70-130 for all analytes	Correct Problem. Reanalyze, re-make, re-verify & re-analyze. If that fails, re-make all standards and repeat calibration
CCV	Daily before sample analysis after tune standard	%Difference ≤ 30	Correct Problem. Reanalyze once. If that fails, see Section 10.3.9 for instruction.
LCS	Each batch or every 20 samples, whichever is sooner.	%Recovery 70-130%	Reanalyze LCS, re-prep and reanalyze LCS, and all associated samples if sufficient sample volume is available. If corrective action not successful, initiate nonconformance report and qualify sample results.
LCSD	Each batch or every 20 samples, only if there is no Sample Duplicate, or per client request	Relative Percent Difference (RPD) ≤ 25%	Reanalyze LCSD, re-prep and reanalyze LCS, LCSD and all associated samples if sufficient sample volume is available. If corrective action not successful, initiate nonconformance report and qualify sample results.
Method Blank	Each batch or every 20 samples, whichever is sooner.	< Reporting Limit	Reanalyze along with associated samples, unless detects for same compounds found in blank are greater than 10X the concentration found in the blank.
Internal Standards	All standards, field and QC samples	+/- 40% area response from last acceptable calibration. RT +/- 0.33 min (20 seconds) from last acceptable calibration.	Inspect system for malfunction. Reanalyze samples. Qualify data.
Sample Duplicate	Each batch or every 20 samples, if volume allows	Relative Percent Difference (RPD) ≤ 25%	Consult with PM. Reanalyze or qualify data.

16.5) In-house LCS limits

Analyte	CAS #	In-House Lower Limit (%R)	In-House Upper Limit (%R)	Relative Percent Difference (%)
Dichlorodifluoromethane	75-71-8	61	142	25
Chlorodifluoromethane	75-45-6	60	147	25
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	71	141	25
Chloromethane	74-87-3	56	141	25
n-Butane	106-97-8	53	151	25
Vinyl Chloride	75-01-4	61	135	25
1,3-Butadiene	106-99-0	58	139	25
Bromomethane	74-83-9	72	124	25
Chloroethane	75-00-3	68	130	25
Isopentane	78-78-4	56	150	25
Bromoethene (Vinyl Bromide)	593-60-2	75	125	25
Trichlorofluoromethane	75-69-4	70	129	25
Pentane	109-66-0	59	152	25
EthylEther	60-29-7	71	143	25
Acrolein	107-02-8	50*	150*	25
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	70	121	25
1,1-Dichloroethene	75-35-4	68	120	25
Acetone	67-64-1	54	154	25
Isopropyl Alcohol	67-63-0	53	142	25
Carbon Disulfide	75-15-0	71	138	25
3-Chloropropene (Allyl Chloride)	107-05-1	50*	150*	25

Acetonitrile	75-05-8	60	154	25
Methylene Chloride	75-09-2	59	137	25
tert-Butyl Alcohol	75-65-0	66	132	25
Methyl tert-Butyl Ether	1634-04-4	70	127	25
trans-1,2-Dichloroethene	156-60-5	69	137	25
n-Hexane	110-54-3	63	138	25
1,1-Dichloroethane	75-34-3	66	130	25
Methyl Ethyl Ketone	78-93-3	72	124	25
cis-1,2-Dichloroethene	156-59-2	72	121	25
Tetrahydrofuran	109-99-9	60	149	25
Chloroform	67-66-3	73	124	25
1,1,1-Trichloroethane	71-55-6	72	127	25
Cyclohexane	110-82-7	76	124	25
Carbon Tetrachloride	56-23-5	71	133	25
2,2,4-Trimethylpentane	540-84-1	68	131	25
1,2-Dichloroethene (total)	540-59-0	73	126	25
Benzene	71-43-2	73	119	25
1,2-Dichloroethane	107-06-2	68	135	25
n-Heptane	142-82-5	60	142	25
Trichloroethene	79-01-6	73	122	25
Methyl Methacrylate	80-62-6	73	129	25
1,2-Dichloropropane	78-87-5	69	128	25
1,4-Dioxane	123-91-1	66	129	25
Dibromomethane	74-95-3	68	126	25
Bromodichloromethane	75-27-4	75	127	25
cis-1,3-Dichloropropene	10061-01-5	74	125	25
Methyl Isobutyl Ketone	108-10-1	58	144	25
n-Octane	111-65-9	62	144	25
Toluene	108-88-3	75	122	25
trans-1,3-Dichloropropene	10061-02-6	74	128	25
1,1,2-Trichloroethane	79-00-5	75	126	25
Tetrachloroethene	127-18-4	70	125	25
Methyl Butyl Ketone	591-78-6	57	143	25
Dibromochloromethane	124-48-1	73	125	25
1,2-Dibromoethane	106-93-4	78	122	25
Nonane	111-84-2	70	131	25
Chlorobenzene	108-90-7	76	119	25
Ethylbenzene	100-41-4	74	122	25
Xylene (m,p)	1330-20-7	76	121	25
Xylene (o)	95-47-6	73	123	25
Styrene	100-42-5	74	125	25
Bromoform	75-25-2	53	149	25
Cumene	98-82-8	73	123	25
1,1,2,2-Tetrachloroethane	79-34-5	74	126	25
Xylene (total)	1330-20-7	75	122	25
n-Decane	124-18-5	68	134	25
n-Propylbenzene	103-65-1	73	127	25
1,2,3-Trichoropropane	96-18-4	71	125	25
4-Ethyltoluene	622-96-8	75	129	25
1,3,5-Trimethylbenzene	108-67-8	72	126	25
2-Chlorotoluene	95-49-8	74	126	25
tert-Butylbenzene	98-06-6	71	125	25
1,2,4-Trimethylbenzene	95-63-6	71	129	25
sec-Butylbenzene	135-98-8	70	128	25
4-Isopropyltoluene	99-87-6	68	130	25
1,3-Dichlorobenzene	541-73-1	69	131	25
1,4-Dichlorobenzene	106-46-7	67	132	25
n-Undecane	1120-21-4	62	136	25
Benzyl Chloride	100-44-7	60	136	25
n-Butylbenzene	104-51-8	65	137	25

1,2-Dichlorobenzene	95-50-1	68	129	25
n-Dodecane	112-40-3	50*	150*	25
1,2,4-Trichlorobenzene	120-82-1	50*	150*	25
1,3-Hexachlorobutadiene	87-68-3	58	130	25
Naphthalene	91-20-3	50*	150*	25
1,2,3-Trichlorobenzene	87-61-6	50*	150*	25
Propylene	115-07-1	50*	150*	25
Vinyl Acetate	108-05-4	59	149	25
Ethyl Acetate	141-78-6	70	131	25
Ethanol	64-17-5	50*	150*	25

*These are poor performing compounds. Limits for poor performing compounds will be no wider than 50-150%.
**The limits in this table are those in effect as of the published date of this SOP. These limits are based on historical data and are subject to change. Current in-house limits are populated in the LIMS database. Contact a laboratory representative for the most current set of limits. Limit Ref: 2019CCTO15

16.6) Appendix A: Terms and Definitions

Acceptance Criteria: Specified limits placed on characteristics of an item, process or service defined in requirement documents.
Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.
Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).
Batch: Environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.
Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.
Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.
Calibration Standard: A substance or reference used to calibrate an instrument.
Continuing Calibration Verification (CCV): An analytical standard gas mixture containing all target analytes and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.
Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.
Cryogen: A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp -195.8°C) or liquid argon (bp -185.7°C).
Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.
Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.
Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.
Initial Calibration Verification (ICV): An analytical standard mixture containing all target analytes and internal standard compounds that are prepared from a source independent of the source of the initial calibration standards. The purpose of the ICV is to verify that the initial calibration is in control.
Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.
Internal Standards (IS): Non-target analytes that are similar to the target analytes but are not expected to be found in environmental media (generally, isotopically labeled target analytes are used for this purpose). IS are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. IS responses are used as the basis for quantitation of target analytes.
Laboratory Control Sample (LCS): A QC sample of known composition spiked with analytes of interest. The LCS evaluates method performance and ability to successfully recover target analytes from a clean matrix. LCS recovery is typically expressed as percent recovery and provides a measure of accuracy. A LCSD is a duplicate LCS prepared and analyzed from a separate canister to provide a measure of replicate precision.
Method Blank (MB): A canister of humidified ultra pure zero air that is treated exactly as a sample. The MBLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.
Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.
Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.
Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.
Stock Gas Mixture: A Commercially purchased concentrated gas mixture containing one or more method analytes

16.7) Appendix B: Standard Preparation Tables

The standard formulations contained in this appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory *SOP52272 Traceability, Handling, and Storage of Standards and Reagents* for further guidance on the preparation of standard solutions.

Prepare all standards using the McMillan Company 80SD mass flow controller. Prepare the standard in zero air, demonstrated to be analyte free. Store the standard at ambient temperature. Unless otherwise specified, assign an expiration date of 30 days from date of preparation unless the parent standard expires earlier, in which case, use the earliest expiration date.

Intermediate Calibration Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Custom Calibration Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15 L Summa Canister. Expiration Period 3months
This standard contains all the target analytes listed in the Routine Compound Table.

Working Calibration Standards

Parent Standard	Calibration Standard	Parent Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Cal Standard 20 ppbv	Cal Standard 0.2 ppbv	20	155	15.46	0.2
Cal Standard 20 ppbv	Cal Standard 0.5 ppbv	20	386	15.46	0.5
Intermediate Calibration Standard	Cal Standard 5 ppbv	200	386	15.46	5
Intermediate Calibration Standard	Cal Standard 10 ppbv	200	773	15.46	10
Intermediate Calibration Standard	Cal Standard 15 ppbv	200	1160	15.46	15
Intermediate Calibration Standard	Cal Standard 20 ppbv	200	1546	15.46	20
Intermediate Calibration Standard	Cal Standard 40 ppbv	200	3092	15.46	40

Prepare in 6 L Summa Canister Expiration Period 3 months
Each calibration standard contains all the analytes listed in the Routine Compound Table at the above concentrations.

Intermediate ICV/LCS Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
ICV Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in Routine Compound Table.

Working ICV/LCS Standard

Parent Standard	Calibration Standard	Stock Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Intermediate ICV/LCS Standard	ICV Standard 10 ppbv	200	773	15.46	10

Prepare in 6L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in Routine Compound Table.

Calibration Level	Working Calibration Standard	Volume Analyzed (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.2 ppbv	40	0.04
Calibration Level 2	Cal Standard 0.2 ppbv	200	0.2

Calibration Level 3	Cal Standard 0.5 ppbv	200	0.5
Calibration Level 4	Cal Standard 5 ppbv	200	5
Calibration Level 5	Cal Standard 10 ppbv	200	10
Calibration Level 6	Cal Standard 15 ppbv	200	15
Calibration Level 7	Cal Standard 20 ppbv	200	20
Calibration Level 8	Cal Standard 40 ppbv	200	40
ICV	Intermediate ICV 10ppb	200	10

Prepare in 6L Summa Canister

Intermediate Ethanol Calibration Standard at 500ppbv/v

1. Fill a 44 ml VOA vial with VOA free water. Remove 197ul of water from the vial.
2. Add 197 ul of >99.5% Ethanol neat material
3. Cap and shake/roll vial for 1 minute
4. Inject 10ul of the prepared water/ethanol mix into a fully evacuated 15 liter summa canister
5. Pump the syringe plunger 5 times to insure complete transfer of material
6. Immediately fill the canister to 22 psig with zero air.

Calibration Level	Working Calibration Standard	Volume added (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.5 ppbv	124	5
Calibration Level 2	Cal Standard 5.0 ppbv	309	10
Calibration Level 3	Cal Standard 10ppbv	464	15
Calibration Level 4	Cal Standard 15 ppbv	618	20
Calibration Level 5	Cal Standard 20 ppbv	1237	40
Calibration Level 6	Cal Standard 40 ppbv	3092	100

DC-SOP49966 Management, Retention, Storage and Archive of Data Records
EHS-SOP47863 Tracking and Collecting of Hazardous Waste
EHS-SOP48269 Procedure for Acceptance and Handling of USDA Regulated Domestic and Foreign Soil
EHS-SOP49961 Burlington Laboratory Corporate Safety Manual Addendum and Contingency Plan
EQ-SOP48255 Canister Cleaning, Certification and Sampling Kit Preparation
EQ-SOP52272 Traceability, Handling, & Storage of Standards & Reagents
ML-SOP49965 Procedures for Control Charts, Trend Analysis and In-House Limits
NC-SOP49967 Nonconformance and Corrective Action Procedures
QA-QM52862 Quality Assurance Manual
SM-SOP49959 Sample Management
TR-SOP52264 Employee Training and Analyst Demonstration of Competence
NDSC-ETHC-SOP43862 Manual Integrations
NDSC-US-EHS-QP46060 NDSC HSE Manual

End of document

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14	03.FEB.2022		
15	21.APR.2023	Conversion to HTML format and full review	
16	29.JAN.2024	SOP Review & Update. Due to database migration, some review by analysts and approval by supervisors are documented outside of D4 in QA SOP directory.	

Title: Analysis of Total and Amenable Cyanide in Water, Drinking water, Wastewater and Soil- Automated by Method EPA SW846 9012B; 335.4 and Standard Method 4500 CN^-C, G

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Methods EPA 335.4, SW846 9012B, Lachat 10-204-00-1-A and SM 4500 CN^-C, G are described in this SOP. The SOP is applicable to water, drinking water, wastewater, soil and waste samples requiring cyanide determination. The method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide, free cyanide and cyanide amenable to chlorination.

1.1.2 The automated method has a detector that is sensitive to approximately to 0.005 mg CN-/L. The laboratory's reporting limit are as follows:

Analyte	Water Reporting Limit (mg/L)	Soil Reporting Limit (mg/Kg) Micro Distillation
Cyanide	**0.01**	**0.24**

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Cyanide is extracted by means of reflux distillation (MICRO DIST apparatus). Sample is distilled in a strongly acidic solution which breaks down cyanide complexes and converts the cyanide to hydrocyanic acid (HCN). HCN is absorbed into a sodium hydroxide absorber solution and the cyanide ion in the absorbing solution is determined by automated UV colorimetry at 570nm.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Interferences can be reduced or eliminated by using the distillation procedure.

4.2 The presence of sulfide in the sample matrix adversely effects the determination of total cyanide. Samples that contain hydrogen sulfide, metals sulfides or other compounds that may produce hydrogen sulfide during distillation should be treated.

4.2.1 Place a drop of the sample on lead acetate test paper (which has been pre-moistened with pH 4 acetate buffer solution) to detect the presence of sulfides. If sulfides are present (test strip turns black), the sample volume

required for the cyanide determination should be increased by 25 milliliters (ml).

4.2.2 The total volume of sample should be treated with powdered cadmium carbonate or lead carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide.

4.2.3 Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper

4.2.4 Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis.

NOTE: Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss of cyanide on the precipitated material.

4.3 Oxidizing agents such as chlorine decompose most cyanides.

4.3.1 To determine if oxidizing agents are present test a drop of sample with potassium iodine-starch paper. If a blue color appears that indicates the need for treatment. Add ascorbic acid, a few crystals at a time, to the sample until a drop of sample produces no blue color on the indicator paper. Add an additional 0.6g of ascorbic acid for each liter of sample volume.

4.4 Nitrates and nitrites may give high biased results. During distillation they form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose to generate HCN. They are interferences when present above 10 mg/L with other specific organic compounds.

4.4.1 Nitrate and nitrite interferences are eliminated by treatment with sulfamic acid just before distillation.

4.5 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/L were not found to interfere.

4.6 Fatty acids, detergents, surfactants may cause foaming during the distillation process, making the endpoint difficult to detect. If this occurs, samples can be acidified with acetic acid (1.6M) to pH 6.0-7.0.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates. Potassium Cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.
Hydrochloric Acid	Corrosive Poison	5 ppm – Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m3 – Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, swelling. Greater exposures cause severe burns with possible blindness.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Sodium Cyanide	Poison Corrosive	5 mg/m3 TWA as CN (skin)	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Silver Nitrate	Poison Corrosive Oxidizer	0.01mg/m3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- MICRO DIST distillation apparatus with sample distillation tube press (Hach).

- Lachat 8000 and 8500 series: Flow injection analysis equipment-designed to deliver and react sample and reagents in the required order and ratios. Lachat 8000 series

  - Multichannel proportioning pump
  - Reaction manifold
  - Colorimetric detector
  - Data system
  - Heating Unit

6.2. Supplies

- Lachat Micro Distillation Tubing, p/n A17117 (collector tubes, caps, membranes, and sample tubes) or equivalent, disposable
- Various Class A volumetric flask and pipettors
- pH-indicator strips (pH 0-14)
- Potassium Iodine-starch paper
- Lead-Acetate paper (pre-moistened with pH 4 acetate buffer solution)
- Analytical balance
- Vacuum pump
- Specimen cups
- Transfer pipettes
- Graduated cylinder (class A)
- Micro-buret

7. Reagents and Standards

7.1. Reagents

7.1.1. Deionized water-18 megohm reagent grade Type II water.

7.1.2. Acetone – store at room temperature; for stability information refer to manufacturer's instructions.

7.1.3. Rhodanine indicator - p-dimethyl-aminobenzalrhodanine: Place 20 mg of p-dimethyl-aminobenzalrhodanine in a 100ml volumetric flask and dilute to the

mark with acetone. Store at room temperature. Stable for six months.

7.1.4. Acetate Buffer - Dissolve 82.0 gram of $NaC_2H_3O_2$ x $3H_2O$ in 100ml DI water. Add sufficient glacial acetic acid and adjust pH to 4.5 (approx. 10 drops). This solution is stable for 6 months, store at 4ºC.

7.1.5. Cadmium Carbonate (powder) ACS reagent –store at room temperature; discard after 5 years from date receipt or earlier if necessary.

7.1.6. Reagents for cyanides amenable to chlorination.

7.1.6.1. Calcium hypochlorite solution, (0.35M), $Ca(OCl)_2$. Dissolve 5 g of calcium hypochlorite in 100 ml of deionized water. Prepare the solution fresh daily, store at room temperature. Solution is stable for 1 month, Note: store in an amber bottle if not used immediately.

7.1.6.2. 0.25 N NaOH. Dissolve 10.0 g NaOH into 1 L of deionized water. Dilute to mark with deionized water and invert to mix. Solution is stable for 6 months, store at room temperature.

7.1.6.3. Sodium Arsenite, NaAsO2. Dissolve 2g of Sodium Arsenite in 100ml of DI water. Prepare solution every month; store at room temperature.

7.1.7. Reagents for cyanide distillation.

7.1.7.1. 0.25 M NaOH. See section 7.1.6.2

7.1.7.2. Sulfamic Acid Solution: Dissolve 9.6 g. H_2NSO_3H into 100ml of deionized water. Solution is stable for 6 months, store at room temperature.

7.1.7.3. Micro Distillation Releasing Agent (7.11 M sulfuric acid/ 0.79M magnesium chloride): In a hood, place a 500ml beaker on a top-loading balance and tare. Then add 110.8g DI water. Then add and dissolve completely 32.2g magnesium chloride hexahydrate ($MgCl_2$ x $6H_2O$) in this water. Slowly add 139g concentrated sulfuric acid a little at a time, swirling and allowing to cool. Solution is stable for 6 months, store at room temperature.

7.1.7.4. Bismuth nitrate Solution: Dissolve 3 g of $Bi(NO)_3$•$5H_2O$ in 10 mls DEIONIZED WATER , add 25 mls glacial acetic acid while stirring and dilute to a 100 ml final volume.

7.1.8. Reagents for automated colorimetric determination.

7.1.8.1. Pyridine-Barbituric acid Reagent: In a fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100 ml DEIONIZED WATER, rinsing down the sides of the beaker to wet the barbituric. Add 75 ml pyridine (C_5H_5N) while stirring and mix until

barbituric acid dissolves. Add 15 ml conc. HCl (12 M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to mark and invert to mix. Prepare weekly, store solution in amber bottle and at room temperature.

7.1.8.2. Chloramine-T: Dissolve 2.0g chloramine-T hydrate in 500 ml deionized water. Prepare fresh daily. Store at room temperature.

7.1.8.3. Phosphate buffer: In a 1 L volumetric flask, dissolve 97.0 g potassium phosphate, monobasic, anhydrous, (KH_2PO_4) in approximately 800 ml of DEIONIZED WATER. Dilute to mark and invert to mix. Solution is stable for one month. Store at room temperature.

7.1.8.4. 0.25 M NaOH (carrier) See section 7.1.6.2.

7.2. Standards

7.2.1. Standard 0.0192N Silver nitrate solution (1 ml = 1 mg CN): Prepared by weighing 3.2647 g of dried $AgNO_3$, dissolved in deionized water and dilute to 1000ml. Solution is stable for 6 months or when QC check is outside the acceptable limits, whichever comes first, store at 4ºC.

7.2.2. Primary (Simple) Cyanide stock solution, 1000 mg/L: This standard is used for the preparation of calibration standards (Sec 10.2). Purchased commercially from Accustandard (catalog number WC-CN-10X-5). Follow manufacturer's instructions for storage and stability. Standardize the solution against 0.0192N silver nitrate solution quarterly.

If prepared in house, prepare as follows: Primary Cyanide stock solution, 1000 mg/L: Dissolve 2.51 gm of KCN and 2.0 gm of KOH in 900 ml of deionized water. Standardize the solution against 0.0192N silver nitrate solution weekly and dilute to the appropriate concentration so that 1ml=1mg CN. Solution is stable for 6 months, store at 4ºC. Standardize the solution as follows::

- Take 5.0 ml of the primary stock cyanide solution (Sec. 7.2.2) and dilute it to 50ml using the 0.25 M NaOH. Add 10-12 drops of the rhodanine indicator (Sec. 7.1.3) to produce a bright yellow color.
- Using the 10 ml micro buret, titrate the stock cyanide solution from a bright yellow color to a salmon hue endpoint. Record the volume of silver nitrate solution used for the titration in the Standardization logbook.
- Titrate a blank using 50mls of 0.25N NaOH, 10-12 drops of rhodanine indicator. Record the volume of silver nitrate solution used for the titration in the Standardization logbook.
- Calculate CN concentration as follows:

Based on the following: $Ag^{+} + 2\ CN \rightarrow [Ag(CN)_2]^{-}$

$$CN^{-}(mg/L) = \frac{A-B}{C} \times N \times \frac{2\ mol\ CN}{1\ eq\ AgNO_3} \times \frac{26.02g\ CN}{1\ mol\ CN} \times \frac{1000mg}{1\ g}$$

Where:
A= ml $AgNO_3$ for titration of sample
B=ml $AgNO_3$ for titration of blank
C= ml or g of sample
N= normality of $AgNO_3$

- Record the preparation in the reagent TALS. Record final concentration in the Standardization logbook.

- **Note***:/standardizedconcentration* .

7.2.3. Secondary (Simple) Cyanide Stock Solution, 1000mg/L: The secondary cyanide stock solution must come from a source different from the primary cyanide stock solution (ERA, cat# 997 or Accustandard, cat# WC-CN-10X-5). Follow manufacturer's instructions for storage and stability. Standardize the solution against 0.0192N silver nitrate solution quarterly. This standard is used for the preparation of ICV and CCV standards (Sec 9.2.1 and 9.2.2).

If prepared in house, prepare as follows: Dissolve 2.51 gm of KCN and 2.0 gm of KOH in 900 ml of deionized water. Note: the secondary cyanide stock solution must be prepared from a different source than the primary cyanide stock solution. Standardize the solution with 0.0192N silver nitrate solution weekly and dilute to the appropriate concentration so that 1ml=1mg CN. The solution is stable for 6 months, store at 4ºC. Follow the standardization procedure in 7.2.2.

Note: */standardizedconcentration* .

7.2.4. Primary Working Solution, 6 mg/L: Take 0.6 mL of the Primary Cyanide Stock Solution (Sec. 7.2.2) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.5. Secondary Working Solution, 6 mg/L: Take 0.6 mL of the Secondary Cyanide Stock Solution (Sec. 7.2.3) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.5.1. Secondary Working Solution for Easy-Dist: 10mg/L: Take 1.0 mL of the Secondary Cyanide Stock Solution (Sec. 7.2.3) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.6. Complex Cyanide stock solution (Potassium Ferrocyanide Trihydrate in 0.5 NAOH), 1000 mg/L: Purchased commercially from ERA (catalog number 998). Follow manufacturer's instructions for storage and stability. This

standard is used for the preparation of QC samples which are distilled prior to analysis (LCS and MS/MSD).

7.2.7. Complex Cyanide Working solution, 6 mg/L: Take 0.6 mL of the Complex Cyanide stock solution (Sec 7.2.6) and dilute to a final volume of 100 ml with 0.25N NaOH. Prepare fresh daily.

7.2.7.1. Complex Cyanide Working solution for Easy-Dist, 10 mg/L: Take 1.0 mL of the Complex Cyanide stock solution (Sec 7.2.6) and dilute to a final volume of 100 ml with 0.25N NaOH. Prepare fresh daily.

7.2.8. Soil Standard Reference Materia (LCSSRM): Purchased commercially from ERA or Phenova. Follow manufacturer's instructions for storage and stability. This standard is used for the preparation of QC samples which are distilled prior to analysis.

8. Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Glass or plastic	200 ml	NaOH, pH > 12; Cool 4 ± 2°C	14 Days	Method 335.4; SW846 Method 9012B
Soils	Glass	5.0 grams	Cool 4 ± 2°C	14 Days	Method 335.4; SW846 Method 9012B

[1] Inclusive of digestion and analysis.

9. Quality Control

9.1. Sample QC: Waters and soils, are separated into different QC batches.

9.1.1. Method Blank (MB): 0.25N NaOH is used for the blank and is carried through the entire sample preparation and analytical process. Add 6ml of 0.25 N NaOH to a disposable distillation sample tube. Results must be less than the reporting limit, or the batch must be redistilled and reanalyzed. One method blank must be analyzed with each set up of samples or every 20 samples, whichever is more frequent.

9.1.2. Laboratory Control Sample (LCS) 0.10 mg/L: A LCS (aqueous batches) or LCSSRM (soil batches) must be analyzed with each distillation batch or every

20 samples, whichever is more frequent. Prepare the solution fresh daily by adding 0.1 mL of the 6 mg/L Complex CN working solution (Sec. 7.2.7) and 5mL of 0.25 NaOH. The results must be within the control limits; if not, the batch must be redistilled and reanalyzed. Control limits: for 9012B ±15% of the true value; for 335.4 ±10% of the true value; and for SM4500CN ±10% of the true value.

- *Soil - Laboratory Control Sample Standard Reference Material (LCSSRM),* weigh out 0.25g of the standard reference material, add 5.0 ml DI water to the disposable distillation tube. The results must be within vendor specified QC limits or the batch must be redistilled and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD) One MS/MSD pair must be analyzed for every 10 samples.

- Aqueous samples (drinking water and wastewater): **MICRO DIST, 0.2 mg/L:** two portions of the same sample (matrix spike and matrix spike duplicate), each 6 ml, are added to separate disposable distillation tubes then add 0.2 mL of the 6 mg/L Complex CN primary working solution (Sec 7.2.7) into each sample. The results must be within 90-110 %..

- Soil samples: **MICRO DIST, 4.8 mg/Kg:** add 0.25g of the sample to two separate disposable distillation tubes (matrix spike and matrix spike duplicate) and add 5.0 ml of deionized water. Spike these samples with 0.2 mL of the 6 mg/L Complex CN primary working solution (Sec. 7.2.7). The results must be within laboratory generated limits.

- Amenable to chlorination: MS/MSD – For aqueous samples, measure 6 ml of sample for both the MS and MSD and add to a 40ml glass vial. Add 0.2 ml of the 6mg/L Complex CN primary working solution (Sec 7.2.7) into each sample; the spiking concentration is 0.20mg/L. For soils samples, weigh out 0.25g of sample and add 5.0ml of water then follow spiking procedure for aqueous samples. The spike must be added before the chlorination step begins. The form of cyanide used for spiking is completely amenable to decomposition with chlorine and therefore, the recovery of the spikes is expected to be 0%. If cyanide remains following the chlorination step, it is evident that the chlorination was not complete.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV), 0.20 ppm: The ICV must be analyzed immediately after an acceptable calibration and before any samples are analyzed. The ICV will be prepared from the secondary Simple CN source (Sec 7.2.3). See Sec 10.2.3.3 for the ICV preparation instructions by Micro Dist. The determined concentration must be within the ICV control limits; if not the analysis should be terminated until the source of the problem is identified and corrected. ICV control limits for 9012B is ±15% of the true

value; for 335.4 ±10% of the true value and for SM4500CN ±15% of the true value.

9.2.2. Continuing Calibration Verification (CCV), 0.20 ppm: A CCV is analyzed after every 10 samples and at the end of the sample run. The CCV will be prepared from the same source as the ICAL. CCV control limits for 9012B is ±15% of the true value; for 335.4 ±10% of the true value and for SM4500CN ±10% of the true value. If the result exceeds the limit, the analysis should be terminated until the source of the problem is identified and corrected and all samples following the last acceptable CCV must be reanalyzed.

9.2.3. Initial Calibration Blank (ICB): An ICB is analyzed immediately after the ICV to verify calibration and acceptable instrument performance. The results must be less than the reporting limit. Use 0.25N NaOH for the ICB.

9.2.4. Continuing Calibration Blank (CCB): A CCB must be analyzed after every 10 samples and at the end of the sample run. The results must be less than the reporting limit. If the CCB result exceeds the reporting limit, all samples following the last acceptable CCB must be reanalyzed. Use 0.25N NaOH for the CCB.

9.2.5. Minimum Reporting Level (MRL), 0.01 mg/L: The MRL is required for 335.4 Drinking water samples only. The MRL must be analyzed immediately after the ICV/ICB pair and before any samples are analyzed. The MRL is prepared in the same manner as the low level standard. Add 5ml of 0.25N NaOH solution to the sample tube, then add 0.01ml of 6ppm Primary Standard (Sec. 7.2.4). Follow procedure for sample prep in Sec. 10.1.3. The recovery for the MRL should be within 50-150% of the true value. If not, re-analyze the entire batch (see exception note below). If the MRL is biased high, non-detect (ND) samples can be reported with flags and add NCM in report.

Note: For Method 335.4, if MRL fails and samples are not drinking water samples, place an 'x' in front of the MRL when data is uploaded into TALS; the MRL will not be reported with the non-drinking water samples.

10. Procedure

10.1. Sample Preparation

10.1.1. Each sample matrix must be spot tested for the presence of interfering ions.

10.1.1.1. Check that pH of each sample is >12 using the pH indicator strip.

10.1.1.2. Check for the presence of chlorine using KI-Starch paper. See section 4.3.1.

10.1.1.3. Check for the presence of sulfide using lead acetate paper (pre-moistened with pH4 acetate buffer solution). See section 4.2.1. – 4.2.4. Method 9012B: If positive add bismuth nitrate to the sample. At the same time add bismuth nitrate to all standards before distillation, see Section 10.2.3.

10.1.2. Procedure for the pretreatment of cyanides amenable to chlorination. **Note**: If total cyanide is detected, the laboratory would need to analyze both the cyanide amenable to chlorination sample and to re-perform (repeat) the total cyanide sample that was originally analyzed through the entire procedure.

10.1.2.1. Divide the sample into two equal aliquots of 6 ml sample and chlorinate one for the determination of cyanide amenable to chlorination. For aqueous samples, measure the 6 ml of sample and add into a 40 ml glass vial. For soils samples, weigh out 0.25g of sample and add 5ml of water. Add calcium hypochlorite solution (Section 7.1.6.1) drop-wise while agitating the mixture using a magnetic stir plate and maintaining the pH at between 11 and 12 with sodium hydroxide solution (Section 7.1.6.2). Cover the vials with aluminum foil to protect the samples from the light.

Note: A MS/MSD must be performed for samples requiring cyanide amenable to chlorination as a negative quality control; see Sec 9.1.3.

CAUTION: Toxic cyanogen chloride is the initial reaction product during an alkaline chlorination. This reaction should take place in a fume hood.

10.1.2.2. Excess residual chlorine must be maintained in the solution for 1 hour while agitating. Monitor pH with pH test paper and chlorine with KI-starch paper. A distinct blue color on the paper indicates sufficient chlorine in solution. If necessary add more calcium hypochlorite and/or NaOH.

10.1.2.3. Following one hour, remove any residual chlorine by dropwise addition of NaAsO2 solution (Sec. 7.1.6.3). Test with KI starch paper to ensure that there is no residual chlorine present as a reducing agent..

10.1.2.4. Test for total cyanide in both the chlorinated and unchlorinated aliquot of the sample. Cyanide amenable to chlorination is determined by the difference between the total cyanide result of the un-chlorinated aliquot and the chlorinated aliquot.

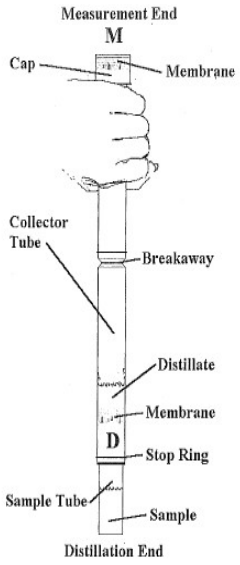
10.1.3. <u>MICRO DIST Distillation Procedure</u>

10.1.3.1. Set the controller to 120 deg C. Allow the block to warm up which takes approximately 40 minutes.

10.1.3.2. With the 'M' end or 'measuring end' up, place the collector tubes for the samples into the tube rack. Add 2 mL of 0.25N NaOH solution and add a membrane and cap. Label the top of the tube with the sample ID. Up to 21 samples tubes can fit on one block.

10.1.3.3. Pipet 6.0ml of aqueous sample into each sample tube or weigh out 0.25 to 0.30 grams of sample plus 5.0 ml of DI water for soil samples. Add 0.25ml of the sulfamic acid solution and 0.75ml of the releasing agent to all tubes. Label the tube with the sample ID.

***NOTE**: Homogenize soil samples properly prior to taking an aliquot for analysis.



10.1.3.4. Immediately push the 'D' end or 'distillation or discarded' end of the collector tube over the open end of each sample tube to start the seal.

10.1.3.5. Place the assembly into the press, putting the sample tube through the hole in the white base. Before pressing, the user should hold the collector tube firmly at the breakaway point to ensure the tube does not shift during the pressing procedure.

10.1.3.6. Press down on the press handle until the stop ring on the sample tube hits the 'D' end of the collector tube.

10.1.3.7. Put on heat resistant gloves and push the sample tube and 'D' end of each tube into the preheated block so that the collector tube stop ring touches the block.

10.1.3.8. Set timer for 30 minutes. Do not pull the tubes out before the 30 minutes to check them. This will cause the sample to suck-back.

10.1.3.9. After the 30 minutes have passed, put on heat resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion. Important: Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur, which may cause low recoveries. Dispose of the sample tube and remaining hot solution by dropping it into the sink or waste bucket.

Note: Do not pull out all the samples at once. Each sample needs to be separated immediately upon removal from the block.

10.1.3.10. Place the tubes in the collector tube rack with the 'M' end up. Parafilm the 'D' end of the tube and allow the tubes to cool for at least 10 minutes.

10.1.3.11. For each collector tube, hold the tube horizontally and rinse the walls with the distillate in order to homogenize it. Slowly roll the distillate around the tube to gather any droplets clinging to the walls. Then slowly return the collector tube to an upright position with the 'D' end up.

10.1.3.12. With the 'D' end up, break the collector tube in half by pulling the 'D' end hard towards the user to break it and twisting and tearing off the 'D' end. Rinse the 'D' end with a small volume of DI water and pour into the 'M' end. The 'D' end can be discarded.

10.1.3.13. Do not invert the sample. With the 'M' end down, place the tube in the collector tube rack. Dilute to the mark with deionized water. Seal the open end with parafilm if the sample is not going to be analyzed immediately.

10.1.3.14. The solution is now ready for semi-automated colorimetry.

10.1.4. <u>MICRO DIST Distillation Procedure using reuse, preclean Microdistillation tube</u>

10.1.4.1. With the D end up, place the collector tubes in the tube rack.

10.1.4.2. In the sample tube, pipet 6.0ml of aqueous sample into each sample tube or weigh out 0.25 to 0.30 grams of sample plus 5.0 ml of DI water for soil samples. Add 0.25ml of the sulfamic acid solution and 0.75ml of the releasing agent to all tubes. Label the tube with the sample ID.

10.1.4.3. Place the sample tube into the press, putting the sample tube through the whole in the white base. Place the D end on top of the sample tube and press until the D end meets the ring around the sample tube.

10.1.4.4. Add 2 mL of 0.25N NaOH solution to the D end which is connected to the with sample tube.

10.1.4.5. Attach a silicone tubing about 1.5" long to the tube labeled M end (labeled with sample ID). Then connect the tube labeled M end to the tube labeled D end (which is connected to sample tube).

10.1.4.6. Put on heat resistant gloves and push the sample tube and 'D' end of each tube into the preheated block so that the collector tube stop ring touches the block.

10.1.4.7. Set timer for 30 minutes. Do not pull the tubes out before the 30 minutes to check them. This will cause the sample to suck-back.

10.1.4.8. After the 30 minutes have passed, put on heat resistant gloves. Remove the first tube from the block and immediately flip the tube upside down and gently tap the tube on the counter a couple times. Remove the D end connected to sample tube. Rinse the D end/sample tube with few drops of DI water and add to the M end tube. The D end/sample tube can now be placed in a bucket for cleaning, refer to SOP ED-GEN-013, Glassware cleaning, for the full procedure.

10.1.4.9. Place the tubes in the collector tube rack with the 'M' end up.

10.1.4.10. Dilute to the mark with deionized water. The solution is now ready for semi-automated colorimetry. Seal the open end with parafilm, if the sample is not going to be analyzed immediately.

10.2. Calibration

10.2.1. All standards (Calibration Standards, calibration blank, ICV/CCV) must be distilled in the same manner as samples. A calibration must be analyzed each day before sample analysis.

10.2.2. Preparation of Standards for samples without sulfide:

10.2.2.1. Use the primary working solution (Sec. 7.2.4) to prepare the calibration standards below.

10.2.2.2. Prepare calibration standards and CCV for Micro Distillation batch as follows; the final volume in collector tube is 6.0 ml.

Volume (mL) of 6 mg/L CN primary working solution	Volume (mL) of 0.25N NaOH added to each Distillation Sample Tube	Concentration CN-(mg/L)
0.40	5	0.40
0.20	5	0.20
0.10	5	0.10
0.05	5	0.05
0.025	5	0.025
0.010	5	0.010
0	5	0.0
CCV	5	0.20

Note: [1] Add a calibration standard of 0.005 CN mg/l as directed on special projects.
[2] Prepare CCV with each distillation batch.
[3] Due to limited volume of the distillate, it may be necessary to prepare and distill two or three CCVs in the batch.

10.2.2.3. Prepare ICV using a secondary Simple CN source (Sec 7.2.3) as follows:

Volume (mL) of 6 mg/L CN secondary working solution (ICV)	Volume (mL) of 0.25N NaOH added to each Distillation Sample Tube	Concentration CN-(mg/L)
0.20	5	0.20

10.2.2.4. Correlation coefficient of 0.995 or greater must be obtained.

10.2.2.5. After running the calibration, recalculate each of the standard's concentration. Review Table 1 of the instrument report specifically the *%Residual* and *Det. Conc. (mg N/L)* column for the resulting concentration. The % Residual of the calibration standards should be within 50% for the first calibration standard, 0.01 mg/L (1 to 2 times the RL), 20% for 0.025 mg/L and 0.050 mg/L standards (3 to 5 times the RL) and 10% the rest of the standards (greater than 5 times the RL). If this criteria is not met, re-analyze the calibration standards (in some cases, new calibration standards may need to be prepared).

10.2.3. Method 9012B: Preparation of Standards for samples with sulfide:

10.2.3.1. From the 6 mg/L primary working standard prepare five standards as above to be distilled in the same manner as the samples using the method of standard additions. Note: Bismuth Nitrate must also be added to the standards. Standards distilled in this way will give a linear curve at lower concentrations but as concentration increased the recovery decreases.

10.2.3.2. Correlation coefficient of 0.995 or greater must be obtained.

10.3. Sample Analysis

10.3.1. System startup:

10.3.1.1. Follow manifold scheme for this method.

10.3.1.2. Turn on heating unit at 60° C for 15 minutes before analysis.

10.3.1.3. Put all reagent lines into deionized water and turn pump on. Check for leaks and slow flow rate.

10.3.1.4. Switch over reagent lines to proper reagent. Let reagent run through manifold for 15 minutes before analysis to allow baseline to stabilize.

10.3.2. Set up tray table with all appropriate QC checks. CCV/CCB every 10 samples and at the end of the run. The following analytical sequence must be used:

Instrument Calibration
ICV (QCS)
ICB
MRL
CCV
CCB
MB
LCS or LCSSRM for soils
8 Samples with Analytical Spikes
CCV (IPC)
CCB
10 samples
CCV (IPC)
CCB
Repeat until run is complete
CCV (IPC)
CCB

10.3.3. Place standards and samples into autosampler cups.

10.3.4. Run tray.

10.3.5. Dilute and reanalyze samples that exceed the calibration curve using the distilled calibration blank or CCB.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples:

Concentration = mg/L

Concentration (mg/L) from instrument is imported to TALS
Samples with dilution factor are calculated in TALS for final results

11.4. Final results calculation in solid samples:

$$\text{Concentration mg/kg} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (mg/L)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams)

NOTE: All dry weight corrections are made in TALS.

11.5. Final results calculation amenable to chlorination:

mg CN^- amenable to chlorination/L = A - B

where:
A: mg CN^-/L: found in unchlorinated portion of sample
B: mg CN^-/L: found in chlorinated portion of sample

11.6. Data Reduction

11.6.1. Cyanide soil and waters are batched separately in TALS Analyst Desktop. Update Cyanide prep batches as follows:.

11.6.1.1. For soils, enter in 0.25-0.30g under the 'initial amount' column and 6ml under the 'final amount' column for each sample including the MB and LCSSRM.

11.6.1.2. For waters, enter 6ml under the columns 'initial amount' and 'final amount' for each sample including the MB and LCS. Under the columns 'chlorinechk' and 'sulfidechk' record the results from the test strip paper. 'N' designates the interference was not present. Record 'pH>12' under the column 'DistillpHchk.'

11.6.1.3. Since the calibration standards are distilled, they will be recorded in the prep batch. Under the column 'Lab ID,' type in "IC' for 7 rows. Enter 6ml under the columns 'initial amount' and 'final amount' for each standard. Enter how many milliliters of the 6ppm primary working solution were used for each calibration standard following the chart in Sec. 10.2.2.1.

11.6.1.4. For both soil and water batches, record reagent information including reagent ID and expiration date on the batch information page. Record the actual time the samples were distilling under 'batch comments.' This should be designated as distillation start and end time.

11.6.2. Instrument data is imported to TALS by following these steps:

11.6.2.1. After the run has finished, click the data icon at the top of the screen. Choose the correct file. Go to 'File" and choose "Export Data' from the drop down menu. A screen will pop up, click 'ok' and then another screen will pop up, click 'ok.'

11.6.3. From the desktop, open the 'LIMSdata' icon on the Lachat 8000 and the 'Exports' icon on the Lachat 8500. Select the correct file, and then right click. Go to 'Send to-> LimsImport ->' Choose the correct method. The data has now been imported to TALS.

11.6.4. All reagent information is recorded in the "Batch Information" page. Use the 'Worksheet tab' if additional pages are necessary.

11.6.5. Record special notes and observations in the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected or diluted).

11.6.6. All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.6.7. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.6.7.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.6.7.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.6.7.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.6.7.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal

restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Pyridine/Hydroxide Solution: Hazardous Waste Liquid, n.o.s. This material is collected at satellite accumulation in 30 gallon Poly containers. When the container is full, the container is transferred to the waste room and held in secondary containment until a waste vendor is called for a pick up.

  Teris Profile Number: 50016717
  Onyx Profile WIP Number: 584650

15.0. References / Cross-References

15.1. Methods for the Determination of Inorganic Substances in Environmental Samples, EMSL-Cincinnati, EPA/600/R-93/100, August 1993, Method - 335.4 Determination of Total Cyanide by Semi-Automated Colorimetry

15.2. Standard Methods for the Examination of Water and Wastewater, American Public Health Association, Washington, DC, Method 4500 CN C, G-2016.

15.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW846 Method 9012B.

15.4. QuikChem Method 10-204-00-1-A, Determination of Cyanide in Waters.

15.5. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.6. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.7. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.9. TestAmerica Edison SOP No. ED-GEN-013, *Glassware cleaning*, current revision.

16.0. Method Modifications:

Item	Method no.	Modification
1	335.4	Sec 4.3.1: This SOP referenced method 335.4 (not SW846) when treating samples suspected to contain chlorine and nitrates/nitrites.

2	QuikChem Method 10-204-001-41-A	Sec 7.1.8.3: Preparation procedure for the Phosphate buffer follows Quikchem method 10-204-001-41-1 (not Method 335.4).
3	SM 4500 CN^- C	Sec 10.1.3.3: Sulfamic acid solution is added to all samples during distillation. Addition of Sulfamic acid is a procedure described in Standard methods 18th ed, Method 4500 CN^- C. Methods SW846 9012B and EPA 335.4 require the addition of Sulfamic acid for samples which are known to contain NO3 and/or NO2.
5	335.4; SW9012B	Sec 7.1.7.2-7.1.7.3; 10.1.4.2; 10.1.4.3- Modified procedure for reagent preparation and amount of reagent added in sample while keeping the reagent sample volume ratio and concentration the same.
6	335.4; SW9012B	*335.4 & 9012B: It is recommended that at least two standards (a high and a low) be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable.* Sec 9.1.2: For Micro Distillation, one LCS (0.10 mg/L) will be prepared for each distillation batch; however calibration standards, ICV and CCV will be distilled in the same manner as the samples.
7	335.4; SW9012B	*Method 335.4; SW9012B: Distill standards for samples with Sulfide.* This SOP follows the procedure described in the Hach Micro Dist User Manual, Method Number 10-204-00-1-X when distilling samples by Micro Dist- *It is imperative that the standards be distilled with the samples.*
8	Micro Dist (Method No. 10-204-00-1-X)	Add between 0.50-1.0g of soil/sludge to 5 ml DI water. This SOP will use 0.25g of soil sample to 5 ml DI water. Improved accuracy with the soil LCS when using 0.25g rather than 0.50g sample weight.

17.0. Attachments

N/A

18.0. Revision History

- Revision 18, dated 08 November 2023
  - Sec. 10.1.3.12 & 10.1.4.8: Revised solution to use when rinsing the CN sample tube after distillation from 0.25N NaOH to DI water.
  - Sec. 10.2.2.5: Added criteria to use when evaluating the calibration curve.

- Revision 17, dated 23 February 2023
  - Sec. 9.2.5: Clarified the applicable matrix, procedure, and corrective action for the QC – MRL.

- Revision 16, dated 22 September 2022
  - Sec. 7.1.6.3 and 10.1.2.3: Replaced ascorbic acid with sodium arsenite to remove residual chlorine.
  - Sec. 9.2.5: Fixed MRL acceptance range to 50-150%.
  - Sec. 10.3.2: Revised instrument tray set up to reflect actual laboratory practices.
  - Sec 15: Revised Standard Method reference to Rev 2016.

- Revision 15, dated 06 January 2021
  - Sec 7.1.6.1: Clarified the stability information for the reagent $Ca(OCl)_2$.

- Revision 14, dated 16 December 2020
  - Sec 3: Revised the Definitions locations as per the most current LQM.
  - Deleted all reference to Easy-Dist (midi-dist) and wipes distillation, not applicable in this SOP.
  - Sec. 7.1.6.1: Revised Calcium Hypochlorite storage requirements and preparation frequency to daily.
  - Sec. 9.1.2: Clarified that only soil LCS should be designated LCSSRM in TALS sample worksheet.
  - 9.1.3: Clarified the MS/MSD procedure for the Cyanide amenable to chlorination.
  - Sec 9.2.5: Added MRL to the list of instrument QC.
  - Sec. 10.1.2: Added requirements that sample which is detected for total cyanide must be re-analyzed and prepared with the cyanide amenable to chlorination sample.
  - Sec 10.1.2.1 & 10.1.2.2: Revised to clarify the procedures for CN amenable to chlorination (i.e. taking identical samples for total and amenable samples; 'excess' added in Sec 10.1.2.2).
  - Sec 10.1.4: Added procedure for setting up distillation using the reuse, preclean distillation tubes.
  - Sec. 11.6.7: Replaced EDS-WI-008, Wetchem Data Review Checklist with TALS DRC; procedures added for DRC.
  - Sec 15.9: Deleted WI EDS-WI-008, not applicable; subsequent sections adjusted.
  - Sec 15.10: Added reference – SOP ED-GEN-013.

- Revision 13.1, dated 23 October 2017
  - Updated SOP header with Eurofins emblem.

- Revision 13, dated 23 October 2017
  - Sec. 9.1.2: Corrected preparation instructions for LCS from 0.20 mg/L to 0.10 mg/L. Updated control limits for Method 9012B to +/-15%.
  - Sec. 9.2.1: Updated the ICV control limits for Method 9012B.
  - Sec. 9.2.2: Updated the CCV control limits for Method 9012B; removed instructions to prepare a CCV with each Micro-Distillation batch.
  - Sec. 10.3.2: Updated the example analytical sequence for Micro Dist and Easy Dist.
  - Sec 16, Item 6 of Method Modification – Deleted text- CCV (0.20 mg/L) will be distilled with each distillation batch.

- Revision 12, dated 26 September 2017
  - Sec. 1.1.2: Updated the Soil RL to 0.24 mg/Kg to reflect the initial soil sample weight change from 0.5g to 0.25g.
  - Sec. 6.2: Added Lachat Micro Distillation tubes and pH test strips to list of supplies.
  - Sec. 7.1.7.2: Revised preparation instructions for sulfamic acid solution; revised to use a double strength concentration sulfamic acid solution, however the amount added in sample is reduced in half.
  - Sec. 7.1.7.3: Replaced 2.5M MgCl2 and 1:1 H2SO4 solution with Microdistillation releasing agent (7.11 M sulfuric acid/ 0.79M magnesium

chloride) to be used instead of the separate H2SO4 and MgCl2 solution; equivalent reagent concentration is added in sample.
  - Sec. 7.2.3: Added ERA cat# 997 as an alternative source for a Simple CN secondary standard.
  - Sec 7.2.4 & 7.2.5: Added preparation instructions for the Micro Dist Primary/Secondary Working standards.
  - Sec 7.2.6: Deleted LCSH
  - Sec 7.2.7: Updated preparation instructions for the Complex CN Working Solution (Micro Dist).
  - Sec 7.2.8: Added soil standard reference material.
  - Sec 9.1.1: Updated preparation instructions for MB from deionized water to 0.25 N NaOH to reflect actual laboratory practices.
  - Sec 9.1.2: Updated preparation instructions for LCS needed for Micro-Dist; removed LCSH prep for Micro Distillation. Revised the LCS sample weight to use from 0.5g to 0.25g. Added Easy Dist prep instructions for LCSSRM.
  - Sec 9.1.3: Updated the matrix spike preparation instructions for Micro Dist for soil and water.
  - Sec 9.2.1: Updated the ICV preparation instructions for Micro-Dist;
  - Sec 9.2.2: Revised the standard source used for preparing CCV; source will be the same source as the calibration standards and will be distilled with each Micro Dist distillation batch.
  - Sec 10.1.1.1: Added instructions to check pH of each sample.
  - Sec 10.1.4.2: Revised the volume to add in collector tube to 2.0ml of 0.25N NaOH.
  - Sec 10.1.4.3: Revised the reagents and amount of reagents added into the sample/distillation tube; Revised the initial weight used for solid samples from 0.5g to 0.25-0.30g. Added note to homogenize soil sample prior to taking aliquot.
  - Sec 10.1.4.12: Added instructions to rinse the D-end and add rinsate to the M-end.
  - Sec 10.2.2: Added instructions to distill all standards used for the analysis of CN by Micro Distillation.
  - Sec 10.2.3.2 & 10.2.3.3: Added preparation instructions for the calibration standards and check standards (ICV,CCV) by Micro Distillation. Added note that CCV must be distilled with each distillation batch.
  - Sec 10.3.2: Clarified analytical sequence for Micro Dist and Easy Dist.
  - Sec 10.3.5: Updated instructions for sample dilutions.
  - Sec 11.7.1: Added instructions for completing Batch information page in TALS.
  - Sec 16: Method modification items 5-8 added.

- Revision 11, dated 11 August 2014
  - Sec. 7.2.2 & 7.2.3: Clarified procedure as to when to apply the standardized value of the CN stock solution.
  - Sec. 9.1.2: LLCS and HLCS will be analyzed for both methods SW9012 and EPA335.4 Deleted text: *'Distillation of two LCSs (LLCS and HLCS) is not a method requirement for Method 335.4. Use HLCS as the QC sample for Method 335.4.*

- Revision 10, dated 12 March 2014
  - Deleted Method 9012A reference throughout the SOP; replaced SW method reference with 9012B.

- Sec 15.3 Added reference to Standard Methods Editorial revision 2011; subsequent sections adjusted accordingly.

- Revision 9, dated 12 November 2012
  - Sec 1.1.2: Revised RL for solids from 0.12 mg/kg to 0.10 mg/kg; sample final volume is 5 ml.
  - Sec 9.1.2 & 9.1.3: Deleted text: *After micro-distillation, volume is brought up to 6 ml with deionized water.* The final volume of the distillate is not adjusted.
  - Deleted Sec 10.1.4.13, final distillate will not to be diluted to 6 ml; subsequent sections adjusted accordingly.
  - Sec 10.1.4.13 (formerly Sec 10.1.4.14): deleted text: *Shake the tube with a gentle whipping motion to mix in diluent water.* Added text: *With the 'M' end down, place the tube in the collector tube rack.*

- Revision 8, dated 25 October 2012
  - Deleted Sec 7.2.4 (5 mg/L primary working solution), standard not applicable; subsequent sections adjusted accordingly.
  - Sec 7.2.2, 7.2.3 & 7.2.6: Clarified to include which QC samples these standards are applicable to.
  - Sec 7.2.6 & 7.2.7: Added complex cyanide standard, 1000 mg/L stock standard and 10 mg/L working solution to be used for distilled QC samples (LCS and MS/MSD).
  - Sec 9.1.2: Added note: *Distillaton of two LCSs (LLCS and HLCS) is not a method requirement for Method 335.4. Use HLCS as the QC sample for Method 335.4.*
  - Sec 9.1.2: Revised the LLCS concentration to 0.10 mg/L; revised to reflect the correct spiking amount (ml) and source standard (complex CN working solution) in the preparation procedure.
  - Sec 9.1.3: Revised the spiking solution from simple to complex CN.

- Revision 7, dated 13 September 2012
  - Sec 1: Revised to include the RL for microdistillation in soils.
  - Sec. 2.0: Revised to include 'MICRO DIST' as one of the ways to distill samples.
  - Sec. 6.0: Updated list of equipment (added MICRO DIST and Lachat 8000 and 8500 series).
  - Sec. 9.1.1: Changed the frequency for the Method Blank analysis from 1 per 10 samples to 1 per 20 samples.
  - Sec. 9.1.2: Changed the frequency for the LLCS/HLCS analysis from 1 per 10 samples to 1 per 20 samples. Also, included the micro distillation preparation procedure for LLCS/HLCS.
  - Sec. 9.1.3: Clarified the requirement of one MS/MSD per 10 samples. Also, included the micro distillation preparation procedure for MS/MSD.
  - Sec. 10.1.4: Added procedure for micro distillation of samples.

- Revision 6, dated 22 March 2012
  - Sec. 1.1.1: added method 4500 CN G to the list of methods.
  - Sec 1.1.3, 12.1 & 12.2: Revised QAM references to reflect the most recent QAM revision.
  - Sec. 7.2.2/7.2.3: Added information on commercially purchased CN stock standard.

- Sec 9.1.3: Added MS/MSD requirement for cyanide amenable to chlorination under sample QC.
- Sec. 10.1.2.1: Added prep for soil samples requiring cyanide amenable to chlorination.
- Sec 11.6: Added calculation formula for CN amenable to chlorination; subsequent sections adjusted accordingly.
- Sec 15.9: Added applicable reference.

- Revision 5, dated 22 November 2010
  - Sec. 1.1.1: Added wipes to list of applicable matrices.
  - Sec. 1.1.2: Added reporting limit for wipes.
  - Sec. 6.2: Added specimen cups, transfer pipettes, graduated cylinder and micro-burets to the list of supplies.
  - Sec. 7.1.4: Changed the amount of acetic acid added from “approximately 100 ml” to “approximately 10 drops” to reflect actual laboratory practices.
  - Sec. 7.1.6.2: Clarified reagent prep
  - Sec. 8.0: Added wipes to section.
  - Sec. 9.1: Added sample QC requirements for wipe batches.
  - Sec. 9.1.1: Included the prep of the method blank for wipe batches
  - Sec. 9.1.2: Revised the prep of the LLCS and HLCS to reflect actual laboratory practices. Also, included prep of the LCS/LCSD for wipe batches.
  - Sec. 9.1.3: Revised MS/MSD prep for aqueous and soil.
  - Sec. 9.2.2: Deleted text “If the CCV is not within the control limits, a second analysis should be performed.”
  - Sec 9.2.3 & 9.2.4: Clarified the type of solution utilized for the ICB and CCB.
  - Sec. 10.1.3.1: Revised step of the distillation procedure to reflect actual laboratory practices.
  - Sec. 10.2.2.1: Revised calibration standards prep Table.
  - Sec. 10.3.2: Added comment on how to set up the analytical sequence for wipe samples.
  - Sec. 11.5: Included calculation for wipe samples.
  - Sec. 11.6: Added steps to import data to TALS.
  - Section 16: Added method revision for addition of wipe matrix to SW846 9012A/9012B.

- Revision 4, dated 28 September 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Incorporated SOP ED-WET-007 (Analysis of Total Cyanide in Drinking water-Automated, Method 335.4) into this SOP; retired SOP ED-WET-007 at the effective date of this SOP.
  - Sec. 7.2.2: Added information for the standardization of the Cyanide Stock. Replace frequency of the standardization from monthly to weekly to comply with Standard methods.
  - Sec 7.2.3 – 7.2.6: Added standards and preparation instructions.
  - Sec 9.1.3: Clarified % recovery limits for water and solid samples.
  - Sec 9.2.1 & 9.2.2: Clarified corrective actions to be taken if QCS/ICV and IPC/CCV results are outside the acceptable limits.
  - Sec 10.3.2: Revised sample analytical sequence to comply with method 335.4.
  - Sec. 11.5: Revised new data reduction procedures in accordance with new TALS.

- Sec. 15: Replace reference 335.2 and 335.3 with the EPA 335.4 reference; added applicable reference.
- Sec 16: Added Method modification to specify actual source of reagent information.

- Revision 3b, dated 13 July 2009
  - Sec 2.1: .For each batch of ten samples or less, a matrix spike and matrix spike duplicate will be distilled and analyzed. (Replaced 20 samples to 10 samples per batch).
  - Sec 9.5.1.3 & 9.5.2.2.: Revised Correlation coefficient from 0.997 to 0.995 as per corporate SOP on Calibration curves.
  - Sec 10.3: Up to 10 samples can be put in a batch. .Revised number of samples in a batch from 20 to 10 samples.

- Revision 3a, dated 13 March 2009
  - Sec 1.1: Delete Methods 335.1/.3; Add Method Lachat 10-204-00-1-A (Methods deleted in response to MUR).
  - Sec 3.6: Delete text: If this occurs, a Dow Corning 544 can be added to prevent the foam from collecting in the condenser. Replace the above procedure with: *If this occurs, samples can be acidified with acetic acid (1.6M) to pH 6.0-7.0.*
  - Sec 5.2: Add: Solution is stable for 6 months or when QC check is outside the acceptable limits, whichever comes first, store at 4ºC.
  - Sec 5.4: Acetate Buffer-Dissolve 82.0g of NaC2H3O2 x 3 H2O in 100ml DI water. Add glacial acetic acid drop by drop until the pH is 4.5. This solution is stable for 6 months, store at 4ºC. (Changed amount of acetic acid added to reflect actual laboratory practices).
  - Sec 5.6: Remove: Dow Corning 544 antifoam agent. Lab does not use this reagent , samples will be acidified with acetic acid.
  - Sec. 5.7.1, 5.8.2, 5.8.3, 5.8.4: Add: Solution is stable for 6 months, store at room temperature.
  - Sec 5.7.2: 0.25M NaOH. Add: Dissolve 10.0g NaOH into 1L deionized water. Solution is stable for 6 months, store at room temperature.
  - Sec. 5.9.1 & 5.9.2: Add: Store at room temperature.
  - Sec. 5.9.3: Phosphate buffer: In a 1L volumetric flask, dissolve 97g of potassium phosphate, monobasic, anhydrous (KH2PO4) in approximately 800ml of deionized water. Dilute to the mark. Solution is stable for one month. Store at room temperature. (Changed reagent preparation to comply with the Lachat method).
  - Sec 6.3: Add: Laboratory Control Sample (LCS): Prepare a 5ppm and 10ppm CN standard by diluting the 1000ppm Primary CN stock. Prepare fresh daily.
    - LLCS: Take 0.25ml of the 5ppm CN standard and bring up to a 50ml final volume with 0.25N NaOH. The true value is 0.025ppm.
    - HLCS: Take 1.0ml of the 10ppm CN standard and bring up to a 50ml final volume with 0.25N NaOH. The true value is 0.2ppm

  - Sec 9.10: Delete text: “Free Cyanide is estimated using the weak acid dissociable procedure following the colorimetric procedure without distillation. For reference refer to SM 4500-CN-A.” Revise the above text to: “*Free Cyanide will be treated following the procedure for the Cyanide Amenable to chlorination and therefore must be distilled.” See Section 9.2*

- Sec. 10.7: MS and MSD Recovery: Delete: The recovery must be within laboratory generated limits. Revise the above text to: *The recovery must be within 90-110% (*Revise MS/MSD recovery to comply with method 335.4).
- Sec. 15.1: References: Replace 335.2 & 335.3 reference with EPA 335.4 reference

Emerging Contaminants

Method Description	Matrix	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units
Per- and Polyfluoroalkyl Substances by LC/MS/MS	Solid	1633	1633_Shake	Perfluorobutanoic acid (PFBA)	375-22-4	0.800	0.200		ug/Kg
				Perfluoropentanoic acid (PFPeA)	2706-90-3	0.400	0.100		ug/Kg
				Perfluorohexanoic acid (PFHxA)	307-24-4	0.200	0.0500		ug/Kg
				Perfluoroheptanoic acid (PFHpA)	375-85-9	0.200	0.0500		ug/Kg
				Perfluorooctanoic acid (PFOA)	335-67-1	0.200	0.0500		ug/Kg
				Perfluorononanoic acid (PFNA)	375-95-1	0.200	0.0500		ug/Kg
				Perfluorodecanoic acid (PFDA)	335-76-2	0.200	0.0500		ug/Kg
				Perfluoroundecanoic acid (PFUnA)	2058-94-8	0.200	0.0500		ug/Kg
				Perfluorododecanoic acid (PFDoA)	307-55-1	0.200	0.0500		ug/Kg
				Perfluorotridecanoic acid (PFTrDA)	72629-94-8	0.200	0.0500		ug/Kg
				Perfluorotetradecanoic acid (PFTeDA)	376-06-7	0.200	0.0500		ug/Kg
				Perfluorobutanesulfonic acid (PFBS)	375-73-5	0.200	0.0500		ug/Kg
				Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	0.200	0.0500		ug/Kg
				Perfluorohexanesulfonic acid (PFHxS)	355-46-4	0.200	0.0500		ug/Kg
				Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	0.200	0.0500		ug/Kg
				Perfluorooctanesulfonic acid (PFOS)	1763-23-1	0.200	0.0500		ug/Kg
				Perfluorononanesulfonic acid (PFNS)	68259-12-1	0.200	0.0500		ug/Kg
				Perfluorododecanesulfonic acid (PFDoS)	79780-39-5	0.200	0.0500		ug/Kg
				1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2	757124-72-4	0.800	0.200		ug/Kg
				1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2	27619-97-2	0.800	0.200		ug/Kg
				1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2	39108-34-4	0.800	0.200		ug/Kg
				Perfluorooctanesulfonamide (PFOSA)	754-91-6	0.200	0.0500		ug/Kg
				N-methylperfluorooctane sulfonamide (NMeFOS	31506-32-8	0.200	0.0500		ug/Kg
				N-ethylperfluorooctane sulfonamide (NEtFOSA)	4151-50-2	0.200	0.0500		ug/Kg
				N-methylperfluorooctanesulfonamidoacetic acid (	2355-31-9	0.200	0.0500		ug/Kg
				N-ethylperfluorooctanesulfonamidoacetic acid (N	2991-50-6	0.200	0.0500		ug/Kg
				N-methylperfluorooctane sulfonamidoethanol (NM	24448-09-7	2.00	0.500		ug/Kg
				N-ethylperfluorooctane sulfonamidoethanol (NEtI	1691-99-2	2.00	0.500		ug/Kg
				Hexafluoropropylene Oxide Dimer Acid (HFPO-D	13252-13-6	0.800	0.200		ug/Kg
				4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	919005-14-4	0.800	0.200		ug/Kg
				Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	0.400	0.100		ug/Kg
				Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6	0.400	0.100		ug/Kg
				9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic	756426-58-1	0.800	0.200		ug/Kg
				11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic	763051-92-9	0.800	0.200		ug/Kg
				Perfluoro (2-ethoxyethane) sulfonic acid (PFEES	113507-82-7	0.400	0.100		ug/Kg
				3-Perfluoropropylpropanoic acid (3:3 FTCA)	356-02-5	1.00	0.250		ug/Kg
				3-Perfluoropentylpropanoic acid (5:3 FTCA)	914637-49-3	5.00	1.25		ug/Kg
				3-Perfluoroheptylpropanoic acid (7:3 FTCA)	812-70-4	5.00	1.25		ug/Kg
				13C4 PFBA	STL00992	2.50			ug/Kg
				13C5 PFPeA	STL01893	1.25			ug/Kg
				13C5 PFHxA	STL02577	0.625			ug/Kg
				13C4 PFHpA	STL01892	0.625			ug/Kg
				13C8 PFOA	STL01052	0.625			ug/Kg
				13C9 PFNA	STL02578	0.313			ug/Kg
				13C6 PFDA	STL02579	0.313			ug/Kg
				13C7 PFUnA	STL02580	0.313			ug/Kg
				13C2 PFTeDA	STL02116	0.313			ug/Kg
				13C3 PFBS	STL02337	0.583			ug/Kg
				13C3 PFHxS	STL02581	0.593			ug/Kg
				13C8 PFOS	STL01054	0.600			ug/Kg
				13C8 FOSA	STL01056	0.625			ug/Kg
				d3-NMeFOSAA	STL02118	1.25			ug/Kg
				d5-NEtFOSAA	STL02117	1.25			ug/Kg
				M2-4:2 FTS	STL02395	1.17			ug/Kg
				M2-6:2 FTS	STL02279	1.19			ug/Kg
				M2-8:2 FTS	STL02280	1.20			ug/Kg
				13C3 HFPO-DA	STL02255	2.50			ug/Kg
				d7-N-MeFOSE-M	STL02277	6.25			ug/Kg
				d9-N-EtFOSE-M	STL02278	6.25			ug/Kg
				d5-NEtPFOSA	STL02704	0.625			ug/Kg
				d3-NMePFOSA	STL02705	0.625			ug/Kg
				13C3-PFBA	STL02680	1.25			ug/Kg
				13C4 PFOA	STL00990	0.625			ug/Kg
				13C2 PFDA	STL00996	0.313			ug/Kg
				13C4 PFOS	STL00991	0.600			ug/Kg
				13C5 PFNA	STL00995	0.313			ug/Kg
				13C2 PFHxA	STL00993	0.625			ug/Kg
				18O2 PFHxS	STL00994	0.593			ug/Kg
				Perfluorodecanesulfonic acid (PFDS)	335-77-3	0.200	0.0500		ug/Kg
				Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	0.400	0.100		ug/Kg
				13C2-PFDoDA	STL02703	0.313			ug/Kg

Per- and Polyfluoroalkyl Substances by LC/MS/MS	NPW	1633	1633_SPE	Perfluorobutanoic acid (PFBA)	375-22-4	8.00	2.13		ng/L
				Perfluoropentanoic acid (PFPeA)	2706-90-3	4.00	1.00		ng/L
				Perfluorohexanoic acid (PFHxA)	307-24-4	2.00	0.500		ng/L
				Perfluoroheptanoic acid (PFHpA)	375-85-9	2.00	0.500		ng/L
				Perfluorooctanoic acid (PFOA)	335-67-1	2.00	0.500		ng/L
				Perfluorononanoic acid (PFNA)	375-95-1	2.00	0.500		ng/L
				Perfluorodecanoic acid (PFDA)	335-76-2	2.00	0.500		ng/L
				Perfluoroundecanoic acid (PFUnA)	2058-94-8	2.00	0.500		ng/L
				Perfluorododecanoic acid (PFDoA)	307-55-1	2.00	0.500		ng/L
				Perfluorotridecanoic acid (PFTrDA)	72629-94-8	2.00	0.500		ng/L
				Perfluorotetradecanoic acid (PFTeDA)	376-06-7	2.00	0.500		ng/L
				Perfluorobutanesulfonic acid (PFBS)	375-73-5	2.00	0.500		ng/L
				Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	2.00	0.500		ng/L

Method Description	Matrix	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units
				Perfluorohexanesulfonic acid (PFHxS)	355-46-4	2.00	0.517		ng/L
				Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	2.00	0.500		ng/L
				Perfluorooctanesulfonic acid (PFOS)	1763-23-1	2.00	0.500		ng/L
				Perfluorononanesulfonic acid (PFNS)	68259-12-1	2.00	0.500		ng/L
				Perfluorododecanesulfonic acid (PFDoS)	79780-39-5	2.00	0.500		ng/L
				1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2	757124-72-4	8.00	2.00		ng/L
				1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2	27619-97-2	8.00	2.00		ng/L
				1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2	39108-34-4	8.00	2.00		ng/L
				Perfluorooctanesulfonamide (PFOSA)	754-91-6	2.00	0.500		ng/L
				N-methylperfluorooctane sulfonamide (NMeFOS	31506-32-8	2.00	0.500		ng/L
				N-ethylperfluorooctane sulfonamide (NEtFOSA)	4151-50-2	2.00	0.500		ng/L
				N-methylperfluorooctanesulfonamidoacetic acid (	2355-31-9	2.00	0.620		ng/L
				N-ethylperfluorooctanesulfonamidoacetic acid (N	2991-50-6	2.00	0.521		ng/L
				N-methylperfluorooctane sulfonamidoethanol (NM	24448-09-7	20.0	5.00		ng/L
				N-ethylperfluorooctane sulfonamidoethanol (NEt	1691-99-2	20.0	5.00		ng/L
				Hexafluoropropylene Oxide Dimer Acid (HFPO-D	13252-13-6	8.00	2.00		ng/L
				4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	919005-14-4	8.00	2.00		ng/L
				Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	4.00	1.00		ng/L
				Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6	4.00	1.22		ng/L
				9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic	756426-58-1	8.00	2.00		ng/L
				11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic	763051-92-9	8.00	2.00		ng/L
				Perfluoro (2-ethoxyethane) sulfonic acid (PFEES	113507-82-7	4.00	1.00		ng/L
				3-Perfluoropropylpropanoic acid (3:3 FTCA)	356-02-5	10.0	2.50		ng/L
				3-Perfluoropentylpropanoic acid (5:3 FTCA)	914637-49-3	50.0	12.5		ng/L
				3-Perfluoroheptylpropanoic acid (7:3 FTCA)	812-70-4	50.0	14.9		ng/L
				13C4 PFBA	STL00992	100			ng/L
				13C5 PFPeA	STL01893	5.00			ng/L
				13C5 PFHxA	STL02577	25.0			ng/L
				13C4 PFHpA	STL01892	25.0			ng/L
				13C8 PFOA	STL01052	25.0			ng/L
				13C9 PFNA	STL02578	12.5			ng/L
				13C6 PFDA	STL02579	12.5			ng/L
				13C7 PFUnA	STL02580	12.5			ng/L
				13C2 PFTeDA	STL02116	12.5			ng/L
				13C3 PFBS	STL02337	24.0			ng/L
				13C3 PFHxS	STL02581	24.0			ng/L
				13C8 PFOS	STL01054	25.0			ng/L
				13C8 FOSA	STL01056	25.0			ng/L
				d3-NMeFOSAA	STL02118	50.0			ng/L
				d5-NEtFOSAA	STL02117	50.0			ng/L
				M2-4:2 FTS	STL02395	50.0			ng/L
				M2-6:2 FTS	STL02279	50.0			ng/L
				M2-8:2 FTS	STL02280	50.0			ng/L
				13C3 HFPO-DA	STL02255	100			ng/L
				d7-N-MeFOSE-M	STL02277	250			ng/L
				d9-N-EtFOSE-M	STL02278	250			ng/L
				d5-NEtPFOSA	STL02704	25.0			ng/L
				d3-NMePFOSA	STL02705	25.0			ng/L
				13C3-PFBA	STL02680	5.00			ng/L
				13C4 PFOA	STL00990	25.0			ng/L
				13C2 PFDA	STL00996	12.5			ng/L
				13C4 PFOS	STL00991	24.0			ng/L
				13C5 PFNA	STL00995	12.5			ng/L
				13C2 PFHxA	STL00993	25.0			ng/L
				18O2 PFHxS	STL00994	24.0			ng/L
				Perfluorodecanesulfonic acid (PFDS)	335-77-3	2.00	0.500		ng/L
				Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	4.00	1.00		ng/L
				13C2-PFDoDA	STL02703	12.5			ng/L
Semivolatile Organic Compounds (GC/MS)	Solid	8270E	3546	1,4-Dioxane	123-91-1	0.0330	0.0289		mg/Kg
				2,4,6-Tribromophenol (Surr)	118-79-6				mg/Kg
				2-Fluorobiphenyl	321-60-8				mg/Kg
				2-Fluorophenol (Surr)	367-12-4				mg/Kg
				Nitrobenzene-d5 (Surr)	4165-60-0				mg/Kg
				Phenol-d5 (Surr)	4165-62-2				mg/Kg
				Terphenyl-d14 (Surr)	1718-51-0				mg/Kg
Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)	NPW	8270E_SIM_MS_ID	3510C_LVI	1,4-Dioxane	123-91-1	0.200	0.0720		ug/L
				1,4-Dioxane-d8	17647-74-4	0.400			ug/L

LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
40	150	30	40	150	30		
40	150	30	40	150	30		
40	150	30	40	150	30		

LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
57	146		57	146	30		
55	152		55	152	30		
58	149		58	149	30		
52	148		52	148	30		
36	145		36	145	30		
67	146		67	146	30		
61	151		61	151	30		
63	152		63	152	30		
61	148		61	148	30		
63	145		63	145	30		
65	139		65	139	30		
58	144		58	144	30		
59	146		59	146	30		
71	136		71	136	30		
69	137		69	137	30		
63	144		63	144	30		
68	146		68	146	30		
55	148		55	148	30		
48	161		48	161	30		
56	156		56	156	30		
46	156		46	156	30		
56	151		56	151	30		
62	129		62	129	30		
63	134		63	134	30		
50	138		50	138	30		
10	130		10	130	30		
40	150		35	150	30		
40	150		55	150	30		
40	150		55	150	30		
30	140		60	140	30		
30	140		55	140	30		
20	140		50	140	30		
20	140		30	140	30		
10	130		10	130	30		
25	150		55	150	30		
25	150		55	150	30		
20	140		45	140	30		
10	130		30	130	30		
10	200		45	200	30		
10	200		10	200	30		
25	200		60	200	30		
25	200		60	200	30		
25	200		50	200	30		
25	160		25	160	30		
10	150		10	150	30		
10	150		10	150	30		
10	130		10	130	30		
10	130		15	130	30		
					30		
					30		
					30		
					30		
					30		
					30		
					30		
51	147		51	147	30		
51	145		51	145	30		
10	150		10	150	30		

30	120	30	30	120	30		
		30			30	24	137
		30			30	48	120
		30			30	31	120
		30			30	38	120
		30			30	39	120
		30			30	25	126

50	142	20	50	142	20		
		20			20	10	150

Title: Method SW846 3060A, The Alkaline Digestion of Soil and Wipe Samples for the Analysis of Hexavalent Chromium

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo 01/07/21	Dan Helfrich 01/07/21
Jasmine Parillo Date	Dan Helfrich Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 01/07/21	01/07/21
Carl Armbruster Date	Mark Acierno Date
Quality Assurance Manager	Laboratory Director

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 SW846 Method 3060A is an alkaline digestion procedure for extracting Hexavalent Chromium [Cr(VI)] from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and similar waste materials. Materials applicable to this method include soil, sediment, debris, brick, concrete, sludge, and precipitate. TestAmerica Edison has modified the method to include digestion of surface wipe samples. To quantify total Cr(VI) in a solid matrix, three criteria must be satisfied: 1) the extracting solution must solubilize all forms of Cr(VI), 2) the conditions of the extraction must not induce reduction of native Cr(VI) to Cr(III), and 3) the method must not cause oxidation of native Cr(III) contained in the sample to Cr(VI). Method 3060A meets these criteria for a wide spectrum of solid matrices.

1.1.2 Method 3060A digestates can be analyzed for hexavalent chromium using Method 7196 (colorimetrically by UV-VIS spectrophotometry). Reference the applicable analytical method SOP for details on reporting limits.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. This method uses an alkaline digestion to solubilize both water-insoluble (with some exceptions) and water soluble Cr(VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during the digestion procedure. The sample is digested using 0.28M Na_2CO_3/0.5M NaOH solution and heating at 90-95ºC for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III).

2.2. Under the alkaline conditions of the extraction, minimal reduction of Cr (VI) or oxidation of native Cr(III) occurs. The addition of Mg^{+2} in a phosphate buffer to the alkaline solution has been shown to suppress oxidation, if observed. The accuracy of the extraction procedure is assessed using spike recovery data for soluble and insoluble forms of Cr(VI) (e.g. K_2Cr2O_7 and $PbCrO_4$), coupled with measurement of ancillary soil properties, indicative of the potential for the soil to maintain a Cr(VI) spike during digestion, such as oxidation reduction potential (ORP), pH, organic matter content, ferrous iron, and sulfides. Recovery of an insoluble Cr(VI) spike can be used to assess the first two criteria, and method-induced oxidation is usually not observed except in soils high in Mn and amended with soluble Cr(III) salts or freshly precipitated $Cr(OH)_3$.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 When analyzing a sample digestate for total Cr (VI), the reducing/oxidizing tendency of each sample matrix can be determined at the client's request. Establishing the tendency of Cr(VI) to exist or not exist in the unspiked sample(s), assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals.

4.1.1 This can be accomplished by characterization of each sample for additional analytical parameters such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.2 Certain substances, not typically found in the alkaline digests of soils, may interfere in the analytical methods for Cr(VI) following alkaline extraction if the concentrations of these interfering substances are high and the Cr(VI) concentration is low. Refer to the SOP for Method 7196A for the specific agents that may interfere with Cr(VI) quantification.

4.3 For waste materials or soils containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(VI) results obtained using this method may be biased high due to method-induced oxidation. The addition of Mg^{2+} in a phosphate buffer to the alkaline extraction solution has been shown to suppress this oxidation

4.4 A sample containing a high buffering capacity may need to be digested using additional digestion solution.

4.5 It is very important to monitor the temperature of the digestion solution during the digestion to maintain a temperature of 90-95^{o}C during the entire time period.

4.6 Do not use Ghost wipe, Whatman, mixed cellulose ester (MCE) or glass fiber filters for sampling wipes because they convert Cr(VI) to Cr(III). Use either PVC filters or binderless quartz fiber filters.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.

6.0 Equipment and Supplies

6.1. Equipment

- Digestion vessel: water bath equipped with shaker (Precision, Model 50)
- pH meter: Orion benchtop or equivalent
- Vacuum filtration apparatus
- Analytical balance capable of accurate weighings to 0.01 grams

6.2. Supplies

- Class A volumetric flasks with stoppers
- Filter paper 0.45 and 0.1 um size.
- Nalgene 115-ml filter disposable unit with 0.45um filter
- Binderless quartz fiber filter, 37 or 47 mm diameter, 0.45 mm thick (for wipe analysis)
- Digestion container: 125ml Nalgene small-mouth polypropylene bottles
- Certified thermometer (NIST traceable)-capable of reading 100ºC
- Finnpipet (1-5 ml) with tips
- Graduated cylinder (class A)
- Specimen cups
- Transfer pipettes
- Wood spatulas

7. Reagents and Standards

7.1. Reagents

7.1.1. The following Reagent grade chemicals are used for all solutions required in this test. For stability information, refer to manufacturer's instructions or 2 years from date opened if no instructions are available. All reagents are stored at room temperature:

7.1.1.1. Concentrated Nitric acid
7.1.1.2. Anhydrous Sodium Carbonate
7.1.1.3. Sodium Hydroxide
7.1.1.4. Potassium Dichromate
7.1.1.5. Magnesium Chloride-$MgCl_2$ (anhydrous)
7.1.1.6. Potassium Phosphate, Dibasic
7.1.1.7. Potassium Phosphate, Monobasic

7.1.2. Phosphate Buffer- 0.5M K_2HPO_4/0.5M KH_2PO_4 buffer at pH 7: Dissolve 87.09g K_2HPO_4 and 68.04g KH_2PO_4 into 700ml reagent water. Bring up to a final volume of 1 liter. Store at room temperature. Stable for 6 months.

7.1.3. Digestion Solution: Weigh 20g of NaOH and 30g of Na_2CO_3 and dissolve in a 1 liter volumetric flask using Type I water. Dilute to the mark and then transfer the solution to a tightly capped polyethylene bottle. Store at room temperature. Prepare fresh monthly. **Note**: Check the pH of the digestion solution to be certain it has a pH of greater than 11.5 before using it. If the pH drops below 11.5, a new solution must be prepared.

7.1.4. 1:1 HNO3: Add 500ml DI to a 1 Liter volumetric flask and then slowly add 500ml of concentrated HNO3. Solution is stable for 6 months. Store at room temperature.

7.2. Standards

7.2.1. Potassium Dichromate Primary Standard Solution (1000 mg/L Cr+6):

Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3093-01 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. Store at room temperature. Solution is stable for 6 months.

7.2.2. Potassium Dichromate Secondary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3090-04 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. **Note**: Prepare the Secondary standard solution from a different source than what was used to prepare the primary standard solution. Stable for 6 months, store at room temperature.

7.2.3. Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 primary standard (Section 7.2.1) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.4. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 Secondary standard (Section 7.2.2) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.5. Lead Chromate-AR Grade; for storage and stability information, refer to manufacturer's instructions

8. Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container[1]	Min. Sample Size	Preservation	Holding Time[3]	Reference
Soils	Glass or plastic	2.50g[2]	Cool 4 ±2ºC	30 Days	SW 846 Method 3060A
Wipes[4]	Glass or plastic	1 wipe	Cool 4 ±2ºC	30 Days	SW 846 Method 3060A

[1] Containers should not contain stainless steel
[2]Additional volume may be required if the sample is chosen for QC or if a re-prep is required.
[3]The alkaline digestate must be analyzed within 168 hours after extraction from the soil or wipe.
[4]In chrome plating environments, wipe samples taken on a PVC filter or an uncoated binderless quartz fiber filter, should be placed in a vial containing 5 mL of an aqueous solution containing 10% Na_2CO_3 with 2% $NaHCO_3$ immediately after sampling to eliminate the interference from the acid used in the chrome plating process. An alternate medium

which does not require extraction in the field is a binderless quartz fiber filter coated with 1% NaOH.

9. Quality Control

9.1. Sample QC - The following quality control samples are prepared daily with each batch of 20 samples or less, whichever is more frequent. Soil and wipe samples must be divided into separate QC batches.

9.1.1. Method Blank:

- *Soil Samples:* One method blank must be digested and analyzed per soil batch. A method blank consists of 50 ml digestion solution and is carried through the entire analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

- *Wipe Samples:* One method blank must be digested and analyzed per wipe batch. The method blank will consist of a blank wipe and 50 ml digestion solution and will be carried through the entire analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

9.1.2. Laboratory Control Sample (Soils and wipes): Laboratory control samples from Secondary sources, shall be digested and analyzed per batch as follows:

9.1.2.1 Soil LCS:

- **LCS Soluble (LCSSRM):** Obtained from an independent source (ERA). Prepare the LCS soluble solution by diluting the concentrated LCS solution as indicated in the manufacturer's instructions. If necessary, prepare a different dilution so that the concentration after analysis falls within the calibration curve. Add 5ml of the diluted LCS soluble solution into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be re-digested and re-analyzed. Document the preparation of the LCS soluble solution in the TALS reagent module.

- **LCS Insoluble (LCSI):** Prepared by adding 0.011g of PbCrO4 into 50ml digestion solution. The results must be within 80-120% of the true value or the entire batch must be re-digested and re-analyzed.

- **LCS matrix matched (LCSSRM):** Obtained from an independent source (ERA). Add 2.5g of the soil LCS into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be re-digested and re-analyzed. This LCSSRM is included in DKQP guidelines however it is not reported to clients.

9.1.2.2 Wipe LCS/LCSD:

For wipe sample batches, a Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate must be prepared using the LCS soluble solution. Add a blank wipe to the digestion bottles for each of the LCS and LCSD and follow the prep for LCS Soluble detailed in Section 9.1.2.1 above.

9.1.3. **Predigestion Matrix Spikes (Soils only):** Both the soluble and insoluble pre-digestion matrix spikes must be analyzed per batch of ≤ 20 soil samples.

9.1.3.1. **Soluble Matrix Spike (MSS):** spike the sample with 1.0 ml of the 100 ppm Cr (VI) primary spiking solution prepared in Section 7.2.3 (equivalent to 40mg/kg Cr (VI)).

9.1.3.2. **Insoluble Matrix Spike (MSI):** to a separate sample aliquot add 0.011g of $PbCrO_4$. It is used to evaluate the dissolution during the digestion process.

9.1.3.3. Both matrix spikes are then carried through the digestion process. The acceptance range for the matrix spike recoveries is 75-125%. If the matrix spike recoveries fall outside these recovery limits, the entire batch must be rehomogenized, redigested, and reanalyzed.

9.1.3.4. If either matrix spike recovery is outside of the acceptable range of 75-125% but, the LCS is within the criteria specified in Section 9.1.2, additional laboratory characterization of each sample in the batch for ORP and pH can be performed at the client's request to determine if the sample exhibits reducing conditions. Further characterization for total sulfides, total organic carbon, chemical oxygen demand, or biological oxygen demand may be required by the client.

9.1.4. **Duplicate (DU) (Soils only):** One duplicate laboratory sample must be analyzed per soil batch. The sample used for the predigestion spike should be used for this purpose. Duplicate samples must have a Relative Percent Difference (RPD) of ≤20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± 2.0mg/kg (laboratory reporting limit) is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

9.1.5. **Post Digestion Spike (PDS) (Soils only):** Following the analysis (colorimetric determination), a post-digestion spike must be analyzed per soil batch. The spike concentration must be equivalent to 40mg/kg or twice the sample concentration, whichever is greater. The post digestion spike must be performed on a field sample, not on a field blank or preparation blank. It would be helpful to perform this analysis on the sample used for the matrix spike. Recovery limits for the post-digestion spike are 85-115%. If the post digestion spike fails to meet the recovery limits, a new aliquot of the sample

must be re-spiked and re-analyzed as described in SOP ED-WET-011 (The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

9.2. Instrument QC

See applicable analytical SOP, ED-WET-011 (The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

10. Procedure

10.1. Sample Preparation

10.1.1. Homogenize soil samples following TestAmerica Edison SOP ED-GEN-007 (Subsampling).

10.1.2. Weigh 2.5g ± 0.10g of sample into a clean labeled 125ml nalgene bottle. Record weight up to two decimal places. For wipe samples, add 1 sample wipe into a clean labeled 125 ml nalgene bottle.

10.1.3. Add 50 +/- 1.0ml of digestion solution to each sample. Then add 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.4. Matrix spike preparation (soils only): Spike the samples designated MS soluble and MS insoluble as detailed in Section 9.1.3. Add 50 +/- 1.0ml of digestion solution to each sample followed by 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.5. LCS preparation: For soil samples, prepare as detailed in Section 9.1.2.1. For wipe samples, prepare as detailed in Section 9.1.2.2. Add 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.6. Place all samples in hot water bath at a temperature of 90-95ºC and begin shaking at 175rpm for 5 minutes. Then release pressure by venting the caps. After the caps are resealed tightly, begin heating and shaking for 60 minutes. Start timing after the water bath has returned to 90-95ºC and maintain this temperature throughout the 60 minute digestion.

10.1.7. Measure and record the digestion start time and temperature, time and temperature after 30 minutes, and the digestion end time and temperature. Use a calibrated thermometer to measure the temperature of one of the samples in the water bath.

10.1.8. Following the heated digestion, gradually cool the solution to room temperature and quantitatively transfer the contents of the bottle to the vacuum filtration apparatus with Type I water rinses. Filter the digestate through a 0.45um filter (commercially purchased filter). Transfer the rinsates and filtrate to a clean labeled 100ml plastic cup. (**Note**: Use a polyethylene bottle if the colorimetric analysis is not following immediately).

10.1.9. Determine the concentration of hexavalent chromium in the sample extract using Method 7196A USEPA SW846 3rd Edition, (TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

10.2. Calibration:

Refer to the applicable analytical SOPs: TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A.

10.3. Sample Analysis

Refer to the applicable analytical SOPs: TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A.

11.0. Calculations / Data Reduction

11.1. Calculations:

Refer to the applicable analytical SOP (ED-WET-011) for calculations.

11.2. Data Reduction:

11.2.1. All sample preparation information is recorded directly in the logbook and in TALS at the time of sample preparation.

11.2.2. Record reagent information in the prep batch information (this can be viewed in the “batch information” page).

11.2.3. Record special comments and observations in the ‘worksheet’ tab.

11.2.4. The logbook pages for the prep are attached to the analytical batch as pdf files.

11.2.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.2.5.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.2.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on ‘Run checklist.’

11.2.5.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose ‘Acknowledge Item.’

11.2.5.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium.

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Sample waste-After analysis, the samples and standards are collected in a polyethylene container labeled 'Cr (VI) waste.' Once the container is full, it is brought to the waste room for disposal.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 3060A.

15.2. Occupational Health and Safety Administration (OSHA) Method W4001, Hexavalent Chromium, Methods Development Team, OSHA Salt Lake Technical Center, Salt Lake City, UT, April 2001.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. Test America Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A, most current revision.

16.0. Method Modifications:

The method has been modified to include procedures specific to the digestion of wipe samples.

17.0. Attachments

N/A

18.0. Revision History

- **Revision 13.1, dated 08 January 2021**
  - Updated SOP header with Eurofins emblem; no procedural change.
  - Sec 3: Revised the Definitions location as per the most current LQM.

- **Revision 13, dated 14 February 2019**

  - Sec. 9.1.2: Added matrix matched LCS applicable to DKQP projects.
  - Sec. 11.2.5: Revised the use of data review paper checklist to TALS DRC review, instructions added.
  - Deleted reference WI# EDS-WI-008 in Sec 15.7, not applicable.
  - Updated approval signatures to current.

- **Revision 12, dated 20 November 2014**

  - Removed references to SW846 7199 throughout the SOP; laboratory no longer perform this method.
  - Sec. 9.1.2: Revised the LCSS limits from +/-15% to vendor's certified limits to reflect actual laboratory practices.
  - Sec. 9.1.3.3: Removed notation that redigestion/reanalysis of batch is only for NJ sites or with PM/client's advice; Re-digestion and reanalysis is performed for all failed QC batch and is a method requirement.
  - Sec. 10.1.7: Revised procedures for checking sample temperature during sample digestion; temperature of the sample will be checked at start, middle and end of digestion.

- **Revision 11, dated 11 December 2012**

  - Sec 1 and 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 9.1.2 (LCSI) and 9.1.3.2 (MSI): Deleted 0.012g to reflect actual laboratory practices.

- **Revision 10, dated 18 November 2010**

  - Section 1.1: included statement regarding the modification of the method for preparation of wipe samples.
  - Section 1.1.2: removed soil RL details and included statement referring reader to the applicable method SOP for RL details.
  - Section 4.1: Oxidizing/reducing parameters will be analyzed if requested by the client.
  - Section 4.6: Added interferences for wipe samples.
  - Section 6.2: Removed bottle top dispenser from list of supplies. Added graduated cylinder, specimen cups, transfer pipettes, and binderless quartz fiber filters to the list of supplies.
  - Section 7.1: Reformatted Reagents Section. Added Potassium Phosphate, Dibasic and Potassium Phosphate, Monobasic to list of reagents.
  - Section 8.0: Added wipes to the sample collection, preservation, shipment, and storage Section.

- Section 9.1: Modified Sample QC Section to specify wipe criteria. Added Sections 9.1.2.1 and 9.1.2.2 to differentiate the LCS procedures for soils and wipes.
- Section 9.1.3.4: Section was revised to specify the additional laboratory characterization only when required by the client.
- Sections 9.1.3, 9.1.4 and 9.1.5: added language to clarify that these QC types apply only to soils (and not to wipes).
- Section 10.1.2: added procedures for wipe sample preparation.
- Section 10.1.5 and 10.1.6: added to detail the QC sample (LCS and matrix spike) prep procedures for soils and wipes.
- Section 11.0: Replaced all calculations with a reference to the applicable analytical method SOP. Add
- Section 15.0: Added reference for OSHA Method W4001, Hexavalent Chromium (wipe sample methodology).
- Section 16.0: added method modification language for addition of wipe samples.

- **Revision 9, dated 03 February 2010**
  - Section 9.1.3.1: Changed MS to MSS to be consistent with TALS naming convention.
  - Section 10.1.3: Added Section (previously part of Section 10.1.4) to clarify that spike should be added directly to the sample aliquot prior to the addition of the digestion solution, magnesium chloride, and phosphate buffer. Subsequent Sections adjusted.
  - Section 10.1.4: Section added to include LCS soluble and LCS insoluble. Subsequent Sections adjusted.

- **Revision 8, dated 25 August 2009**
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1.2: Added reporting limit.
  - Section 7.1.8: Added prep information.
  - Removed midpoint calibration checks standard (CCS) and Calibration blank in the QC Section, both QC samples are not applicable to this SOP.
  - Deleted pH adjustment procedure, previously named Section 9.6; this procedure will be included in the analytical SOP.
  - Section 15: Added applicable references.

- **Revision 7b, dated 30 April 2009**
  - Section 5.6: Added stability and storage information.
  - Section 5.7: Added stability information
  - Section 5.8: Added stability information

- Section 10.8: Added information about the pH adjusted PS to conform to method requirements.
- Section 15: Added applicable references.

- **Revision 7a, dated 10 February 2009**

  - Section 4.4: Add filter size to Nalgene 115-ml disposable filter.
  - Section 4.10: Add Bottle top dispenser use for adding the digestion solution.
  - Section 4.11: Add Wood spatulas; Use wooden spatulas instead of the stainless steel spatulas.
  - Section 6.1: Delete pre-made standard purchased from Inorganic Ventures. Cr6+ Primary Standard will be prepared in the lab.
  - Section 6.2: Specified source of the 1000 ppm Cr6+
  - Section 6.3 and 6.6: Delete 10mg/l Cr6+ standards; Working standard 10mg/L Cr+6 is not utilize for soil analysis.
  - Section 10.3: expanded LCS information to include preparation information for both LCSs.
  - Section 10.4.2: Specified the exact amount of PbCrO4 added to the insoluble matrix spike.
  - Section 6.5: Added storage information.
  - Section 6.4: Added text: source must be different from the primary standard.
  - Section 9.3: Expanded Section to include the spiking of LCS and MS.
  - Section 9.4: Expanded Section to include recording specific information for the water bath.

- **Revision 7, dated March 2007**

  - Section 10.3: Revised to specify the types of LCS used including their source and acceptance limits.
  - Section 10.6: Deleted the requirement that ‘CCS is taken through the entire digestion process to reflect actual laboratory practices.

- **Revision 6, dated 30 October 2006**

  - Section 9.6.3: Corrected referenced SOP number from EDS-WET-010 to EDS-WET-011.
  - Section 10.5:Revised first sentence to read “One duplicate laboratory sample per batch *must* be analyzed.” (‘Must’ previously read ‘should’).
  - Section 10.4.3: The text in italics was added to the following sentence in Section 10.4.3:
    “NOTE: If the spiked sample concentration is greater than 4x the predigestion spike concentration, no redigestion/reanalysis is required unless the samples are from a NJ site in which case the lab project manager should be contacted for further guidance.”
  - Section 10.8: Added the following text:
    “If the Post Digestion Spike fails to meet recovery limits, a new aliquot of the sample must be re-spiked and re-analyzed.

Title: The Analysis of Digestates for Hexavalent Chromium by EPA SW846 7196A

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This SOP is applicable to the determination of hexavalent chromium digestates of solid samples using SW846 Method 7196A. Solid samples must be first digested according to Method 3060A USEPA SW846 3rd Edition, TestAmerica Edison SOP ED-WET-010 (The Alkaline Digestion of Soil Samples for the analysis of hexavalent chromium).

1.1.2 The laboratory's reporting limit for hexavalent chromium in soil is 2.0 mg/kg.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Solid samples are digested utilizing SOP ED-WET-010, alkaline digestion of soil samples via Method 3060A. Following the sample preparation and digestion procedure, dissolved hexavalent chromium is determined spectrophotometrically at 540 nm by reaction with diphenylcarbazide in acid solution.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

4.2 Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of Cr (VI) present.

4.3 Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

4.4 Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time; however, interference is checked by post digestion spike samples.

4.4.1 The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon

(TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.5 For waste materials or soils containing soluble Cr (Ill) concentrations greater than four times the laboratory Cr (VI) reporting limit, Cr (VI) results obtained using this method may be biased high due to method-induced oxidation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at an elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

6.0 Equipment and Supplies

6.1. Instrument

- Spectrophotometer for use at 540 nm, providing a light path of 1cm or longer
- pH meter, Orion benchtop or equivalent

6.2. Supplies

- Nalgene 115-ml disposable filter unit with 0.45um filter
- Membrane filters, 0.45um and 0.1 um
- Specimen cups (plastic)
- Eppendorf or Finnpipettes, varying volumes
- Class A volumetric flasks
- Class A graduated cylinder
- Vacuum pump
- Magnetic stirrer (Teflon coated)
- Transfer pipettes

7. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric acid (10% v/v): Measure 100 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water. Stable for six months, store at room temperature.

7.1.2. Indicator solution (DPC): Place 5.0 g 1,5-Diphenylcarbazide (AR Grade) in an amber 1000 ml volumetric flask and dilute to mark with acetone. Prepare monthly or when solution becomes cloudy. Store at room temperature.

7.1.3. Acetone: reagent grade; for stability and storage information refer to manufacturer's instructions.

7.1.4. 1:1 HNO3: Add 500ml DI to a 1 Liter volumetric flask and then slowly add 500ml of concentrated reagent grade HNO3. Solution is stable for 6 months. Store at room temperature.

7.1.5. 1N NaOH: Place 4.0g reagent grade NaOH in a 100ml volumetric flask and dilute to the mark with deionized water. Stable for six months, store at room temperature.

7.2 Standards

7.2.1 Potassium Dichromate Primary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3093-01 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. Stable for 6 months, store at room temperature.

7.2.2 Potassium Dichromate Secondary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3090-04 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. **Note**: Prepare the secondary standard from a source or lot different from the primary standard solution. Stable for 6 months, store at room temperature.

7.2.3 Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6): Prepare by adding 10 ml of 1000 mg/L Cr+6 primary standard (Sec 7.2.1) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.4 Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 secondary standard (Sec 7.2.2) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.5 Lead Chromate: AR Grade; for stability and storage information refer to manufacturer's instructions.

8. Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container [1]	Min. Sample Size[2]	Preservation	Holding Time [3]	Reference
Soils	Glass, plastic	2.5 grams	Cool 4 ± 2°C	30 Days - *from sampling to extraction*	SW846 Method 3060A
				168 Hours – *from extraction to determinative analysis*	SW846 Method 7196A

[1] Containers should not contain stainless steel
[2] Additional volume may be required if the sample is chosen for QA or if a re-prep is required.
[3]The alkaline digestate must be analyzed within 168 hours after extraction from the soil.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of 20 samples or less.

9.1.1. Method Blank: One method blank must be digested and analyzed per batch. A method blank consists of 50 ml digestion solution and is carried through the entire digestion and analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

9.1.2. Laboratory Control Sample: Two secondary source (LCS) shall be digested and analyzed per batch.

- **LCS soluble (LCSSRM)** is obtained from an independent source (Phenova). Prepare the LCS soluble solution by diluting the concentrated LCS solution as indicated in the manufacturer's instructions. If necessary, prepare a different dilution so that the concentration after analysis falls within the calibration curve. Add 5ml of the diluted LCS soluble solution into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be redigested and re-analyzed. Document the preparation of the LCS soluble solution in the Reagent module in TALS.

- **LCS insoluble (LCSI)** is prepared by adding 0.010g-0.020g of PbCrO4 into 50ml digestion solution. The results must be within 80-120% of the true value or the entire batch must be redigested and re-analyzed. (Typical amount of $PbCrO_4$ added to LCSI is 0.011g, this is equivalent to 708 mg Cr (VI)/Kg).

9.1.3. Predigestion Matrix Spikes: Both a soluble and insoluble pre-digestion matrix spikes must be analyzed per batch of ≤ 20 field samples.

9.1.3.1 Soluble matrix spike (MSS): spike the sample with 1.0 ml of the 100 ppm Cr (VI) primary spiking solution prepared in Sec 7.2.3 (equivalent to 40mg/kg Cr(VI)).

9.1.3.2 Insoluble matrix spike (MSI): to a separate sample aliquot add 0.010g- 0.020g of $PbCrO_4$. It is used to evaluate the dissolution during the digestion process. Typical amount of $PbCrO_4$ added to LCSI is 0.011g, this is equivalent to 708 mg Cr (VI)/Kg.

9.1.3.3 Both matrix spikes are then carried through the digestion process. The acceptance range for the matrix spike recoveries is 75-125%. If the matrix spike recoveries fall outside these recovery limits, the entire batch must be rehomogenized, redigested, and reanalyzed.

- Note: If redigesting/reanalyzing the batch, the Soluble matrix spike (MSS) is spiked at twice the sample concentration or 40mg/kg Cr(VI) whichever is greater.

9.1.3.4 If upon reanalysis, the matrix spike is not within the recovery limits of 75%-125%, but the LCS is within the criteria specified in Section 9.1.2, additional laboratory characterization of each sample in the batch for ORP (see TestAmerica Edison SOP ED-WET-066, Redox by SM2580) and pH (see TestAmerica Edison SOP ED-WET-061, pH Soil by SW846 9045C) may be required by the client to determine if the sample exhibits reducing conditions. Further characterization for total sulfides, Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), or Biochemical Oxygen Demand (BOD) may also be required by the client.

9.1.4. Duplicate (DU): One duplicate laboratory sample must be analyzed per batch. The sample used for the predigestion spike should be used for this purpose. Duplicate samples must have a Relative Percent Difference (RPD) of ≤20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± 2.0mg/kg (laboratory reporting limit) is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

9.1.5. Post Digestion Spike (PDS): Following the analysis (colorimetric determination), a post-digestion spike must be analyzed per batch. The spike concentration must be equivalent to 40mg/kg or twice the sample concentration, whichever is greater. To spike the extract with 40mg/Kg Cr

(VI), add 0.50 ml of 100 ppm Cr (VI) primary standard to a 50.0 ml alkaline extract, pour out 45.0 ml of the spiked sample and add color indicator and adjust pH (see Sec 10.3.4 & 10.3.5); bring the final volume to 50ml with deionized water. The post digestion spike must be performed on a field sample, not on a field blank or preparation blank. If possible perform the post digestion spike analysis on the sample used for the matrix spike. Recovery limits for the post-digestion spike are 85-115% of the true value. If the post digestion spike fails to meet the recovery limits, a post spike must be re-analyzed using the alkaline extract. Adjust the pH of the sample Duplicate (alkaline extract) back up to 8.0-8.5 using 1N NaOH; then respike and reanalyze following Sec. 10.3.4-10.3.5.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV (0.50 mg/L Cr (VI)) is analyzed immediately after an acceptable initial calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within ±10% of the expected value. The ICV is prepared and analyzed similar to the calibration standards; prepare by adding 0.5 ml of 100 ppm Cr (VI) Secondary Spiking Solution (Sec. 7.2.4) into a specimen cup containing 50 ml of digestion fluid. If the measured concentration exceeds the ±10% limit, a second analysis should be performed. If the result still exceeded the ±10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.2. Continuing Calibration Verification (CCV): A CCV is a mid-point calibration Cr (VI) standard (0.5ppm) from a different source than the calibration standards. Add 0.5 ml of 100 ppm Cr (VI) Secondary Spiking Solution (Sec. 7.2.4) into a specimen cup containing 50 ml of digestion fluid. The CCV is analyzed after every 10 samples (20 readings including backgrounds), and after reading the last sample. Acceptance criteria for the CCV are 90-110% of the true value. If the result is not within the acceptance limits, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial Calibration Blank and Calibration Check Blank (ICB,CCB): An initial calibration blank (ICB) must be analyzed immediately following the calibration curve and a continuing calibration blank (CCB) is analyzed after each CCV. The sample consists of 50 ml digestion solution and is carried through the entire analytical procedure. The concentration of the CCB must be below the reporting limit if not, all samples following the last acceptable CCB must be re-analyzed.

10. Procedure

10.1. Sample Preparation:

10.1.1. Just prior to analysis, SLOWLY adjust the pH of the sample extract to a pH of 7.0-8.0 with constant stirring using 1:1 nitric acid. Record pH and time. If a flocculent precipitate forms after the addition of 1:1 HNO3, filter the sample through a 0.45 um membrane filter, a larger size filter may be used to pre-filter the sample. Monitor the pH with a calibrated pH meter. If the pH drops below 7.0, discard the sample extract and re-digest the sample.

10.1.1.1. Remove and rinse the stir bar. Quantitatively transfer the sample extract to a 100 ml volumetric flask and dilute to the mark with deionized water.

10.1.1.2. If necessary, the solution can be diluted to eliminate the effects of color on the analytical determination. Use the smallest dilution necessary and record results.

10.2. Calibration

10.2.1. Prepare fresh calibration standards everyday or before each analysis.

10.2.2. Prepare a set of 6 calibration standards. Prepare 0.0, 0.05, 0.1, 0.5, 0.75, and 1.25 mg/l $K_2Cr_2O_7$ standards by aliquoting 0, 0.05, 0.1, 0.5, 0.75 and 1.25 mls of 100ppm Cr (VI) Primary Spiking Solution (Sec 7.2.3) respectively into the specimen cups containing 50mls of digestion fluid. At the same time prepare the mid-point Initial Calibration Verification and Initial Calibration Blank, see Sec 9.2.

10.2.3. Adjust pH to 7.5±0.5 with 1:1 HNO_3 and dilute to 100 ml with DI water. Record pH reading and analysis time in the logbook.

10.2.4. Develop the color for the standards as explained in Section 10.3 of this SOP.

10.2.5. Following the preparation of the calibration standards, “zero” the spectrophotometer with the 0 ppm standard. Analyze the standards.

10.2.6. The correlation coefficient must be 0.995 or better.

10.2.7. Immediately following calibration of the spectrophotometer and before reading samples, verify the calibration of the spectrophotometer by analyzing an ICV and ICB.

10.3. Sample Analysis

10.3.1. Warm up spectrophotometer and set to 540nm.

10.3.2. Prepare two rows of plastic cups and label them accordingly (first row with sample no. and the 2nd row as background).

10.3.3. Pour out 45.0 ml of the sample extracts, which have already been pH adjusted to between 7.0 and 8.0, into the appropriately labeled cups. Do the same for the background cups using 10 ml of the sample extract.

10.3.4. Add 1.0 ml indicator solution and swirl. (Do not add reagent to background sample).

10.3.5. Slowly add 10% H_2SO_4 (Sec 7.1.1) dropwise while swirling and monitoring pH until pH is 2 ±0.5. Record reading in the appropriate logbook.

10.3.6. Transfer sample to 50 ml volumetric flask and bring to volume with deionized water. Empty contents into labeled specimen cup to mix.

10.3.7. Let stand 5-10 minutes for full color development.

10.3.8. Read calibration standards, QC samples, and samples in a 1 cm cell on a spectrophotometer at 540 nm. (Note: Calibration standards and samples must be read within the same timeframe following full color development).

10.3.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

10.3.10. Dilute samples that exceed calibration curve using deioinized water and analyze following Sec. 10.3.3-10.3.7. Add dilution factor to TALS under "dilution" column. Note: The dilution should be made from the sample extract that has not had the indicator added.

10.3.11. After every twenty readings (10 samples) and after reading the last sample in the batch, verify the spectrophotometer calibration by reading a calibration check standard (CCV) and calibration check blank (CCB).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration = Hexavalent Chromium in mg/Kg = $\frac{A \times B \times E * 100}{C \times D}$

Where:

A = Concentration from the calibration curve in mg/L
B = Final digested volume in Liters
C = Wet sample weight in kg
D = percent solids
E = Dilution (if necessary)

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

11.4. Trivalent Chromium Calculation: When required, the calculation for trivalent chromium is completed using TALS Method '7196a_CR3.' Samples must be logged in under this method as well as the methods for Total chromium (metals) and Hexavalent chromium. Upon completion of the total and hexavalent chromium analyses the analyst may process the trivalent chromium results as follows:

- In order for TALS to perform an automated trivalent chromium calculation enter '1' (for 'yes') in trivalent chromium batch information page in response to the prompt 'automatically perform calculation.' As long as both methods are at 2nd level review status in TALS the program will automatically pull in the metals results for chromium and the hexavalent chromium results to perform the automated calculation. TALS will display an 'ok' message on the worksheet tab if the Cr6 result is less than Total Cr result.
- In cases where Total Cr results are not linked to TALS method 7196a_CR3, trivalent chromium is calculated manually. Enter '0' (for 'no') in batch information page 'Perform calculation.' Results will be entered manually.

11.5. Data reduction:

11.5.1. All data is recorded directly in TALS and recorded in the logbook at the time the analysis is performed.

11.5.2. Attach the prep and analytical logbook pages and calibration curve to the batch as a pdf file.

11.5.3. On the worksheet tab, enter the sample absorbance under 'Uncorrected Abs' and background absorbance under 'Color Blank Abs.' Also, enter the pH between 7-8 as 'initial pH',' pH between 1.5-2.5 as 'Final pH' and the background pH under 'Notes.'

11.5.4. Record special notes and observations in the "worksheet" tab and record reagent information in the prep batch information page (see "view batch information" page of ADII).

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.5.5.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

11.5.6. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. After the batch is second level reviewed, the checklist is filed in wetchem department and scanned in the network drive.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are

disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.
- Sample waste-After analysis, the samples and standards are collected in a polyethylene container labeled 'Cr6+ waste.' Once the container is full, it is brought to the waste room for disposal.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

15.2. Test America Edison SOP ED-WET-010, The Alkaline Digestion of Soil Samples for the Analysis of Hexavalent Chromium, most current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. TestAmerica Edison SOP ED-WET-066, Redox, Analysis of Oxidation-Reduction Potential, most current revision.

15.7. TestAmerica Edison SOP ED-WET-061, Analysis of pH for soil and organic samples electrochemically, Method 9045C, most current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16. Method Modifications:

N/A

17. Attachments

N/A

18. Revision History

- **Revision 12, dated 17 June 2020**
  - Updated SOP header with Eurofins emblem.
  - Sec. 9.1.2: Changed LCS source vendor from ERA to Phenova.
  - Sec. 11.5.5: Revised data review procedure to include TALS DRC procedures.
  - Sec 15.8: Deleted Wetchem Data Review Checklist Work Instruction # EDS-WI-008, not applicable.

- **Revision 11, dated 05 March 2018**
  - Sec 11.4: Added procedure for Trivalent chromium calculation using TALS method 7196a_CR3; subsequent section adjusted accordingly.

- **Revision 10, dated 23 February 2016**
  - Sec 9.1.2 and 9.1.3.2: Revised the allowable amount of PbCrO4 to add to the QC sample LCSI to reflect the range referenced in the Method. Also included the typical amount of $PbCrO_4$ that is added to the QC sample (LCSI and MSI).
  - Sec 9.1.3.3: Added spiking instructions for MSS when redigesting a batch; MSS should be spiked at 2x sample concentration or 40mg/kg, whichever is greater.

- **Revision 9, dated 25 September 2013**
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec. 9.1.2: Changed nomenclature to LCSSRM and revised the acceptance limits from 85-115% to vendor specified QC limits.
  - Sec. 9.1.3.3: Deleted text: '*Note: If the spiked sample concentration is greater than 4X the predigestion spike concentration, no redigestion/reanalysis is required unless the samples are from a NJ site in which case the project manager should be contacted for further guidance.'*

    Per method 3060a, all batches must be rehomogenized, redigested and reanalyzed if MSS or MSI fail regardless of location.
  - Sec. 10.3.5: Changed pH range from 1.6-2.2 to pH 2.0 +/-0.5 as per the method.
  - Sec. 11.4.3: Changed pH range from 1.6-2.2 to pH 1.5-2.5 as per the method.

- **Revision 8, dated 06 September 2011**
  - Sec 3: Revised the LQM reference for the list of definitions.
  - Sec 9.1.3.4: Clarified lab procedures for failing matrix spikes.
  - Sec. 9.2.2: Added procedure if the CCV is not within acceptance limits.
  - Sec. 9.2.3: Added procedure if CCB is not below reporting limit.
  - Sec. 11.4: Revised data reduction section to reflect actual laboratory practices.
  - Sec 15.9: Reference added.

- **Revision 7, dated 25 August 2009**

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 1.1: added laboratory's reporting limit; change the range of analysis to 0.05-1.25mg/L to reflect actual laboratory practices.
  - Sec 9.1.5: Added preparation instruction for Post digestion spike.
  - Sec. 9.2: added ICV in the Instrument QC.
  - Sec 10.1: Added procedure for adjusting pH of sample extract; procedure formerly described in the Digestion SOP (ED-WET-010).
  - Sec 15: Added applicable references.

- **Revision 6a, dated 21 April 2009**

  - Sec.4.3: Added membrane filters, 0.45um and 0.1um.
  - Sec 4.4: Delete Erlenmeyer flasks and beakers
  - Sec 4.8:Add: Eppendorf or Finnipettes; varying volume
  - Sec 5.1, 5.2 & 5.4: Add stability and storage information.
  - Sec 5.5: Added reagent used for the pH adjusted PS: 1N NaOH: Place 4.0g NaOH in a 100 volumetric flask and dilute to the mark with deionized water. Solution is stable for 6 months. Store at room temperature.
  - Sec. 6.1: Potassium Dichromate Primary Standard Solution (1000mg/L Cr+6): Weight 0.2829g of dried (105ºC) potassium dichromate and dissolve in deionized water in a 100ml flask. Dilute to the mark. Store at room temperature. Solution is stable for 6 months.
  - Sec 6.1: Deleted pre-made standard purchased from Inorganic Ventures. Cr6+ Primary Standard will be prepared in the lab.
  - Sec 6.2: Specified source of the 1000 ppm Cr(VI).
  - Sec 6.3 and 6.6: Deleted the 10mg/L Cr (VI) working standard for soil analysis.
  - Sec 6.5: Add storage information.
  - Sec 6.4: Add text: Source must be different from the primary standard.
  - Sec 9.2.2: Replace 100 ppm stock standard with 100 ppm Cr6+ Primary spiking solution.
  - Sec. 9.2.3: Add text: Record pH reading in the logbook.
  - Sec. 9.3.8: Add text: Make the dilution from the sample that has not had the indicator added.
  - Sec 9.3.6: Add text: Before reading samples, verify the calibration of the spectrophotometer by analyzing a CCV and CCB.
  - Sec. 10.7: Added procedure for reanalyzing Post Spike sample to conform to method requirements: *If the post digestion spike fails to meet the recovery limits, a post spike must be reanalyzed using the alkaline extract. Adjust the pH of the sample Duplicate (alkaline extract) back up to pH 8.0-8.5 using 1N NaOH; then*

respike and reanalyze.

- **Revision 6, dated March 2007**

  - Section 7.2: Section deleted (start color development for digested samples within one hour after adjusting pH between 7.5±0.5); section not applicable.
  - Section 9.3.7: The text in italics was inserted to clarify the time period for measuring sample absorbance.

    "Note: Calibration standards and samples must be read within the same timeframe following full color development."
  - Section 10.2: Deleted the requirement that 'CCS is taken through the entire digestion process' to reflect actual laboratory practices.
  - Section 10.4. Revised to specify the types of LCS used including their source and acceptance limits.

- **Revision 5, dated 30 October 2006**

  - Section 9.2.2: Revised the high point of the calibration curve from 1.0 ppm to 1.25 ppm. Standards preparation instructions revised accordingly.
  - Section 9.2.2: Revised to include instructions for preparation of a second source mid-point Calibration Check Standard.
  - Section 10: Inserted a new Section 10.1 explaining the QA batching system and inserted a new section 10.4 detailing the LCS requirements. All existing sections renumbered accordingly.
  - Section 10.6 (previously Section 10.5): The italicized text was added to the following sentence:

    "NOTE: If the spiked sample concentration is greater than 4x the predigestion spike concentration, no redigestion/reanalysis is required *unless the samples are from a NJ site in which case the lab project manager should be contacted for further guidance."*
  - Section 10.6 (previously Section 10.5): Added STL Edison SOP and method references for ORP and pH.
  - Section 10.7 (previously Section 10.6): Added the following text: "If the post digestion spike fails to meet the recovery limits, a new aliquot of the sample must be re-spiked and re-analyzed."
  - Section 10.8 (previously Section 10.7): Revised first sentence to read "One duplicate laboratory sample per batch *must* be analyzed." ('Must' previously read 'should').

Title: The Analysis of Waters for Hexavalent Chromium by EPA SW846 Method 7196A

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1.0 Scope and Application

1.1. SW846 Method 7196A may be used to determine the hexavalent chromium content of groundwater samples or digestates of solid samples. This SOP is applicable for the determination of hexavalent chromium in groundwater samples. Groundwater samples associated with regulated drinking water samples (i.e. potable water wells) are not applicable to this Method.

1.2. The range of analysis by this method is 10-200 ug/l hexavalent chromium.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Hexavalent chromium is determined spectrophotometrically at 540nm by reaction with diphenylcarbazide in acid solution. For each batch of samples a method blank, a laboratory control sample, matrix duplicate, and a post verification spike are analyzed and compared to method limits and laboratory control limits.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

4.2. Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of hexavalent chromium present.

4.3. Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

4.4. Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time; however, interference is checked by a post verification spike samples.

4.4.1. The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.5. For waste materials containing soluble trivalent chromium concentrations greater than four times the laboratory hexavalent chromium reporting limit, the hexavalent chromium results obtained using this method may be biased high due to method-induced oxidation.

5.0. Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

6.0. Equipment and Supplies

6.1. Instrumentation

- pH meter, Orion benchtop or equivalent
- Spectrophotometer for use at 540 nm with a light path of 1 cm (Hach DR2800 or equivalent).

6.2. Supplies

- Membrane filters, 0.45 um and 0.1 um (NALGENE disposable)
- Glasswares: volumetric pipets (class A) and volumetric Flasks (class A)
- Graduated Cylinder (class A)
- Transfer pipettes
- Calibrated pipettes: Eppendorf or equivalent
- Vacuum pump
- Specimen Cups (plastic)
- Magnetic stirrer (teflon-coated)

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric acid (10% v/v) - Measure 100 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water. Reagent is stable for 6 months. Store at room temperature.

7.1.2. Indicator solution - Place 5.0 g 1, 5-Diphenylcarbazide (AR Grade) in an amber 1000ml volumetric flask and dilute to mark with acetone. Prepare monthly or when solution becomes cloudy. Store at room temperature.

7.1.3. Acetone (reagent grade) - Store at room temperature. For stability information, refer to manufacturer's instructions or 2 years from date opened if no instructions are available.

7.2. Standards

7.2.1. Primary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.2.2. Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of the 1000 mg/L Primary Potassium Dichromate Standard (see Section 7.2.1) to a 100ml volumetric flask and dilute to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.3. Hexavalent Chromium Primary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Standard (see Section 7.2.1) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.4. Secondary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). *The source of this standard must be from a different lot or manufacturer than the Primary Potassium Dichromate Stock Standard detailed in Section 7.2*.1. Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.2.5. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of 1000 mg/L Cr+6 Secondary Standard (see Section 7.2.4) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.6. Hexavalent Chromium Secondary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Secondary Standard (see Section 7.2.4) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

8.0. Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE[2]	250 mL	Cool 4 ±2°C	24 Hours	SW 7196A

[1] Samples must be analyzed within 24 hours from time of collection. Store samples at 4°C before analysis.
[2] High-density polypropylene container

9.0. Quality Control

9.1. **Sample QC** - - The following MB, LCS, and PVS are analyzed with each batch of 20 samples or each time samples are setup, whichever is more frequent. The DU is analyzed at a frequency of 1 per 10 samples or each time samples are set up, whichever is more frequent.

9.1.1. Method Blank (MB): Use deionized water for the method blank. Analyze using all reagents in the same volumes as used in the analysis of samples. The blank value should fall below the reporting limit if not, all samples associated with the method blank must be re-analyzed. Do not subtract the blank value from sample values.

9.1.2. Post Verification Spike (PS): The PVS concentration must be two times the concentration of Cr(VI) found in the sample or 30.0 ug/L (whichever is greater). If necessary, dilute the PS to fall within the range of the calibration curve. If the PS recovery is not within 85-115%, dilute the sample and re-analyze.

Note: The post spike is identified as “MS” in TALS QC.

9.1.3. Matrix Duplicate (DU): A duplicate is prepared and analyzed in exactly the same manner as the original sample. The relative percent difference (RPD) between the sample and duplicate should be less than or equal to 20%.

9.1.4. Laboratory Control Sample (LCS): The hexavalent chromium LCS is a certified QC standard purchased from Phenova (Catalog WP

Hexavalent Chromium). The LCS is used to monitor the accuracy of the entire analytical process. The LCS recovery must be within vendor specified QC limits or all samples associated with the LCS must be reanalyzed.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV (75.0 ug/L Cr (VI)) is analyzed immediately after an acceptable initial calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within ±10% of the expected value. The ICV is prepared by adding 0.375ml of a 10 mg/L Cr6+ Hexavalent Chromium Secondary Spiking Solution (Section 7.2.6) into a 50 ml volumetric flask and diluting to 50ml with deionized water. If the measured concentration exceeds the ±10% limit, a second analysis should be performed. If the result still exceeded the ±10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.2. Calibration Check Verification (CCV): A midpoint Calibration Check Verification spiked at 75.0 ug/L is prepared in the same way as the ICV (See Section 9.2.1). The CCV is analyzed after every 10 samples (20 readings including backgrounds) and after reading the last sample. The result must be within ± 10% of the true value. If not, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): Initial Continuing Calibration Blank (ICB) must be analyzed after the ICV. Continuing Calibration Blank (CCB) must be analyzed after every 10 samples or after each Calibration Check Verification (CCV). Use deionized water for the ICB/CCB. The value of the ICB/CCB should be less than the RL. If not, all samples following the last acceptable ICB/CCB must be re-analyzed.

9.2.4. Minimum Reporting Level (MRL), 10ppb: The MRL must be analyzed daily before samples are analyzed. Prepare the MRL by adding 0.05ml of a 10 mg/L Hexavalent Chromium Primary Spiking Solution (Section 7.2.3) into a 50 ml volumetric flask and diluting to 50ml with deionized water. The results must fall within 50-150% of the true value.

10.0 Procedure

10.1. Calibration

10.1.1. Prepare fresh calibration standards everyday before analysis.

10.1.2. Initial Calibration Standards: (6 points). Place about 25 mL of deionized (DI) water in six 50 ml flasks. Prepare the six points as follows:

Cr(6+) Standard conc. (ug/L)	Volume (ml) of 10 mg/L Cr+6 (Section 7.2.3) to add	Final volume (ml) using Deionized water

0.0	0.0	50
10.0	0.05	50
50.0	0.25	50
75.0	0.375	50
100	0.50	50
200	1.0	50

10.1.3. Analyze the calibration standards in the manner detailed in Sections 10.2.1 thru 10.2.7. Zero the spectrometer with the 0 ug/L standard.

10.1.4. Perform linear regression on data resulting from analysis of the initial calibration. The correlation coefficient must be 0.995.

10.1.5. Analyze the Initial Calibration Verification (ICV) standard in the same manner as the initial calibration standards detailed above. If the ICV meets the criteria listed in Section 9.2.1, the analyst may continue with sample analysis (Section 10.2).

10.2. Sample Analysis

10.2.1. Warm up spectrophotometer and set to 540 nm.

10.2.2. Place 45.0 mL of sample into a 100 ml specimen cup. Also, place 45 mL of sample in a separate cup for background.

10.2.3. Add 1.0 ml indicator solution (see Section 7.1.2) and swirl. (Do not add indicator to background sample).

10.2.4. Slowly add 10% H_2SO_4 (See Section 7.1.1) dropwise to sample and background while measuring pH until pH is 1.5-2.5. Record reading.

10.2.5. Place sample in a 50 ml volumetric flask and bring to volume with deionized water. Pour back into original cup and swirl.

10.2.6. Let stand 5-10 minutes for full color development.

10.2.7. Read calibration standards, QC samples, and samples on spectrophotometer at 540 nm. Analyze the calibration check standard and calibration check blank immediately following the calibration standards.

10.2.8. Dilute samples that exceed calibration curve using deionized water. Recolorize and read again on spectrophotometer noting dilution factor.

10.2.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

10.2.10. After every 10 samples and after reading the last sample in the batch, verify the calibration by reading Calibration Check Verification (CCV) and Continuing Calibration Blank (CCB).

11. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculate the hexavalent chromium result for each aqueous sample as follows:

Hexavalent Chromium in ug/L = A x E

A = Concentration from the calibration curve in ug/L
E = Dilution (if necessary)

11.4. Trivalent Chromium Calculation: When required, the calculation for trivalent chromium is completed using the TALS method '7196a_CR3' which must be logged for the samples being processed along with methods for total chromium (metals) and hexavalent chromium. Upon completion of the total and hexavalent chromium analyses the analyst may process the trivalent chromium results as follows:

- To have TALs perform an automated trivalent chromium calculation: on the trivalent chromium batch information page enter '1' (for 'yes') in response to the prompt to automatically perform the calculation. As long as both methods are at 2nd level review status in TALS the program will automatically pull in the metals results for chromium and the hexavalent chromium results to perform the automated calculation. TALS will display an 'ok' message on the worksheet tab if the Cr6 result is less than Total Cr result.

- If the analyst intends to manually enter trivalent chromium results an entry of '0' (for 'no') should be made the and the results entered manually.

11.5. Data Reduction

11.5.1. Record analytical data directly into TALS and in the 'Hexachrome Run Logbook.'

11.5.2. Record Reagent information on the "batch information" page.

11.5.3. Special comments or observations should be recorded on the "worksheet" tab. Record Sample absorbances and pH readings on the "worksheet" tab.

11.5.4. Attach the calibration curve plot and the logbook pages for the analysis to the analytical batch as pdf files.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.5.5.1 Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2 Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3 Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.4 Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are generated as a result of this analysis:

- Water Retain Samples -These materials are originally received aqueous samples. These solutions are then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9 with sodium bicarbonate (Siedler Chemical SC-0219-25). This solution is discharged into the municipal sewer system.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

- Revision 11, 10/01/2021
  - Sec. 9.2.4: Added criteria to analyze a Minimum Reporting Level (MRL) daily.
- Revision 10, 14 May 2020
  - Updated SOP header with Eurofins logo.
  - Sec 1.1: Clarified matrix applicability for this SOP.
  - Sec. 9.1: Revised the frequency of Matrix Duplicate from 1/20 samples to 1/10 samples.
- Revision 9, 12 February 2019
  - Sec. 9.1.4: Changed LCS source vendor from ERA to Phenova.
  - Sec. 11.5.5: Replaced Wetchemistry Data review checklist (EDS-WI-008) to TALS Data Review Checker (DRC); instructions added.
  - Deleted Sec 15.6, WI EDS-WI-008, not applicable; subsequent sections adjusted accordingly.
- Revision 8, 30 Nov 2016
  - Sec. 11.4: Added Cr3 calc procedure
- Revision 7, 26 November 2012
  - Section 9.1.2: Revised the minimum concentration for PS from 15 ug/L to 30 ug/L to comply with Method 7196A.
- Revision 6, 04 October 2012
  - Section 1&12: Updated LQM reference to reflect the most current LQM revision.
  - Sec 5: Updated to comply with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP)
  - Sec. 9.1.1: Revised to include corrective action procedure when MB is greater than the RL.
  - Sec. 9.1.4: Changed limits from 85-115% to vendor specified QC limits.
  - Sec. 9.2.2 & 9.2.3: Revised to include corrective action procedure when CCV and CCB are outside the acceptance limits.
- Revision 5, 7 September 2010
  - Section 2.0: Removed text 'Dissolved;' this SOP is only applicable to aqueous samples for method SW7196A which are not filtered.
  - Sec 3: Revised to update LQM reference for the list of definitions.
  - Section 6.2: Added graduated cylinders (class A) and transfer pipettes to the list of supplies.
  - Removed Nitric acid on list of reagents, not applicable for the analysis of Cr6+ waters.

- Section 9.1.1: Revised method blank criteria from <MDL to <RL.
- Section 9.1.4: Replaced LCS vendor specified limits to 85-115%.
- Section 9.2.2: Removed text: “immediately after analyzing the calibration standards.” Renamed CCS to CCV.
- Section 9.2.3: Revised CCB criteria from <MDL to <RL.
- Section 10.1.4: Revised the correlation coefficient criteria from ≥0.997 to ≥0.995.
- Section 13 & 14: Revised to comply with the current Method SOP format (CW-QS-002, Writing a Standard Operating Procedure (SOP)).
- Section 11.4: Revised Data Reduction section in accordance with TALS.
- Section 15: Added applicable references.

- Revision 4, January 22, 2008

  - Updated to new format as per TestAmerica’s SOP format.
  - Added the shelf-life and storage requirements to reagents in section 7, Reagents and standards.
  - Added J.T.Baker (Catalog Nos. 3090, 3093 or equivalent) as source of primary and secondary standards. Deleted reference to Inorganic Ventures as a source of the primary standard.
  - Replaced the source standard used for the preparation of 10 mg/L Cr+6 (primary and secondary spiking solution) from 100 mg/L Cr+6 to 1000 mg/L Cr+6.
  - Added the control limits for Post Verification Spike (PVS).
  - Added ICV as Instrument QC.
  - Added the preparation procedure and control limits for the Calibration Check Standard (CCS).
  - Deleted Matrix spike (matrix spike low and matrix spike high) as sample QC requirement.
  - Section 10.3.3: Revised pH range from 1.6-2.2 to 1.5-2.5 to reflect method requirements.
  - Standardized all analytical units to ug/L and mg/L.
  - Added the following to the References section: TestAmerica Edison SOP ED-GEN-022 (Training) and TestAmerica Laboratory Quality Manual (ED-QA-LQM).

Title: Trace Metals Analysis for Water, Wastewater, Soil and Sediment Samples by ICP-MS EPA Method 200.8

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes the procedures used to determine the concentration of various elements in surface, drinking, and ground water, wastewater, by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) using EPA Method 200.8.

The routine target analytes and reporting limits are as follows:

Table 1

Analyte List and Method Reporting Limits (RL)

Element	Drinking Water RL	Non-Drinking Water RL
	(ug/L)	200.8 NPDES (ug/L)
Aluminum (Al)	50	40
Antimony (Sb)	0.4	2
Arsenic (As)	1	2
Boron (B)	20	80
Barium (Ba)	2	4
Beryllium (Be)	0.3	0.8
Cadmium (Cd)	0.5	2
Calcium (Ca)	250	500
Cobalt (Co)	2.5	4
Chromium (Cr)	0.9	4
Copper (Cu)	2.5	4
Iron (Fe)	250	120
Lead (Pb)	2.0	1.2
Magnesium (Mg)	250	200
Manganese (Mn)	25	8
Molybdenum (Mo)	2.5	4
Nickel (Ni)	0.5	4
Potassium (K)	250	200
Selenium (Se)	8	2.5
Silver (Ag)	2.5	2
Sodium (Na)	250	500
Strontium (Sr)	2.5	4
Thallium (Tl)	0.3	0.8
Tin (Sn)	4	16
Titanium (Ti)	2.5	4
Vanadium (V)	2.5	4
Zinc (Zn)	20	16

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

For total recoverable analysis of aqueous samples containing undissolved material and for drinking water samples with turbidity $\geq$ 1 NTU, samples must undergo digestion prior to analysis. Refer to TestAmerica Edison SOP Nos. ED-MTP-001. For the determination of dissolved analytes in a filtered aqueous sample and in a drinking water sample with turbidity < 1 NTU, samples don't require digestion prior to analysis. Samples are ready for analysis after the addition of nitric acid.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. Isobaric polyatomic/molecular ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest and which cannot be resolved by the mass spectrometer in use. Most isobaric interferences that could affect the ICP-MS analysis for elements in this SOP have been identified. These can be managed by the selection of an alternate isotope, the use of the collision/reaction cell (used for non-potable samples only), or by the use of elemental interference equations when the collision/reaction cell is not in use.

4.2. For Drinking Water analysis (standards, samples, and relating QC) the collision/reaction cell can not be used; therefore, the following interference equations should be used to reduce interferences:

Mass	Equation
6:	(6)*1 - (7)*0.082
44:	(44)*1 - (88)*0.015
51:	(51)*1 – (53)*3 + (52)*0.34

66: (66)*1 – (69)*0.00141
75: (75)*1 – (77)*2.9 + (82)*2.23 – (83)*2.23
82: (82)*1 – (83)*1
111: (111)*1 – (108)*1.073 + (106)*0.712
115 (115)*1 – (118)*0.016
208: (208)*1 + (206)*1 + (207)*1 (used for both drinking and non-drinking water analysis)

The basic elemental interference equations are based on natural isotopic abundances. The most precise coefficients for an instrument must be determined from the ratio of the net isotope signals that are observed for a known standard solution at a concentration sufficient to produce suitable counting statistics.

4.3. Krypton affects the determination of both arsenic and selenium but can be greatly reduced with the use of high purity Krypton free argon. Krypton must be analyzed for every sample and standard.

4.4. Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. These changes in matrix can cause significant signal suppression or enhancement. Dissolved solids can deposit on nebulizer tips and interface cones (reducing the orifice size and the instrument's performance). Internal standards can be used to correct for physical interferences if they are carefully matched to the analyte so that both elements react similarly to the matrix changes.

4.5. Memory interferences can occur when analytes from a previous sample contribute to signals measured from subsequent samples. The memory effects can result from analyte deposition of sample on the sample tubing, joints, nebulizer, spray chamber, torch, and/or interface cones. Routine maintenance on the sample introduction system is necessary in order to minimize the memory interferences. The memory effects must be taken into account when setting up a suitable rinse times. The evaluation of a minimum of three replicate integrations will help to determine memory problems.

4.6. Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Agilent 7800 and Agilent 7900: inductively coupled plasma mass spectrometer with data system (microprocessor, monitor, printer) and autosampler. Mass Range 2-260 amu. Vacuum purged spectrophotometer with an axial plasma torch. Collision/Reaction Cell.

- Heat exchanger –PolyScience Model 6106T Recirculating Chiller or equivalent
- Autosampler – Agilent SPS 4 Autosampler

6.2. Supplies

- Reagent Water -18 megohm Reagent grade Type II water
- Volumetric Flasks (Class A): 50 mLs, 100 mLs, 500mls & 1000mls
- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid (HNO3) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.2. Concentrated Hydrochloric Acid (HCL) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.3. Argon supply - 99.9% (Liquid)

7.1.4. Helium and Hydrogen supply – 99.999% (Gas)

7.1.5. 18 megohm Reagent grade Type II water.

7.1.6. Reagent water, 2% HNO3 + 0.5% HCl by volume: Add 200ml of concentrated HNO3 and 50ml of concentrated HCl to deionized water and bring to 10 liter volume with deionized water. For potable samples not requiring digestion, the reagent water is prepared without HCL. Note: Always add acid to water. Record preparation in the TALS Reagent Module Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.1.7. 5% HNO3 + 5% HCl: Add 1 liter of concentrated HNO3 and 1 liter of concentrated HCl to deionized water and bring to 20 liter volume with deionized water. For potable samples not requiring digestion, the reagent water is prepared without HCl. Varying amounts of this reagent may be made (e.g., 10L) by proportionally adjusting the volume of acids used.

Note: Always add acid to water. Record preparation in the TALS Reagent Module Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.2. Standards

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. Certificate of analysis are filed in Metals managers office.

Storage requirements: all standards are stored at room temperature

Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 6 months
Working cal standards – two weeks
Working Initial calibration verification standard – quarterly
Interference Check Standards (A&B) - weekly
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: Final concentrations for the calibration standards are given in Attachment 1. All standards must be prepared in reagent water (Sec. 7.1.6 & 7.1.7).

Standards must be prepared in accordance to the shelf life (Sec 7.2) "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

All standards must be prepared in reagent water (Sec. 7.1.6 and 7.1.7). Final concentrations for the calibration standards are given in Attachment 1. Varying amounts of these standards may be made by proportionally adjusting the volume of standards and reagent water used.

7.2.1. Calibration Standards: ICPMS-Cal (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures and from CPI International. Refer to manufacturer's instructions for stability and storage information.

6020B RL Standard contains: 20 mg/L Ca, K, Mg, Na
12 mg/L Fe
8 mg/L B
4 mg/L Al
1.6 mg/L Sn, Zn
0.8 mg/L Mn
0.4 mg/L Ba, Co, Cr, Cu, Mo, Ni, Sr, Ti, V
0.25 mg/L Se
0.2 mg/L Ag, As, Cd, Sb,
0.12 mg/L Pb
0.08 mg/L Be, Tl

TA-CAL-1 contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL-2 contains: 1,000 mg/L Al, Ca, Fe, K, Mg, Na

Single Element Standard: Ag 1,000 mg/L

Single Element Standard: B 1,000 mg/L

Single Element Standard: Zn 1,000 mg/L

200.8 Potable RL Std contains: 25000 ug/L Ca, Fe, K, Mg, Na
2500 ug/L Al, Mn,
2000 ug/L B
800 ug/L Se
400 ug/L Sn, Zn,
250 ug/L Ag, Co, Cu, Mo, Sr, Ti, V
200 ug/L Ba, Pb
100 ug/L As
90 ug/L Cr
50 ug/L Cd, Ni
40 ug/L Sb
30 ug/L Be, Tl

7.2.2. Working Calibration Standards:

For Non-Drinking Waters:

CAL5: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.4ml of TA-CAL-1 and 5ml of TA-CAL-2. Add 10 ml of MS-Cal-Int. Bring to volume with 5% HNO3 / 5% HCl.

CAL4/CCV: Add 10ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 50ml polypropylene tube. Add 10ml of working Cal5 and mix.

CAL3: Add 15ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 50ml polypropylene tube. Add 5ml of working Cal5 and mix.

CAL2: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 10ml of 6020B RL Standard. Bring to volume with 5% HNO3 / 5% HCl.

CAL1: Add 10ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 50ml polypropylene tube. Add 10ml of working Cal2 and mix.

MS-Cal-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 2ml each of B and Zn. Add 0.4ml of Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7).

For Drinking Waters:

CAL5: Add 100ml of reagent water (Sec 7.1.6) to a clean 200ml flask. Add 0.2ml TA-CAL-1, 0.5ml of TA-CAL-2, 0.04ml of 1000ppm B, and 0.02ml each of 1000ppm Ag & Zn. Bring to volume with reagent water (Sec 7.1.6).

CAL4/CCV: Add 10ml of reagent water (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 10ml of Cal5 (drinking water) and mix.

CAL3: Add 15ml of reagent water (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 5ml of Cal5 (drinking water) and mix.

CAL2: Add 18ml of reagent water (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 2ml of Cal5 (drinking water) and mix.

CAL1: Add 100ml of reagent water to a clean 200mL flask. Add 2 mL of the 200.8 Potable RL Std. Bring to volume with reagent water (Sec 7.1.6). This standard is used as the Reporting Limit standard (CRI).

Note: see Attachment 1 for final elemental concentrations.

7.2.3. Initial Calibration Verification Stock standards: purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Standards must be from a different source than those used for the calibration standards.

TA-CAL1-SS contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL2-SS contains: 1,000 mg/L Al, Ca, Fe, K, Mg, Na

2nd Source Single Element Standard: Ag 1,000 mg/L

2nd Source Single Element Standard: B 1,000 mg/L

2nd Source Single Element Standard: Zn 1,000 mg/L

7.2.4. ICV Working Standard:

7.2.4.1. ICV Potable: The ICV Working Standard must be at a concentration different from the concentrations used for the CCV working standard. Prepare by adding 100ml of reagent water (Sec 7.1.6) to a clean 500ml flask. Add 0.2ml TA-CAL-1-SS, 0.5ml of TA-CAL-2-SS, 0.04ml of 1000ppm B (2nd source std), and 0.02ml each of 1000ppm Ag & Zn (both 2nd source std). Bring to volume with reagent water (Sec 7.1.6). The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh quarterly or as needed and stored at room temperature.

7.2.4.2. ICV Non-Potable: The ICV Working Standard must be at a concentration different from the concentrations used for the CCV working standard. Add 10ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 50ml polypropylene tube. Add 10ml of working Interm_ICV (Sec 7.2.4.2.2) and mix. The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh quarterly or as needed and store at room temperature.

7.2.4.2.1. MS-ICV-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 2ml each of 2nd Source: B & Zn. Add 0.4ml of 2nd Source: Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7).

7.2.4.2.2. Interm ICV: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.32ml of TA-CAL-1-SS and 1.6ml of TA-CAL-2-SS. Add 8 ml of MS-ICV-Int (see Sec 7.2.4.2.1). Bring to volume with 5% HNO3 / 5% HCl

7.2.5. Continuing Calibration Verification standard, Non-potable: see CAL4/CCV (Sec. 7.2.2).

7.2.6. Continuing Calibration Verification standard, Potable: see CAL4/CCV (Sec. 7.2.2).

7.2.7. Internal Standards and EPA Tune Check:

The following elements are used as the internal standards: Bi, In, Li6, Sc, Tb, and Ge.

7.2.7.1. Internal Standards Stock Solutions**:**

7.2.7.1.1. Testamerica-1 (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 250 mg/L each: Li6, Sc; 50 mg/L each: Bi, In, Tb.

7.2.7.1.2. MSGE-100PPM (Custom Calibration Standard Stock Solution) purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Ge 100 mg/L.

7.2.7.1.3. Triton X-100 (Standard Stock Solution: Non-ionic Surfactant) purchased from Fisher Scientific, p/n BP151-100. Refer to manufacturer's instructions for stability and storage information. According to the ICPMS manufacturer (Agilent Technologies), Triton-X 100 will reduce the 3 replicate %RSD during analysis.

7.2.7.2. Working Internal Standard Solution: *For Non-Potable*: Add 100ml of reagent water to a clean 1000ml flask. Add 1 ml of Testamerica-1 and 1 ml of MSGE-100PPM, and 0.1 ml of Triton X-100. Bring to volume with reagent water. *For Potable*: Add 100ml of reagent water to a clean 250ml flask. Add 0.5 ml of Testamerica-1, 0.5 ml of MSGE-100PPM, and 0.05 ml of Triton X-100. Bring to volume with reagent water.

The following are the recommended analysis masses, tune steps, and internal standards:

Element	IS
Be9	Li6
B11	Li6
Na23	Sc45
Mg24	Sc45
Al27	Sc45
K39	Sc45
Ca40	Sc45
Ti47	Sc45
V51	Sc45
Cr52	Sc45
Mn55	Sc45
Fe56	Sc45
Co59	Sc45
Ni60	Sc45
Cu63	Sc45
Zn 66	Sc45
As75	Sc45
Se78	Sc45
Sr88	In115
Kr83**	(none)
Mo95	In115
Ag107	In115
Cd111	In115
Sn118	In115
Sb121	In115

Ba137	Tb159
Tl205	Bi209
Pb 208*	Bi209

*Pb 208 = Pb 208 + Pb 207 + Pb 206
**Kr83 is used for monitoring Krpyton levels only.

Different masses and internal standards may be utilized, as matrix issues deem necessary. Non-drinking waters are analyzed using collision/reaction cell technology (helium/hydrogen gas mode). Typical non-drinking water Collision/Reaction Cell usage: Calcium, Iron, and Selenium are analyzed in the Hydrogen reaction mode; Beryllium and Boron Titanium are analyzed in the No Gas mode; all other elements are analyzed in the helium collision mode. For drinking water analyses, collision cell technology is not allowed; therefore, the no-gas mode is utilized. Use masses Ca44, Fe57, Cu65, and Se82 instead of their respective masses listed above.

Tune check criteria:

Instrument tunes must be performed daily, before calibration. A solution at ~10ppb of Be, Mg, Co, In, and Pb is analyzed, and the precision, mass calibration, and resolution are checked. Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.

The following limits are used to evaluate mass calibration, resolution, and instrument stability:

The following limits are used to evaluate the tune:

Mass calibration: +/-0.1 amu
Resolution check: ≤0.9 amu at 10% peak height. Note: This also satisfies Method 200.8, 1.0 amu at 5% peak height)
Stability (5reps): <5%

7.2.7.3. EPA Tune Check Stock Standard: 2008TS purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 10 ug/mL each: Be, Co, In, Mg, Pb.

7.2.7.4. Working EPA Tune Check Standard: add 100ml of reagent water to a clean 1000ml flask. Add 1 mL of 2008TS. Bring to volume with reagent water. This solution contains 10 ug/L of Be, Co, In, Mg, and Pb

7.2.8. Reporting Limit (CRI) Check standard:

For Non-Drinking Water: See Section 7.2.2, CAL1
For Drinking Water: See Section 7.2.2, CAL1.

7.2.9. ICP Interference Check Solutions: Stock solutions for ICSA and ICSAB, purchased from Inorganic Ventures (6020ICS-8A & 6020ICS-8B) and CPI International (TA-ICPMS-ICSA & TA-ICPMS-ICSAB), contains the following elemental concentrations:

TA-ICPMS-ICSA contains: 10000 mg/L Chloride; 2000 mg/L Carbon; 1000 mg/L ea: Al, Ca, Fe, K, Mg, Na, P, S; 20 mg/L ea: Mo, Ti

TA-ICPMS-ICSAB contains: 100 mg/L W; 10 mg/L ea: As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Si, Sn, Sr, V; 5 mg/L ea: Sb, Tl;

Single Element Standard: Ag 1,000 mg/L

Single Element Standard: Zn 1,000 mg/L

7.2.10. Working Interference Standards (ICSA/ICSAB):

For Non-Drinking Waters:

ICSA: Add 50ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 100ml flask. Add 10ml of TA-ICPMS-ICSA stock solution. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

ICSAB: Add 50ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 100ml flask. Add 10ml of TA-ICPMS-ICSA, 2 ml of TA-ICPMS-ICSAB, and 0.02 ml each of 1000 mg/L Ag and Zn. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

For Drinking Waters:

ICSA: Add 50ml of reagent water (Sec 7.1.6) to a clean 100ml flask. Add 2.5ml of TA-ICPMS-ICSA stock solution. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

ICSAB: Add 50ml of reagent water (Sec 7.1.6) to a clean 100ml flask. Add 2.5ml of TA-ICPMS-ICSA, 0.5ml of TA-ICPMS-ICSAB, and 0.005ml each of 1000 mg/L Ag and Zn. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

7.2.11. ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK: purchased from High-Purity Standards. Refer to manufacturer's instructions for stability and storage information. Refer to attachment 2 for final elemental concentrations. Stock solution contains the following elemental concentrations:

Element	Conc. Of Stock (mg/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

8.0. Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container	Min. Sample Size[2]	Preservation	Holding Time[1]	Reference

Matrix	Sample Container	Min. Sample Size[2]	Preservation	Holding Time[1]	Reference
Metals Waters except Hg	Polyethylene or Glass	250 mLs	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [3,4]	6 months	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.
[2] Drinking water samples may require 1000ml sample volume.
[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24 hrs, verify ph<2).
[4] Aqueous samples may be stored at room temperature.

Note: If drinking water samples are properly preserved with nitric acid to a pH<2 and the turbidity is <1 NTU, then acid digestion is not required. The laboratory must maintain documentation of the turbidity reading.

Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered. Properly filtered and nitric preserved samples requiring analysis for dissolved analytes don't require digestion prior to analysis.

9.0. Quality Control

Note: If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.

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

Quality Control	Frequency	Control Limit
Preparation blank	One per batch of twenty samples or less	**200.8**: < RL or <10% sample concentration.
Lab Control Sample Water (LCSW)	One per batch of twenty samples or less	**200.8**: +/- 15%
Matrix Duplicate	One in 20 or fewer samples	20% RPD
Matrix Spike (200.8)[1]	One, if 10 or less samples in batch; two, if 11 or more samples	70-130%
Matrix Spike Duplicate	When requested by client	70-130%; If MS and MSD are both ≥ 5X CRDL, then 20% RPD. If MS and MSD

Quality Control	Frequency	Control Limit
		are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Post Digestion Spike	One per batch of twenty samples or less	See Section 9.1.6.4

[1] The sample for MS is randomly selected, unless specifically requested by a client.

9.1.1. Preparation Blank/Method Blank: One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. results must be less than the reporting limit (RL) or less than 10% of the determined analyte concentration for a sample. If any analyte concentration in the blank is above the control limit, the batch must be prepared again for the element in question and the samples reanalyzed.

9.1.2. Laboratory Control Sample Water (LCS): A laboratory control sample must be analyzed with each group of samples digested. Refer to Water Matrix Spike concentration in Attachment 1 for final elemental concentrations. Results must be within the acceptable control limits, if not, all samples prepared in association with the LCS must be redigested and reanalyzed.

- Method 200.8: wastewater samples - prepare the LCS by adding 0.25 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 50 ml deionized water. Results must be within ± 15% of the true value.
- Method 200.8: drinking water samples - prepare the LCS by adding 0.10 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 50 ml deionized water. Results must be within ± 15% of the true value.
- For dissolved aqueous samples not undergoing a preparation procedure, the CCV may serve as the LCS.

9.1.3. Matrix Duplicate: A duplicate is analyzed for each batch of samples digested. The relative percent difference between the results of the sample and the duplicate must fall within 20% RPD for samples greater than five times the reporting limit.

9.1.5. Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples digested.

- For aqueous MS sample, spike 0.25 ml of ICP-MS LCS/SPK (Sec 7.2.11) into 50 ml sample.
- For drinking water MS sample spike 0.10 ml of ICP-MS LCS/SPK into 50 ml sample.
- Method 200.8: a recovery of 70-130% is required.
  An exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more. If the recovery is not within specified limits a post digestion spike is required to be analyzed

at a concentration between 10 to 100 times the instrument detection limit. If the Post digestion spike recovery is not recovered within 85-115% for 200.8, a matrix effect should be suspected. See Attachment 1 for final Matrix spike concentration for water and soil.

9.1.6. Post-Digestion Spike: To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). Post-Digestion Spike sample should be evaluated if serial dilution fails.

9.1.6.1. Transfer 10mL of a digestate to a suitable vial.

9.1.6.2. For Non-Potable samples, spike the sample with 0.05ml of ICP-MS LCS/SPK. For Potable samples, spike the sample with 0.01ml of ICP-MS LCS/SPK. Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed). The final concentration of the post digestion spike is as follows:

Element	Non-Potable Post Spike concentration (ug/L)	Potable Post Spike concentration (ug/L)
Aluminum (Al)	2500	500
Antimony (Sb)	25	5
Arsenic (As)	50	10
Barium (Ba)	50	10
Beryllium (Be)	25	5
Boron (B)	100	20
Cadmium (Cd)	25	5
Calcium (Ca)	2500	500
Chromium (Cr)	50	10
Cobalt (Co)	25	5
Copper (Cu)	50	10
Iron (Fe)	2500	500
Lead (Pb)	25	5
Magnesium(Mg)	2500	500
Manganese (Mn)	250	50
Mercury (Hg)	2.5	0.5
Molybdenum (Mo)	50	10
Nickel (Ni)	50	10
Potassium (K)	2500	500
Selenium (Se)	50	10
Silver (Ag)	25	5
Sodium (Na)	2500	500
Strontium (Sr)	50	10
Thallium (Tl)	20	4
Tin (Sn)	50	10

Element	Non-Potable Post Spike concentration (ug/L)	Potable Post Spike concentration (ug/L)
Titanium (Ti)	50	10
Vanadium (V)	50	10
Zinc (Zn)	50	10

9.1.6.3. Calculate the percent recovery of the post digestion spike as follows:

$$\%REC = \frac{Cps - Cs}{C2} \otimes 100$$

Where: Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = concentration of spike (ug/L

9.1.6.4. Limits for post digestion spike recoveries: 85-115%. Results must be within the acceptable limits, if not, repeat analysis and/or re-prepare the sample and reanalyze. If necessary dilute the QC sample (DU, MS, and PDS) to compensate for the matrix effect and reanalyze.

The post digestion spike must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgment when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyze to determine the extent of the matrix interferences. If necessary, adjust the interference corrections and reanalyze the samples.

9.1.7. Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. See Sec 9.1.5 for spiking procedures and acceptance limits.

- If matrix spike sample and matrix spike duplicate are both ≥ CRDL, then 20% RPD. If matrix spike sample and matrix spike duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.2. Instrument QC

Table 2 below describes the frequency, criteria, and corrective actions for the calibration and Quality control samples.

Table 2: Calibration, Quality control and Corrective Action Summary

QC Item	Frequency	Criteria	Corrective Action
Tune validation-Mass Calibration	Daily	Within 0.1amu from mass unit	Terminate analysis, fix problem and repeat
Tune validation-Resolution Check	Daily	≤0.9amu full width at 10% peak height	Terminate analysis, fix problem and repeat
Instrument Stability Check	Daily	<5% RSD for five replicates	Terminate analysis, fix problem and repeat
Initial Calibration: Multi-point-minimum 5 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	+/- 10%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within +/- 10% of the true value	Fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the RL.	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning of an analysis and after every 12 hours of analysis	Pay attention to false positives and false negatives for elements not present in the solutions. Results of the spiked elements should fall within +/- 20%.	If an element is consistently out of the +/- 20% range in the ICSAB solution, the problem should be investigated.
Serial Dilution (1/5 dilution)	One per batch of twenty samples or less	See Section 9.2.6	
Reporting Limit Check Standard (CRI)	At the beginning of analysis run	For *non-drinking water* analysis: Recovery +/- 50% of the true concentration. For *drinking water* analysis: Recovery +/- 50% of the true concentration for Sb, Ba, Be, Cd, Cr, Ni, Tl. All others +/- 15% of the true concentration	Stop the analysis, fix the problem and reanalyze the affected samples
Internal Standards	Analyzed with every standard and sample	200.8- 60-125%	Standard-terminate the analysis and recalibrate Sample- dilute (The analyst should check the IS response for the bracketing CCV/CCBs to see if there is a similar response to the sample.

QC Item	Frequency	Criteria	Corrective Action
			Recalibration may be required.)

9.2.1. Initial Calibration Verification (ICV): Analyze an initial calibration verification solution at the beginning of the run. ICV solution must have the same acid matrix as the calibration standard and it must come from a source other than the calibration standard source. The final concentrations of the various elements are listed in Attachment 1. The results for the target elements in the initial calibration verification (ICV) must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem and recalibrate the instrument. See Sec 7.2.4 for the ICV standard preparation instructions.

9.2.2. Continuing Calibration Verification (CCV): The calibration of the ICP-MS must be verified every 10 samples and at the end of the analysis run by analyzing the QC Check Solutions (CCV). For non-drinking waters, use Cal4/CCV. For drinking waters, use Cal3/CCV. The concentration of the CCV standard must be at or near mid-range levels of the calibration. The final concentrations of the various elements are listed in Attachment 1. The results for the target compounds must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples following the last good CCV.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. Use reagent water for the blank solution.

9.2.4. Reporting Limit Check Standard (CRI): At the beginning of the analysis run verify the accuracy at the RL by analyzing a solution at the RL. RL/PQL Check Solution is analyzed to demonstrate that the ICPMS is capable of detecting the target compounds at the reporting limit (RL). For non-drinking water analysis, the determined concentration must be within +/-50% of the true concentration. For drinking water analysis, the determined concentration must be within +/-50% of the true concentration for Sb, Ba, Be, Cd, Cr, Ni, and Tl; for all others, +/-15% of true concentration. See Attachment 1 for list of Rep Lim standard concentration in ug/L. Refer to Sec 7.2.8 for the RL/PQL standard preparation instructions.

9.2.5. Interference Check Sample (ICSA & ICSAB): Verify the inter-element and background corrections by analyzing the interference check solutions (ICSA & ICSAB) at the beginning of the analysis run and after every 12 hours of analysis. The ICS is analyzed in order to demonstrate that proper corrections are being utilized for known interferences. Analyst must evaluate these solutions in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for

elements not present in the interference check solutions. Results should be within 20% of the true value for each element. See Attachment 2 for list of elements and the corresponding concentrations in ppb.

9.2.6. Serial Dilution QC Check: A 1/5 dilution is prepared and analyzed on one sample per batch (one for every ten samples for Method 200.8, or whichever is greater) to determine if matrix interferences are present.

9.2.6.1. Select a sample digestate that contains one or more target analytes at concentrations greater than 10X the reporting limit.

9.2.6.2. Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

9.2.6.3. Compare the results of the diluted and un-diluted aliquots of sample digestate.

9.2.6.4. If the results of the dilution are within ±10% of the results of the undiluted sample, no matrix interference is present. If not, matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike.

9.2.7. Determination of Linear Range of the ICP-MS

Determine the linear dynamic range of the instrument by analyzing a minimum of three high standards at the upper limit of the instrument. The analytically determined concentration of these standards must be within 10% of the true value. The linear ranges should be redetermined annually or if the instrument is significantly changed.

9.2.7.1. Prepare the standard at concentrations that are expected to define the linear range of the instrument. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids. See Attachment 1 for Linear Range concentrations in ug/L.

9.2.7.2. Analyze the standard(s) after the initial calibration is validated.

9.2.7.3. Compare the concentration of the linear range standard with its true concentration using the following equation:

$$\text{Percent Difference} = \left|\frac{Ccal - Ctrue}{Ctrue}\right| \otimes 100$$

Where:

Ccal = concentration determined from analysis
Ctrue = true concentration of the standard

Note: If the percent difference is less than or equal to 10%, the linear range is confirmed at that concentration. If the percent difference is greater than 10%, repeat the analysis with a lower concentration. For elements validated in this manner you may report data up to 90% of that linear range before a dilution is required.

10.0. Procedure

10.1. Sample Preparation

10.1.1. For non-potable samples to be analyzed for total analytes by Method 200.8, follow TestAmerica SOP No. ED-MTP-001.

10.1.2. For Drinking water samples, if turbidity is ≥1, follow the digestion procedure for Total recoverable in TestAmerica SOP No. ED-MTP-001.

10.2. Calibration

10.2.1. Turn the ICP-MS on and initiate the tune screen. Start the tune screen to allow the instrument to become thermally stable before beginning to analyze the calibration standards. While the instrument is warming up, aspirate the interference check solution (or similar solution) to precondition the cones.

10.2.2. Check the tune parameters for the proper sensitivities, precision, oxides, and double charged values following the instrument manufacturer's recommendations. Refer to sections *Preparations Before Analysis*, *Plasma ON/Executing a Startup*, and *Appendix (Recommended Values for Tuning Parameters)* of the MassHunter Workstation User Guide (*Agilent Technologies 7800/7900, p/n G7201-90403).*

10.2.3. Prior to calibration, run an EPA Tune check solution. Refer to Sec. 7.2.7 for the Tune check criteria.

10.2.4. The instrument is required to be calibrated daily or once every 24 hours. For Non-Drinking Water analysis, use instrument method *BM New Method.icpms.template.* For Drinking Water analysis, use instrument method *DW New Method.icpms.template*. These are programs in the Mass Hunter Workstation software containing calibration standards, check tables, etc. Calibrate the instrument using a blank and five standards. Refer to the section *Creating a Method* in the instrument manufacturer's instructions *Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide (p/n G7201-90403)*.

10.2.5. Internal standards are added to the calibration standards, blank and samples in-stream by the use of T-fitting.

10.2.6. Analyze the calibration standards and calibrate the ICP-MS in accordance with the manufacturer's recommendations and the TestAmerica Corporate

SOP No. CA-Q-S-005 Calibration Curves (General).

10.2.7. Calibration Curve Read-Back:

10.2.7.1. Low-Level readback – evaluate Cal 1 which is at the LLOQ. The %RE (relative error) must be +/- 50% (see Sec 11.4); if not, stop the analysis and recalibrate.

10.2.7.2. Mid-Level readback – evaluate Cal 4. The %RE (relative error) must be +/- 10% (see Sec 11.4); if not, stop the analysis and recalibrate.

10.3. Sample Analysis

10.3.1. All sample measurements must be made within 90% of the linear range of the instrument. Preparation of all reference materials used for calibration must be documented.

10.3.2. All potable and non-potable samples are typically analyzed undiluted. Dilutions must be prepared in reagent water (Sec 7.1.6 for Potable; Sec 7.1.7 for Non-Potable).

10.3.3. The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.3.4. The samples are analyzed in a sequence as follows:

Typical Analytical Sequence
INSTRUMENT WARM-UP (approx. 30 minutes)
TUNE CHECK
STANDARDIZATION/CALIBRATION (Blank, 5 Cal Stds)
ICV
ICB
CRI
ICSA
ICSAB
LRC-A (6020B)
LRC-B (6020B)
Rn Chk
Rn Chk
CCV
CCB
10 Samples
CCV
CCB
10 Samples
CCV
CCB
{continue until done; after 12 hrs run ICSA/ICSAB}

Note: The analytical sequence must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.5. Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.5.1. If the sample concentration is greater than 90% of the linear range, the sample digestate must be diluted and reanalyzed.

10.3.5.2. The amount of sample digestate needed to prepare the desired dilution is determined from the following equation:

$$\text{V digest} = \frac{Vf\ vol}{DF}$$

Where:
Vf vol = final volume of diluted sample (mL)
V digest = volume of sample digestate used to make the dilution (mL)
DF = Dilution Factor

10.3.5.3. The dilution factor is calculated as follows:

$$\text{DF} = \frac{Vf\ vol}{V\ digest}$$

Where: Vf vol = final volume of diluted sample extract (mL)
V digest = volume of sample extract used to make the dilution (mL)

Note: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE

A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP-MS. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$\text{DF} = \frac{100ml}{1.0ml} = 100$$

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation:

$$\text{Concentration = ug/ L} = \frac{C \times V1 \times D}{V2}$$

Where:

C= Element concentration from instrument (ug/L)

V1= Final volume of sample digested (in liters)

D= Dilution performed on sample)

V2= Initial volume of sample digested (in liters).

11.4. Relative Error (%RE):

$$\text{\% Relative Error} = (100)\,(MC-TC)/TC$$

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Reduction

11.5.1. All data is recorded directly into the TALS Analyst Desktop II program.

11.5.2. Standard and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers.

11.5.3. Generate Raw Data PDF file for TALS

11.5.3.1. Using a scanner, scan all raw data, i.e., instrument printouts. This includes, the calibration, calibration curve info, and all samples analyzed (includes instrument and batch QC). Attach the pdf to the Docs section of the analytical batch in TALS (ADII).

11.5.4. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.5.4.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.
11.5.4.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'
11.5.4.3. Acknowledge by filling in responses to all unacknowledged findings.
11.5.4.4. Fill in appropriate comments in the response box, then hit 'OK.'
11.5.4.5. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

The IDL for EPA Method 200.8 is defined as the concentration equivalent to the analyte signal which is equal to 3 times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the selected mass(es) and should be determined annually.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization. The neutralization system consists of a 55-gallon poly open top container. There is also a local snorkel exhaust for venting any noxious odors. Once the liquid is transferred into the container, the contents are sparged with air and a representative pH is measured and the amount of material is recorded in the discharge log. The pH value is recorded in the discharge log, and the liquid is neutralized to a pH of 6-9 SU using food grade sodium bicarbonate. Once the secondary pH has been taken, confirmed and recorded, the discharge valve is opened to the municipal sewer system and the neutralization process is complete. The date, type of waste stream, and volume discharged are recorded on a discharge log.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  TSDF under Veolia Profile Number: 590598

- Soil Retain Samples - These containers are stored in the main sample-receiving laboratory cooler until they are designated for disposal. Once designated for disposal, the samples are transferred to the waste disposal area and placed into cubic-yard disposal containers, e.g., Jupiter-boxes. This waste is shipped directly to the TSDF under Veolia Profile Number 402535 and is subject to incineration.

15.0. References / Cross-References

15.1. Methods for the Determination of Metals in Environmental Samples; US EPA Office of Research and Development. Washington, DC, May 1994. Method 200.8, Revision 5.4.

15.2. TestAmerica Corporate SOP No. CA-Q-S-005, *Calibration Curves (General)*, most current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.6. TestAmerica Edison SOP ED-MTP-001, *Digestion of Aqueous Samples for Analysis by ICP and ICPMS USEPA Method No(s). 200.2, 200.7 (Rev 4.4), 200.8 & CLP SOW ISM01.2*, most current revision.

15.7. Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide, p/n G7201-90403, Rev. A, June 2017

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Standards and Matrix Spiking concentrations – Drinking water and non Drinking water.
Attachment 2: Interference Check standard elemental concentrations - Drinking water and non Drinking water.

18.0. Revision History

- **Revision 14, dated 15 January 2023**
  - Sec 1.1, Table1: Updated reporting limit for Al and Zn in Drinking water, and Ca & Na for NPW to reflect current laboratory limits.
  - Sec 2.0 & 8.0: Clarified instructions for the analysis of dissolved samples (i.e., digestion not required for the analysis of dissolved samples).
  - Sec 7.2.2: Updated the preparation instructions for the Working calibration standards (drinking water and non-drinking water) to reflect current lab practices.
  - Sec 7.2.4.2.2: Updated the preparation instructions for the ICV Non-Potable.
  - Sec 7.2.4.2.2: Added preparation instructions for Interm_ICV std
  - Sec 7.2.6: Replaced CAL3/CCV with CAL4/CCV.
  - Sec 7.2.7.2: Updated preparation instructions for the potable Working Internal standard; Added titanium to the list of analytes in the no-gas mode; Updated

concentration of tune check analytes.
- Sec 7.2.7.4: Updated preparation instructions for the Working EPA Tune Check Std.
- Sec 9.2, Table 2: Revised the minimum number of standards to 5 and 1 blank.
- Sec 10.1: Revised to specify 'total analytes' for samples which require digestion prior to analysis.
- Sec 14.2: Updated waste management information to reflect current practices.
- Attachment 1, Table for Drinking water standards: Updated Cal-3 standard concentrations to reflect current lab practices.

- **Revision 13, dated 08 October 2020**
  - Sec 1.1, Table1: Updated the Drinking water reporting limit concentrations for Sb, Ba, Be, Cd, Cr, Ni, Se, and Tl to reflect current laboratory limits.
  - Sec 3: Updated the Definitions locations as referenced in the Laboratory Quality Manual.
  - Sec 7.1.6: Updated the preparation instructions for the reagent water.
  - Sec 7.2: Added instructions to make varying amounts of standards
  - Sec 7.2.1: Removed stock standards TANJ-STD-1, TANJ-STD-2, TA-88, STLNJ-STD-1, STLNJ-STD-2, and TA-72REV2 from this SOP; Added stock standard 200.8 Potable RL standards.
  - Sec 7.2.2: Updated preparation instructions for Drinking Water standards.
  - Sec 7.2.3: Deleted stock standard ICV1ICPMS and ICV2ICPMS, no longer applicable.
  - Sec 7.2.4.1: Updated preparation instructions for ICV (Potable).
  - Sec 7.2.7.2: Added preparation instructions for the Potable working internal standard.
  - Sec 7.2.9: Deleted Interference stock standards 6020ICS-8A & 6020ICS-8B.
  - Sec 7.2.10: Updated preparation instructions for ICSA/ICSAB drinking water.
  - Sec 9.1.6.2: Updated Non-Potable PDS spiking instructions; updated Non-Potable PDS concentrations in Table.
  - Sec 9.2, Table 2 & Sec 9.2.4:Updated reporting limit (CRI) criteria limits for Sb, Ba, Be, Cd, Cr, Ni, Tl.
  - Sec 18.0, Attachment 1 (Potable): Updated calibration, ICV, CCV concentrations.
  - Sec 18.0, Attachment 2 (Potable): Updated ICSA & ICSAB concentrations

- **Revision 12, dated 18 June 2020**
  - Updated SOP header to Eurofins emblem.
  - Throughout the SOP removed all SW846 Method 6020 references.
  - For Non-Drinking Water preparations: Throughout the SOP, replaced all references to 2%HNO3/0.5%HCl with 5%HNO3/5%HCl.
  - Sec 1.1, Table 1: Removed references to typical dilutions and updated the RLs for Non-Drinking Water.
  - Sec 6.1: Added two ICPMS instruments, Agilent 7800 & 7900; removed reference to resolution and grating; added instrument mass range. Updated the models of the heat exchanger and autosampler.
  - Sec 7.1.7: Added preparation instructions for 5%HNO3/5%HCl.
  - Sec 7.2.1: Added CPI stock standard concentrations; CPI International is added as the vendor for the stock cal stds.

- Sec 7.2.2: Updated preparation instructions for the non-drinking water working cal standards and added preparation instructions for MS-Cal-Int.
- Sec 7.2.3: Updated the Initial calibration verification stock standards.
- Sec 7.2.4.2: Updated preparation instructions for the non-potable ICV working standard.
- Sec 7.2.4.2.1: Added preparation instructions for MS-ICV-Int.
- Sec 7.2.7.1.2: Replaced Inorganic Ventures with CPI International as the vendor source for the Ge stock standard.
- Sec 7.2.7.2: Updated preparation instructions for the Internal standard. Changed recommended mass for Ca (non-potable) from 44 to 40. Updated typical referenced internal std for As & Se from Ge to Sc. Updated typical analysis mode for non-potable Ca & Fe. Added Ca44 as the typical mass used for Calcium during potable analysis.
- Sec 7.2.9 & 7.2.10: Added ICSA/ICSAB stock standards from CPI International and updated Non-Potable preparation instructions for working ICSA & ICSAB.
- Sec 9.1 & 9.11: Updated control limit criteria for the Preparation blank.
- Sec 9.1.2 & 9.1.5: Updated preparation instructions for the LCS water samples and aqueous matrix spikes (MS).
- Sec 10.2.2: Added reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900. Removed references to ICPMS1&2 (includes Chemstation software and relating Operation Manuals) from this SOP.
- Sec 10.2.4: Added instrument method program and reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900.
- Sec 10.2.7.1 & 10.2.7.2: Added Low-Level readback and Mid-Level readback evaluation instructions.
- Sec 10.3.2: Updated typical dilution instructions.
- Sec 10.3.4: Updated Typical Analytical Sequence table to reflect current laboratory practices.
- Sec 11.3.2: Added final results calculation instructions.
- Sec 11.5: Added %Relative Error calculation equation.
- Sec 11.6.5: Added instructions for the Data Review Checker (DRC); removed reference to the Metals data review checklist EDS-WI-146, not applicable.
- Sec 15.3, 15.11, & 15.12: Deleted references for the Agilent 7500 Manuals, not applicable.
- Sec 15.13: Deleted reference to the ICPMS Data Review Checklist, CA-Q-WI-044.
- Sec 15.14: Added reference for the Agilent ICPMS 7800/7900 User Guide.
- Sec 18.0, Attachment 1 (Non-Potable): Updated calibration, ICV, CCV concentrations; removed dilution instructions.
- Sec 18.0, Attachment 2 (Non-Potable & Potable): Updated ICSA & ICSAB analyte list and concentrations.

- **Revision 11, dated 26 April 2018**
  - Sec 7.2.7.4: Added Magnesium as one of the working tune check solution's constituents.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix.

- **Revision 10, dated 15 November 2016**

- Sec 1.1, Table 1: Changed the Drinking water RL from 2.5 ug/L to 2 ug/L. Removed reference to Method 3005.
- Sec 2.0: Removed reference to method 3005 and replaced SOP ED-MTP-007 with SOP ED-MTP-001.
- Sec 7.1.6: Reagent recording in ICPMS Reagent Dilution logbook is replaced with TALS Reagent Module.
- Sec 7.2.1: Replaced stock standard TA-72 with standard TA-72REV2.
- Sec 7.2.2: Replaced standard TA-72 with TA-72REV2 for CAL1 standard-Drinking Water.
- Sec 7.2.7.2: Tune check criteria: updated the resolution check criteria to reflect laboratory practices.
- Sec 7.2.10: Added the preparation instructions for ICSA & ICSAB standards used when analyzing drinking water samples.
- Sec 9.2, Table 2: Updated Tune validation-Resolution Check criteria to reflect laboraotry practices
- Sec 10.1: Replaced SOP ED-MTP-007 reference with SOP ED-MTP-001.
- Sec 11.5.4: Replaced checklist CA-Q-WI-004 with Work Instruction EDS-WI-146.
- Attachment 1, Drinking water: Replaced the Calibration concentration and RL Std.Conc. of Pb from 2.5 to 2 ug/L.
- Attachment 2: Added ICSA & ICSAB elemental concentrations for Drinking water analysis.

- **Revision 9, dated 02 April 2015**

  - Throughout the SOP: Removed all reference to SOP# ED-MTP-002.
  - Sec 7.2.1: Added calibration standards TA-88 and TA-72.
  - Sec 7.2.2: Added the new low-level cal std (CAL2) for non-potable water and updated the preparation instructions for the potable cal standards.
  - Sec 7.2.7.1.3: Added Triton X-100 to the list of internal standard stock solutions.
  - Sec 7.2.7.2: Updated the preparation instruction of the working internal standard solution to include Triton X-100; Revised note to state that Selenium is now analyzed under the Hydrogen reaction mode.
  - Sec 7.2.8: CAL1 is now used as the RL check standard for drinking water analysis.
  - Sec 9.1.6.4: Added PDS recovery limits (85-115%) for method 200.8. Clarified the procedure when PDS is outside the acceptable limits.
  - Sec 9.1 and 9.1.7: Added Matrix Spike Duplicate (MSD) to the list of Sample QC.
  - Sec 11.5.4: Updated the metals data review checklist form number.
  - Sec 15.13: Added TestAmerica Corp form No. CA-Q-WI-044 to the list of references.
  - Attachment 1 (non-Drinking Waters): Updated the calibration concentrations for all metals; updated the matrix spike concentration for Zinc (water & soil).
  - Attachment 1 (Drinking Waters): Updated the calibration concentrations for As, B,Sn and Zn.

- **Revision 8, dated 05 December 2012**

  - Sec 1 and 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 2.0: Deleted the exception that silver analysis does not undergo digestion. Silver may be directly analyzed for drinking waters provided the turbidity <1.
  - Sec 7.1.6: Revised to include the reagent water prep for potable samples not

requiring digestion. Revised preparation frequency to 12 months to reflect actual laboratory practices.
- Sec 7.2.2 & 7.2.3: Sections deleted to remove Hg stock standard info and the preparation instructions of Hg intermediate standard; subsequent sections adjusted accordingly.
- Sec 7.2.2 (formerly Sec 7.2.4): Removed all references to Hg. Clarified M1CAL3 as the reporting limit for Drinking Water standard. Clarified M1CAL2 is not the reporting limit for Boron.
- Sec 7.2.3 and 7.2.4 (formerly Sec 7.2.5 & 7.2.6): Hg references have been removed.
- Sec 7.2.6: Section added; subsequent sections adjusted accordingly.
- Sec 7.2.7 (formerly Sec 7.2.8): Added 'EPA Tune Check' to the section header. Removed the use of gold standard from this SOP. Hg is not analyzed by this method.
- Sec 7.2.7.1. Added internal standard stock solutions.
- Sec 7.2.7.2: Added internal standard preparation instructions. Also clarified that drinking water samples are analyzed without the use of collision cell technology.
- Sec 7.2.7.3: Added Tune Check stock standard
- Sec 7.2.7.4: Added preparation instructions of the EPA Tune Check standard.
- Sec 7.2.11 (formerly Sec 7.2.12): Changed standard vendor for the Matrix spike stock solution to High-Purity Standards.
- Sec 8: Revised the wait time for sample preparation after in-lab preservation to 24 hours to comply with the updated 40CFR Part 136.
- Sec 9.1: Preparation Blank's control limits have been updated to reflect method requirements.
- Sec 9 and 10: Revised section references of standard solutions where applicable.
- Sec 10.1.2: Revised SOP number reference to reflect actual laboratory practices.
- Sec 10.2.2: Included Tuning Handbook reference for ICPMS2.
- Sec 10.2.4: Included Operation Manual for Masshunter software to ICPMS2.
- Sec 11.5: Added instructions for data recording, raw data PDF creations and Metals Data Review Checklist.
- Sec 15.4: Deleted SOP No. ED-MTP-007 reference, not applicable; subsequent sections adjusted accordingly.
- Sec 15.11 through 15.13: Added applicable references.
- Sec 17.0: Attachments 3 and 4 have been removed.
- Attachment 1: Deleted 'Linear range conc.' column; new linear range studies are updated on instruments. Updated MS concentration for Boron.

- **Revision 7, dated 16 July 2010**

  - Sec 1.1 Table 1: Revised Reporting Limits for drinking water and Soil.
  - Sec 3: Revised definitions reference to Appendix 2 of ED-QA-LQM.
  - Sec 7.2.1: Added STLNJ-STD-1 and STLNJ-STD-2 to the list of Calibration Stds
  - Sec 7.2.4: Revised working calibration standard including preparation procedure to reflect actual lab practices.
  - Sec 7.2.9: Revised prep procedure for RL check standard. Also deleted RL/PQL working standard, previously section 7.2.10. Subsequent sections adjusted accordingly.
  - Sec 18.0 Attachment 1: Table updated to reflect correct calibration standard concentrations and drinking water reporting limits.

- **Revision 6, dated 11 September 2009**

- Sec 9.2 Table 2 & Sec. 9.2.4: Revised Reporting limit criteria for drinking water analysis to ± 15%.

- **Revision 5, dated 08 July 2009**

  - Sec 4.1: Expanded section to include the isobaric polyatomic/molecular ion interferences; clarified using collision/reaction cell for non-potable samples only.
  - Sec 4.2: Added equations used for drinking water analysis when collision/reaction cell can not be used.
  - Sec 4.3: Added Krypton interference which affects arsenic and selenium, and the use of high purity Krypton free argon.
  - Sec 6.1 Instrumentation: Add collision/reaction cell.
  - Sec 7.2.4: Added a low calibration standard (M1CAL1) increasing the number of calibration standards to five; five cal standards will only be analyzed for elements Be, Fe, Pb and Mn.
  - Sec 7.2.8: Added Kr83 in the list of elements in order to monitor Kr levels.
  - Added text in Tune check criteria, Sec 7.2.8: *Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.*
  - Sec 8: Clarified holding time and procedure for acidifying unpreserved samples.
  - Sec 9: Added text: *If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.*
  - Sec 9.1 & 9.1.2: Changed LCS control limit for Method 6020 from ± 25% to ± 20%.
  - Sec 9.2.2: CCV standard- changed solution to M1CAL4.
  - Sec 10.2.4: Calibration- changed the number of standards from three to four standards.
  - Sec 10.3.2: Added in the procedure the requirement for diluting samples 5x (water) and 20x (soil) prior to analysis; subsequent section numbers were adjusted accordingly.
  - Sec 10.3.5.1: revised to include that samples will be diluted if the concentration is >90% of the linear range.
  - Attachment 1: Revised calibration standard concentration specifically Cal std 2 to reflect the correct concentration. Additionally, a fifth cal standard was added to the four elements (Be, Fe, Pb, and Mn) so the RL will be at or above the low level standard; Reporting limits were also revised for these elements.
  - Attachment 3: Revised dilution requirement for soil analysis from 10x to 20x.

- **Revision 4, dated 24 March 2009**
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1: Relocated Table 1 to this section. Updated 6020 Soil RLs. Revised several RLs for Drinking Water and Digested Water
  - Sec. 2: Added the method and SOP references for digestion of samples to be analyzed for metals via ICPMS.
  - Sec 6.1: Added Heat exchanger and autosampler to the list of instruments.
  - Sec 7.1.6: Added reagent water and its prep instructions to the list of reagents.
  - Sec 7.2 Standards: Included vendor and storage information of standards.
  - Sec 7.2.2: Revised the vendor source and stock concentration of the Hg standard from 1000 ppm to 10 ppm; revised preparation instructions of the Intermediate standard (Hg-Cal-Int).

- Sec 7.2.4: Revised prep instructions for the working calibration standards (i.e. amount of spiking solution and final volume).
- Sec 7.2.5: Deleted ICV stock standards STLNJ-Cal 6 & 7 and added ICV stock standards ICVICPMS and ICV2ICPMS.
- Hg Secondary stock solution in Sec 7.2.5: Revised the vendor source and stock concentration of the Hg secondary standard from 1000 ppm to 10 ppm; revised preparation instructions of the Intermediate standard (Hg-ICV-Int).
- Sec. 7.2.8: added statement: "Drinking Water Analysis: All tune steps = 3 (thereby eliminating the use of the Collision/Reaction Cell.) Use masses Fe57, Cu65, and Se82 instead of their respective masses listed above."
- Sec 7.2.9: Deleted ICPMS RL stock standard and added custom standards (STLNJ-STD-1 & 2) for Reporting Limit Check standard.
- Sec 7.2.10: Revised prep instructions for the RL/PQL Working standard.
- Sec 7.2.11: Revised prep instructions for the Working Interference check standards (final volume reduced from 100ml to 50ml; spiking amount adjusted).
- Sec 8: Sample collection, preservation and storage: Reformat section into Table format and included minimum sample size and reference information.
- Sec. 9.1: Added prep instruction to the QC samples (MB, LCS, BS and MS).
- Deleted Single point method of standard addition; previously identified Sec.9.2.8.
- Sec 10.1: Clarified the digestion procedure utilized for digesting different matrices (wastewater, drinking water and soil).
- Sec 15: Added applicable references.
- Sec 17: Added Attachment 2 (Interference Check Standard elemental concentrations).

- **Revision 3, dated March 2007**
  - Section 16. Attachment 1. Modified the 'Linear Range Concentration' of 11 elements (Sb, Be, B, Cd, Co, Pb, Se, Ag, Sr, Tl, V) to reflect the most recent LDR study.

- **Revision 2**, **dated February 2007**
  - Revised various sections of the SOP to include the specific sections in the Operator's manual where applicable information can be found. These sections are Sec. 5.3, 10.1.2., 10.1.3, Appendix A – Analytical Sequence Table.
  - Section 6.3 Calibration Standards. Modified the frequency for the preparation of calibration standards from six months to two weeks as per Method 200.8.
  - Section 6.4. Linearity Check Standards. Revised the HNO3 acid concentration used in the preparation of linearity check solutions to '2% by volume' to reflect actual laboratory practices.
  - Section 6.5 (Internal Standards). Revised the concentration of the element Au, both spiking and resulting concentrations.

    '*A solution of 1 mg/L (internal standard elements) and 2 mg/L of Au is prepared and added in-stream by the use of a T-fitting. The resulting concentration (in-stream) of the standard is ~100 ug/L (Au) and ~50 ug/L (internal standard elements).'*

  - Section 7.4. Section deleted; contents not applicable to this SOP.
  - Section 8.1.1 (MDL and IDL determination). Modified the solution used in the analysis of IDL to 'reagent blank solution' to reflect actual laboratory practices.
  - Section 8.1.2 (MDL and IDL determination). Added an SOP reference to further describe MDL determination.

- Section 10.3.2. The name of the Reporting limit solution in the analysis sequence was changed from 'Detection Limit Check Solution' to 'Reporting Limit Standard (RL).'
- Section 15.1, deleted; reference not applicable. Subsequent section numbers adjusted accordingly.
- Section 15.1 (previously identified as section 15.2), was revised to include the specific method in the reference information, in this case method 6020.
- Section 15.2 (previously identified as section 15.3), was revised to include the specific method in the reference information, in this case method 200.8.
- Section 15.6. Added Operator's instructions manual in the list of references. The section previously identified as 15.6 is now 15.5.
- Section 16. Attachment 1. Modified the 'Linear Range Conc. (ug/L)' to reflect the most recent LDR study. Updated the 'RL Std. conc.' for Boron, Iron, Tin and Zinc to reflect actual laboratory's reporting limits for these elements.
- Appendix A (Sample Preparation). Specified the name of the SOP used in the digestion of samples.
- Appendix A (Calibration, Quality Control and Corrective action summary). Revised the criteria for the ICSA/ICSAB to clarify that ICSA when used as linear range sample the determined analyte concentrations >90% must be diluted and reanalyzed.
- Appendix B (ICV Solution). Added the frequency for the preparation of the ICV solution.

- **Revision 1, dated January 2007**

  - Specified Operator's manual '*Agilent 7500 ICP-MS Chemstation (G1834B)*' in various sections: Sections 5.3, 10.1.2, 10.1.3, Appendix A – Analytical Sequence section.
  - All references to '6020A' are updated to '6020' to reflect actual method reference (i.e. sections 1.1, 2.2, 8.1.2 etc.).
  - Section 10.1.3. Filename '2008.M' is specified as the program/filename used in the ChemStation software.
  - Section 16 Table 1. Updated Reporting limits of 8 elements (As, Cd, Co, Cu, Ni, Ag, Sr, Sn) to reflect current standard concentrations and laboratory's reporting limits.
  - Section 16 Attachment 1. Revised Matrix spike concentrations for water and soil to include the actual final expected concentrations of the matrix spike.
  - Appendix B – Calibration, Quality Control and Corrective action summary. Criteria for internal standards was changed from >30% to 30-120% to reflect actual method criteria limits.
  - Appendix B – ICP Interference Check Solutions for SW846 6020. Added the frequency of preparation for the Working Interference Standards ICSA and ICSAB, 'these standards are made fresh weekly.'
  - Appendix B. Added data qualifiers for ICP-MS
  - Section 15.4. Added USEPA CLP ILM05.3 to the list of references.

Attachment 1

STANDARDS AND MATRIX SPIKING CONCENTRATIONS
For Non-Drinking Waters

Name	Mass	Cal1 CRI	Cal-2	Cal-3	Cal-4/CCV	Cal-5	ICV	*LCS/MS Conc*
		(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	*(ug/L)*
Be	9	0.8	4	50	100	200	80	*25*
B	11	80	400	125	250	500	200	*500*
Na	23	200	1000	12500	25000	50000	20000	*2500*
Mg	24	200	1000	12500	25000	50000	20000	*2500*
Al	27	40	200	12500	25000	50000	20000	*2500*
K	39	200	1000	12500	25000	50000	20000	*2500*
Ca	40	200	1000	12500	25000	50000	20000	*2500*
Ti	47	4	20	50	100	200	80	*50*
V	51	4	20	50	100	200	80	*50*
Cr	52	4	20	50	100	200	80	*50*
Mn	55	8	40	50	100	200	80	*250*
Fe	56	120	600	12500	25000	50000	20000	*2500*
Co	59	4	20	50	100	200	80	*25*
Ni	60	4	20	50	100	200	80	*50*
Cu	63	4	20	50	100	200	80	*50*
Zn	66	16	80	125	250	500	200	*250*
As	75	2	10	50	100	200	80	*50*
Se	78	2.5	12.5	50	100	200	80	*50*
Sr	88	4	20	50	100	200	80	*50*
Mo	95	4	20	50	100	200	80	*50*
Ag	107	2	10	25	50	100	40	*25*
Cd	111	2	10	50	100	200	80	*25*
Sn	118	16	80	50	100	200	80	*50*
Sb	121	2	10	50	100	200	80	*25*
Ba	137	4	20	50	100	200	80	*50*
Tl	205	0.8	4	50	100	200	80	*20*
Pb	208	1.2	6	50	100	200	80	*25*

STANDARDS AND MATRIX SPIKING CONCENTRATIONS
For Drinking Waters

Name	Mass	Cal1 CRI	Cal-2	Cal-3	Cal-4/CCV	ICV	*LCS/MS Conc*
		(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	*(ug/L)*
Be	9	0.3	10	25	100	40	*5*
B	11	20	20	50	200	80	*100*
Na	23	250	500	1250	5000	2000	*500*
Mg	24	250	500	1250	5000	2000	*500*
Al	27	25	500	1250	5000	2000	*500*
K	39	250	500	1250	5000	2000	*500*
Ca	44	250	500	1250	5000	2000	*500*
Ti	47	2.5	10	25	100	40	*10*
V	51	2.5	10	25	100	40	*10*
Cr	52	0.9	10	25	100	40	*10*
Mn	55	25	10	25	100	40	*50*
Fe	57	250	500	1250	5000	2000	*500*
Co	59	2.5	10	25	100	40	*5*
Ni	60	0.5	10	25	100	40	*10*
Cu	65	2.5	10	25	100	40	*10*
Zn	66	4	10	25	100	40	*50*
As	75	1	10	25	100	40	*10*
Se	82	8	10	25	100	40	*10*
Sr	88	2.5	10	25	100	40	*10*
Mo	95	2.5	10	25	100	40	*10*
Ag	107	2.5	10	25	100	40	*5*
Cd	111	0.5	10	25	100	40	*5*
Sn	118	4	10	25	100	40	*10*
Sb	121	0.4	10	25	100	40	*5*
Ba	137	2	10	25	100	40	*10*
Tl	205	0.3	10	25	100	40	*4*
Pb	208	2	10	25	100	40	*5*

Attachment 2

Interference Check Standard (ICSA & ICSAB) Elemental Concentrations				
ELEMENT	Non-Potable		Potable	
	ICSA (ug/L)	ICSAB (ug/L)	ICSA (ug/L)	ICSAB (ug/L)
Sodium	100000	100000	25000	25000
Magnesium	100000	100000	25000	25000
Aluminum	100000	100000	25000	25000
Potassium	100000	100000	25000	25000
Calcium	100000	100000	25000	25000
Titanium	2000	2000	500	500
Iron	100000	100000	25000	25000
Molybdenum	2000	2000	500	500
Carbon	200000	200000	50000	50000
Chloride	1000000	1000000	250000	250000
Phosphorus	100000	100000	25000	25000
Sulfur	100000	100000	25000	25000
Silver	n/a	200	n/a	50
Arsenic	n/a	200	n/a	50
Barium	n/a	200	n/a	50
Beryllium	n/a	200	n/a	50
Cadmium	n/a	200	n/a	50
Cobalt	n/a	200	n/a	50
Chromium	n/a	200	n/a	50
Copper	n/a	200	n/a	50
Lithium	n/a	200	n/a	50
Manganese	n/a	200	n/a	50
Nickel	n/a	200	n/a	50
Lead	n/a	200	n/a	50
Antimony	n/a	100	n/a	25
Selenium	n/a	200	n/a	50
Silicon	n/a	200	n/a	50
Tin	n/a	200	n/a	50
Strontium	n/a	200	n/a	50
Thallium	n/a	100	n/a	25
Vanadium	n/a	200	n/a	50
Tungsten	n/a	2000	n/a	500
Zinc	n/a	200	n/a	50

Title: SW846 Method 8082A, Analysis of Polychlorinated Biphenyls by Gas Chromatography

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Approvals (Signature/Date):

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1.0 Scope and Application

This method is used to quantify specific polychlorinated biphenyls (PCBs) as Aroclors (see Table 1 below) in extracts from aqueous, soil, sludge, leachate, wipe or oil matrices by direct injection dual capillary column gas chromatography using SW846 Method 8082A. An electron capture detector (ECD) is employed for detection.

1.1 Analytes, Matrix(s), and Reporting Limits

The specific analytes determined by this method are identified in Table 1.

Table 1
Polychlorinated Biphenyls

Compound Name	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

The routine Eurofins Edison reporting limits (RLs) by analyte and matrix are summarized in Table 2 (below).

Table 2
Reporting Limits by Matrix

Parameter	Soil	Soil	Water	Leachate	Oil	Wipe
	Reporting Limits (ug/kg) LOW Level	Reporting Limits (ug/kg) MED Level	Reporting Limits (ug/L)	Reporting Limits (mg/L)	Reporting Limits (ug/kg)	Reporting Limits (ug/wipe)
Aroclor-1016	67	500	0.50	0.0050	1000	0.40
Aroclor-1221	67	500	0.50	0.0050	1000	0.40
Aroclor-1232	67	500	0.50	0.0050	1000	0.40
Aroclor-1242	67	500	0.50	0.0050	1000	0.40
Aroclor-1248	67	500	0.50	0.0050	1000	0.40
Aroclor-1254	67	500	0.50	0.0050	1000	0.40
Aroclor-1260	67	500	0.50	0.0050	1000	0.40
Aroclor-1262	67	500	0.50	0.0050	1000	------
Aroclor-1268	67	500	0.50	0.0050	1000	------

The most current MDLs and RLs for this method can be found in the active Eurofins LIMS (TALS) SW846 8082A Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8082A. A measured volume or weight of sample (15 g for soil, 1 g for oil, 250 ml for water and TCLP/SPLP/ASTM leachates) is extracted using the appropriate matrix-specific sample extraction technique (reference the applicable Organic Sample Prep SOPs listed below). The extract is exchanged into hexane and concentrated to a final volume between 1 and 20 ml depending upon the prep technique used.

2.1.1. Aqueous and leachate samples are extracted at a neutral pH using SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C).*

2.1.2. Wipe samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B*)

2.1.3. Solid samples are extracted using SW846 Method 3546 (*SOP No. ED-ORP-0044: Procedure for the Microwave Extraction of Solids,SW846 Method 3546*).

2.1.4. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A*).

2.1.5. Extract cleanup steps are employed as need depending on the nature of the matrix interferences encountered. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*), SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) and SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*) for heavy organic interferences.

2.2. After cleanup, the extract is analyzed by injecting a known volume of sample into a gas chromatograph equipped with a dual wide-bore fused silica capillary columns and dual electron capture detectors (GC/ECD). The GC is temperature programmed to separate and detect the analytes recovered during the extraction step. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point initial calibration (six points for second order regression). Specific calibration and quality control steps are detailed in this SOP and meet the specification of SW846 Method 8082A.

2.3. Samples are analyzed only after all the necessary calibration and QC checks have been performed.

2.4. Acquired data from sample analysis is manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences from phthalate esters introduced during sample preparation can pose major difficulties for PCB determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCBs. Sulfur contamination should be expected with sediment samples. Employ SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) for removal of sulfur.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*),

4.4. Interferences from other organic compounds can effectively be removed using a sulfuric acid treatment, SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*). This destructive technique can be employed only when the sample extract is being analyzed solely for PCBs (i.e., it is not to be used prior to analysis for pesticides).

4.5. Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD) will cause interference. A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples. A System Performance Check (DDT/Endrin) is analyzed daily to assist in evaluation of potential DDT interference with this Aroclor 1254 peak.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas Chromatograph:

6.1.1 Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC), equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides and PCB's. All operations are as automated as possible with the equipment utilized.

6.1.2 Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles a single sample between the sample tray and the injector turret.

6.1.2.1 The sample is injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.3 Liners: The injection port is each fitted with a replaceable, heavy-walled glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.4 Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.4.1 Two dissimilar columns are used for analysis. A Restek StxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column (or equivalent) is used for sample quantitation. The secondary column is a Restek StxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column (or equivalent).

6.1.5 Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.5.1 Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas is fed from a supply other than the injection port.

6.2 Data System:

6.2.1 The data systems consist of Agilent Technologies GC Chemstation Revision A.08.02 and Agilent Technologies Enviroquant Chemstation G1701AA Version A.03.00 upgraded to A.03.02 which is used for acquisition and Eurofins Chrom (chromatography data processing software).

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Ultra high purity (99.999%) Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Nitrogen is used as make-up gas. (Alternatively, ultra-high purity Helium with P-5 make-up gas may be used). Make-up gas is introduced to the GC via the make-up gas adapter at the end of the capillary column. Gases are supplied at tank pressures of 2000-2400 psig for a 300 cft tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the G.C. The traps are as follows:

- Hydrocarbon trap
- H_2O (moisture) trap
- O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction, clean up procedures and dilutions include Hexane, Methylene Chloride, and Acetone that are exchanged to Hexane prior to analysis. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis.

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions. Standard compounds or mixtures for this analysis include an Aroclor 1016/1260 mix, Aroclor 1221, 1232, 1242, 1248, 1254, 1262, 1268 and the surrogate compound Decachlorobiphenyl (DCB) (packaged with the Tetrachloro-m-xylene (TCMX), a surrogate used in pesticide analysis).

NOTE: Two independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent.

7.2.2. Standards mixes and sources: *

Standard Name	Source	Concentration
TCMX/DCB Surrogate Calibration Mix	Restek 32000	200 ug/ml
TCMX/DCB Surrogate Spike Mix	Supelco 861275	10 ug/ml
Aroclor 1016 Calibration Standard	Supelco 48097	1000 ug/ml
Aroclor 1221 Calibration Standard	Restek 32007	1000 ug/ml
Aroclor 1232 Calibration Standard	Restek 32008	1000 ug/ml
Aroclor 1242 Calibration Standard	Restek 32009	1000 ug/ml
Aroclor 1248 Calibration Standard	Restek 32010	1000 ug/ml
Aroclor 1254 Calibration Standard	Restek 32011	1000 ug/ml
Aroclor 1260 Calibration Standard		1000 ug/ml
Aroclor 1262 Calibration Standard	Restek 32409	1000 ug/ml
Aroclor 1268 Calibration Standard	Restek 32410	1000 ug/ml
Aroclor 1660 Mix (Arolcors 1016 & 1260)	Restek 32039	1000 ug/ml
Aroclor 1016/1260 Calibration Standard (Second Source)	Restek 32039.sec	1000 ug/ml
1-Bromo-2-nitrobenzene (internal standard)	Restek 32279	1000 ug/ml

*Suppliers with equivalent standards may be used.

7.2.3. Aroclor 1016/1260 & Surrogate Calibration Standard Solution Preparation

Five levels of calibration standards are prepared using the above referenced Aroclor 1660 Mix (Restek – 23039) and TCMX/DCB Surrogate Calibration standard mix (Restek 32000). They are prepared as follows:

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (1000 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
50 ppb (25 ppb DCB) [1]	5	6.25	100
500 ppb (50 ppb DCB)	50	25	100
1000 ppb (100 ppb DCB)	1000	500	1000
1500 ppb (150 ppb DCB)	150	75	100

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (1000 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
2500 ppb (200 ppb DCB)	250	100	100

(1): The low level Aroclor 1016/1260 standard is 50 ppb and 12.5 ppb for the surrogate DCB (prepare by making a 2x dilution of the 100 ppb standard in hexane).

7.2.4. Surrogate Spiking Solution (soil and water)

A TCMX/DCB Surrogate Spike Mix is prepared by diluting 10 ml of Restek 32000 (see Table 1 above) to 200 ml of Acetone. Final solution concentration is 10 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 10 ug/ml solution to 100 ml acetone with a final solution concentration of 2 ug/ml.

7.2.5. Aroclor 1016/1260 Spiking Solution (soil, water and wipe)

An Aroclor 1660 Mix is prepared by diluting 10 ml of Restek 32039 (See Tables 1 and 2 above) to 100 ml acetone. Final solution concentration is 100 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 100 ug/ml solution to 200 ml with a final solution concentration of 20 ug/ml. used spiking soils and waters as received from Supelco without further dilution.

7.2.6. Individual Aroclor Calibration Solutions (1221, 1232, 1242, 1248, 1254, 1262 & 1268)

A 1000 ppb calibration standard is prepared for each remaining Aroclor from the stock standards detailed in Section 7.2.1. 200ul of 1000 ug/ml individual Aroclor solution and 100ul of 200 ug/ml TCMX/DCB is diluted to 200ml with Acetone. The final concentration of surrogates is 100 ppb.

7.2.7. Aroclor 1016/1260 Initial Calibration Verification (ICV) Standard Solution Preparation

A mid-point Aroclor 1016/1260 ICV standard is prepared using the second source Aroclor 1016/1260 Calibration Standard (Restek 32039.sec) detailed in Section 7.2.2. 1000 ul of 1000 ug/ml standard along with 500 ul of 200 ug/ml TCMX/DCB surrogate standard (Restek 32000) is diluted to 1000 ml with acetone for a final ICV concentration of 1 ug/ml (1000 ppb).

7.2.8. PCB Internal Standard Spike Mix (1 ug/ml)

The PCB 1 ug/ml internal standard spike mix is prepared by dilution 500ul of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 500 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.9. System Performance Solution (3,3-DDT and Endrin at 0.25 ug/ml):

The DDT/Endrin performance solution is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane.

8. Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	SW846

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8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.2. Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

9. Quality Control

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

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard	every sample[3]	Response within -50% to +100% of most recent cal standard.

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

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. **Laboratory Control Sample (LCS):** A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to assess method performance and serves to determine whether the methodology is in control at the time of preparation and analysis. The recoveries of the LCS must fall within lab generated acceptance criteria. If the LCS recovery results are outside QC limits, the extract is reanalyzed. If upon reanalysis the recoveries remain outside of recovery limits the following evaluations are made:

- If LCS results fall outside the laboratory generated limits with low recoveries (refer to the current TALS Method Limit Group database), the LCS and all associated samples should be re-extracted and re-analyzed.

- If LCS results fall outside the laboratory generated limits with high recoveries (refer to the current TALS Method Limit Group database), the LCS and associated sample results may be reported with an Non-Conformance Memo (NCM) detailing the issue.

9.1.3. **Matrix Spike/Matrix Spike Duplicate (MS/MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. **Surrogate Standards:** All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the DCB surrogate standard is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database). (Note: the surrogate must pass CCV criteria to be reportable and must be reported from the same

column as sample target analyte results). If the DCB recovery is outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzne) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.2. Instrument QC

9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.1.1. *Initial Calibration Range*: Aroclors 1016/1260 and the surrogate (DCB) are calibrated using a five-point calibration range using a minimum of five (5) peaks per Aroclor. The reporting limit (RL) is equal to the low point of the calibration range. The initial calibration block must include at least one level with Aroclor 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration). If the 1016/1260 calibration meets the required average RF criteria all other Aroclors are then calibrated at the anticipated midpoint of the calibration range with a single point calibration using a minimum of five (5) peaks for each Arolcor (minimum of 3 peaks for Aroclor 1221). All peaks selected must be at least 25% the peak height of the largest peak in the Aroclor (except for Aroclor 1268 where the requirement is 10%). The following Aroclors can be analyzed together: 1016/1260, 1221/1254, 1232/1262 and 1242/1268. Standards are prepared following the instructions in Section 7.2.

9.2.1.2. *Initial Calibration Verification (ICV):* An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.7 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.2. Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260. If an Aroclor other than Aroclors 1016/1260 is detected a CCV of the identified Arolcor must be analyzed within 12 hours of the sample. If that CCV does not meet criteria the sample must be reanalyzed under an acceptable CCV using the detected Arolcor. The response factors for the CCV must be within +/- 20 % of the initial calibration RF.

9.2.3. Calibration Acceptance Summary

9.2.3.1. Retention Time Windows: Retention time (RT) windows must be determined for all analytes.

9.2.3.1.1 Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.3.1. Initial Calibration Range. The internal standard calibration technique is employed for this method. The response factor (defined as the ratio of the area to the standard concentration) is calculated for each characteristic peak in the Aroclor 1016/1260 standard at each calibration concentration. The percent relative standard deviation (% RSD) of the response factors for each individual peak in the Aroclor 1016/1260 mix on each column is then determined (both columns must pass calibration criteria).

9.2.3.1.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

9.2.3.1.2. Linear Calibration: If the % RSD is less than 20% over its working range for at least five peaks in the Aroclor 1016/1260 mix, the linearity of the range is assumed for all Aroclors over the same analytical range. Each individual peak's response factor is used for quantitation of all the samples and verification standards. The average of the value calculated for each individual peak is used to report the concentration in the samples.

9.2.3.1.3. Linear Calibration Using Least Squares Regression: If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r squared value must be > 0.99 for the calibration to be acceptable

9.2.3.1.4. Relative Standard Error: The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative

Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (*r*) and Coefficient of Determination (r_2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.3.2. Initial Calibration Verification (ICV):

9.2.3.2.1. After the initial calibration range has been analyzed, an Initial Calibration Verification standard is analyzed on each column to verify the validity of the initial calibration range. The ICV standard must be from a standard lot independent of the standards used in the initial calibration range. The verification standard for PCBs is the mid-range Aroclor 1016/1260 standard at 1000ppb. (See Section 7.2.7 for details on the preparation of the ICV).

9.2.3.2.2. At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20%, the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

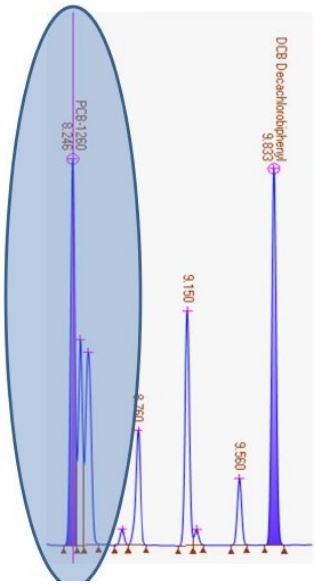
9.2.3.3. Continuing Calibration Verification (CCV) and System Performance Check:

9.2.3.3.1. On each day of analysis the System Performance Check (see Section 7.2.9) is analyzed to assist in the evaluation of possible DDT interferences with the last major peak in Aroclor 1254.

9.2.3.3.2. A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260.

9.2.3.3.3. If there are Aroclor hits in the associated samples then the calculated concentration of the CCV must be within ±20%D of the expected concentration on both columns. If there are no Aroclor hits in the associated samples one column may exceed the ±20%D criteria on the hight side (alternatively a low level Aroclor 1016/1260 standard may be analyzed to demonstrate adequate sensitivity. Should the foregoing criteria not be met the analyst must take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. The surrogate (DCB) must also meet CCV criteria on both columns.

9.2.3.3.4. Resolution (Aroclor 1260): The CCV level Aroclor 1016/1260 standard must meet the following resolution critieria: Resolution (degree of overlap) for the triplet towards the end of the 1260 chromatogram must be <75% on <u>one</u> of the two columns used. The resolution requirement must be met for peak 1/2 and peak 2/3:



The circled triplet of peaks is observed towards the end of the 1260 pattern on columns such as CLP 1. Minimum resolution (degree of overlap) requirement between peak 1 / 2 and peak 2 / 3 is <75%. This chromatogram shows overlap of about 50% between peak 2 and 3, and 30% between peak 1 and 2.

Resolution (degree of overlap) is calculated as

[Height of the valley / (Sum of the two peak heights / 2)] x 100%

The acceptance criteria for the Initial Calibration Range, the ICV and the CCV are detailed in the table below.

Step	Standards	Type	Control Limit	Frequency
Method # 8082A				
Initial Calibration Range	50, 500, 1000, 1500 and 2500 ppb for Aroclor 1016/1260, 1000 ppb for all remaining Aroclors	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r^2 ≥0.990*	*Initially and as required when ICV or CCV do not meet requirements*
ICV	*1000 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*1000 ppb*	*Average*	*± 20%D (see Section 9.2.3.4.2 for exceptions); Acceptable Resolution*	*Every 20 samples*
System Performance Standard			*For evaluation of DDT intereference with Ar1254*	*Once daily*

10. Procedure

10.1. Gas Chromatograph (GC) Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Instrument Operating Conditions:

10.1.2.1. **Injection System**: A splitless injection port with electronic pressure control (EPC) is used. Seventy-five seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the pressure Ramp mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
25 psi	0.50 min	20psi/min	15 psi	2.00 min
		8 psi/min	12 psi	6.60 min
		10.0 min	16 psi	2.00 min

10.1.2.3. For PCB analysis the normal operating conditions of the injection port are as follows:

Injection Temperature:	250^{o}C
Injection Port Pressure: Column flow:	25ml/min 33.2 ml/minute
Split vent flow:	60.0 ml/minute
Purge vent flow:	1.2 ml/minute
EPC:	Ramp pressure mode

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. This liner/glass wool combination provides many functions.

10.1.2.5. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.6.1. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.6.2. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all

column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.6.3. The septa should be changed each time the injection port is opened. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column.

10.1.2.6.4. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.7. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.7.1. The oven program and pressure ramping for PCB analysis is employed for all columns as follows:

Initial Temp	Hold Time 1	Rate1	Temp1
164°C	0.0min	12°/min	234°C
Hold Time2	**Rate2**	**FinalTemp**	**FinalTime**
2.4 min	40°/min	325°C	1.5min

10.1.2.8. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.9. Chemstation: The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.10. Eurofins Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to Eurofins LIMS (TALS) which is used to generate all reports.

10.2. Analytical Sequence

10.2.1. The instrument operating conditions should be set as detailed in Section 10.1.

10.2.2. Once instruments conditions have been established, the Initial Calibration Range, calibration verifications and retention time windows must be established Section 9.2.

10.2.3. The analytical sequence is established via the "SEQUENCE" macro of the Chemstation data system. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the calibration and/or calibration verification standards, evaluate the instrument status, and, finally, (if all meet criteria) complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro

10.2.4. An idealized analytical sequence including an Initial Calibration Range is presented in the table below.

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Idealized Analytical Sequence with Initial Calibration Range	
Injection Number	**Identification**
1	Hexane
2	Instrument Blank
3	Aroclor-1660 Level 1 Cal Std (50 ppb)
4	Aroclor-1660 Level 2 Cal Std (500 ppb)
5	Aroclor-1660 Level 3 Cal Std (1000ppb)
6	Aroclor-1660 Level 4 Cal Std (1500 ppb)
7	Aroclor-1660 Level 5 Cal Std (2500 ppb)
8	Aroclor-1221 Level 3 Cal Std (1000 ppb)
9	Aroclor-1232 Level 3 Cal Std (1000 ppb)
10	Aroclor-1242 Level 3 Cal Std (1000 ppb)
11	Aroclor-1248 Level 3 Cal Std (1000 ppb)
12	Aroclor-1254 Level 3 Cal Std (1000 ppb)
13	Aroclor-1262 Level 3 Cal Std (1000 ppb)
14	Aroclor-1268 Level 3 Cal Std (1000 ppb)
15	Initial Calibration Verification (Aroclor 1660)
16	Hexane
17	Continuing Calibration Verification (Aroclor1660)
18 thru 37	Client samples and QC Samples (MS/MSD, LCS, Method Blank)
19	Continuing Calibration Verification (Aroclor1660) (*every 20 samples*)

10.2.5. After each 20 samples a CCV standard mix must be analyzed. If this standard fails the criteria listed in Section 9.2.3.4, all samples analyzed during the previous period must be re-analyzed with a passing CCV.

10.2.6. PCB Data Reporting: The Chrom data system calculates the concentrations of the selected Aroclor Peaks. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.2.7.1. The quantitative values for all confirmed analytes must agree within 40% between the primary column and the confirmation column.

10.2.7.2. If the quantitative values do not agree within 40%, the discrepancy must be noted in the report with a qualification

10.3 Dual Column Approach

NOTE: Data generated under the NJDEP DKQP requires the reporting of the higher concentration in all cases unless it can be demonstrated that interfering compounds are the cause of the higher results in which case the lower value can be reported with a narrative explanation. Other programs may also require a dual column reporting approach different than the one described below in which case the lab will report as required by that program.

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. Results are reported from the primary column unless the difference in concentration between the two columns results in ≥40% RPD in which case the lower concentration is reported (unless the client or program requirements dictate otherwise).

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*.

10.3.4. If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the

compliant column. If the recovery falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4 Extract Cleanup

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Sulfuric Acid Permanganate and/or TBA sulfite. Refer to Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision and Eurofins Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision. Blanks must also undergo cleanup following the same procedures as samples..

10.4.3. Cleanup of all soil samples is conducted using TBA sulfite and Sulfuric Acid Permanganate. Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Cleanup using Sulfuric Acid Permanganate effectively destroys the majority of organic material in the sample extract and should be used

only when PCB is the only analysis to be performed on the sample extract.

10.5 Documentation

10.5.1 Before the analysis sequence is initiated the GC Performance and Repairs logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, column A flow, column B flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2 After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor method, job number, QA number, extraction date, lab prep batch, target batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)

Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2 Solid Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml

11.3.3 Wipe Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(W)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
W	=	Wipe

The 1000 in the denominator represents the number of ul in 1 ml

11.4 Relative Response Factors

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

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5 Percent Relative Standard Deviation (% RSD):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6 Percent Difference (% D):

$$\overline{\%\ D} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7 Relative Standard Error (RSE)

$$\%\ RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

11.8 Percent Recovery (% R): Surrogates and Spikes

Concentration (or amount) found

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) added}}{\text{Concentration (or amount) added}} \times 100$$

11.9 Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL) The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification: The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.
  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This

material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624
Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, "Method 8082A, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. Eurofins Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. Eurofins Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. Eurofins Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. Eurofins Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision.

15.8. Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.9. Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.10. Eurofins Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision.

15.11. Eurofins Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.12. Eurofins Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Approach

18.0. Revision History

Revision 7, dated 21 Oct 2022

- Section 4.5 added which details possible DDT and analogs interference with the last major DDT peak.
- Section 7.2.9: Added System Performance Standard (DDT/Endrin) for use in evaluating potential interference of DDT with last major Aroclor 1254 peak.
- Section 9.2.3.3.1: Added requirement to analyze System Performance standard daily for evaluation of DDT interference with Aroclor 1254 quantitation.

Revision 6 dated 17 Oct 2022

- Updated to Eurofins branding, removed TestAmerica references
- Added Section 8.2 to include requirement to treat samples from chlorinated sources with sodium thiosulfate
- Section 9.2.3.1.4: Section 9.2.4.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
- Section 11.7: added formula for calculation of RSE.
- Section 12.2 added: details the annual LLOQ verification requirement.

Revision 5 dated 01 Apr 2020

- Updated to Eurofins branding
- Section 7.2.5: added wipe matrix
- Added Section 11.3.3 – calculation for wipe samples

Revision 4 dated 15 Dec 2016

- Section 7.2.8: clarified text to more accurately describe the PCB Internal Standard Mix.

Revision 3 dated 16 May 2016

- Section 2.1: removed SW846 3550 as a prep method for solids and added it as a prep method for wipe samples.
- Section 2.1.1 and 15.0: removed reference to SW846 Method 3520C (SOP No. ED-ORP-003: *Extraction of Semi-Volatile Organic Compounds in Water by Continuous Liquid-Liquid Extraction, SW846 Method 3520C).*
- Section 2.2: expanded to include reference to internal standard calibration.
- Section 7.1.1: revised to clarify that the primary carrier:make-up gas combo is hydrogen and nitrogen.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to the table
- Added Section 7.2.8 which describes the preparation of the internal standard solution.
- Section 9.1: added internal standard to 'Sample QC' table
- Section 9.1.2: completely re-written to clarify LCS evaluation and corrective action.
- Section 9.1.4: clarified that surrogates must pass CCV criteria in order to be reportable and must be reported from the same column as target analyte results.
- Added Section 9.1.5 which describes acceptance criteria for internal standards (retention time and response)
- Section 9.2.1.1: added following sentence for clarity and compliance with TestAmerica PCB Minimum Requirements document (WI No. CA-T-WI-003): "All other Aroclors are calibrated using a single point calibration up to 8 peaks at the anticipated midpoint of the calibration range if the 1016/1260 calibration meets the required average RF criteria." Added language detailing which Aroclor standards can be analyzed together.
- Section 9.2.1.1: added this sentence: "The initial calibration block must include at least one level with 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration)."
- Section 9.2.1.1: clarified that a minimum of five peaks are required for quantitation of each Aroclor (except for Aroclor 1221 which requires a minimum of three peaks). Added requirement that selected peaks must be at least 25% of height of largest peak in Aroclor (except Aroclor 1268 where the requirement is 10%).
- Section 9.2.2: Revised first sentence to read: "A mid-point Continuing Calibration Verification (CCV) standard must be analyzed after every 20 samples at minimum," (i.e., removed the closing CCV requirement and 12 hour requirement).
- Section 9.2.2: clarified that the CCV typically consists of a 1016/1260 standard. Also added the following sentence: "If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260."
- Section 9.2.2: added requirement to analyze a CCV for each detected Aroclor within 12 hours of detection.

- Table 2: updated with the expected RT acceptance criteria from Minimum Requirements document (0.03 minutes).
- Section 9.2.3.2: Revised 'External standard' to 'Internal Standard'. Emphasized that both columns must pass calibration criteria.
- Section 9.2.3.2.2: revised to require that 5 peaks must be selected (was 3) and evaluated for initial calibration (per corporate Minimum Requirements).
- Section 9.3.3.3.1: corrected section reference for preparation of ICV (was Section 7.2.9; now Section 7.2.7);
- Section 9.2.3.3.2: revised first sentence to read: "At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification." (it had previously required three peaks).
- Section 9.2.4.3.1: updated text reflect corporate Minimum Requirements document.
- Added Section 9.2.3.4.2: more fully describes CCV acceptance criteria and corrective actions. Updated table with calibration criteria.
- Added Section 9.2.3.4.3: details resolution requirements per the corporate Minimum Requirements document.
- Section 10.2.5: revised to remove 12 hour requirement (replaced solely with 20 sample requirement) and bracketing requirement. Corrected section reference for CCV acceptance criteria.
- Section 11: completely re-written to include internal standard result calculations and other required QC calculations.
- Section 15 (References): added following document reference: TestAmerica Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.
- Attachment 1: revised completely.

- Revision 2, dated 08 Jun 2015

  - Section 1.1: Corrected Method Limit Group (MLG) reference to 8082 (was incorrectly listed as 8081B,
  - Table 2: updated RLs for leachates from 0.0050 mg/L to 0.00050 mg/L.
  - Throughout document: updated the default initial volumes for aqueous preps (250 ml) and leachate (TCLP/SPLP/ASTM) preps (250 ml).
  - Throughout document: removed any notes referencing option for 'reduced volume' extractions since this is now standard as defined in SOP.
  - Section 2.2.2: replaced SW846 3541 (Soxtherm) prep with SW846 3546 (Microwave) prep.
  - Section 6.2.1 (and throughout document): replaced Target software references with references to TestAmerica's Chrom chromatography data processing software.
  - Section 7.2.2 and throughout document: updated source of standards from Supelco to Restek.
  - Section 7.2.3 and throughout document revised concentration of low calibration standard to 50 ppb.
  - Section 7.29: updated ICV prep instructions using Restek standards.
  - Section 8: updated sample container from 1000ml to 250ml; updated minimum sample size to 250ml.
  - Section 10.3: added note explaining NJDEP DKQP requirement to report the higher concentration in all cases.

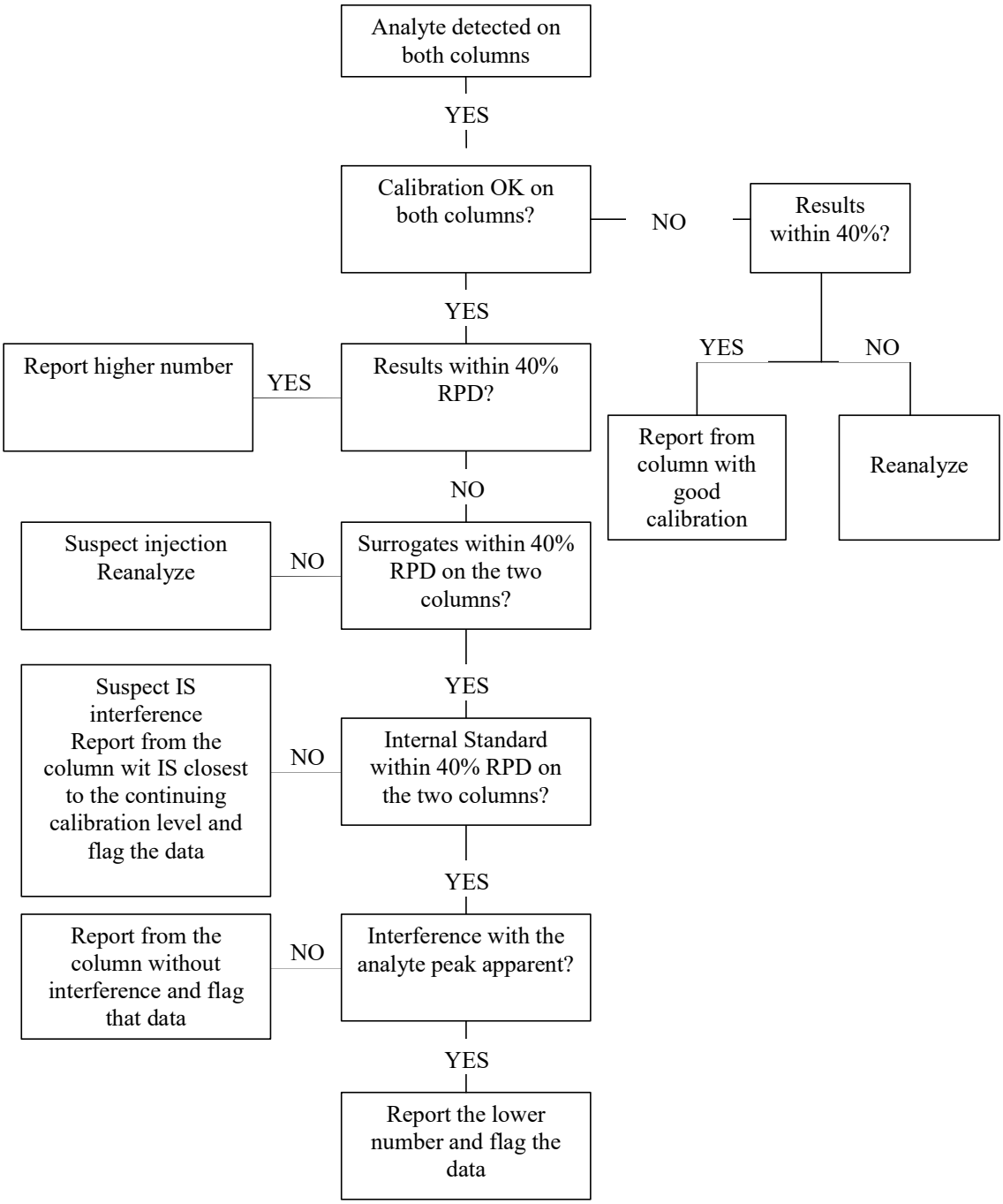
- Section 10.3 and Attachment 1: revised to reflect current TestAmerica dual column reporting rules.
- Section 15: removed outdated references.

- Revision1, dated 11 October 2012

  - Throughout document: Revised LQM section references to reflect the most current LQM revision
  - Revised Table 2 to reflect RLs for reduced initial/final volume prep option.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) extraction option.
  - Section 2.1.2 and Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
  - Section 7.2.3: added procedure for prep and analysis of a lower level Aroclor 1016/1260 standard (50 ppb) and for the surrogate DCB (12.5 ppb) when analyzing samples prepped using the reduced initial/final volume procedure.
  - Section 9.2.3.4.1: Added reference to 50 ppb 1016/1260 ICAL standard for the reduced initial/final volume method.
  - Section 10.2.4: Added 50 ppb 1016/1260 ICAL standard to the sequence for the reduced initial/final volume method

- Revision 0, dated 02/16/2011: NEW

Attachment 1
Dual Column Approach


Analyte detected on both columns
YES
Calibration OK on both columns?
NO
Results within 40%?
YES
NO
YES
Report higher number
YES
Results within 40% RPD?
Report from column with good calibration
Reanalyze
NO
Suspect injection Reanalyze
NO
Surrogates within 40% RPD on the two columns?
YES
Suspect IS interference Report from the column wit IS closest to the continuing calibration level and flag the data
NO
Internal Standard within 40% RPD on the two columns?
YES
Report from the column without interference and flag that data
NO
Interference with the analyte peak apparent?
YES
Report the lower number and flag the data

Title: SW846 Method SW8081B, Analysis of Organochlorine Pesticides by Gas Chromatography

Approvals (Signature/Date):

Catalina Dalangin SVOA GC Manager	10/17/2022 Date	Dan Helfrich Health & Safety Manager	10/17/2022 Date
Carl Armbruster Quality Assurance Manager	10/17/2022 Date	Mark Acierno Laboratory Director	10/17/2022 Date
Sylvanus Klusey Operations Manager - Organics	10/17/2022 Date		

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using dual fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). The list of analytes and their corresponding reporting limits are as follows:

Parameter		Soil	Water	Leachate
	CAS Registry No.	Reporting Limits (ug/Kg)	Reporting Limits (ug/L)	Reporting Limits (mg/L)
Aldrin	309-00-2	6.7	0.02	---
Alpha-BHC	319-84-6	2.0	0.02	---
Beta-BHC	319-85-7	2.0	0.02	---
Delta-BHC	319-86-8	2.0	0.02	---
Gamma-BHC (Lindane)	58-89-9	6.7	0.02	0.00050
Chlordane	57-74-9	67	0.50	0.0050
4,4' –DDD	72-54-8	6.7	0.02	---
4,4' –DDE	72-55-9	6.7	0.02	---
4.4' –DDT	50-29-3	6.7	0.02	---
Dieldrin	60-57-1	2.0	0.02	---
Endosulfan I	959-98-8	6.7	0.02	---
Endosulfan II	33213-65-9	6.7	0.02	---
Endosulfan sulfate	1031-07-8	6.7	0.02	---
Endrin	72-20-8	6.7	0.02	0.00050
Endrin aldehyde	7421-93-4	6.7	0.02	---
Endrin ketone	53494-70-5	6.7	0.02	---
Heptachlor	76-44-8	6.7	0.02	0.00050
Heptachlor epoxide	1024-57-3	6.7	0.02	0.00050
Methoxychlor	72-42-5	6.7	0.02	0.00050
Toxaphene	8001-35-2	67	.50	0.0050
Gamma- Chlordane	5103-74-2	6.7	0.02	---
Alpha-Chlordane	5103-71-9	6.7	0.02	---
Mirex	2385-85-5	6.7	0.010	---

The most current MDLs and RLs for this method can be found in the active Eurofins LIMS (TALS) SW846 8081B Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8081B. A measured volume or weight of sample (15g for soil, 1 g for waste, 250 ml for water, and 250 ml for TCLP) is extracted using the appropriate matrix-specific sample extraction technique. (Reference the applicable Organic Sample Prep SOPs listed below). The effective final volume is usually between 5 and 20 ml in hexane.

2.1.1. Aqueous samples are extracted using SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel*).

2.1.2. Solid samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction*) or SW846 Method 3546: Microwave (SOP No. ED-ORP-044: *Procedure for the Microwave Extraction of Solids,SW846 3546).*

2.1.3. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs*).

2.1.4. Extract cleanup steps are employed depending on the nature of the matrix interferences. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*) and SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*).

2.2. After cleanup, a small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to an Electron Capture Detector (ECD) which is used to detect the compounds eluting from the GC.. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8081B.

2.3. For pesticide analysis a system performance check (DDT/Endrin breakdown) and a calibration verification standard must be run prior to analysis. Failure of either will generally indicate the need for injection port/column maintenance and/or recalibration.

2.4. Samples are analyzed after all the necessary checks have been performed. Samples analyzed for pesticides require an additional post analysis Quant Report to be printed and attached to the chromatographic report.

2.5. All samples are then manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. If sulfur is encountered, employ the sulfur removal procedures detailed in SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*). Note that the recover of Endrin Aldehyde is adversely affected by the TBA cleanup procedure detailed in this method. Accordingly, this compound must be determined prior to sulfur cleanup.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*).

4.3.1. Check Florisil prior to use to assure quantitative recovery of targeted analytes. Duplicate checks are required for each new lot or every three hundred samples whichever is more frequent.

4.3.2. Check Florisil by spiking 1ml of the Pest Std Mix A midpoint (Supelco Catalog No. 47977) and 0.5 ml of trichlorophenol (Absolute Standardsl Catalog No. 20024) onto the cartridge and concentrating to final volume of 1 ml. Inject 1 ul onto a capillary column, conducting the elution and analyzing the extract. Recovery is acceptable if all pesticides are recovered at 80 - 110% and the recovery of trichlorophenol is <5% and co-eluting interfering peaks are absent from the extract.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. Gas Chromatograph: The system used is an HP and an Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC). Each GC is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles samples between the sample tray and the injector turret.

6.1.2.1. The samples are injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.2.2. Liners: The injection port is each fitted with replaceable, heavy-walled siltek-coated glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.2.3. Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.2.3.1. Two dissimilar columns are used for analysis. A Restek RtxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column is used for sample quantitation. The secondary confirmation column is a Restek RtxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column.

6.1.2.4. Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.2.4.1. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas (P-5 & Nitrogen) is fed from a supply other than the injection port.

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. P-5 and Nitrogen) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. Hydrogen is supplied via a Parker Balston H2 Generator. Nitrogen & P-5 is supplied by Air Gas

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction and cleanup procedures include n-hexane, methylene chloride, and acetone that are exchanged to n-hexane prior to analysis.

7.1.3. Hexane is required in this procedure. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2).

NOTE: Independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.2.2.1.

7.2.2. Standard mixes and sources *.

Standard Name	Source
Organochlorine Pesticide Mix AB #3	Restek Catalog No. 32415
Organochlorine Pesticide Mix AB #3.sec	Restek Catalog No. 32415.sec (second source)
Pesticide Surrogate Mix	Restek Catalog No. 32000
Endrin/DDT	Supelco Catalog No 48282
Chlordane	Restek Catalog No. 32021
Toxaphene	Restek Catalog No. 32005
Toxaphene (different pattern)	Restek Catalog No. 32071
Mirex	Accustandard Cat. No. P-066S-10X
1-Bromo-2-nitrobenzene (internal standard)	Restek Cat. No. 32279

*Suppliers with equivalent standards may be substituted..

The components of each standard mix are as follows (note: 'sec' indicates second source):

Parameter	Supplier	Catalog No.	Concentration of Standard (ug/ml)
Aldrin	Restek	32415 & 32415.sec	2000
Alpha-BHC	Restek	32415 & 32415.sec	2000
Beta-BHC	Restek	32415 & 32415.sec	2000
Delta-BHC	Restek	32415 & 32415.sec	2000
Gamma-BHC (Lindane)	Restek	32415 & 32415.sec	2000
Alpha -Chlordane	Restek	32415 & 32415.sec	2000
Gamma -Chlordane	Restek	32415 & 32415.sec	2000
Technical Chlordane	Restek	32021	1000
4,4' –DDD	Restek	32415 & 32415.sec	2000
4,4' –DDE	Restek	32415 & 32415.sec	2000
4.4' –DDT	Restek	32415 & 32415.sec	2000
Dieldrin	Restek	32415 & 32415.sec	2000
Endosulfan I	Restek	32415 & 32415.sec	2000
Endosulfan II	Restek	32415 & 32415.sec	2000
Endosulfan sulfate	Restek	32415 & 32415.sec	2000
Endrin	Restek	32415 & 32415.sec	2000
Endrin aldehyde	Restek	32415 & 32415.sec	2000
Endrin ketone	Restek	32415 & 32415.sec	2000
Heptachlor	Restek	32415 & 32415.sec	2000
Heptachlor epoxide	Restek	32415 & 32415.sec	2000
Methoxychlor	Restek	32415 & 32415.sec	2000
Toxaphene	Restek	32005	1000
Toxaphene	Restek	32071	5000
Mirex	Accustandard	861428-U	1000
4,4-DDT	Supelco	18282	500
Endrin	Supelco	48282	500
Decachlorobiphenyl (DCB)	Restek	32000	200
Tetrachloro-m-xylene (TCmX)	Restek	32000	200
1-Bromo-2-nitrobenzene (internal standard)	Restek	32279	1000

7.2.2.1. Standards Preparation

7.2.2.1.1. Calibration Mix (Organochlorine Pesticide Mix)

The 5 point calibration standards are prepared as detailed in the following table using volumetric glassware and hexane as the diluent:

Initial Calibration Standards Prep (Organochlorine Pesticide Mix)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Organochlorine Mix AB#3 ***(2000 ug/ml)*** *Restek (Cat No..32415)in Hexane:Toluene*	Volume brought up to 200 ml hexane: *0.25 ul*	Volume brought up to 200 ml hexane: *5.0 ul*	Volume brought up to 500 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *50 ul*
Initial Calibration Standards Prep (TCMX/DCB Surrogate Mix)					
Stock Std	**25 ppb***	**50 ppb**	**100 ppb**	**150 ppb**	**200 ppb**
Tetrachloro-m-xylene/ Decachlorobiphenyl Surrogates Mix (200 ug/ml) *Restek (Cat, No, 32000) in Acetone*	Volume brought up to 200 ml hexane: *6.25 ul*	Volume brought up to 200 ml hexane: *50 ul*	Volume brought up to 500 ml hexane: *250 ul*	Volume brought up to 200 ml hexane: *150 ul*	Volume brought up to 200 ml hexane: *200 ul*
Initial Calibration Standards Prep (Mirex)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Mirex ***(1000 ug/ml)*** *Accustandard (Cat. No. P-066S-10x) in Methanol*	20x dilution of 50 ppb	10x dilution of 500 ppb	5x dilution of 500 ppb	2x dilution of 500 ppb	Volume brought up to 100 ml hexane: *50 ul*

Note: 20 ul of 5 ug/ml Internal Standard solution is added to all calibration standards prior to analysis.

7.2.2.2. **Pesticide Surrogate Spike Mix (10 ug/ml**) : The Pesticide 10ug/ml surrogate spiking solution is prepared by diluting 10 ml of 200 ug/ml of the Pesticide Surrogate Mix (Restek-32000) in to 200 ml of Acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.3. **Pesticide Surrogate Spike Mix (2 ug/ml) for the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described in 7.2.2.2 to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.4. **Pesticide Internal Standard Spike Mix (5 ug/ml):** The Pesticide 5 ug/ml internal standard spike mix is prepared by

dilution 1 ml of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 200 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.2.5. **Pesticide Spiking Standard (20 ug/ml):** The Pesticide Spiking Mix containing the single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul of the Organochlorine Pesticide Mix AB#3 (Restek 32415 to a 50 ml final volume with acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.6. **Pesticide Spiking Standard (4 ug/ml) for the reduced volume extraction option**: prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared in Section 7.2.2.4 above to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.7. **System Performance Solution (Breakdown Check)** 3,3-DDT and Endrin at 0.25 ug/ml): The breakdown check is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane

7.2.2.8. **Technical Chlordane Calibration Solution** (1.0 ug/ml solution w/ surrogates TCMX & DCB at 0.10 ug/ml): 100 ul of 1000 ug/ml Technical Chlordane Standard (Restek Catalog No 32021) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. ***NOTE:*** ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.9. **Toxaphene Calibration Solution** (1.0 ug/ml solution w/surrogates TCMX & DCB at 0.1 ug/ml) : 20 ul of 5000 ug/ml Toxaphene stock (Restek Catalog No 32071) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. ***NOTE:*** ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.10. **Initial Calibration Verification (ICV)** Preparation: follow the instructions above for midpoint standards substituting the second source standard (catalog no. suffix = sec) for the primary standards.

8. Sample Collection, Preservation, Shipment and Storage

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.2. Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2ºC	14 days to extraction; Analyze within 40 days of extraction	SW846

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

9. Quality Control

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

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard [1]	Every sample[3]	Response within -50% to +100% of most recent cal standard

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

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall

below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The recoveries of the LCS must fall within lab generated acceptance criteria (refer to the current TALS Method Limit Group database). If the LCS recovery results are outside of these limits, the extract is reanalyzed. If LCS recoveries are still outside of QC limits after extract reanalysis but recoveries for the Matrix Spike/ Matrix spike Duplicate (MS/MSD) are within QC limits, the data is reported and a Non Conformance Memo (NCM) is written.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database*)*. Minimum requirements for surrogate evaluation:

- Both surrogates must have reportable results that meet the acceptance criteria;
- Reported surrogates must be from a column with a passing CCV;
- At least one surrogate must pass on any column from which target analytes are identified and reported.

If both TCMX and DCB recovery are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzene) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.1.6. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification: The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to

demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

9.2. Instrument QC

9.2.1. GC System Performance Check

9.2.1.1. **Endrin/4,4'-DDT Breakdown:** Prior to performing any standards or sample analysis, a daily check is made on the chromatographic performance of the system. This performance check is made by injecting a standard of Endrin and DDT, each at a 250-ppb level (see Section 7.2), and calculating the percentage breakdown for each compound.

9.2.1.2. Ideally, only two peaks will be seen (one for Endrin and one for DDT). As a rule, this is not the case. It is normal to observe up to six peaks. Three peaks are attributable to Endrin and its degradation products: Endrin Aldehyde (EA) and Endrin Ketone (EK). Three peaks are attributable to DDT and its degradation products: DDE and DDD. Calculate the percentage breakdown as follows:

Endrin:

$$\frac{\text{(Areas of EA + EK)}}{\text{(Areas of EA + EK + Endrin)}} \times 100 = \%\ \text{breakdown Endrin}$$

DDT:

$$\frac{\text{(Areas of DDE + DDD)}}{\text{(Areas of DDE + DDD + DDT)}} \times 100 = \%\ \text{breakdown DDT}$$

9.2.1.3. If the percentage breakdown for either Endrin or DDT is greater than 15%, the system CANNOT be used for pesticide analysis. If the Endrin/DDT performance check fails, injection port/column maintenance must be performed. Usually, changing the glass wool/liner will cure most breakdown problems in the injection port. Depending upon the nature of the samples, the entire injection port will occasionally need to be cleaned. This cleaning is best done with 1:1 Acetone: Hexane. Another routine maintenance operation to improve column performance is the removal of the

first 3 cm of the column. (Note: the septa should be changed each time the injection port is opened).

9.2.1.4. After injection port/column maintenance has been performed, and the columns have been given time to equilibrate (baseline back down to normal) the Endrin/DDT must be re-injected and the system performance re-evaluated.

9.2.2. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.2.1. ***Initial Calibration Range*:** Single component pesticides are calibrated using a five-point calibration range. Multi-component pesticides are calibrated using a single point calibration at the anticipated midpoint of the calibration range. Standards are prepared following the instructions in Section 7.2.

9.2.2.2. Single response Pesticide Calibration: All single component pesticides and two surrogates are calibrated with a minimum of 5 concentrations. Single component pesticides are analyzed at 10, (5 ppb for the 125 ml initial volume method) 5, 100, 250 and 500 ppb. Surrogate standards are analyzed at 25, 50, 100, 150 and 200 ppb. See Section 7.2 for details on standard prep.

9.2.2.3. Multi-response Pesticide Calibration: Chlordane (technical) and Toxaphene initial calibration is accomplished by analysis of a single point at 1000 ppb (see Section 7.2 for details on standard prep).

9.2.2.4. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3. Continuing Calibration Verification (CCV): For single component pesticides, a mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent). 'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… For multi-response pesticides a CCV must be analyzed within 12 hours of any multi-response pesticide detects.

9.2.3.1 Analysis of Replicate CCVs: Occasionally dual, sequential CCVs may (for a variety of reasons) be included in an analytical sequence. When such replicate CCVs are injected both must be evaluated as detailed in the table below (reference Eurofins Document CA-Q-W-008, 'Technical Guidance on the Use and

Evaluation of Replicate Continuing Calibration Verification (CCV)').

Dual CCV Evaluation Decision Tree		
Inejection	**QC Acceptance**	**Action**
CCV1 **CCV2**	**Pass** **Pass**	Continue analytical sequence. Data acceptable based on calibration.
CCV1 **CCV2**	**Pass** **Fail**	Analyses before CCV1 may be accepted. Re-analyze all samples that were analyzed after failed CCV2.
CCV1 **CCV2**	**Fail** **Pass**	Re-analyzed all samples that were analyzed before CCV1 and after the previous compliant CCV. Analyses after CCV2 may continue.
CCV1 **CCV2**	**Fail** **Fail**	Re-analyze all samples since the last acceptable CCV. Perform maintenance and/or recalibration prior to re-analysis.

9.2.3.2 In the event that replicate CCVs are included in a sample injection sequence, the acceptance of the associated sample analyses must be properly evaluated and the evaluation process must be documented. Under no circumstances is it allowable to accept sample data based on the evaluation of only 1 of the CCV replicates.

9.2.3.3 Clear documentation of the evaluation of the CCV must be included in the analytical sequence run log (i.e., 'Pass' or 'Fail' in the comment section for each CCV). Additionally, clear documentation of corrective action taken in the event of CCV failures must be included in the run log (see table above for actions that need documentation).

Note: It is acceptable to use the CCV solution as a primer at the beginning of an analytical sequence however the injection should be documented as a 'Primer' in the sequence rather than as a CCV.

9.2.4. Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability (for more detail on the following procedures refer to

Eurofins Corporate Technical Services Policy No. CA-T-P-005, “Policy for Determining RT Windows for GC/ECD Tests” and Eurofins Corporate Technical Services Work Instruction No. CA-T-WI-010, “Further Guidance on the RT Window Policy No. CA-T-P-005”).

9.2.4.1.1. Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.4.1.1.2. Use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCSs in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

9.2.4.1.1.3. Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 minutes, in which case the window is set at 0.01 minutes. For example, if the maximum RT deviation for a specific analyte is 0.008 minutes, then the RT window is set at ±0.012 minutes.

NOTE: For the multi-component analytes, for example Aroclors, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations.

9.2.4.1.1.4. Retention time windows for analytes of interest must not overlap for GC analysis.

9.2.4.1.2. Ongoing evaluation of retention time windows

9.2.4.1.2.1. Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

9.2.4.1.2.2. Matrix spike analytes may fall outside of the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike.

9.2.4.1.2.3. If any analytes fall outside of the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

9.2.4.1.2.4. Retention time windows should be reliably narrower than ±0.03 minutes. If RT windows wider that this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. A response factor is calculated for each analyte at each calibration concentration.

Response factor = $((A_x)\ (C_{is}))/((A_{is})\ (C_x))$

Where:
A_x = area of the compound
C_x = Concentration of the compound
A_{is} = area of the internal standard
C_{is} = Concentration of the internal standard

9.2.4.2.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

Where:
RF = Response Factor

9.2.4.2.2. If the % RSD across the 5 point range is <20% for any given compound the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.4.2.3. If the % RSD is >20% for any given compound, a first order linear regression may be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.990 for the calibration to be acceptable. Calibration is checked every

12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.4.2.4. Chlordane and Toxaphene Calibration: Chlordane and Toxaphene are multiple response pesticides and are calibrated with a minimum of 5 points as required (i.e, within 12 hours of either analyte being detected in a sample). Three to eight peaks are used for calculation of response factors and the same criteria detailed above is applied to determine acceptability of calibration.

9.2.4.2.5. Resolution: All single component analyte peaks must exhibit at least 80% chromatographic resolution. The analyst performs a visual check of the mid-level initial calibration standard and all subsequent calibration checks (ICV/CCV). If the resolution requirement is not met instrument maintenance should be performed followed by re-calibration. Percent resolution can be calculated when necessary as follows:

$$\% \text{ Resolution} = V/H \times 100$$

Where:

V= Depth of the valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks

H = Height of the shorter of the adjacent peaks

9.2.4.3. **Relative Standard Error:** The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r_2) for

testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.4.4. Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.2.4. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5)

9.2.4.5. Continuing Calibration Verification (CCV): A CCV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.3.. The calculated concentration of the CCV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5).

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Method # 8081B				
GC System Performance Check	*Endrin/DDT, 250 ppb*	*Performance check*	*15% breakdown*	*At beginning of 12 hour clock and after system maintenance*
*Initial Calibration**	2.5, 50, 100, 250 and 500 ppb for single response, 1000 ppb for multi response	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r ≥0.990 or <20%RSE*	*As required when ICV or CCV do not meet requirements*
ICV	*100 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*100 ppb*	*Average*	*± 20%D*	*Every 12 hrs or 20 samples, whichever is more frequent*

10. Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Operating Conditions

10.1.2.1. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the ramp pressure mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
12 psi	2.5 min	7 psi/min	4 psi	1.50 min
		5 psi/min	9 psi	1.40 min
		9 psi/min	13 psi	2.00 min

10.1.2.3. For pesticide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	250^{o}C
Column flow:	12.3 ml/minute
Split vent flow:	5 ml/minute
EPC:	Pressure Ramp
Detector temperature	330C

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.5. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. The glass wool will be changed when changing the liner. The changing of the glass wool/liner is based upon the breakdown of

an Endrin/DDT standard. This is covered in further detail in section 10.2.1.

10.1.2.7. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.8. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.9. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.10. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.11. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.11.1. A standard oven program for pesticide analysis is employed for all columns as follows:

Initial Temp	**Hold Time1**	**Rate1**	**Temp1**
160°C	0.62 min	30°/min	244°C
Hold Time2	**Rate 2**	**Final Temp**	**Final Time**
2.5min	21°/min	315°C	3.0min

10.1.2.12. Detectors: Detectors operate at 330°C and need to be supplied with 60 ml/min total flow. They are essentially maintenance free

on a day-to-day basis. They are routinely baked out at 330°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).

10.1.2.12.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.13. Chemstation: HP Chemstation software is used for automation of runs and data acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.14. Eurofins Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to Eurofins LIMS (TALS) which is used to generate all reports..

10.2. Analytical Sequence

10.2.1. The analytical sequence for performance checks, initial calibration, calibration verifications and sample analysis is described in the following sections.

10.2.2. Before calibration standards are analyzed the GC Performance Check Standard (see Section 9.2.2.1) must first be analyzed and evaluated to check the performance of the injection port and column with regard to catalytic active sites. The breakdown for both Endrin and DDT in the Performance Check Standard must be less than 15%. If the performance check fails this criteria system maintenance must be performed and the check successfully reanalyzed before proceeding with calibration.

10.2.3. A five point initial calibration (ICAL) is analyzed and evaluated for each of the 21 single response pesticides plus surrogates as described in Section 9.2.4.2. When needed a 5 point ICAL is also analyzed for Mirex.

10.2.4. Calibration for Technical Chlordane and Toxaphene: Chlordane and Toxaphene are multiple response pesticides containing at least 3-8 primary peaks each. A single point calibration check standard at a concentration of

1000ug/l is analyzed for Chlordane and Toxaphene. A full 5 point calibration range is analyzed for these compounds should they be detected in client samples..

10.2.5. A second source initial calibration verification (ICV) is analyzed and evaluated for each of the 21 single response pesticides as described in Section 9.2.4.3. When needed an ICV is also analyzed for Mirex. **NOTE**: The 12 hour time clock for Pesticides commences with the injection of the first Pesticide Calibration Standard or Verification.

10.2.6. GC Performance Check Standard is analyzed and the breakdown of Endrin/DDT is measured again before samples are analyzed and at the beginning of each subsequent 12 hour shift. If the breakdown check fails, then injection port/column maintenance is required

10.2.7. Client samples and QC samples may be analyzed after the analysis of the performance check. Sample analysis may proceed for up to 20 samples or 12 hours prior to analysis of another calibration verification (whichever is more frequent).

10.2.8. A Continuing Calibration Verification (CCV) for the 21 single response pesticides plus surrogates must be analyzed and evaluated every 12 hours or 20 samples (whichever is more frequent) as described in Section 9.2.4.4.. This is accomplished by running the midpoint calibration standard as a CCV (Pest Mix 100 ppb check standard and Mirex 100 ppb check standard; see Section 7.2.2.1). The calculated concentration for each compound in the CCV must be +/- 20 % of the expected concentration. Any samples analyzed after a failing CCV must be reanalyzed under a passing CCV. If, after performing instrument maintenance, the reanalysis of a CCV fails criteria, a new initial calibration range must be analyzed (see Section 9.2.4.2). Any data reported against a failing CCV must have a Non-Conformance Memo detailing the issue.

10.2.9. Analytical Sequence: The automation of GC runs is accomplished via the "SEQUENCE" macro of the Chemstation. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the check standards, evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

Example Analytical Sequence	
1. Hexane	12. Mirex 1 (10 ppb) (if needed)
2. Instrument Blank	13. Mirex 2 (20 ppb) (if needed)
3. Endrin/DDT Breakdown	14. Mirex 3 (100 ppb) (if needed)
4. Pesticide Mix 1 (2.5 ppb)	15. Mirex 4 (250 ppb) (if needed)
5. Pesticide Mix 2 (50 ppb)	16. Mirex 5 (500 ppb) (if needed)

Example Analytical Sequence	
6. Pesticide Mix 3 (100 ppb) 7. Pesticide Mix 4 (250 ppb) 8. Pesticide Mix 5 (500 ppb) 9. Chlordane (1000 ppb) 10. Toxaphene (1000 ppb) 11. Pesticide ICV (100 ppb)	17. Mirex ICV (100 ppb) (if needed) 18. Instrument Blank 19. Endrin/DDT Breakdown 20. Pest Mix 3 (100 ppb)…CCV 21. Mirex 3 (100 ppb) …CCV (if needed) 22. 20 or fewer samples or 12 hours 23. Instrument Blank 24. Endrin/DDT Breakdown 25. Pest Mix 3 (100 ppb)…CCV 26. Mirex 3 (100 ppb) …CCV (if needed) 27.20 or fewer samples or 12 hours

10.3. Dual Column Approach

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows. Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*. **Exception:** NJDEP DKQP protocols require reporting the higher concentration in all instances.

10.3.4. If the surrogates on one column are >40% RPD compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on

both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4. Extract Cleanups

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Florisil (Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision) and TBA sulfite (Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.3. Cleanup of all soil samples is conducted using Florisil (Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision) and, if needed, TBA sulfite (Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Check Florisil prior to use to assure quantitative recovery of target analytes. (see Section 4.3.2 above and Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B,

10.5. Data Processing and Documentation

10.5.1. Before the analysis sequence is initiated the GC Maintenance logbook must be filled out. It should contain the following information: date, injector temp,

oven temp, detector temp, injector flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job number, QA number, extraction date, lab prep batch, Chrom batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

10.5.3. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and %moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.5.4. Any compound concentration that exceeds the concentration of the calibration range must be diluted and re-analyzed.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.1.2 Solid Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

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

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

11.7. Percent Difference (% D):

$$\%\ D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.8. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.9. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification:

The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution

prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710

Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method SW8000C: Determinative Chromatographic Separations,”, Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, “Method 8081B, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. Eurofins Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. Eurofins Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. Eurofins Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. Eurofins Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision.

15.8. Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.9. Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.10. Eurofins Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.11. Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), most current revision.

15.12. Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), most current revision.

15.13. Eurofins Document CA-Q-W-008, ‘Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version.

15.14. Eurofins Corporate Quality SOP No. CA-Q-P-004, “Reporting Results for Methods that Require Second-Column Confirmation”, most current revision.

15.15. Eurofins Corporate Work Instruction No. CA-T-WI-008, “Minimum Requirements for Pesticide Analysis by SW846 Method 8081B”, most current revision.

15.16. Eurofins Corporate Policy CA-T-P-004, "Policy for Determining RT Windows for GC/ECD Tests", most current revision.

15.17. Eurofins Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005", most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Reporting Flowchart

18.0. Revision History

Revision 7, dated 10/172022:

- Updated to Eurofins branding throughout document.
- Revised Section 8 to include requirement to treat samples from chlorinated sources with sodium thiosulfate
- Section 9.2.4.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
- Section 11.6: added formula for calculation of RSE.
- Section 12.2 added: details the annual LLOQ verification requirement.

Revision 6, dated 03/25/2021:

- Added Section 10.5.3 which details how the reporting limit is established.
- Added Section 10.5.4 which discusses requirement to dilute analytes into range of calibration.

Revision 5, dated 10/12/2020:

- Edited throughout to reflect Eurofins branding and policy.
- Updated Lab Quality Manual section references as needed.

Revision 4, dated 09/13/2016:

- Section 9.2.4.1: completely rewrote this section to reflect requirements of Eurofins Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010
- Section 15 (References): added references to TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD

Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005".
- Attachment 1: RT Windows for Single Analytes/Surrogates: DELETED and renamed subsequent attachments.

Revision 3, dated 01/05/2016:

- Section 2.2: expanded to include summary of internal standard calibration.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to table.
- Added Section 7.2.2.4 which describes the preparation of the internal standard solution. All subsequent sections renumbered accordingly.
- Section 7.2.2.1.1: added footnote to ICAL standard prep table detailing requirement to spike each standard with internal standard solution.
- Section 7.2.2.4: describe internal standard spiking protocol (20 ul of 5.0 ug/ml solution into all standards, QC extracts and field sample extracts.
- Section 9.1: added internal standard to 'Sample QC' table.
- Section 9.1.4: added following minimum requirements for surrogate evaluation and reporting: a) Both surrogates must have reportable results that meet the acceptance criteria; b)reported surrogates must be from a column with a passing CCV; c)At least one surrogate must pass on any column from which target analytes are identified and reported. (per corp "Minimum Requirements" document).
- Section 9.1.5: added this section which describes acceptance criteria for internal standards (retention time and response).
- Section 9.2.3: Added the following sentence: "'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… ". Deleted the phrase 'and at the end of every sequence'. (both changes reflect 'Minimum Requirements for Pesticide Analysis' document).
- Added Section 9.2.4.1.3: "Updating absolute retention times: Update retention times with the retention times found in the opening CCV for that 12 hour period."
- Section 9.2.4.2: replaced 'external standard' with 'internal standard'. Added formula for calculation of response factors by internal standard method. Deleted text regarding calibration acceptance criteria from this section as it is duplicated in subsequent sections.
- Section 9.2.4.2.2: added phrase 'for any given compound.'
- Added section 9.2.4.2.5: describes resolution check.
- Sections 9.2.4.3 and 9.2.4.4: added statement concerning 80% resolution requirement.
- Section 9.2.4.4: changed 'ICV' to 'CCV' in last sentence.
- Section 10.1.2.13: Revised first sentence to read: ": HP Chemstation software is used for automation of runs and data acquisition."
- Section 10.2.2: corrected reference to Section 9.2.2.1 (was incorrectly listed as 7.2.2.1).
- Section 10.2.8: revised to clarify that CCV bracketing is not required and that only samples analyzed after a failing CCV must be reanalyzed (i.e., samples analyzed immediately prior to a failing CCV need not be reanalyzed).
- Section 10.3.2: added the following regarding updating retention times: "Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period."

- Section 11: added formulas for calculation of sample concentrations as well as %RSD, %D, % Recovery, RRF and dry weight correction.
- Section 15: removed reference to SOP No. ED-ORP-016*: Automated Soxhlet Extraction of Solid Samples – Pesticides/PCBs, SW846 Method 3541* as it is no longer is use.
- Section 15: added reference to TestAmerica Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation"
- Section 15: added reference to TestAmerica Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B".
- Section 15: added reference to TestAmerica Corporate Policy Memorandum No. CA-Q-QM-006, "Technical Guidelines for Analysis of Complex GC/ECD Chromatograms".

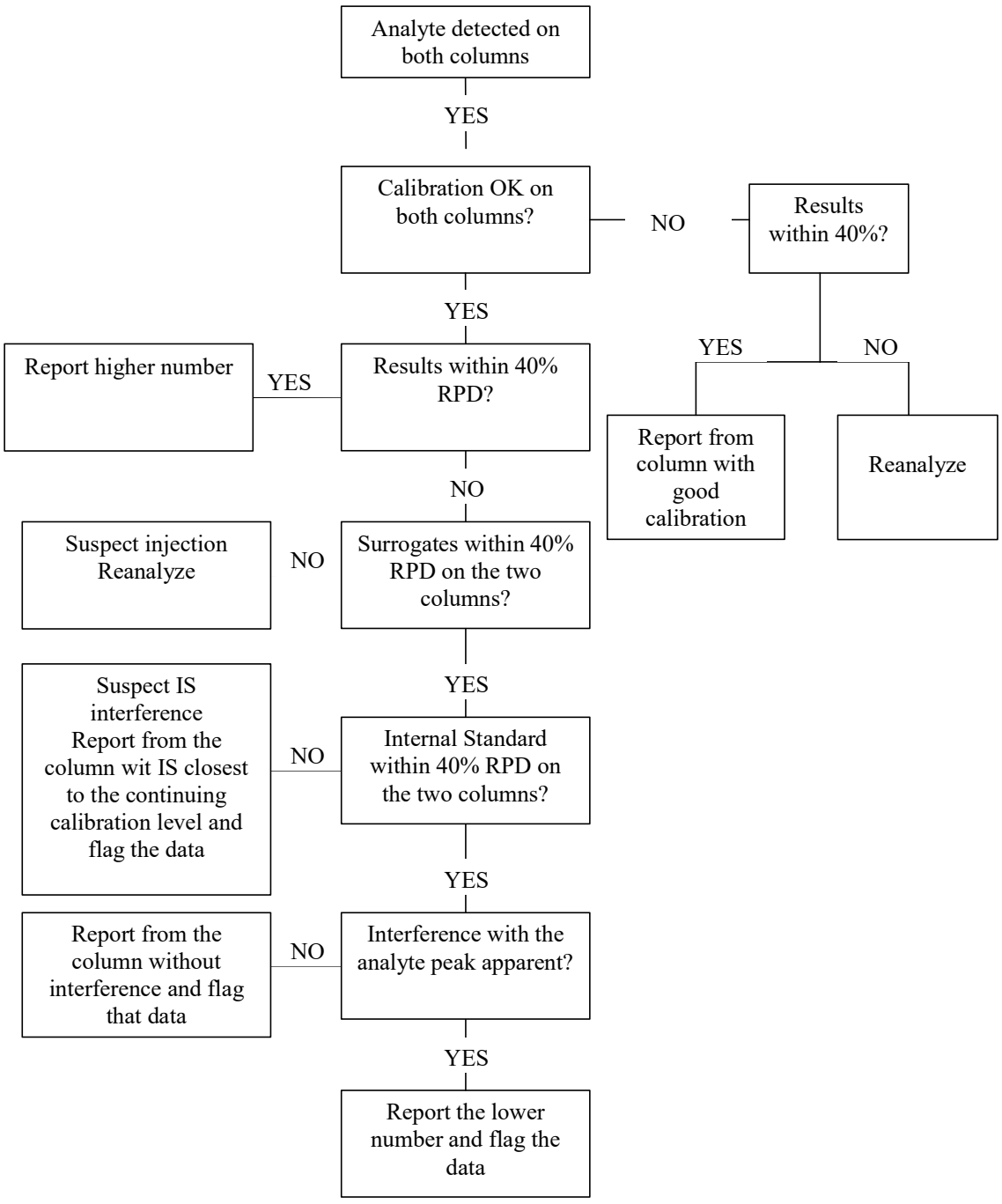
- Revision 2, dated 13 May 2015:
  - Section 1.1: updated RLs to reflect current lab practice.
  - Section 2.1 and throughout: revised initial aqueous/TCLP sample volumes to reflect current lab practice
  - Section 2.1.1: removed note describing option for Reduced Volume Extraction (RVE) as this is now standard lab practice.
  - Section 2.1.2 and throughout document: removed reference to prep by SW3541 as this method is no longer in use at Edison lab.
  - Section 4.3.2 and throughout: updated source of standards used for Florisil check.
  - Section 6.1.2.3.1: updated name of analytical GC columns currently in use.
  - Section 6.1.2.4.1: updated make-up gas to P-5 & Nitrogen.
  - Section 7.1.1: updated carrier gas to Hydrogen and make-up gas to P-5&Nitrogen.
  - Section 7.2.2 and throughout document: updated source and catalog numbers for analytical standards (Restek is now primary source of standards).
  - Section 7.2.2.1: updated standards prep instructions. Removed outdated references to standards and spiking mixes. Removed notes pertaining to RVE since this procedure is now standard and incorporated into instructions.
  - Section 7.2.2.10: added instructions for preparation of ICVs.
  - Section 8.1: adjusted water sample container from 1000ml to 250ml.
  - Section 9.2.2: removed references and instructions pertaining to the Pesticide Resolution Check (a CLP requirement we no longer perform).
  - Section 9.2.3.1: Added extensive discussion regarding the analysis of replicated CCVs.
  - Section 9.2.4.4: updated table to include new concentration for low standard (2.5 ppb).
  - Section 10.1.2.14 and throughout document: replaced references to Target with TestAmerica Chrom.
  - Section 10.2: made extensive revisions to discussion of the analytical sequence (removing duplicate entries and adjusting to reflect current procedures).
  - Section 10.3: added note concerning NJ DKQP requirements for reporting highest concentration of dual columns.
  - Section 15 (References): updated to include "Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version."
  - Added Sections 7.2.2.11 and 7.2.2.12 reflecting the addition and preparation of Internal standard

- Revision 1, dated 09 October 2012
  - Throughout: Revised LQM section number references to reflect the most current LQM revision.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) option.
  - Section 2.1.2: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
  - Section 7.2.2.1.1 added preparation of 5ppb standard for the reduced volume method.
  - Section 9.2.2.2 Added 5ppb standard for the reduced volume method
  - Section 9.2.4.4 Added 5 ppb standard to the initial calibration for the reduced volume method
  - Section 10.2.6 Added 5 ppb to the Analytical sequence for the low level method Section 15.0: Removed reference to TestAmerica Edison SOP EDS-GEN-019, *Organic Calculations,* most current revision.
  - Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

- Revision 0, dated 02/17/2011: New

ATTACHMENT 1

Dual Column Reporting Flowchart
(Note: NJDEP DKQP requires reporting the higher concentration in all instances)



Title: Procedure for the Microwave Extraction of Solids, SW846 3546

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey Organic Operations Manager	3/12/18 Date	Dan Helfrich Health & Safety Manager	3/12/18 Date
Carl Armbruster Quality Assurance Manager	3/12/18 Date	Mark Acierno Laboratory Director	3/12/18 Date

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1.0 Scope and Application

The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 100^{o} -115^{o}C and 50 - 175 psi) in a closed vessel containing the sample (soil, sediment and sludge) and organic solvent(s) to achieve analyte recoveries equivalent to those from Automated Soxhlet extraction (Method 3541), using less solvent and taking significantly less time than the Automated Soxhlet procedure.

This method is applicable to the extraction of semi-volatile petroleum products in soil, sediment and sludge which may then be analyzed by a variety of chromatographic procedures. The method may also be applicable for the extraction of additional target analytes, provided that the analyst demonstrates adequate performance for the intended application as further described in TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

This method only is applicable to solid samples with small particle sizes. If practical the sample may be air dried and ground to a fine powder prior to extraction.

1.1 **Analytes, Matrix(s), and Reporting Limits:** This standard operating procedure details the procedures used by TestAmerica Edison for the microwave extraction of soils, sediments, sludges, and waste solids for semivolatile organic compounds by SW846 Method 3546 for subsequent analysis of:

- Diesel Range Organics (C10-C28 & C8-C44) by SW846 Methods 8015B, 8015C and 8015D (see TestAmerica Edison SOP Nos. ED-GCS-009 and ED-GCS-020);
- TPH by NJDEP-OQA-QAM-025 (see TestAmerica Edison SOP No. ED-GCS-011);
- Organochlorine Pesticides and PCBs by SW846 8081A/B and 8082/A (see TestAmerica Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017)
- Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS) by SW846 8270 and 8270D (TestAmerica Edison SOPs ED-MSS-002 and ED-MSS-009)
- PCBs by GC/MS using EPA Method 680 (TestAmerica Edison SOP ED-MSS-010)

1.2 The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in TestAmerica Edison SOP ED-GCS-012 (current revision).

1.3 For a complete discussion of analytes and reporting limits (RLs) please refer to the applicable TestAmerica Edison analytical SOPs referenced in Section 1.1 and in References (Section 14.0).

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A representative soil, clay, sediment, sludge, solid or waste sample is weighed and placed into a 75mL Teflon vessel and extracted using an appropriate solvent in a microwave extractor. The extraction vessel containing the sample and solvent system is heated to the extraction temperature and extracted for 10 minutes. The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined. The extract is then dried, concentrated using a nitrogen blow down device, and as necessary, solvent exchanged for use in clean up, fractionation, or determinative methods.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may contribute to interferences with sample analysis. All materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing a method blanks.

4.2 Phthalate esters are commonly extracted from laboratory items. Plastics in particular, must be avoided because phthalates are easily extracted from these materials. The presence of these contaminants in extracts is problematic because it will give false positive results for the methods that list phthalates as target compounds or it will interfere with the target compounds of other methods that are co-extracted with them.

4.3 All glassware used must be scrupulously cleaned before use in trace analysis. Refer to TestAmerica Edison SOP No. ED-GEN-013 (*Glassware Cleaning*), current revision.

4.4 Use caution if sodium sulfate is used for this method. Salts are known to super heat when used in microwave. Typically samples are analyzed with the addition of 5.0 grams sodium sulfate.

5.0 Safety

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

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

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The use of the microwave to extract samples creates excessive pressure and temperature within the digestion vessels very rapidly. Cooling the sample after digestion is extremely important.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Zymark Turbovap II concentrator
- Electronic Balance, capable of weighing to 0.01g.
- N-Evap Analytical Evaporator
- Fisher 8 Position Steam Bath
- MARS 5 microwave oven with temperature sensor (CEM Corp) capable of sensing

temperature within 2.5°C and adjusting microwave field output within 2 sec of sensing.
- MARS 40 position carousel (CEM Corp)
- Visiprep DL column holder

6.2 Supplies

- 10, 50 and 200 mL Concentrator tubes.
- Vials, - 1ml, 4ml, and 10ml with PTFE lined snapcaps.
- Glass syringes, 25ul, 50ul, 100ul, 500ul, 1000ul
- Pastuer pipets
- Glass Buchner funnels (stainless steel funnels may be substituted) – Fisher Scientific.
- 500 ml Kuderna-Danish (KD) flask
- Stainless steal spatulas
- Glass wool- Contaminant-free (silane treated or oven baked at 400^{O}C)
- 75mL Teflon Express vessels with stopper and cap (CEM Corp.)
- Glass fiber filter paper - #1 185mm (Whatman)
- Weigh boats
- Tongue depressors, wood

7.0 Reagents and Standards

Note: Each solvent lot is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1. Reagents

7.1.1 Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.2 Methylene Chloride, JT Baker Resi-analyzed, P/N 9254-03 (or equivalent)

7.1.3 Hexane – JT Baker Ultra-Resi 73513-42-5 or equivalent

7.1.4 Acetone, J.T. Baker Ultra- ResiAnalyzed, Catalog No. 9264-03 (or equivalent)

7.1.5 Nitrogen, high-purity

7.1.6 Construction Sand (Home Depot or Lowes), free from organic compounds. (Baked in the muffle furnace for four hours at 400^{O}C and stored in a dessicator prior to use.)

7.1.7 Sodium Sulfate Anhydrous Powder, Mallinckrodt MA8020-06 or equivalent (Must be baked in the muffle furnace for four hours at 400^{O}C and stored in a dessicator prior to use.)

7.2. Standards

7.2.1 Stock target analyte standard spiking solutions are purchased as prepared solutions (see table below). Stock solutions are diluted (in volumetric glassware) to a working concentration using methylene chloride ($MeCl_2$) hexane or Acetone as the diluent as indicated below below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: Second sources (from separate lots) are used for quanitation standards and spiking.

Stock Standards (Methods 8015B,8015C and 8015D DRO & NJDEP OQA-QAM-025)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
TPH Mix 3	1000 ppm	Sigma Aldrich	861394U	*NJDEP OQA-QAM-025
o-Terphenyl	10000 ppm	Sigma Aldrich	47580U	*SW846 8015B, 8015C &8015D DRO *NJDEP OQA-QAM-025
Chlorobenzene	10000 ppm	Sigma Aldrich	4 6860U	*NJ DEP OQA-QAM-025
Diesel Fuel #2	50000 ppm	Restek	31259	*SW846 8015B, 8015C &8015D DRO *NJDEP OQA-QAM-025

Stock Standards - Pesticides/PCBs* (Methods SW846 8081A/B and 8082/A)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
Pesticide Surrogate Spike Mix ("Pest/PCB Surrogate")	10 ug/ml	Supelco	861275	SW846 8081/8082
Aroclor Spike Mix ("PCB Spike")	100 ug/ml	Supelco	861274	SW846 8082
SS CLP Organochlorine Pesticides Mix ("Pest Spike")	2000 ug/ml	Supelco	4S7426-U	SW846 8081
Chlordane (technical) ("Technical Chlordane Spike")	1000 ug/ml	Supelco	48065-U	SW846 8081
Toxaphene	1000 ug/ml	Supelco	48065-U	SW846 8081

*May be substituted with equivalent standards from alternate sources.

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081A/B and 8082/A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Decachlorobiphenyl (DCB)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
Aroclor 1016	Supelco - 861274	PCB Spike	100

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081A/B and 8082/A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Aroclor 1260	Supelco - 861274	PCB Spike	100
Aldrin	Supelco - 4S7426-U	Pest Spike	2000
Alpha-BHC	Supelco - 4S7426-U	Pest Spike	2000
Alpha-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Beta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Delta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Dieldrin	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan I (Alpha)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan II (Beta)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan Sulfate	Supelco - 4S7426-U	Pest Spike	2000
Endrin Aldehyde	Supelco - 4S7426-U	Pest Spike	2000
Endrin Ketone	Supelco - 4S7426-U	Pest Spike	2000
Endrin	Supelco - 4S7426-U	Pest Spike	2000
Gamma-BHC (Lindane)	Supelco - 4S7426-U	Pest Spike	2000
Gamma-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor Epoxide (Isomer B)	Supelco - 4S7426-U	Pest Spike	2000
Methoxychlor	Supelco - 4S7426-U	Pest Spike	2000
4,4’-DDD	Supelco - 4S7426-U	Pest Spike	2000
4,4’DDE	Supelco - 4S7426-U	Pest Spike	2000
4,4’-DDT	Supelco - 4S7426-U	Pest Spike	2000
Chlordane (technical)	Supleco – 48065-U	Technical Chlordane Spike	1000
Toxaphene	Supelco 48103	Toxaphene Spike	1000

Stock Standards – BNAs (Method 8270x)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
8270 List 1/ Std#1 MegaMix	500/1000/2000	RESTEK	567672
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000	RESTEK	567676
8270 List 1/ Std#2 Amines	2000	RESTEK	567673
8270 List 1/ Std#3 Benzoic Acid	2000	RESTEK	567674
Custom SVO Mix	2000	SPEX	SVO-TANJ-16-5
Bisphenol A	1000	SPEX	S-509-MC
8270 List 1/ Std#8	2000	RESTEK	568724
8270 Surrogate Standard	5000	RESTEK	567685
Aromatic Amines Custom Mix	2000	Supelco	21467482

Stock Standards – PCB Homologues (Method 680)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
Retention Time Calibration Standard Mixture	Varies by component (100 to 200)	Ultra	CB-682MN
Concentration Calibration Standard Mix	Varies by component (50 to 250)	Ultra	CB-681MN
13C12-Decachlorobiphenyl Surrogate	40	Cambridge	EC-1410-3
Lindane13C6	100	Cambridge	CLM-1282-S

Components of PCB Homologue Mixes (Methods 680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
PCB-104	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
PCB-208	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
PCB-77	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
Total Nonachlorobiphenyls	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
DCB Decachlorobiphenyl	Ultra CB-681MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50

7.2.2 Spiking Standards Prep for GC FID (DRO/QAM): The table below provides instruction in preparation of the various working spiking standards for use in this preparation method using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with the indicated solvent.

Working Spiking Standards Preparation					
Standard Name	**Vendor/ Cat #**	**Initial Conc. (ug/ml)**	**Vol. of Standard (ml)**	**Vol. of solvent mls (solvent)**	**Final Conc. (ug/ml)**
DRO Spike/ QAM-025 LCS Spike Diesel Fuel #2	Restek/31259	50000	4	100 ($MeCl_2$)	2000
DRO Surrogate -o-terphenyl	Supelco/47580U	10000	1.0	500 (Acetone)	20
QAM-025 MS/MSD Spike TPH Mix 3	Sigma/861394-U	1000	1.47	10 ($MeCl_2$)	147
QAM-025 Surrogate -Chlorobenzene -o-terphenyl	Supelco/46860-U Supelco/47580-U	10000 10000	1.0 1.0	500 500	20 20

7.2.3 Spiking Standards Prep for GC ECD(8081/8082):

7.2.3.1 All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.2.3.2 Pesticide Spiking Standard (LCS/MS/MSD): The Pesticide Mix BS/MS/MSD solution with 20 single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul (0.50 ml) of the 2000 ppm Pest Spike stock solution (Supelco 4S7426-U, see tables in Section 7.2.1) to a 50 ml final volume with acetone. For spiking instructions refer to Section 10.

7.2.3.3 PCB Spiking Standard (LCS/MS/MSD): The PCB spiking solution (Supelco – 861274, see Tables 1 and 2) is a custom mix received from the vendor at its working concentration of 100 ug/ml in Acetone. For spiking instructions refer to Section 10.

7.2.3.4 Technical Chlordane Spiking Standard (LCS/MS/MSD): A technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Technical Chlordane Spike solution (Supelco 48065-U, see tables in Section 7.2.1) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.2.3.5 Toxaphene Spiking Standard (LCS/MS/MSD): A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Toxaphene Spike solution (Supelco 48103, see tables in

Section 7.2.1) to a final volume 10 mL.using acetone. For spiking instructions refer to Section 10.

7.2.3.6 Pesticide/PCB Surrogate Spiking Standard: The Pesticide/PCB surrogate spiking solution is a concentrated 10 ml solution of DCB/TCMX at a concentration of 10 ug/ml. (Supelco Catalog # 861275, see tables in Section 7.2.1). The concentrate is used without further dilution prior to the spiking of samples.

7.2.4 Spiking Standard for BNA: For use in spiking Soils samples prepared for BNA analysis by SW846 Method 8270. Prepare the spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#2 Amines	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#3 Benzoic Acid	2000ppm	10 ml (methanol)	100 ppm
Custom SVO Mix	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#8	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

** *As needed based on clients requirement.*

7.2.4.1 BNA Surrogate Standard Spiking Solution: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 m

7.2.5 Spiking Standard for PCB Homologues (EPA 680): For use in spiking LCS and MS/MSD prior to extraction. Prepare by adding 1.0ml

of the Retention Time Calibration Standard and 1.0ml of the Concentration Calibration Standard Mix to hexane in volumetric glassware and dilute to final volume of 100ml. 1.0mL of this solution is spiked into LCS/MS/MSD prior to extraction.

7.2.5.1 PCB Homologue Surrogate Standard Spiking Solution (EPA 680): (2.0ug/mL and 2.5ug/mL) – Add 3.125mL of $^{13}C_{12}$-Decachlorobiphenyl and 1.0mL of Lindane13C6 and dilute to a final volume of 50mL in hexane in volumetric glassware. 1.0mL of this solution is spiked into all blanks, samples and QC items prior to extraction.

7.2.6 The preparation of all standards must be documented in a standard preparation logbook or in the TALS reagent module. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass	4 oz	Cool 4 ± 2°C	14 Days	a) NJDEP-OQA-QAM-025; b)SW846 Method 8000

Samples are collected in amber jars with Teflon lined caps.
[1]All sample extracts must be analyzed within forty (40) days of extraction.

9.0 Quality Control

9.1. Sample QC – The following quality control samples are prepared with each batch of samples.

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS/LCSD)[1]	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Method Blank (MB)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
MS\MSD[2] (Client specified)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Surrogates[3]	All Samples including QC	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract except for NJDEP EPH 10/09 where LCS/LCSD is required.
[2] The sample selections for MS/MSD are randomly selected, unless specifically requested by a client. For NJDEP EPH 10/09, only MS is required (MSD for NJDEP EPH 10/09 is by client request only).
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.2. Instrument QC

N/A

10.0 Procedure

10.1. Sample Preparation

10.1.1 Decant and discard any water layer on a sediment sample. Discard any foreign objects such as rocks, leaves and sticks. Mix sample thoroughly with wooden tongue depressor prior to subsampling

10.1.2 The initial amount and treatment of soil samples is method dependent:

- For QAM-025, DRO (8015): 15 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures). Where practical samples should be air dried and ground prior to extraction. Drying should be performed in a hood to avoid contamination.
- For 8270/ 8081/8082: 15 grams of soil is weighed into an aluminum weighing dish (the weight is recorded to two significant figures). 15 mls of acetone is added to the dish and the soil/acetone mixture is stirred well with a wooden tongue depressor to create a slurry. The soil/acetone slurry is transferred to a 75 ml Teflon microwave vessel. The dish is then rinsed with 15 ml of methylene chloride which is also added to the microwave vessel.
- For EPA 680: 30 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures).

10.1.3 For laboratory blanks and control spikes, an equal portion of clean sand is prepped along with samples.

10.1.4 All samples, blanks, and spikes are fortified with an appropriate amount of the applicable method surrogates. All matrix spikes and lab

control spikes are fortified with an appropriate amount of the applicable method spiking solution. See Attachment 1 for general spiking instructions. (Refer to Section 7.2 for information on preparation of spiking solutions):

- PCBs (8082): add 50 ul of the PCB Spike solution to each LCS and designated MS/MSD;
- Pesticides (total, 8081): 100 ul of the Pest Spike solution to each LCS and designated MS/MSD;
- Technical Chlordane: 100 ul of the Technical Chlordane Spike mix to each LCS and designated MS/MDS;
- Toxaphene: 100 ul of the Toxaphene Spike mix to each LCS and designated MS/MSD;
- BNA (8270): 500ul of spike/Surrogate solution to each LCS and designated MS/MSD);
- PCB Homolgues (EPA 680): 1.0 ml each of Spiking solution (see Sec.7.2.5) and Surrogate solution (see Sec. 7.2.5.1) to each LCS and designated MS/MSD.

NOTE: When spiking the samples, it is critical to remove all bubbles from the syringe.

10.1.5 Add the following solvent to each sample vessel depending upon the fraction (**note**: for Pesticides and PCBs by methods 8081 and 8082 the solvent addition is addressed in Section 10.1.2 above)

- 30mL of methylene chloride for DRO (8015)/QAM-025;
- 30 mL of Methylene Chloride/Acetone blend (50:50) for BNA (8270)

30 mL of Hexane for PCB homologues by EPA 680

10.1.6 A stopper is placed over the opening along with the vessel cap. The vessel is then tightened using the MARS capping wrench. This capping wrench tightens the cap to the appropriate torque.

10.1.7 The sample vessels are placed into the 40-position carousel. Each position has an insulator sleeve that the vessel slides into – slide the vessel into the appropriate sleeve – and press firmly down to ensure a complete connection.

10.1.8 Place the carousel (with samples and vessels) into the microwave unit.

Start Run: (load method – using key pad on face of unit)
Press: Home

Press Select: load method
Press Select: from user directory
Press Select: 3546 Microwave – Xpress method
Press Start.

Microwave Operating Parameters				
Power Max	**Power %**	**Oven Ramp**	**Degrees Celsius**	**Hold Time**
1200W	100	10.0 minutes	110	10.0 minutes
1600W[1]	100	20.0 minutes	115	10.0 minutes

[1] For pesticide/PCB and BNA samples only

10.1.9 A 5-minute cool down will close out the run.

10.1.10 Remove vessels from carousel and allow cooling (either at room temperature or in a cold water bath).

10.1.11 When cool down is complete, remove carousel from microwave.

10.1.12 Pre-wash a funnel filled with sodium sulfate with three successive washings of 30 ml acetone. Pour the extract carefully through the sodium sulfate funnel (to remove any moisture) into the 200mL concentrator tube. Rinse the 75mL vessel with 10 mL methylene chloride three times to ensure quantitative transfer.

10.1.13 Set the water temperature in the concentrator to 35°C and adjust the pressure to around 5psi of nitrogen.

10.1.14 Place the tube into the apparatus and slowly raise the pressure to 20psi. If splashing occurs, start with a lower pressure and raise it when the solvent gets lower.

10.1.15 Concentrate to just under the 1mL mark on the nipple of the tube. Remove from the bath and transfer to an auto sampler vial with a glass Pasteur pipette..

10.1.16 Add a few drops of clean methylene chloride to the tube, rinsing the narrow part and add to the vial to adjust to 1mL or other method specific final volume. If solvent exchange to hexane is required, add approximately 30mL (mix well) and return to concentrator set at 35°C. See Attachment 2.

10.1.17 Total concentration time should be approximately 20 minutes for methylene chloride. If a sample takes a longer than usual amount of time to concentrate or if the extract becomes viscous the final volume should be adjusted to a larger volume and documented.

10.1.18 The extract is now ready for the appropriate clean up and or analysis.

10.2. Calibration

10.2.1 The balance calibration is checked daily prior to use. Refer to TestAmerica Edison SOPs ED-GEN-010, *Calibration of Analytical Balances*, current revision and ED-GEN-006, *Standard Operating Procedure for Preventive Maintenance and Calibration Procedures for All Analytical Instruments and Ancillary Equipment*, current revision.

11.0 Calculations / Data Reduction

N/A

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0 Pollution Control

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- *Mixed Solvent Waste*: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- *Auto sampler vials and expired standards*: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3546, Microwave Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Laboratory Manual, Physical/Chemical Methods, February 2007.

15.2 United States Environmental Protection Agency, “Method SW8015B, Non-Halogenated Organics, Test Methods for Evaluating Solid Wastes, SW846 Third Edition,Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.3 United States Environmental Protection Agency, Method SW8015C, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, February 2007.

15.4 United States Environmental Protection Agency, Method SW8015D, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, June 2003.

15.5 NJDEP Method No. OQA-QAM-025, “Quantitation of Semivolatile Petroleum Products in Water, Soil, Sediment and Sludge” (current revision)

15.6 United States Environmental Protection Agency, “Method 8081A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.7 United States Environmental Protection Agency, “Method 8082, Polychlorinated Biphenyls PCBs) by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.8 United States Environmental Protection Agency, “Method 8081B, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.9 United States Environmental Protection Agency, “Method 8082A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.10 United States Environmental Protection Agency, “Method SW8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.11 United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, February 2007.

15.12 TestAmerica Edison SOP No. ED-GCS-008, *Preparation and Analysis of Diesel Range Organics (DRO C10-C28) in Soil and Water Samples by SW846 Methods 3510C (Separatory Funnel), 3541 (Automated Soxhlet Extraction) and 8015B(GC/FID),* current revision.

15.13 TestAmerica Edison SOP No. ED-GCS-009, *Preparation and Analysis of Diesel Range Organics (DRO C10-C44) in Soil and Water Samples by SW846 Methods 3510C (Sep Funnel), 3541 (Automated Soxhlet) and 8015B (GC/FID),* current revision.

15.14 TestAmerica Edison SOP No. ED-GCS-011,*NJDEP OQA-QAM-025 Quantitation of Semivolatile Petroleum Products in Water Soil Sediment and Sludge,* current revision.

15.15 TestAmerica Edison SOP No. ED-GCS-003, *Analysis of Organochlorine Pesticides by Gas Chromatograph, SW846 Method 8081A*, current revision.

15.16 TestAmerica Edison SOP No. ED-GCS-004, *Analysis of Polychlorinated Biphenyls by Gas Chromatograph, SW846 Method 8082*, current revision

15.17 TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by Gas Chromatograph, SW846 Method 8081B*, current revision.

15.18 TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by Gas Chromatograph, SW846 Method 8082A*, current revision

15.19 TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS, EPA Method 680,* current revision.

15.20 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.21 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.22 TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing& Approval*, current revision.

15.23 TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.24 TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning,* current revision

15.25 TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision

15.26 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

15.27 TestAmerica Edison SOP No. ED-GEN-006, *Standard Operating Procedure for Preventive Maintenance and Calibration Procedures for All Analytical Instruments and Ancillary Equipment,* current revision.

15.28 TestAmerica Edison SOP No. ED-GEN-010, *Calibration of Analytical Balances,* current revision.

15.29 TestAmerica Edison SOP No. ED-GEN-007, *Subsampling*, current revision.

15.30 Operation Manuals for the Zymark Turbo Vap II Concentrator.

15.31 Operating manual for the MARS 5 Microwave unit.

16.0 Method Modifications:

N/A

17.0 Attachments

Attachment 1: Spiking information
Attachment 2: Specific Extraction Conditions

18.0 Revision History

Rev 11, March 12, 2018

- Updated Section 10.1 to revise procedures for the microwave extraction of solids for subsequent analysis by EPA methods 8081, 8082 and 8270.
- Updated to remove all references to addition of sodium sulfate to samples prior to microwave extraction.
- Fixed section numbering and formatting errors.

Rev 10, May 16, 2017

- Updated throughout to add procedures for microwave extractions of solids for subsequent analysis by EPA Method 680 (PCBs by GC/MS).

Rev 9, Dec 15, 2016

- Updated throughout to remove procedures for microwave extraction of solids for NJ EPH method. Those procedures are fully covered in TestAmerica Edison SOP ED-GCS-012, Preparation and Analysis of Extractable Petroleum Hydrocarbons (EPH) in Solid and Water Samples using NJDEP EPH Method 10/08, August 2010 (Rev. 3): Analysis of Extractable Petroleum Hydrocarbon Compounds (EPH) in Aqueous and Soil/Sediment/Sludge Matrices (current revision)
- Added following text to Section 1.2: The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in TestAmerica Edison SOP ED-GCS-012 (current revision).
- All references to NJDEP EPH methodology removed.

Rev 8, Jan 22, 2015

- Updated to include procedures for microwave extraction of solids for analysis my SW846 Methods 8270C and 8270D (Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS). Specifically
    - Added 8270 method references and SOP numbers to Sections 1.1 and 15.0
    - Added a table to Section 7.2.1 detailing the BNA standard mixes.
    - Added new Section 7.2.4 detailing the preparation of BNA spiking standards

- Added Section 7.2.4.1 detailing the preparation of BNA surrogate spiking standards.
- Added details of 8270 spiking and extraction to Section 10.0 as appropriate.
- Added BNA Spiking details to Attachments 1 and 2. COAs for BNA spiking not included in SOP but are on-file in the department.

Rev 7, June 14, 2013

- Section 1.1: updated the DRO method references to include SW8015C and SW8015D. Also update the SOP references for the DRO analytical methods.
- Section 7.2.1: updated stock standards table to include SW8015C and 8015D. Revised source for o-Terphenyl stock. Removed TPH Mix 1 from list. Removed 8015x DRO from the list of methods for which TPH Mix 3 is used. Added 8015x DRO to the list of methods for which Diesel Fuel #2 is used.
- Section 7.2.2.1: Revised DRO surrogate spike source and prep instructions. Revised DRO and QAM-025 spiking standard source and prep instructions.
- Section 15.0: References: added references for methods SW8015C and 8015D.
- Attachment 1: updated spiking information for DRO.

Rev 6, October 10, 2012

- Throughout document:: Revised LQM reference sections to reflect the most current revision
- Throughout document: updated as detailed below to include prep instructions for solid samples undergoing analysis by SW846 8081A/B and 8082/A.
- Section 1.1: added references to the 8081/8082 SOPs: TestAmerica Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017.
- Section 6.2: added weigh boats and wooden tongue depressors.
- Section 7.2.1: added acetone as a diluent and added tables detailing the Pestcide and PCB standards.
- Section 7.2.2: added “GCFID (DRO/EPH/QAM)” to section header.
- Section 7.2.3: added instructions for prep of Pest/PCB spiking standards.
- Section10.1.4: Added Pest/PCB spiking instructions.
- Section 10.1.5: added instructions for addition of extraction solvent (Methylene Chloride:Acetone for Pest/PCB).
- Section 10.1.8: updated table to include Microwave Parameters for Pest/PCB prep.
- Section 10.1.12: added a methylene chloride rinse.

- Section 15: added method and SOP references for SW846 8081A/8081B and SW846 8082/8082A.
- Attachments: updated to include Pest/PCB information.

Rev 5, August 26, 2011

- Section .6.2: added vendor and part number details for silica gel columns (Phenomenex Part No:8L-S012-JCH)
- Section 10.1.2: Added details for the pre-washing of sodium sulfate funnels with three successive 30 ml volumes of acetone. Added details for the rinsing of the 75 ml vessel with three successive volumes of methylene chloride.

Rev 4, April 21, 2010

- Section 7.2.1: Corrected the footnotes in the tables 'Stock Standards – Aliphatics (Method NJDEP EPH 10/08)' and 'Stock Standards-Aromatics (Method NDJEP EPH 10.08)'.
- Attachment 3: Added this attachment which consists of select certificates of analysis for Method NJDEP EPH 10/08.
- Section 7.2.2.1: Table 'Working Spiking Standards Preparation'" replaced asterisks (*) with dashes (-) in order to clarify that these are simply bullets rather than referencing footnotes.

Rev 3, April 19, 2010

- Section 7.2.2.1: added procedure for preparation of a 500 ppm Aliphatic Neat Spike Mix.
- Section 7.2.2: Spiking Standards Prep: revised the procedure for prep of the EPH Aliphatic Spike in the 'Working Spiking Standards Preparation' table as follows:
    - Replaced 'MA EPH-n-Hydrocarbons (Absolute/91488) with the 'Florida TRPH Standard (Restek/31266)'
    - Included used of the intermediate 500 Aliphatic Neat Spike Mix.

Rev 2, February 9, 2009

- Section 3.0, Definitions – updated to reflect current location of definitions (Appendix 2 of Lab Quality Manual
- Section 5.2, Primary Materials Used – added 'hexane' to table (used in the NJDEP EPH method).
- Section 7.2.1: added stock standards associated with NJDEP EPH 10/08 method.
- Section 7.2.2: Spiking Standards Prep: updated 'Working Spike Standards' table to include revised NJDEP EPH 10/09 spiking standards; revised other as required.
- Section 9.1, Sample QC: updated with revise NJDEP EPH 10/09 QC requirements.
- Section 10.1, Sample Preparation: revised initial sample weight to 15 grams.
- Section 10.1.8: added Microwave Operating Parameters for the NJDEP EPH 10/09 method.
- Section 10.2.1: changed the specifications of the silica column.

- Section 10.2.5: changed the volume of the hexane rinsate.
- Section 10.2.7: changed the volume of the methylene chloride rinsate.
- Section 10.2.11: Added text describing procedures for NJDEP EPH 10/09 Demonstration of Fractionation Capability.
- Section 14.1 and Section 15.0 : Removed reference to TestAmerica Edison SOP No. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* (SOP is now retired).
- Section 15.0: updated references to NJDEP EPH method as well as TestAmerica Edison SOP for NJDEP EPH analysis.
- Section 17.0, Attachments: corrected descriptions of attachments.

Rev 1, June 29, 2009

- Section 1.0, Scope and Application – corrected typo in the temperature range 100 – 115^{o}C
- Section 5.2, Primary Materials Used – removed ‘sodium hydroxide’ and ‘sulfuric acid’ from table as they are not used in this method.
- Section 6.1, Instrumentation – added Visiprep DL column holder to the instrumentation list
- Section 6.2, Supplies – added 5 g silica gel column (20 ml capacity)
- Sections 7.2.1, 7.2.2, Attachment 1 and Attachment 2: corrected a typo in the abbreviation for Methylene Chloride ($MeCl_2$).
- Section 10.1.1.2 - sentence correction to show actual process and typo error correction. Phrase ‘may also’ changed to ‘shall’.
- Section 10.1.12 – replaced the word ‘salt’ with ‘Sodium Sulfate’ to show actual reagent being used.
- Section 10.1.17 – typo error correction to ’20 minutes’
- Section 10.2.5 – Added the following text to the end of the section: “The amount on hexane will need to be adjusted with every lot of silica columns. The Hexane amount should never exceed 20 mls.”
- Corrected carbon ranges for the reference DRO SOPs throughout the document.

Rev 0, March 27, 2009: New

Attachment 1

Spiking Information – Microwave Extraction

Method	Surrogate Amount (all samples and QC)	Spike Amount (LCS/MS/ MSD)	Final volume	Initial Solvent	Final Solvent
DRO (8015)	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
QAM-025	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
Pest/PCB (8081/8082)	0.05 mL	0.1/0.05 mL	10 mL	MeCl2/Acetone (50:50)	Hexane Exchange from MeCl2/Acetone
BNA (8270)	500uL	500uL	1 mL	MeCl2/Acetone (50:50)	$MeCl_2$
PCB Homologues (EPA 680)	1.0	1.0	1 mL	Hexane	Hexane

Attachment 2

Specific Extraction Conditions

Method	Initial extraction pH	Secondary extraction pH	Final Solvent	Volume of extract required for cleanup(mL)	Final extract volume for analysis (mL)
DRO (8015)	as received	NA	$MeCL_2$	NA	1.0
QAM-025	as received	NA	$MeCL_2$	NA	1.0
Pest/PCB (8081/8082)	as received	NA	Hexane	3	10.0
BNA (8270)	as received	NA	$MeCL_2$	NA	1.0
PCBs (EPA 680)	as received	NA	Hexane	NA	1.0

Title: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Methods 8270E

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

USEPA Method 8270E is an analytical method which employs the use of GC/MS to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples

Eurofins Edison has the capability to analyze and report the compounds listed in Table 1 via Method 8270E.

Table 1

Compound	CAS No.	Compound	CAS No.
1,1'-Biphenyl	92-52-4	Anthracene **(1)**	120-12-7
1,2,4,5-Tetrachlorobenzene	95-94-3	Atrazine	1912-24-9
1,2,4-Trichlorobenzene	120-82-1	Benzaldehyde	100-52-7
1,2-Dichlorobenzene	95-50-1	Benzidine	92-87-5
1,2-Diphenylhydrazine	122-66-7	Benzo[a]anthracene **(1)**	56-55-3
1,3-Dichlorobenzene	541-73-1	Benzo[a]pyrene **(1)**	50-32-8
1,3-Dimethylnaphthalene	575-41-7	Benzo[b]fluoranthene **(1)**	205-99-2
1,4-Dichlorobenzene	106-46-7	Benzo[g,h,i]perylene **(1)**	191-24-2
1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1	Benzo[k]fluoranthene **(1)**	207-08-9
1,4-Dioxane **(1) (2)**	123-91-1	Benzoic acid	65-85-0
1-Methylnaphthalene	90-12-0	Benzyl alcohol	100-51-6
1-Naphthylamine	134-32-7	Bis(2-chloroethoxy)methane	111-91-1
2,2'-oxybis[1-chloropropane]	108-60-1	Bis(2-chloroethyl)ether **(1)**	111-44-4
2,3,4,6-Tetrachlorophenol	58-90-2	Bis(2-ethylhexyl) phthalate	117-81-7
2,3,7,8-TCDD	1746-01-6	Bisphenol-A	80-05-7
2,3-Dihydroindene	496-11-7	Butyl benzyl phthalate	85-68-7
2,3-Dimethylaniline	87-59-2	Caprolactam	105-60-2
2,4,5-Trichlorophenol	95-95-4	Carbamazepine	298-46-4
2,4,5-Trimethylaniline	137-17-7	Carbazole	86-74-8
2,4,6-Tribromophenol **(Surrogate)**	118-79-6	Chrysene **(1)**	218-01-9
2,4,6-Trichlorophenol	88-06-2	Chrysene-d12 **(ISTD)**	1719-03-5
2,4-Dichlorophenol	120-83-2	Coumarin	91-64-5
2,4-Dimethylphenol	105-67-9	Dibenz(a,h)anthracene **(1)**	53-70-3
2,4-Dinitrophenol	51-28-5	Dibenzofuran	132-64-9
2,4-Dinitrotoluene	121-14-2	Diethyl phthalate	84-66-2
2,4-Xylidine	95-68-1	Dimethyl phthalate	131-11-3
2,6-Dinitrotoluene	606-20-2	Di-n-butyl phthalate	84-74-2
2-Chloronaphthalene	91-58-7	Di-n-octyl phthalate	117-84-0
2-Chlorophenol	95-57-8	Fluoranthene **(1)**	206-44-0
2-Ethylaniline	578-54-1	Fluorene **(1)**	86-73-7
2-Fluorobiphenyl **(Surrogate)**	321-60-8	Hexachlorobenzene **(1)**	118-74-1
2-Fluorophenol **(Surrogate)**	367-12-4	Hexachlorobutadiene	87-68-3
2-Methylnaphthalene	91-57-6	Hexachlorocyclopentadiene	77-47-4
2-Methylphenol	95-48-7	Hexachloroethane	67-72-1
2-Naphthylamine	91-59-8	Indeno[1,2,3-cd]pyrene **(1)**	193-39-5
2-Nitroaniline	88-74-4	Isophorone	78-59-1
2-Nitrophenol	88-75-5	n,n'-Dimethylaniline	121-69-7
2-tertbutyl-4-methylphenol	2409-55-4	Naphthalene **(1)**	91-20-3
2-Toluidine	95-53-4	Naphthalene-d8 **(ISTD)**	1146-65-2

Table 1

Compound	CAS No.	Compound	CAS No.
3 & 4 Methylphenol	15831-10-4	n-Decane	124-18-5
3,3'-Dichlorobenzidine	91-94-1	Nitrobenzene	98-95-3
3,4-Dimethylaniline	95-64-7	Nitrobenzene-d5 **(Surrogate)**	4165-60-0
3,5-di-tert-butyl-4-hydroxytol	128-37-0	N-Nitrosodimethylamine **(1)**	62-75-9
3-Nitroaniline	99-09-2	N-Nitrosodi-n-propylamine	621-64-7
4,6-Dinitro-2-methylphenol **(1)**	534-52-1	N-Nitrosodiphenylamine	86-30-6
4-Bromophenyl phenyl ether	101-55-3	n-Octadecane	593-45-3
4-chloro-2-methylaniline	95-69-2	o-Toluidine-d9 **(Surrogate)**	194423-47-7
4-Chloro-3-methylphenol	59-50-7	Pentachloronitrobenzene	82-68-8
4-Chloroaniline	106-47-8	Pentachlorophenol **(1)**	87-86-5
4-Chloroaniline–d4 **(Surrogate)**	191656-33-4	Perylene-d12 **(ISTD)**	1520-96-3
4-Chlorophenyl phenyl ether	7005-72-3	Phenanthrene **(1)**	85-01-8
4-Methylphenol	106-44-5	Phenanthrene-d10 **(ISTD**)	1517-22-2
4-Nitroaniline	100-01-6	Phenol	108-95-2
4-Nitrophenol	100-02-7	Phenol-d5 **(Surrogate)**	4165-62-2
Acenaphthene **(1)**	83-32-9	Phenyl ether	101-84-8
Acenaphthene-d10 **(ISTD)**	15067-26-2	Pyrene **(1)**	129-00-0
Acenaphthylene **(1)**	208-96-8	Pyridine	110-86-1
Acetophenone	98-86-2	Terphenyl-d14 **(Surrogate)**	1718-51-0
Aniline	62-53-3	Total Cresols	STL00160
Aniline-d5 **(Surrogate)**	4165-61-1		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).
(2) Compound can also be analyzed by Isotope Dilution/SIM.

1.2 For a listing of method detection limits (MDLs) and Reporting Limits (RLs) please refer to the currently active Method 8270 Method Limit Groups in TALS (Eurofins LIMS).

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work)*, and Section 19 (*Test Methods and Method Validation)* in Eurofins Edison's Quality Assurance Manual (Eurofins Edison Document No. ED-QA-LQM).

1.4 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP ED-GEN-003. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 This method is used for the analysis of aqueous and solid matrices for semi-volatile base, neutral and acid organic compounds that are extracted from the sample matrix with an organic solvent.

2.2 An aliquot of sample containing surrogate spiking compounds is extracted with an organic solvent. The extract is concentrated on a steam bath to a suitable volume. Internal standards are added to the extract.

2.3 Sample extraction techniques are specified for each matrix in the following Eurofins Edison SOPs:

- ED-ORP-002 (*Extraction of Semivolatile Organic Compounds in Water by Separatory Funnel, SW846 Method 3510C*);
- ED-ORP-043 (*SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270);*
- ED-ORP-0044 (*Microwave Extraction for Solids, SW846 Method 3546*);

2.4 A small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to a mass spectrometer (MS) which is used to detect the compounds eluting from the GC. The detected compounds are fragmented with an electron beam to produce a mass spectrum which is characteristic of the compound introduced into the MS. Identification of target analytes is accomplished by comparing their mass spectra with the electron ionization spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion (quantitation ion) relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8270E as applicable.

2.5 The standard preparation procedure for aqueous samples involves use of a Reduced Volume Extraction (250 ml) (RVE) followed by analysis using a Large Volume Injection (LVI). Optionally, a full volume (1000 ml nominal) may be employed. The details of the extractions are outlined in the applicable prep SOPs while the analytical details for 8270E is presented in this SOP.

2.6 These methods are also applicable to the analysis of samples by Selected Ion Monitoring (SIM) for the purpose of obtaining lower reporting limits for the following compounds:

Table 2 – SIM Analytes

SIM Analytes	CAS #
1,4-Dioxane	123-91-1
4,6-Dinitro-2-methylphenol	534-52-1
Acenaphthene	83-32-9

Table 2 – SIM Analytes	
SIM Analytes	**CAS #**
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Benzo[b]fluoranthene	205-99-2
Benzo[g,h,i]perylene	191-24-2
Benzo[k]fluoranthene	207-08-9
Bis(2-chloroethyl)ether	111-44-4
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Indeno[1,2,3-cd]pyrene	193-39-5
Naphthalene	91-20-3
N-Nitrosodimethylamine	62-75-9
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Pyrene	129-00-0

2.7 An isotope dilution selected ion monitoring (SIM) technique for the analysis of 1,4-dioxane in water at a reporting level of 0.2 ug/l is also described in this SOP. Using this technique 1,4-dioxane-d8 is added prior to sample extraction and is used as an internal standard to calculate the concentration of 1,4-dioxane present. Additionally, 1,4-dichorobenzene-d4 is added to the extract prior to analysis to monitor the recovery of 1,4-dioxane-d8.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of the Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Analysts must take steps to determine the source of the interference and take corrective action to eliminate the problem.

4.1.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce

carryover, the sample syringe is automatically rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Alternately, verify that the sample analyzed after the high concentration sample does not show any carryover through inspection of chromatogram and target results.

4.1.2 Contaminants from the extraction process detected in the method blank should be evaluated to determine the impact on the analysis. Interferences from any target analyte must not be present in the method blank above the reporting limit for that compound. If these types of interferences occur, corrective action is required. The source should be identified and corrective action initiated to eliminate the interference from the extraction process. Affected samples must be re-extracted and re-analyzed.

4.1.3 The analyst must take precautions to make sure that contaminants do not enter the analytical system. These precautions include systematic procedures designed to eliminate interferences.

4.2 Some compounds analyzed by this method are unstable or sensitive to extraction and/or instrument conditions:

- Benzidine is easily oxidized during extraction. Neutral extraction may enhance the recovery of this compound.
- Hexachlorocyclopentadiene breaks down photochemically and can decompose from high temperatures, particularly in the injection port of the GC. This compound can also react with acetone in solution.
- 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene.
- Phenols are sensitive to active sites and can give a low response or exhibit poor chromatography by tailing. Therefore, it is important the GC is maintained in the best possible condition. See Section 10.1 for proper daily maintenance.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3 and 4-methylphenol.
- Pyridine may perform poorly at the GC injection port temperatures listed in this SOP. Lowering the injection port temperature may reduce the amount of degradation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Dimethyl-dichloro-silane	Flammable	none	Can be corrosive to the respiratory tract causing severe irritation and tissue damage. Harmful if absorbed through the skin. May cause severe irritation and systemic damage. Severely irritating to the skin and eyes. Harmful if swallowed. Can cause abdominal discomfort, nausea, vomiting, diarrhea, and irritation to the mouth, throat and stomach.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph: An Agilent/HP 6890/7890/900 Intuvo (or equivalent) houses the capillary column. The GC provides a splitless injection port and allows the column to be directly coupled to the mass spectrometer. The oven is temperature programmable to meet the requirements of the method. An HP/Agilent 7673/7683/7963 autosampler (or equivalent) with a 10 ul syringe provides automatic injection of sample extracts while the instrument is unattended.

6.1.2 Analytical Column: 30m x 0.25mm ID, 0.25 um film thickness, Restek Rxi-5Sil MS, Catalog #13623

6.1.3 Mass spectrometer: Agilent (HP) 5972, 5973, 5975 or 5977A Mass Selective Detector (MSD) Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts electron energy in the electron ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 50 ng of decafluorotriphenylphosphine (DFTPP) which meets the criteria in Section 9.2.1 when 2 ul of the 25 ug/ml GC/MS tuning standard is injected through the GC.

6.1.4 GC/MS interface: Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

6.1.5 Data system: The data system is interfaced to the mass spectrometer and accommodates continuous acquisition and storage of GC/MS data throughout the duration of the chromatographic program. The data system consists of a Hewlett-Packard Chemstation equipped with Mustang software used for instrument control and data acquisition. This, in turn, is interfaced to Eurofins's Chrom software for data processing. Data from sample extract analysis can be accessed in real-time, while sample data reports and library searches can be performed on data files from previously run samples. The software is also capable of searching any GC/MS data file for ions of a specific mass whose abundances can be plotted versus time or scan number which allows integration of abundances for any extracted ion between specified times or scan-number limits. Library searches utilize a NIST 02.1 Mass Spectral Library.

6.2 Bottles, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

6.3 Injection port liners, splitless

6.4 Injection port septa

6.5 Injection port graphite seals

6.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.9 Vials, 2ml amber screw cap with Teflon liner

6.10 Wheaton microvials 100ul (or equivalent)

6.11 Volumetric Flasks, Class A with ground glass stoppers (2ml - 100ml)

6.12 Analytical balance, ASP Model SP-180 (or equivalent), capable of accurately weighing to 0.0001 gr.

7.0 Reagents and Standards

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the MSDS prior to the use of any reagent or standard.

The preparation of standards, surrogates and spiking solutions is documented in the TALS Reagent Module. Formulary reports can be generated upon request.

7.1. Reagents:

7.1.1. Methylene Chloride: J.T.Baker Resi-Analyzed, used for Organic Residue Analysis (P/N 9266-V8 or equivalent).

7.1.2. Methanol: J.T.Baker Purge and Trap Grade (P/N 9077-02 or equivalent).

7.1.3. Sylon-CT: Supelco (P/N 33065-U or equivalent). Sylon-CT is a highly reactive silanizing reagent consisting of 95% Toluene and 5% Dimethyldichlorosilane (DMDCS).

7.1.4. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards:

7.2.1. **Calibration Standards (Full Scan Analysis)**: Stock analytical standard solutions are purchased mainly from Restek Corporation. Other standards are prepared in the laboratory as needed using neat compounds or prepared solutions purchased from Agilent, SPEX CertiPrep, Chem Service,

Accustandard, Supelco or other suppliers. Standards prep instructions are detailed for the following full scan analyte list options:

- Full Volume Aqueous Prep; and,
- Reduced Volume Aqueous Prep and Soils

Secondary dilutions are either made from purchased stock solutions as listed below or from prepared solutions as listed in the following table:

NOTE: Second sources (from certified separate lots) are used for ICV standards.

Table 3 – Full Scan Stock Standards			
Target Analyte Standard Name	**Conc. (PPM)**	**Vendor**	**Catalog #**
1,2,3,4-TCDD	50	SPEX	SVO-TANJ-12
Agilent Mix (contains compounds listed in Table 4 below)	2000 *	Agilent	Cus 0456
8270 List 1/ Std #1 Megamix	Varied	Restek	571995
8270 List 1/ Std#9	2000	Restek	569730
8270 List 1/ Std#11	2000	Restek	569732
8270 Surrogate Standard	5000*	Restek	567685
8270 Internal Standard	2000	Restek	567684
8270 List 1/ Std#10	2000	Restek	569731
Bisphenol-A	1000	Agilent	Cus-0457

*Agilent Mix, 8270 list1/std#9 and 8270 Surrogate standard are diluted to 100ppm prior to the preparation of the 1.0ppm and 0.5ppm standards.

Table 4	
Agilent Mix Catalog No. Cus-0456	
Analyte	**Concentration (PPM)**
Pentachloronitrobenzene	2000
2 -tert-butyl-4-Methylphenol	2000
2,6-Di-tert-butyl-4-Methylphenol	2000
Coumarin	2000
Phenyl ether	2000
N,N'-Dimethylaniline	2000
N-Methylaniline	2000
Carbamazepine	2000
Benzonitrile	2000
1,3-Dimethylnaphthalene	2000

7.2.1.1. Individual calibration standards for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 5
Full Volume Aqueous Prep and Soils
Working Standards Preparation

Solution Name	120 PPM	80 PPM	50 PPM	20 PPM	10 PPM	5 PPM	2 PPM	1 PPM	0.5 PPM
8270 List 1/ Std #1 Megamix	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	50ul	25ul
8270 List 1/ Std #9	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
8270 List 1/ Std #10	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	-	-	-
Agilent custom Mix	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
1,2,3,4-TCDD	-	-	500ul	-	-	-	-	-	-
8270 Surrogate Standard	600ul	400ul	500ul	100ul	50ul	50ul	20ul	500ul*	250ul *
8270 Internal Standard	500ul	500ul	1000 ul	500ul	500ul	1000 ul	1000 ul	1000 ul	1000 ul
Bisphenol-A	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	-	-
8270 List 1/ Std #11	400ul	300ul	500ul	200ul	125ul	125ul	50ul	25ul	-
Final Volume (ml)	**25**	**25**	**50**	**25**	**25**	**50**	**50**	**50**	**50**

Note: The 1.0ppm and 0.5pmm standards (above) are prepared using the 100ug/ml standard for Agilent custom Mix, 8270 List1/std#9 and 8270 Surrogate Standard.

Table 6
Reduced Volume Extraction/LVI
Working Standards Preparation

Solution Name	24 PPM	16 PPM	10 PPM	4 PPM	2 PPM	1 PPM	0.4 PPM	0.2 PPM	0.1 PPM
120 ppm (see Table 5)	2.0mL								
80 ppm (see Table 5)		2.0 mL							
50 ppm (see Table 5)			2.0 mL						
20 ppm (see Table 5)				2.0 mL					

Table 6 Reduced Volume Extraction/LVI Working Standards Preparation									
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**	**0.4 PPM**	**0.2 PPM**	**0.1 PPM**
10 ppm (see Table 5)					2.0 mL				
5.0 ppm (see Table 5)						2.0 mL			
2.0 ppm (see Table 5)							2.0mL		
1.0 ppm (see Table 5)								2.0 mL	
0.5 ppm (see Table 5)									2.0mL
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**

7.2.1.2. **Initial Calibration Verification (full scan)**: Second source ICVs for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of ICVs for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 7 8270/625 ICV Working Standards Preparation	
Solution Name	**25 PPM**
8270 List 1/ Std #1 Megamix (2nd Lot)	250ul
8270 List 1/ Std #9 (2nd Lot)	125ul
8270 List 1/ Std #10 (2nd Lot)	125ul
Agilent custom Mix (2nd Lot)	125ul
8270 Internal Standard	200ul
8270 List 1/ Std#11	125ul
Bisphenol-A (2nd Lot)	250ul
Final Volume (ml)	**10**

7.2.1.3. **Surrogate Standards (Full Scan Analysis):** A 5000ppm Surrogate Standard is purchased from Restek for use in spiking blanks, samples and associated QC prior to extraction (reference the applicable sample prep SOPs for spiking instructions).

Table 8 Full Scan Surrogate Standards Solution Restek Catalog No. 567685	
Surrogate Standard Compounds	**Concentration (PPM)**
Nitrobenzene-d5	5000
p-Terphenyl-d14	5000
2,4,6-Tribromophenol	5000
Phenol-d5	5000
2-Fluorobiphenyl	5000
2-Fluorophenol	5000

7.2.1.4. **Internal Standards** (**Full Scan Analysis**): The Internal Standards Solution at 2000ppm is purchased from Restek (Catalog # 567684). The Internal Standard solution is stored in 10ml amber screw cap bottles with Teflon liners in the dark at 4°C. The Internal standard solution is used in preparing all analytical standards. Inject 20ul of this solution (2000ppm) per ml of sample extract prior to analysis resulting in a concentration of 40ppm (ug/ml) in the extract.

Table 9 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
1,4-Dichlorobenzene-d4	2000
Phenanthrene-d10	2000
Naphthalene-d8	2000
Chrysene-d12	2000
Acenaphthene-d10	2000
Perylene-d12	2000

7.2.2. **Calibration Standards (SIM analysis)**: The Edison lab currently analyzes only a select list of compounds by 8270E SIM (see Sections 1.0 and 2.0). Stock analytical SIM standard solutions are purchased mainly from Agilent. Working standards are prepared from these solutions as listed in the tables in Section 7.2.2.1:

Table 10 Stock SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
Pentachlorophenol	1000ppm	AGILENT	PH-180-1
n-Nitrosodimethylamine	100ppm	AGILENT	NS-100-1
Hexachlorobenzene	100ppm*	AGILENT	CH-151-1
PAH Mix	100ppm	AGILENT	PAH-605-1
Bis(2-chloroethyl)ether	100ppm*	AGILENT	BEC-110-1
4,6-Dinitro-2-methylphenol	1000ppm**	AGILENT	PH-150
1,4-Dioxane	1000ppm**	AGILENT	NV-152-1

*Hexachlorobenzene and Bis(2-chloroethyl)ether are diluted to 10ppm prior to SIM Standards prep

** 4,6-Dinitro-2-methylphenol and 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

NOTE: Second sources (from separate lots are used for ICV standards).

7.2.2.1 Individual calibration standards for SIM analysis are prepared in one of two ways depending upon the technique (full volume aqueous prep or reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 11
Full Volume Aqueous Prep – SIM
Working Standards Preparation

	0.025 PPM	0.05 PPM	0.1 PPM	0.5 PPM	1.0 PPM	5.0 PPM
Pentachlorophenol	2.5uL	2.5uL	12.5uL	10uL	20uL	50uL
n-Nitrosodimethylamine	25uL	25uL	125uL	100uL	200uL	500uL
PAH mix	6.25uL	5uL	25uL	50uL	100uL	200uL
Hexachlorobenzene	25uL	25uL	250uL	1000uL	2000uL	500uL*
Bis(2-chloroethyl)ether	25uL	25uL	250uL	1000uL	2000uL	500uL*
4,6-dinitro-2-methylphenol	50ul	50ul	250ul	200ul	400ul	1000ul
1,4-Dioxane	25ul	50ul	250ul	200ul	400ul	1000ul
ISTD	500uL	200uL	500uL	200uL	200uL	200uL
Final Volume (ml)	**25**	**10**	**25**	**10**	**10**	**10**

*For Hexachlorobenzene and Bis(2-chloroethyl)ether the 5.0 ppm level is prepared using the 100ppm standard.

Table 12
Reduced Volume Extraction/LVI – SIM
Working Standards Preparation

	0.005 PPM	0.01 PPM	0.02 PPM	0.10 PPM	0.20 PPM	1.0 PPM
0.025 PPM Std (see Table 11)	1.0 mL					
0.05 PPM Std (see Table 11)		1.0 mL				
0.1 PPM Std (see Table 11)			1.0 mL			
0.5 PPM Std (see Table 11)				1.0 mL		
1.0 PPM Std (see Table 11)					1.0 mL	
5.0 PPM Std (see Table 11)						1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.2.2 Initial Calibration Verification (SIM): A 0.1 ppm separate lot SIM ICV is prepared as detailed in Table 13 using the stock standards detailed in Section 7.2.2 (above)

Table 13 0.1ppm SIM ICV preparation	
Pentachlorophenol	25uL
n-Nitrosodimethylamine	25uL
PAH mix	5uL
Hexachlorobenzene	5uL
1,4-Dioxane	5ul
4,6-Dinitro-2-methylphenol	100ul
ISTD	**100uL**
Final Volume	**5 ml**

7.2.2.3 Internal Standard solution (SIM): A 50 ppm Internal Standard solution for SIM analysis is prepared by adding 125ul of the 2000ppm stock ISTD (see Section 7.2.1.4) and bringing to volume with Methylene Chloride in a 5ml volumetric flask.

7.2.2.3.1 For SIM analysis inject 20ul of this solution (50ppm) per ml of sample extract prior to analysis resulting in a concentration of 1ppm (ug/ml) in the extract.

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7.2.3. Calibration Standards (Isotope Dilution SIM – 1,4-Dioxane): The Edison lab currently analyzes only for 1,4-dioxane by 8270E isotope dilution SIM (see Sections 1.0 and 2.0). Stock analytical isotope dilution SIM standard solutions are purchased mainly from Accustandard and Restek. Working standards are prepared from these solutions as listed in the tables below.

Table 14 - Stock 1,4-Dioxane Isotope Dilution SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane	1000ppm*	Accustandard	APP-9-096

* 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

Table 15 - Stock Labeled 1,4-Dioxane SIM Surrogate/Internal Standard (added at prep)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane-d8	2000ppm	Restek	30614

Table 16 - Stock 1,4-Dioxane Isotope Dilution SIM Internal Standard (added to extract)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dichlorobenzene-d4	2000ppm	Accustandard	AZ-014J-3

7.2.3.1 Individual calibration standards for 1,4-dioxane isotope dilution SIM analysis are prepared at the concentrations detailed in the following tables. Prepare by combining the appropriate volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 17 Reduced Volume Extraction/LVI – 1,4-Dioxane Isotope Dilution SIM ICAL Standard Concentrations (ug/ml)									
	Lev 1	**Lev 2**	**Lev 3**	**Lev 4**	**Lev 5**	**Lev 6**	**Lev 7**	**Lev 8**	**ICV***
1,4-Dioxane	0.02	0.04	0.1	0.2	0.5	1	2	10	0.2
1,4-Dioxane-d8	4	4	4	4	4	4	4	4	4
1,4-Dichlorobenzene-d4	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2

***:** The ICV is prepared from the second source stock in Table 13.

7.2.4. GC/MS Instrument Performance Check (DFTPP): The DFTPP standard is prepared by is prepared at 25 ppm by adding 2.5ml of EPA 8270 GC/MS Tuning Solution II (Restek Catalog # 31615) to a 100ml volumetric flask and bringing to volume with Methylene Chloride.

7.2.5. Information on prepared standard solutions must be recorded in the TALS Reagent Module. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Standards must be remade every 6 months, or sooner, if the standards expire or begin to show signs of unacceptable degradation. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.6. Please refer to Eurofins Edison SOP No. ED-GEN-008, Standard Operating Procedure for Preparation, Purity and storage of Reagents and Standards.

- Shelf Life of Standard: 1 year after preparation or stock standard manufacture expiration, whichever comes first;
- Storage Requirements: Stock standards are stored at 4°C and Working Standards stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

8.2 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.3 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.4 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml or 250 ml (1)	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270E
Solids	Wide mouth glass, 8 or 16 oz.	50g	Cool 4 ± 2ºC	14 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270E

(1) : Reduced volume extraction (RVE) LVI option

9.0 Quality Control

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

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every sample	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are determined annually and are updated into TALS limit group..

9.1.1. Method blanks are extracted with every sample batch on each day that samples are extracted. To be considered acceptable, the method blank must contain less than the reporting limit of all target compounds except for phthalates, which can be present at up to 5x the MDL. For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.

If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

9.1.1.1. Surrogate recoveries for the method blank are compared to laboratory generated limits. If two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference.. If any surrogate is still outside limits, all samples and QC samples associated with that method blank must be re-extracted (volume permitting).

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared and extracted concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria. See the current active TALS 8270 Method Limit Group for QC limits. The MS/MSD spiking solution should the same as used for the calibration standards.

9.1.2.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.2.2 An LCS/LCSD may be substituted for the MS/MSD if insufficient sample volume is available.

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.3.2 Spike recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.4. Surrogate Standards: All full scan samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.1.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits).

If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

9.1.4.1 Surrogate recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.5. Internal Standards: The response (area count) of each internal standard in the sample must be within -50 +100% of its corresponding internal standard in the CCV or, the ICAL midpoint for samples analyzed under the initial calibration range. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.2. Instrument QC

9.2.1 GC/MS Instrument Performance Check (DFTPP): (**Note**: the DFTPP performance check applies only to full scan analyses and is not evaluated for SIM analysis). The GC/MS system is tuned using Perfluortributylamine

(PFTBA) such that an injection of 50ng of Decafluorotriphenylphosphine (DFTPP) meet the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all DFTPP key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. Daily tune verification is not required for 8270E CCV.

DFTPP Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
51	30-60% of mass 198
68	<2% of mass 69
69	reference only
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base Peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

9.2.1.1. Evaluate DFTPP using three scan averaging and background subtraction techniques. Select the scan at the peak apex, add +1 scan from the apex and -1 scans from the apex.

9.2.1.2. The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. Background subtract DFTPP by selecting a scan for subtraction ≤20 scans before the apex scan of DFTPP.

9.2.1.3. Check column performance using pentachlorophenol and the benzidine peaks (these compounds are included in the DFTPP solution). Benzidine & Pentachlorophenol should respond normally without significant peak tailing (Tailing Factor should be <2 measured at 10% peak height). If responses are poor and excessive peak tailing is present, corrective action for the GC/MS instrument may be required. Corrective actions may include:

9.2.1.3.1 Retune the GC/MS;
9.2.1.3.2 Clip the injector end of the GC column;
9.2.1.3.3 Replace the septum and injection port liner;
9.2.1.3.4 Change the injection port seal;

9.2.1.3.5 Replace the GC column;
9.2.1.3.6 Clean the injection port with MeCl2
9.2.1.3.7 Clean the MS ion source;
9.2.1.3.8 Place a service call.

9.2.1.4. The breakdown of 4, 4-DDT into 4,4-DDD and 4,4'DDE may also be used to assess GC column performance and injection port inertness. If so evaluated the breakdown must be <20%.

9.2.1.5. DFTPP parameter settings are stored in a tune file, which will be used in all subsequent analysis of standards and sample extracts.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. Initial Calibration: The initial calibration range consists of a minimum of five concentration levels of analytical standards (six for second order regression) prepared as described in Section 7.2. and analyzed once the DFTPP instrument performance check has met the criteria in Section 9.2.1. .

9.2.2.2. Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range.

9.2.3 Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the DFTPP instrument performance check (when applicable).. The CCV is prepared as detailed in Section 7.2. (typically, 50 ug/ml for full volume aqueous and soils, 10 ug/ml for LV, 0.02 ug/ml for LVI SIM) and 0.2 for isotope dilution SIM). Additionally a Low Level Continuing Calibration Verification (LLCCV) is analyzed after the CCV for full scan analysis. The LLCCV is the same as the lowest calibration level analyzed with the initial calibration range (See Section 7.2).

9.2.4 Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. Obtain the retention time for all compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window by using the absolute retention time for each analyte, internal standard and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the

same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. For qualitative identification to be acceptable the retention time of the relative retention time (automatically calculated in Chrom) must be within 0.8 - 1.2 RRT units of its assigned internal standard. The relative retention times of each compound in the five calibration standards must agree within .06 relative retention time units.

9.2.4.2. Initial Calibration Range: Internal standard calibration is employed for this method. After the initial calibration range has been analyzed the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

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

Where:
A_x = Area characteristic ion (see Table 21) for the compound
Ais = Area characteristic ion (see Table 21) of associated internal standard
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound. Minimum response factors must be met for each of the compounds listed in Table 18 (below). Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity in the analytical batch to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.

Table 18:
Minimum Response Factors

Compound	Minimum Response Factor
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl) ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalene	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroanailine	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalene	0.010

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010
Pentachloronitrobenzene	0.050

9.2.4.2.2. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

9.2.4.2.3. The % RSD of the RRF's must be ≤20% for each target analyte listed in Table 18. The % RSD of each target analytes must be ≤20% in order for the calibration range to be acceptable. Additionally for 8270E, the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit or do not meet the minimum correlation coefficient (0.99) or Relative Standard Error (≤20%) for alternate curve fits (see below) then appropriate corrective maintenance action must be performed. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit **AND** do not meet the minimum correlation coefficient (0.99)) or Relative Standard Error (≤20%) then recalibration is necessary.

9.2.4.2.4. If the above listed criteria is met, the system can be assumed to be linear and sample analysis may begin and the average RF from the initial calibration range is used to quantitate all samples.

9.2.4.2.4.1 Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.2.5. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.4 Calculate the first order linear regression for any compound which did not meet the 20% criteria. First order linear regression calibration may be employed if alternative average response calibration procedures were not applicable. The r value (Correlation Coefficient) of the equation must be ≥0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.2 Second order regression calibration can be used for any compound that has an established history as a non-linear performer.

9.2.4.2.5.3 If second order regression calibration is used a minimum of six (6) calibration levels must be analyzed.

9.2.4.2.5.4 If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be ≥ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.5 Any compound that fails to meet the 20% RSD or 0.99 correlation coefficient or RSE (≤20%) criteria must be flagged as estimated for detects (or must be noted in the narrative). If there are non-detects the compounds may be reported if there is adequate sensitivity to detect at the quantitation limit. To demonstrate adequate sensitivity analyze the low level point of the initial calibration in each analytical batch (LLCCV) The criteria for demonstrating adequate sensitivity is detection in the LLCCV using the standard qualitative identification criteria.

9.2.4.2.5.6. When calculating the calibration curve using the linear calibration model a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration back into the curve. The recalculated concentration of the low calibration point should be within ±50% of the

standard's concentration. This evaluation can be checked using the Initial Calibration %Drift Report in Chrom. Any detects for analytes calibrated using the linear model and failing this readback criterion must be flagged as estimated or detailed in the narrative.

9.2.4.3. **Calibration Point Read-back Criteria**: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be <50%.

- See Section 11.8 for the Calculation of Percent (%) Error.

9.2.4.4. **Initial Calibration Verification (ICV):**.Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 7.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds with the exception of the poor performing compounds listed in Attachment 1 which are allowed to be within 50-150% : An NCM must be initiated to denote any ICV non-conformances.

9.2.4.5. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed these criteria as long as their recoveries are within 65-135%. For the poor performers (see Attachment 1) the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.6. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a DFTPP instrument performance check (not required for 8270E), and analysis of a calibration verification standard. **Note:** Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.5.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of DFTPP. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For 8270E analysis only, tune verification is required just prior to ICAL.

9.2.4.5.2 Calibration Verification: Analyze the calibration verification standard immediately after a DFTPP that meets criteria. Daily analysis of the DFTPP is not required as part of the CCV for 8270E analysis. .When samples are analyzed after an ICAL the last ICAL standard may be used as the starting time reference for evaluation. Use the mid point calibration standard (approximately 50ug/l). NOTE: The calibration standard contains internal standards; Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12} at 40ug/l (0.1ug/L for SIM). The calibration check standard must also include all the target analytes from the original calibration.

9.2.4.5.3 The RFs must meet the criteria for the compounds in Table 18. Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met

9.2.4.5.4 The percent difference (when using average response factor) or percent drift (when using linear regression) of the compounds in Table 18 must be ≤20% for at least 80% of the total analyte list. If more than 20% of the compound list fail to 20% difference or drift criterion then appropriate corrective action must be taken prior to the analysis of the samples. Any individual compound that fails must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative identification criteria in the method must be met.

9.2.4.5.5 **CCV Poor Performers**: Refer to Attachment 1 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

9.2.4.5.6 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.5.7 The response (area count) of each internal standard in the calibration verification standard must be within 50 - 100% of its corresponding internal standard in the mid-level calibration standard of the active calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.2.4.5.8 The relative retention times of each compound in the calibration verification standard must agree within .06 relative retention time units of its value in the initial calibration.

9.2.4.5.9 Use the average response factors from the original five-point calibration for quantitative analysis of target analytes identified in field samples.

9.2.4.5.10 Prepare a calibration summary or list indicating which compounds did not meet the 20% average percent difference criteria. Record this information in that run log.

9.2.4.7. Low Level Continuing Calibration Verification (LLCCV): An LLCCV consisting of the low level standard from the initial calibration range is analyzed every 12 hours of instrument operation after the CCV. The purpose and evaluation of the LLCCV is described in Section 9.2.4.4.4.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The sequence of events for GC/MS analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed.

10.1.2. Preparation of the Injection Port Liner and Installation Procedure: Prior to the start of initial calibration and each daily analysis of sample extracts, a new liner for the injection port must be prepared. Once a liner has been used it is no longer inert and will cause serious chromatography problems with phenols and other compounds. When preparing the liner, proper laboratory protection must be worn and the liner must be prepared in a well-ventilated hood. When the procedure is completed all traces of toluene, Sylon-Ct and methanol will be removed immediately so that extraction solvents and preparation of sample extracts will not come into contact with these solvents and become contaminated.

10.1.2.1 Remove one liner from a 40ml VOA bottle containing other liners immersed in Sylon-Ct solution. Rinse off the liner with Toluene and wipe dry. Insert 1cm of pre-silanized glass wool partially into one end of the liner and trim neatly. Push the glass wool into the center of the liner so that it is 1 1/4" from the bottom. Do not use glass wool or solvents that are dirty (i.e. suspended particles) or use liners which are chipped on the ends, deformed or fractured. Inspect the glass wool for cleanliness after it has been inserted.

10.1.2.2 Using a Pasteur pipette flush out the interior of the liner containing the glass wool with Sylon-Ct. Rest the liner horizontally on a small beaker and allow the Sylon-Ct to re-deactivate the interior surfaces and the glass wool. There should be no air bubbles caught in the glass wool. After several

minutes flush out the Sylon-Ct with toluene and finally with methanol. Dry the outer surface of the liner and rest it on the injection port housing until the remaining methanol is boiled off

10.1.2.3 Insert the liner with the newly silanized glass wool plug into the injection port. Verify that the column extends up into the injection port and is perpendicular. Inspect the graphite seal and replace it if the edges are knife-shaped.

10.1.2.4 The septum is always replaced daily. Bake out the column at 300^{O}C for 15 minutes after the vacuum in the analyzer has returned to normal.

10.1.2.5 Performance may enhanced by clipping a small portion of the column at the injection port end. Document this activity in the maintenance record.

10.1.3. Prior to calibration or sample analysis always verify that the analyzer is under sufficient vacuum and that the column has proper carrier gas flow.

10.1.4. Establish the following GC/MS operating conditions:

10.1.4.1 Full Scan Operating Mode

Full Scan Mode – Standard Injection Volume
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse, splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

Full Scan Mode – Large Volume Injection (LVI)
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse, splitless
Injection Volume: 5ul
Splitless Valve Time: 0.3 minutes

10.1.4.2 SIM Operating Mode

SIM Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.5 minutes
Initial Column Temperature and Hold Time: 40^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 3 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

10.1.4.3 Isotope Dilution Selected Ion Monitoring Mode :

SIM Parameters

Group 1
Plot 1 Ion: 74.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	42.0 50	43. 0 50	68.0 50
	74.0 50	128.0 50	129.0 50
	136.0 50	150.0 50	152.0 50
	93.0 50	66.0 50	
	58.0 50		
	88.0 50		

Group 2
Group Start Time: 6.00
Plot 1 Ion: 152.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	151.0 50	152.0 50	153.0 50
	154.0 50	162.0 50	164.0 50
	165.0 50	166.0 50	

Group 3
Group Start Time: 7.80
Plot 1 Ion: 188.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	94.0 50	101.0 50	142.0 50
	178.0 50	179.0 50	188.0 50
	202.0 50	264.0 50	266.0 50
	284.0 50		

Group 4
Group Start Time: 10.50
Plot 1 Ion: 228

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	120.0 50	228.0 50	229.0 50
	240.0 50		

Group 5
Group Start Time: 12.00
Plot 1 Ion: 252.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	138.0 50	139.0 50	252.0 50
	253.0 50	260.0 50	264.0 50
	267.0 50	276.0 50	278.0 50

Table 19: Target Compound - Primary and Monitoring Ions

Compound	1	2	3

Compound	1	2	3
1,4-Dioxane-d8	96	64	62
1,4-Dioxane	88	58	57
1,4-Dichlorobenzene-d4	152	150	

10.1.5. The above listed instrument conditions are used for all analytical standards for calibration and for all sample extracts analyzed by this method.

10.1.5.1 The column conditions, scan start time, and splitless valve time for analysis of DFTPP only are as follows are as follows:

Initial Column Temperature and Hold Time: 140^{o}C for 0.5 minutes
Column Temperature Program: 140^{o} to 320^{o}C at 22^{o}C/minute
Final Column Temperature Hold: 320C for 0.5 minutes
Scan Start Time: approx. 5 minutes
Splitless Valve Time: 0.3 minutes
Injection Volume: 2 ul

10.2. Analytical Sequence

10.2.1. Dilutions are made based on initial GC/MS analysis. Dilutions are made in 1-ml vials using microsyringes. Calculate the dilution factor using the equation below:

DF= Ph / 5 x Is
Where:
DF = Dilution Factor
Ph = Sample Peak Height
Is = Internal Standard Peak Height

When DF >1 but <2, combine 500ul of sample extract with 500ul methylene chloride in a 1 ml amber vial, add20 ul internal standard and crimp seal

Use **Table 20** to determine dilution and internal standard amount.

Table 20 Dilution Factor Calculations			
DF Value	**Volume of Sample (ul)**	**Volume of Methylene Chloride (ul)**	**Volume of ISTD (ul)**
<1	1,000	None	None
>1, <2	500	500	10
>4, <5	200	800	16
>10, <20	100	900	36
>20	500*	500	10
*Prepare this dilution by serially diluting the >10, <20 dilution			

10.2.2. Instrument Performance and Calibration Sequence

10.2.2.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.2.2.2. Analyze the Instrument Performance Check Standard (DFTPP) as discussed in Section 9.2.1.

10.2.2.3. Initially and as required, analyze the Initial Calibration Range (minimum 5 points, six points for second order regression) as detailed in Sections 7.2.1 and 9.2.4.2. Evaluate the acceptability of the Initial Calibration Range as detailed in Section 9.2.4.2.

10.2.2.4. Immediately after the Initial Calibration Range only, analyze the Initial Calibration Verification (ICV) as detailed in Sections 7.2. and 9.2.4.3. Evaluate the acceptability of the ICV as detailed in Section 9.2.4.3.

10.2.2.5. Every 12 hours, reanalyze and evaluate the Instrument Performance Check Standard (DFTPP), not required for 8270E followed by the Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV) as detailed in Section 9.2.3, 9.2.4.4 and 9.2.4.5. Evaluate the acceptability of the CCV and LLCCV as detailed in Section 9.2.4.4

10.2.2.6. Client samples and QC samples are analyzed (as detailed in Section 10.2.3) after acceptable Instrument Performance and Calibration Checks and until the 12 hour clock expires. Repeat the sequence as required. The automation of GC/MS runs is accomplished via the “SEQUENCE” macro of the ChemStation.

10.2.3. Sample Analysis Sequence

10.2.3.1. Sample extracts are normally prepared on the same day as analysis. The GC/MS operator will prepare the extracts that will be run on his or her instrument. Volume adjustments to the extracts will be made at the discretion of the supervisor.

10.2.3.2. Prior to the start of sample analysis the GC/MS operator will generate a sequence program containing the list of the sample extracts to be analyzed, the position on the autosampler tray, and the proper acquisition and tune methods that are to be used. This sequence program contains all the necessary information on the samples to be analyzed and how the GC/MS system is to analyze them. The sample extracts are loaded onto the autosampler (ALS) tray. Their position is verified by checking them against the ALS number on the sequence. This batch analysis will be performed automatically over the 12-hour period.

10.2.3.3. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.3. Data Processing

10.3.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled. Chrom's auto-processing system queries TALS (LIMS) for each sample's processing parameters (including target compounds lists) and downloads the required processing methods from LIMS to analyze data. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.3.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.3.3. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW 8270E are listed in Table 21.

10.4. Interpretation and Qualitative Identification: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The

characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.4.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.4.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.4.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.4.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.4.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.4.1.7. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.4.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.4.2.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.4.2.2. The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.4.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

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

10.4.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.2.6. If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc.).

10.5. Data Reporting

10.5.1. Final Report. The Chom data system automatically produces a data report consisting of hardcopy reports corresponding to specific data reporting requirements, which is uploaded to the TALS LIMS System for the report production group.

10.5.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.5.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.5.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.5.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.5.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.5.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

10.6. The low-level calibration standard establishes the reporting limit. All reported data must be at a concentration at or above the low concentration standard. Any quantitative values below the report limit must be qualified as estimated.

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11.0. Calculations/Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method (see the formula in Section 11.3).

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3. See Section 9.2.4 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method (see formula in Section 11.3). For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Internal Standard Calculation:

11.3.1. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2. Solid Samples

$$\text{Concentration } (\mu g/KG) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

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

Where:

A_x = Area characteristic ion for the compound (see Table 21)
Ais = Area characteristic ion of associated internal std (See Table 21)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.4 (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Calculation of Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level

11.9. Calculation of Percent (%) Error

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount

11.10. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the Eurofins corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-

up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3.4 Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0. Pollution Control

13.1 It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

14.1. Pollution Prevention

14.2.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Laboratory Manual, Physical/Chemical Methods, Revision 5, July 2014..

15.2. United States Environmental Protection Agency, “Method SW8270E, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Update IV, Laboratory Manual, Physical/Chemical Methods, Revision 6, June 2018.

15.3. United States Environmental Protection Agency, "Method SW8000D: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012..

15.4. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5. Eurofins Edison SOP No. ED-ORP-002, *SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel,* current revision.

15.6. Eurofins Edison SOP No. ED-ORP-043, *SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270,* current revision.

15.7. Eurofins Edison SOP No. ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW3546, current revision.*

15.8. Eurofins Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.9. Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.10. Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.11. Eurofins Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision.

15.12. Eurofins Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.13. Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision.

15.14. Eurofins Edison SOP No. ED-ORP-001, *Extraction of Semivolatile Organic Compounds in Water, EPA Method 625.1,* current revision.

15.15. Eurofins Edison SOP No. ED-GEN-022, *Training,* current revision.

15.16. Eurofins Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.17. Eurofins Corporate Quality SOP No. CA-Q-S-006, *Detection and Quantitation Limits,* current revision.

16.0. Method Modifications:

Method 8270E requires the DFTPP tune standard to be analyzed once prior to an ICAL

and not daily prior to sample analysis. Until such time as 8270D is removed from lab capabilities and in order to satisfy both 8270D and 8270E The laboratory will analyze the DFTPP tune daily, prior to QC and sample analysis. The laboratory will use the tighter criteria from Methods 8270C/8270D for tune evaluation, rather than the criteria suggested in Table 3 of Method 8270E.

17.0. Attachments

Attachment 1 Poor Performing Analytes

18.0. Revision History

- Revision 10, date 10/18/2022
    - Updated to Eurofins branding throughout.
    - Section 8.1: updated to include procedure for sampling chlorinated water sources.
    - Section 9 updated throughout to include ICAL evaluation with Relative Standard Error (RSE).
    - Section 11.8: added formula for calculation of Relative Standard Error (RSE)

- Revision 9, date 03/15/2021
    - Updated as needed to reflect 1,4-dioxane RL of 0.2 ug/l.
    - Updated Tables 11 and 12 to reflect new low ICAL standard concentration of 1,4-dioxane..

- Revision 8, date 06/29/2020
    - Updated to Eurofins branding.
    - Updated throughout to include 8270E requirements.
    - Removed references to SW846 3550B/C prep methods (no longer in use for this method at Edison lab).
    - Update equipment listed in Section 6.0.Updated analytical column in Section 6.1.2.
    - Updated, deleted and renumbered tables as required.
    - Made extensive updates to Standards (sources and preparation) in Section 7.2.
    - Removed all references to Aromatic Amines. Deleted all tables specific to Aromatic Amine analysis. Renumbered remaining tables in document and updated text references.
    - Throughout document clarified tune requirements for 8270E.
    - Following added to Section 9.1.1: For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.
    - Calibration Point Read-back Criteria was added to Section 9.2.4.3. The calculation for percent error was added to Section 11.8.
    - Section 9.2.4.2.3: added following for 8270E: the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%.
    - Section 9.2.4.2.5.6: added ‘The recalculated concentration of the low calibration point should be within ±50% of the standard’s concentration.”

- Section 12.1 revised to reflect the updated MDL procedure.
- Added Section 12.3: annual Lower Limit of Quantitation Verification
- Added Corporate SOP CA-Q-S-006, Detection and Quantitation Limits to references.
- Section 16.0: added a Method Modification regarding tuning check requirements.

- Revision 7, date 06/08/2018

  - Section 2.3: revised to clarify that RVE/LVI is lab standard procedure.
  - Section 9.1.3: removed statement regarding allowance for up to five analytes to recover outside of lab acceptance limits in LCS/LCSD.
  - Section 9.2.4.3: Replace table 'ICV Poor Performers (50-150% Recovery) with expanded list of 'Poor Performing Analytes' in Attachment 1.
  - Added Section 9.2.4.4.5: CCV Poor Performers
  - Corrected number in section 9.2.4.5
  - Added Attachment 1 – Poor Performing Analytes

- Revision 6, date 01/12/2018:

  - Section 7.2.5 included to specify reagent and standard storage conditions.
  - Revised Section 9.1.3 to clarify requirements for specific LCS/LCSD evaluation criteria regarding the # of out of criteria analytes.
  - Revised Section 9.2.4.3 to add 2,4-Dimethylphenol as a poor performing analyte, increased the range for the poor performers to 50-150 and also expanded the guidelines for flagging the ICV outliers.

- Revision 5, dated 09/29/2017:
  - Revised Section 9.1.1 to clarify requirements for surrogate recovery in method blanks.

- Revision 4, dated 08/21/2017:
  - Updated throughout to add a procedure for the analysis of 1,4-dioxane by isotope dilution selected ion monitoring (SIM)
  - Added tables for isotope dilution SIM standards. Renumbered all tables as necessary.
  - Section 7.2.1: added a list of full scan calibration list options.
  - Table 3: Renamed 'Full Scan Stock Standards'.
  - Section 9.2.1: noted that DFTTP applies only to full scan analysis.
  - Section 9.2.3: updated CCV concentrations
  - Added reference to GC/MS Tuning Policy in Section 15.16.

- Revision 3, dated 01/07/2016:
  - Tables 1 and 2: added SIM as option for 1,4-Dioxane.
  - Section 2.3: removed SW3541 (Soxtherm) as option for soils prep (lab has discontinued use of this method). Also removed SW3541 SOP reference from Section 15.0.
  - Tables 19 and 20: added source and prep instructions for 1,4-Dioxane SIM standard. Updated source and prep instructions for 4,6-Dinitro-2-methylphenol.
  - Table 22: added prep instructions for 1,4-Dioxane and 4,6-Dinitro-2-methylphenol

SIM ICV standard.
- Corrected the information in the 'DFTPP Key Ions and Abundance Criteria' table in Section 9.2.1 to match the info found in SW846 8270C.
- Section 10.1.4.2: updated "SIM Parameters" to included ion masses/dwell times for 1,4-Dioxane.

- Revision 2, dated 01/28/2015:
  - Extensively reformatted the SOP. Placed tables that had been in rear of document into the body of the text. Renumbered tables as applicable and fixed text references to tables.
  - Section 1.1, Table 1: Revised table to include all current analytes. Also footnoted those compounds which are currently analyzed by SIM.
  - Section 2.3: added options for extraction of solids by SW846 3456 (Microwave Extraction) and by SW3580A (Waste Dilution) and added SOP references. Deleted reference to SOP ED-ORP-005 (SW3550B – Low Level); Updated Section 15 (References).
  - Section 2.5: added text detailing the RVE/LVI options.
  - Section 2.6: added table which includes all analytes routinely analyzed by SIM.
  - Section 6: updated to include newer GC, MS and autosampler models currently in use.
  - Section 6.1.3: added Zebron ZB column as an option.
  - Section 7.2: extensively revised standards information to reflect switch to Restek standards.
  - Table 3:Added Custom Aromatic Amine Surrogate Standard and revised Table 8 to include initial calibration prep instructions for the Aromatic Amine surrogates.
  - Throughout document: removed references to Target and replaced with Chrom.
  - Section 7.2.1: Added reference to section 10.2.1.2 for LVI.
  - Added Section 7.2.1.3.1 and Table 17A both of which discuss use of Aromatic Amine surrogates.
  - Section 7.2.1.2: Added reference to Tables 9,10 and 11 (ICV Preparation)
  - Section 8.0: Added Sample container and minimum sample size (250 ml) for Reduced volume extraction.
  - Sections 9.1.2, 9.1.3, 9.1.4 and 9.2.4: added statement that certain state regulatory programs have defined recovery limits which, where applicable, are used for spike and calibration evaluations.
  - Section 9.1.2: Deleted sentence "A minimum of 16 spiked analytes are reported to in client reports (the full list is reported at least once during each 2 year period because we employ full spiking list.
  - Section 9.1.4: Added note regarding use of Aromatic Amine Surrogates.
  - Section 9.2.2.2: Added reference to ICV Preparation tables in Section 7.2.
  - Section 9.2.3: added more specific info as to the concentration of the CCVs for all techniques.
  - Section 9.2.4.2.1: Changed to reflect that each analyte should meet minimum RF's, not the average across the calibration. Added LLCCV requirement.
  - Section 10.3.1: added explanation of Chrom's interaction with TALS. Removed references to Target.
  - Section 9.2.4.2.5.5: Added: (or can be noted in the narrative)
  - Section 9.2.4.2.5.6: Revised last sentence to read: "This evaluation can be checked using the Initial Calibration %Drift Report in Chrom."
  - Section 9.2.4.3: Removed 65-135% criteria and added "poor performing" analyte list and associated criteria of 60-140%.

- Section 9.2.4.4.3: Added LLCCV criterion for RFs
- Section 9.2.4.4.4: Added LLCCV criterion for %D
- Section 10.1.4: Updated GC/MS operating conditions for full scan, SIM and DFTPP.
- Section 10.1.4.1: added a table detailing operating conditions for LVI option.
- Table 2: Added 2-ethylaniline, 2,4-dimethylaniline, 3,4-dimethylaniline, 2,3-dimethylaniline, 2,4,5-trimethylaniline and 4-chloro-o-toluidine to Working Standards preparation information.
- Table 25: updated to include all current analytis/surrogates/internal standards and associated ions.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision.

- Revision 1, dated 11/07/2011

  - Section 1.1, Table 1: Added Pentachloronitrobenzene and associated CAS# to the analyte list.
  - Section 7.2.1: Added Pentachloronitrobenzene standard information.
  - Table 2: Added Pentachloronitrobenzene to Working Standards preparation information.
  - Table 4: Added Pentachloronitrobenzene and associated minimum RF.
  - Table 8: Added Pentachloronitrobenzene and associated ions.

- Revision 0, dated 02/22/2011: NEW

Table 21
Characteristic Ions Of Semi-Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
1,1'-Biphenyl	154	153,76
1,2,4,5-Tetrachlorobenzene	216	214, 179
1,2,4-Trichlorobenzene	180	182, 145
1,2-Dichlorobenzene	146	148, 111
1,2-Diphenylhydrazine	77	105, 182
1,3-Dichlorobenzene	146	148, 111
1,3-Dimethylnaphthalene	156	141, 115
1,4-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene d4 **(ISTD)**	152	150, 115
1,4-Dioxane	88	58, 43
1-Methylnaphthalene	142	141, 115
1-Naphthylamine	143	115, 116
2,2'-oxybis[1-chloropropane]	45	77, 121
2,3,4,6-Tetrachlorophenol	232	131, 230
2,3,7,8-TCDD (screen)	320	322, 324
2,3-Dihydroindene		
2,3-Dimethylaniline	106	129

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
2,4,5-Trichlorophenol	196	198, 200
2,4,5-Trimethylaniline	102	55, 56
2,4,6-Tribromophenol **(Surrogate)**	330	132, 141
2,4,6-Trichlorophenol	196	198, 200
2,4-Dichlorophenol	162	164, 98
2,4-Xylidine	121	120, 106
2,4-Dimethylphenol	122	107, 121
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
2-Ethylaniline	106	122,104
2-Fluorobiphenyl **(Surrogate)**	172	171
2-Fluorophenol **(Surrogate)**	112	64
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
2-Naphthylamine	143	115, 116
2-Nitroaniline	65	108, 138
2-Nitrophenol	139	109, 65
2-tert-butyl-4-Methylphenol	149	121, 91
2-Toluidine	107	106, 77
3,3'-Dichlorobenzidine	252	254, 126
3,4-Dimethylaniline	106	129, 127
3,5-Di-tert-butyl-4-Hydroxytol	205	220, 145
3-Nitroaniline	138	108, 65
4,6-Dinitro-2-methylphenol	198	51, 105
4-Bromophenyl phenyl ether	248	250, 141
4-chloro-2-methylaniline	106	144, 142
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	127	129
4-Chloroaniline-d4 **(Surrogate)**	131	133
4-Chlorophenyl phenyl ether	204	206, 141
4-Methylphenol	108	107
4-Nitroaniline	138	108, 65
4-Nitrophenol	139	109, 65
Acenaphthene	154	153, 152
Acenaphthene d10 **(ISTD)**	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Aniline	93	66
Aniline-d5 (**Surrogate**)	98	71,42
Anthracene	178	176, 179
Atrazine	200	173,215
Benzaldehyde	77	105,106
Benzidine	184	92, 185

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic Acid	122	105, 77
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-ethylhexyl)phthalate	149	167, 279
Bisphenol-A	213	228, 119
Butyl benzyl phthalate	149	91, 206
Caprolactam	113	55,56
Carbamazepine	193	236, 135
Carbazole	167	166, 139
Chrysene	228	226, 229
Chrysene d12 **(ISTD)**	240	120, 136
Coumarin	146	118, 63
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Diethylphthalate	149	177, 150
Dimethylphthalate	163	194, 164
Di-n-butylphthalate	149	150, 104
Di-n-octylphthalate	149	167, 43
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95,138
Kepone	272	237, 355
N,N-Dimethylaniline	120	122, 104
Naphthalene	128	129, 127
Naphthalene d8 **(ISTD)**	136	68
n-decane	43	57
Nitrobenzene	77	123, 65
Nitrobenzene-d5 **(Surrogate)**	82	128, 54
N-Nitrosodimethylamine	42	74, 44
N-Nitroso-di-n-propylamine	170	42,101,130
N-Nitrosodiphenylamine	169	168, 167
n-Octadecane	57	43, 85
o-Toluidine-d9 **(Surrogate)**	114	112, 42
Pentachloronitrobenzene	237	214,295
Pentachlorophenol	266	264, 268

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Perylene d12 **(ISTD)**	264	260, 265
Phenanthrene	178	179, 176
Phenanthrene d10 **(ISTD)**	188	94, 80
Phenol	94	65, 66
Phenol-d5 **(Surrogate)**	99	42, 71
Phenyl ether	170	77, 115
Pyrene	202	200, 203
Pyridine	79	52, 51
Terphenyl-d14 **(Surrogate)**	244	122, 212

Attachment 1
Poor Performing Compounds

1,2,4,5-Tetrachlorobenzene
1,4-Dioxane
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2-Chloroaniline
2-Naphthylamine
3&4-Methylphenol
3'3-Dichlorobenzidine
4,6-Dinitro-2-methyl- phenol
4-Chloroaniline
4-Nitrophenol
Aniline
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Diphenylamine
Hexachlorocyclopentadiene
Hexachloroethane
n-Decane
n-Nitrosodimethylamine
o,o,o-Triethylphosphorothioate
o-Toluidine
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

These analytes are exempt from the ICV and CCV criteria as detailed in this SOP

Title: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) by SW846 Method 8260D

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 USEPA SW846 Method 8260D is used for the determination of volatile organic compounds in a variety of aqueous and solid matrices by purge and trap gas chromatography (GC)/mass spectrometery (MS). The methods are applicable to the compounds listed in Table 1 (below). Actual target compound lists are determined through regulatory or project specifications. Method performance criteria for each target analyte will be determined prior to sample analysis.

1.1.2 This SOP also describes the optional procedure for analyses of compounds using 8260D Selected Ion Monitoring (SIM). SIM analyses is specific to target compounds: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-Dioxane. Benzene and Chloroform if needed.

Table 1: Method Analytes

Compound	CAS #	Compound	CAS #
1,1,1,2-Tetrachloroethane	630-20-6	cis-1,2-Dichloroethene	156-59-2
1,1,1-Trichloroethane	71-55-6	cis-1,3-Dichloropropene	10061-01-5
1,1,1-Trifluoro-2,2-dichloroethane	306-83-2	Cyclohexane	110-82-7
1,1,2,2-Tetrachloroethane	79-34-5	Cyclopentene	142-29-0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Dibromomethane	74-95-3
1,1,2-Trichloroethane	79-00-5	Dichlorobromomethane	75-27-4
1,1-Dichloroethane	75-34-3	Dichlorodifluoromethane	75-71-8
1,1-Dichloroethene	75-35-4	Dichlorofluoromethane	75-43-4
1,1-Dichloropropene	563-58-6	Dimethylnaphthalene (total)	28804-88-8
1,1-Difluoroethane	75-37-6	Epichlorohydrin	106-89-8
1,2,3-Trichlorobenzene	87-61-6	Ethanol	64-17-5
1,2,3-Trichloropropane **(1)**	96-18-4	Ethyl acetate	141-78-6
1,2,3-Trimethylbenzene	526-73-8	Ethyl acrylate	140-88-5
1,2,4,5-Tetramethylbenzene	95-93-2	Ethyl ether	60-29-7
1,2,4-Trichlorobenzene	120-82-1	Ethyl methacrylate	97-63-2
1,2,4-Trimethylbenzene	95-63-6	Ethylbenzene	100-41-4
1,2-Dibromo-3-Chloropropane **(1)**	96-12-8	Ethylene Dibromide **(1)**	106-93-4
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	Hexachlorobutadiene	87-68-3
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	Hexane	110-54-3
1,2-Dichlorobenzene	95-50-1	Indan	496-11-7
1,2-Dichloroethane	107-06-2	Iodomethane	74-88-4
1,2-Dichloroethene, Total	540-59-0	Isobutyl alcohol	78-83-1
1,2-Dichloropropane	78-87-5	Isopropyl acetate	108-21-4
1,3,5-Trichlorobenzene	108-70-3	Isopropyl alcohol	67-63-0
1,3,5-Trimethylbenzene	108-67-8	Isopropyl ether	108-20-3
1,3-Dichlorobenzene	541-73-1	Isopropylbenzene	98-82-8
1,3-Dichloropropane	142-28-9	Methacrylonitrile	126-98-7
1,3-Dichloropropene, Total	542-75-6	Methyl acetate	79-20-9

Compound	CAS #	Compound	CAS #
1,4-Dichlorobenzene	106-46-7	Methyl acrylate	96-33-3
1,4-Dioxane **(1)**	123-91-1	Methyl methacrylate	80-62-6
1-Chloropropane	540-54-5	Methyl tert-butyl ether	1634-04-4
2,2,4-Trimethylpentane	540-84-1	Methylcyclohexane	108-87-2
2,2-Dichloropropane	594-20-7	Methylene Chloride	75-09-2
2,4,4-Trimethyl-1-pentene	107-39-1	Methylnaphthalene (total)	1321-94-4
2-Butanone (MEK)	78-93-3	Monochloropentafluoroethane	76-15-3
2-Chloro-1,3-butadiene	126-99-8	m-Xylene & p-Xylene	179601-23-1
2-Chloroethyl vinyl ether	110-75-8	Naphthalene	91-20-3
2-Chloropropane	75-29-6	n-Butanol	71-36-3
2-Chlorotoluene	95-49-8	n-Butyl acetate	123-86-4
2-Hexanone	591-78-6	n-Butyl acrylate	141-32-2
2-Methyl-1,3-butadiene	78-79-5	n-Butylbenzene	104-51-8
2-Methyl-2-propanol	75-65-0	n-Heptane	142-82-5
2-Nitropropane	79-46-9	n-Propyl acetate	109-60-4
2-Octanol	123-96-6	N-Propylbenzene	103-65-1
2-Octanone	111-13-7	o-Xylene	95-47-6
4-Chlorotoluene	106-43-4	p-Diethylbenzene	105-05-5
4-Ethyltoluene	622-96-8	Pentane	109-66-0
4-Isopropyltoluene	99-87-6	Propene	115-07-1
4-Methyl-2-pentanone (MIBK)	108-10-1	Propionitrile	107-12-0
Acetaldehyde	75-07-0	sec-Butylbenzene	135-98-8
Acetone	67-64-1	Styrene	100-42-5
Acetonitrile	75-05-8	Tert-amyl methyl ether	994-05-8
Acrolein	107-02-8	Tert-butyl ethyl ether	637-92-3
Acrylonitrile	107-13-1	tert-Butylbenzene	98-06-6
Allyl chloride	107-05-1	Tetrachloroethene	127-18-4
Amyl acetate (mixed isomers)	628-63-7	Tetrahydrofuran	109-99-9
Benzene **(1)**	71-43-2	Toluene	108-88-3
Benzyl chloride	100-44-7	Total BTEX	STL00431
Bromobenzene	108-86-1	trans-1,2-Dichloroethene	156-60-5
Bromoform	75-25-2	trans-1,3-Dichloropropene	10061-02-6
Bromomethane	74-83-9	trans-1,4-Dichloro-2-butene	110-57-6
Butadiene	106-99-0	Trichloroethene	79-01-6
Butyl Methacrylate	97-88-1	Trichlorofluoromethane	75-69-4
Camphene	79-92-5	Vinyl acetate	108-05-4
Camphor	76-22-2	Vinyl chloride	75-01-4
Carbon disulfide	75-15-0	Xylenes, Total	1330-20-7
Carbon tetrachloride	56-23-5	1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1
Chlorobenzene	108-90-7	1,4-Dioxane-d8 **(ISTD)**	17647-74-4
Chlorobromomethane	74-97-5	2-Butanone-d5 **(ISTD)**	24313-50-6
Chlorodibromomethane	124-48-1	Chlorobenzene-d5 **(ISTD)**	3114-55-4
Chlorodifluoromethane	75-45-6	Fluorobenzene **(ISTD)**	462-06-6
Chloroethane	75-00-3	TBA-d9 **(ISTD)**	25725-11-5
Chloroform **(1)**	67-66-3	1,2-Dichloroethane-d4 **(Surrogate)**	17060-07-0
Chloromethane	74-87-3	4-Bromofluorobenzene **(Surrogate)**	460-00-4

Compound	CAS #	Compound	CAS #
Chlorotrifluoroethene	79-38-9	Dibromofluoromethane **(Surrogate)**	1868-53-7
Chlorotrifluoromethane	75-72-9	Toluene-d8 **(Surrogate)**	2037-26-5
2-ethylhexyl acrylate	103-11-7		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).

1.1.3 Method 8260D can be used to quantitate most volatile organic compounds that have boiling points below 200°C, and that are insoluble or slightly soluble in water. Water-soluble compounds can be included in this method, but quantitation limits will be higher due to poor purging efficiency.

1.1.4 The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve (1 ug/l for most compounds). For a complete list of method detection limits (MDLs) and RLs, please see reference the current TALS (LIMS) active Method Limit Group database.

1.1.5 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 20 (*Test Methods and Method Validation*) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

1.1.6 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 Method 8260D is used to determine volatile organic compounds in aqueous, non-aqueous and solid matrices. Sample preparation techniques vary, depending on the matrix and the level of contamination expected. Purge and trap techniques are used to introduce the sample to the GC/MS system. Refer to Eurofins Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision and ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

2.2 All soil samples are screened by GC/FID static headspace or MS analysis to provide the analyst with appropriate initial dilution factors. Aqueous samples without historical results are also screened. For additional details see Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

2.3 An aliquot of sample containing internal standard and surrogate spiking solution is purged with nitrogen in a closed sparging vessel. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent column is heated and backflushed with helium to desorb the volatiles onto a gas chromatograph column.

2.4 Analytes eluted from the capillary chromatograhy column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

2.5 For aqueous VOA samples submitted for New Jersey Groundwater Quality Standard (NJ GWQS) evaluation, a full scan analysis is initially performed using the 8260 methodology No further analysis by SIM is required if all of the following compounds are present above the full scan RL: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-dioxane, chloroform, vinyl chloride and benzene. If any of these compounds are undetected in the undiluted, full scan analysis, the sample must be analyzed via 8260D SIM for those compounds.

2.6 In order to meet lower reporting limits of 0.5ug/L for most analytes, 2.5 ug/L for ketones and generally lower limits for other non-routine analytical compounds, samples must be analyzed against an initial calibration with a low point at those levels. The corresponding TALS login method for low level aqueous analysis is 8260_LL. See Table 3b for initial calibration levels and spike amounts.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method is susceptible to contamination from a number of sources, including organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatiles analytical laboratory is housed in a separate building, away from the organic extraction lab area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatiles laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 Trip Blanks are shipped to clients with aqueous bottle ware as requested. The purpose of the trip blank is to detect and identify any VOC contamination of the samples while in transit to and from the lab. The blank is created at the laboratory by completely filling the volatile vial container with lab grade organic free deionized water and sealing the container Trip Blanks accompany bottle ware and samples through the sampling, storage and analysis stages as a check on contamination that may occur at these points.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.7 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.4 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.5 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551, 4100 Automatic Sampler/4660,4760 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4660,4760 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;
- Archon Autosampler/EST Encon concentrator.
- Archon/EST Evolution

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.3 An OI analytical purge trap #10 is used for the OI 4560,4660 and 4760 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: 30M long x 0.25mm ID, 1.4um film thickness, 20M x 0.18mm x 1um DB-624 and 20M long x 0.18 mm ID Restek Rtx-VMS capillary column with 1um film thickness or similar phase.

6.1.8 Mass Spectrometer (Agilent 5973/5975/5977): scanning from 35-260 amu every 0.9 seconds, utilizing 70 volts (nominal) electron energy in the electron ionization mode and producing a mass spectrum which meets all EPA performance criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

6.1.9 GC/MS Interface: transfer lines heated to 180°C .

6.1.10 Data system: HP Chemstation II for data acquisition and Eurofins Chrom for data processing.

6.2 Supplies

- Microsyringes: 10 ul to 1000 ul.
- Syringes: 5 ml to 25 ml gas-tight.

- Injection port liners: HP 18740-80200 or equivalent
- Volumetric flasks: Class "A" glassware, 5 ml to 500 ml.
- VOA vials: 20-ml and 40-ml glass with PTFE – faced septum.
- Vials: 2-ml amber glass with screw cap with Teflon-faced septa.
- Top loading analytical balance.
- Spatula: Narrow, stainless steel.
- Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.1.1) Reagent water is obtained from Millipore system. Other methods of preparing reagent water are acceptable, provided that the water produced meets method blank criteria.

7.1.2 Methanol: Ultra Resi-Analyzed, purge and trap grade, purchased from JT Baker or equivalent. (Cat # 9077-02)

7.1.2.1 Each lot of methanol is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 **Calibration Standards** Stock target compound analytical standard solutions are purchased mainly from Restek, Supelco, Inc, Absolute Standards and Spex although standards of similar quality from other suppliers may be substituted as required. Standards noted with an asterisk (*) are custom mixes made especially for Eurofins Edison.

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 / Std #3 Gases*	2500 ppm	Restek	569722
8260 List 1 / Std #3 Gases – (SS)*	2500 ppm	Restek	569722 sec
8260 List 1 / Std #1 MegaMix*	1250-62500 ppm	Restek	569720
8260 List 1 / Std #1 MegaMix (SS)*	1250-62500 ppm	Restek	569720 sec
8260 List 1 / Std #2 Ketones *	12500 ppm	Restek	569721
8260 List 1 / Std #2 Ketones * (SS)	12500 ppm	Restek	569721 sec
8260 List 1 / Std #5 Acrolein *	20,000 ppm	Restek	568720
8260 List 1 / Std #5 Acrolein (SS)	20,000 ppm	Restek	568720 sec
8260 List 1 /Std #4 2 CEVE *	2500 ppm	Restek	569723

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 /Std #4 2 CEVE (SS) *	2500 ppm	Restek	569723 sec
8260 List 1 /Std #6 Vinyl Acetate *	5000 ppm	Restek	569724
8260 List 1 /Std #6 Vinyl Acetate (SS) *	5000 ppm	Restek	569724 sec
8260 List 2 / Std #1 Additions *	2500-62500ppm	Restek	568725
8260 List 2 / Std #1 Additions (SS) *	2500-62500 ppm	Restek	568725 sec
8260 List 3 / Std #1 Polar Additions *	2500-100000ppm	Restek	568728
8260 List 3 / Std #1 Polar Additions (SS) *	2500-100000 ppm	Restek	568728 sec
VOC Extra Standard 2015 *	2500-5000 ppm	Absolute	98593
VOC Extra Standard 2015 * (SS)	2500-5000 ppm	Absolute	98593
Epichlorohydrin	1000 ppm	Absolute	70377
Acrolein	5000 ppm	Restek	91980
Acrolein *	Neat	Sigma	110221
2-Freon Mix quote # 12258 *	2500ppm	Absolute	12258
2-Freon Mix quote # 12258 * (SS)	2500ppm	Absolute	12258
1,4-Dioxane	Neat	Sigma	360481
Epichlorohydrin	Neat	Sigma	45340
2-Chloroethylvinyl ether	Neat	Sigma	109983
1,4-Dioxane	1000 ppm	Absolute	70373
1,4-Dioxane	10000 ppm	Absolute	92785
Benzene	1000 ppm	Absolute	70025
Chloroform	1000 ppm	Absolute	70076

(1): The separate source for this material is not available as a distinct catalog number. Analyst must ensure that a separate lot of the material is selected and used as required.

An asterisk (*) indicates a custom standard mix.

7.2.1.1. Prepare stock solutions at volumes and concentrations indicated in Table 2 (Working Standards Preparation) by combining the indicated volumes of each stock solution into a volumetric flask corresponding to the total final volume. Dilute to the volume marker with methanol.

7.2.1.2. Prepare individual calibration standards as applicable per Section 9.2.2.1, Table 3, Initial Calibration Standards Preparation, Low Level Soil, Table 3a, Initial Calibration Standards Preparation (Low Level), Aqueous or Table 3B Initial Calibration Standards Preparation, Aqueous.

7.2.1.3. The 'Second Source' standards listed are used in the preparation of the Initial Calibration Verification (ICV) standard (see Tables 4 and 4a for ICV preparation instructions) and the Laboratory Control Standard (LCS) (see Section 9.1.3 and Tables 4 and 4a).

7.2.2 **Surrogate Standards:** Surrogate standard solutions are prepared from the stock solution (2500ppm)

Surrogate Standard Name	Concentration	Vendor	Catalog #
4-Bromofluorobenzene	2500ppm	Restek	567650
Toluene-d8			
1,2-Dichloroethane-d4			
Dibromofluoromethane			

7.2.2.1 A primary surrogate stock solution (2500 ppm each) is prepared from the neat standards as follows:

7.2.2.2 Secondary surrogate standard solutions are prepared at two (2) levels using the 2500 ppm primary stock solution as detailed in the table below:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume in MeOH/Total volume of MeOH
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	250ppm	10mL 9.0mL TV/M
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	50ppm	50mL 9.0mL TV/M

7.2.2.3 Methanol/Surrogate solution (2.5ug/mL): For methanol sampling field kits. Prepared by adding 1mL of 2500 ug/ml primary surrogate stock solution (see Section 7.2.2.1) to 1 L purge and trap grade methanol.

7.2.3 **Internal Standards:** Internal Standards Solutions are purchased from Restek:

Standard Name	Concentration	Vendor	Catalog #
8260 Internal Standard Mix: *Chlorobenzene-d5 *1,4-Dichlorobenzene-d4 *Fluorobenzene *1,4-Dioxane-d8 *TBA-d9	250-5000ppm	Restek	567649

7.2.4 **Internal Standard/Surrogate Mix (125 ppm each):** A solution containing both Internal Standards and Surrogates at 125 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500

ppm surrogate stock solution prepared in Section 7.2.2.1 and the 2500 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 20ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (125 ppm) For Aquatek Autosampler	2500 ppm Surrogate Mix	1.0ml	125 ppm each component
	250 Internal Std Mix	10 ml	

7.2.5 **Internal Standard/Surrogate Mix (SIM) (2.5/50 ppm each):** A solution containing both Internal Standards and Surrogates at 25 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500 ppm surrogate stock solution prepared in Section 7.2.2.1 and the 250 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 10ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (25 ppm) (SIM)	2500 ppm Surrogate Mix	10ul	2.5/50 ppm each component
	250 Internal Std Mix (Restek)	10ul	
1,4-Dioxane-d8	10000 ppm	50ul	

7.2.6 **GC/MS Instrument Performance Check (BFB):** The instrument performance check solution consists of 4-Bromofluorobenzene in addition to the other three surrogates in methanol. Prepare the solution at **50ppm as specified in section 7.2.2.2**. Assign an expiration date of 6 months.

7.2.7 All standards preparation information must be logged into the TALS Reagent Module. All pertinent information must be entered: Date prepared, Lot #'s, Expiration dates, Solvents used, Lab Lot # (expiration date), Manufacturer and Verification signature. Additionally, all prepped standards are typically given a unique Lot# and all information pertaining to standard preparation is entered into the GC/MS VOA Standard Preparation Log Book. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.8 Please refer to Eurofins Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision. For Method 8260D:

- Shelf Life of Standard:
  - Stock standards (Non-gases) - 6 months after opening vendor stock of up to 2000ppm, 3 years for 10,000ppm, 5 years for over 50,000ppm, or manufacturer's expiration date whichever comes first.
  - Stock Standards (Gases) - 2 months after opening vendor stock, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Non-gases) – 6 months after preparation, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Gases) – 1 week from the date of preparation for 50ppm and 2 weeks for 500ppm, or manufacturer's expiration date whichever comes first.
  - Daily Calibration Standards – 24 hours after preparation.

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- Storage Requirements:
  Aqueous standards are stored at 4°C and Methanol standards are stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

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

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass 40 ml vials	40 mLs	HCl, pH < 2; Cool 4 °C ± 2°C	14 Days / preserved 7 Days / unpreserved	SW846 Method 5030
Waters	Glass 40 ml vials	40mLs	TSP,pH>11 Cool 4 °C ± 2°C	14 Days / preserved	SW846 Method 5030
Soils (Low)	Encore or Terracore (40 ml vials)	5 grams in 5 mls DI H_2O	Frozen Stored -7°C to -20°C	14 Days	SW846 Method 5035A

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils (Med)	Encore or Terracore (40 ml vials)	5 grams in 10 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030
Soils (High)	Glass (Lab Prepared Kits)	10 grams in 25 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030

8.1.1 There are several methods of sampling soil. The recommended method is to take samples using an EnCore™ sampler or using a Terra Core™ sampling kit. At specific client request, unpreserved soil samples in 4oz jars may be accepted. For EnCore and Terra Core sampling, a separate jar is required for percent solids/moisture determination, unless one is supplied for another analysis.

8.1.2 For EnCore™ samplers, the 5g sample is extruded into a pre-weighed 40mL vial containing 5mL of methanol (medium level analysis) or reagent water (for low level, <50 µg/kg, analysis). The exact samples weight is determined as the difference between the vial + preservative weight and weight after the sample is added.

- Samples must be transferred (extruded from the sampler) and preserved within 48 hours of sampling.
- Water preserved samples are then frozen at <10°C. Methanol preserved samples may be stored at > 0.0 °C but < 6° °C or frozen.
- Methanol preserved samples are shaken for at least 2 minutes, and a portion of the methanol extract after settling may be transferred to a smaller Teflon-lined capped vial for storage below 6 °C
- Normally one (1) medium level and two (2) low-level samples are taken and preserved.
- One vial with a clean matrix of each preservation type is prepared at the same time as samples, to be used for LCS analysis. Spikes are not added until the time of analysis.
- Samples are spiked with internal standards and surrogates at the time of analysis.

8.1.3 Terra Core™ sampling kits are pre-preserved for use and immediate samples preservation in the field. Kits are shipped that include one (1) methanol preserved and two (2) reagent water preserved vials, along with a 4oz jar for solids/moisture analysis volume.

- Terra Core™ vials are immediately placed in the freezer (<-10°C) upon receipt at the lab. Methanol preserved vials are shaken for at least two (2) minutes to break up the solid and create the methanol extract.
- Terra Core™ vials are labeled with the weight of the vial and preservative. The vials are re-weighed prior to analysis to determine the weight of the solid sample added. It is important that labels NOT

be added to these vials prior to weighing, because the weight of the label will add to the sample weight. Vials may be marked with indelible marker, or placed in a labeled, sectioned box until ID labels can be added after weighing.

8.2 Unpreserved soils - At client request, unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. A 5g portion of the sample is transferred to a 40mL vial and mixed with reagent water and/or methanol for analysis. Since this procedure is not compliant with SW5035A an NCM and case narrative statement describing the non-conformance must be included with any resulting data reported to the client.

8.3 Aqueous samples are stored in 40mL glass vials with Teflon lined septa at >0 and ≤ 6.0°C. Vials are required to have no headspace larger than a small pea.

8.3.1 Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources

8.3.2 Regulatory requirements for 2-Chloroethyl vinyl ether:

- 2-Chloroethyl vinyl ether: The stability of this compound is reduced when subjected to low pH, therefore samples for analysis to include 2-CEVE must be taken without acid preservation. Unpreserved samples must be analyzed within 7 days.

- SW846 Update V removed special preservation requirements for Acolein and Acylonitrile. These compounds may be analyzed for using a preserved sample vial.

8.4 Soil samples and water samples preserved to pH <2 with HCl have a maximum holding time is 14 days from sampling until the sample is analyzed. If water samples are known to be unpreserved, the holding time is 7 days from sampling to analysis.

8.4.1 Preserved water samples are checked to confirm the preservation pH AFTER analysis because the vials must not be opened prior to analysis. If the pH is found to be >2, this must be addressed in the case narrative.

8.5 Medium level solid methanol extracts, if taken at the time of preservation, are aliquoted into 4 mL glass vials with Teflon lined caps and stored at > 0.0 °C but ≤6.0°C or frozen. The extracts are stored with minimum headspace.

8.6 Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After 1-2 weeks, the storage blanks are removed and analyzed. Additional details can be found in Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every samples	Response within -50% to +100% of CCV

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

9.1.1. **Method blanks** are analyzed every 12 hours immediately after successful calibration verification (ICV and CCV) and before any samples are analyzed during the 12 hour clock. Analyze the blank in the same manner as the associated samples.

9.1.1.1. Prepare an aqueous blank by filling a 40 mL vial with reagent water and placing it in the autosampler. The autosampler will add the internal standard and/or surrogate standard.

9.1.1.2. Prepare a medium or high level blank in a 50 mL volumetric flask by adding 1.0 mL of purge and trap grade methanol to reagent water and bringing up to volume with the reagent water. The appropriate volume of this mix is added to the purge vessel. The autosampler will automatically internal standard and/or surrogate standard.

9.1.1.3. Prepare a low- level soil blank in a 40 ml VOA vial by adding a magnetic stir bar and 5 ml of reagent water and placing the vial in the autosampler tray. An additional 5mL of reagent water plus 1uL of 250ppm Internal Standard/Surrogate Mix (see Section 7.2.4) will be added by the Archon prior to purging.

9.1.1.4. To be considered acceptable, the method blank must not have any target analytes above the reporting limit. For method 8260D method blank is acceptable when target analyte concentrations are less than one-half of the reporting limit. Method blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected or sample

concentrations/responses are >10x the blank If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Re-analysis is not necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed. Method blanks, trip blanks and other field blanks must be carried out through all stages of sample preparation and analysis.

9.1.1.5. Surrogate recoveries for the method blank must be within the laboratory generated limits. (Method 8260D requires the use of a minimum of three (3) surrogates. Since we are spiking with four (4) surrogates, either 1,2-Dichloroethane-d4 or dibromofluoromethane can be recovered outside of control limits without corrective action) .Internal standard area counts in the method blank must be within method specified limits. If any surrogate or internal standard is outside the limits, the method blank must re-analyzed.

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch which may contain up to 20 samples, and additional samples can be added to the batch for 14 days after the first sample was analyzed). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared (as described in Section 9.1.2.1) concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria which are updated annually. For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database.

9.1.2.1. Prepare the MS/MSD as follows:

9.1.2.1.1 Low Level Soil: The low level soil MS/MSD is prepared as detailed in the following table. This is prepared in duplicate (one for the MS, the other for the MSD) in a 5 ml syringe filled with reagent water. Once prepped the solution is added to separate 40 ml vials each containing 5 gram aliquots of the sample to be spiked :

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
Gas Mix Li	50ppm	2	20

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
8260 combined	50ppm	2	20
Acrolein	500 ppm	3	300
Propenes	50ppm (varied)	2	20 (varied)
Freons	50 ppm	2	20

9.1.2.1.2 Aqueous Samples: The MS/MSD for aqueous samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with an aliquot of sample to be spiked. Once prepped the solution is poured into a 40 ml VOA vial and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Sample	Final Concentration(ug/L)
Gas Mix Li	50ppm	20	20
8260 combined	50ppm	20	20
Acrolein	500 ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50	20	20

9.1.2.1.3 Medium & High Level Soils: The MS/MSD for medium/high level soils is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with reagent water which has been previously spiked with the methanol sample extract. Once prepped the solution is poured into a 40 ml VOA vial, the and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Reagent Water containing sample methanol extract	Final Concentration (ug/L)
Gas Mix Li	50ppm	20	20
8260 conbined	50ppm	20	20
Acrolein	500ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50 ppm	20	20

9.1.2.1.4 SIM: The MS/MSD for SIM samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 100 ml

volumetric flasks filled with an aliquot of sample to be spiked. Once prepped, two separate 10ml solution is poured into 40 ml VOA vials, 2ul of SIM IS/S is then added to each vial and loaded onto the purge and trap autosampler:

Standard Solution	Concentration	Volume of Standard Added to 100 ml of Sample (ul)	Final Concentration (ug/L)
8260SIM Mix1	10ppm	0.5	0.05
1,4-Dioxane	50ppm (varied)	10	5
Benzene/Chloroform	10ppm	0.5	0.05

9.1.2.2. An Laboratory Control Sample (LCS) /Laboratory Control Sample Duplicate (LCSD) may be substituted for the MS/MSD if insufficient sample volume is available (see Section 9.1.3).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be prepared analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (see For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference. If the LCS recovery results are outside the method specified, the LCS is reanalyzed. If, upon reanalysis, the LCS is it is still outside of limits the entire batch must be reanalyzed. For 8260D, when an LCS is prepared in the same manner as CCV, the same standard can be used as both the LCS and CCV.

9.1.3.1 For LCS preparation instructions please refer to Section 9.1.2.1 for low level soil introduction technique (note: use reagent water only, no solid matrix is used when preparing the LCS) and Sections 9.1.2.1.2 and 9.1.2.1.3 as applicable for aqueous/medium or high level solids introduction (note: use reagent water only, no sample or sample extract is used when preparing the LCS).

9.1.3.2 The LCS for SIM samples is prepared as detailed in the following table. This is prepared in a 200 ml volumetric flasks filled with organic free reagent water. Once prepped, 10ml of the solution is poured into a 40 ml VOA vial and 2ul IS/SS added manually and loaded onto the purge and trap autosampler

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260 Mix1	10ppm	1	0.05
1,4-Dioxane	50ppm	20	5
Benzene/Chloroform	10ppm	1	0.05

9.1.3.3 A Laboratory Control Sample Duplicate (LCSD) is analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a four (4) component surrogate standard mix (see Section 7.2.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database).

9.1.4.1. Surrogate recovery limits are lab generated and are updated annually.

9.1.4.2. Surrogate recoveries are calculated for the blank, samples, and QC samples. Surrogate recovery is calculated as:

$$\frac{\text{Concentration found}}{\text{Concentration added}} \times 100 = \%\ \text{RECOVERY}$$

9.1.4.3. If the surrogate recoveries of any blank, sample, or QC sample fails to meet the current recovery criteria, the sample must be re-analyzed. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary. Method 8260D requires the use of a minimum of three (3) surrogates. As we spike with four (4) surrogates, one can be recovered outside of control limits without corrective action.

9.1.5. Internal Standards: All samples, blanks, standards and QC samples are spiked with a five (5) component internal standard mix (See Section 7.2.3). The response (area count) and retention time of each internal standard in all samples, standards, blanks and QC samples are monitored.

9.1.5.1. The internal standard responses must be within -50 +100% of its corresponding internal standard in the mid-level calibration standard or the active calibration curve. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.1.5.2. Internal standard retention time is evaluated immediately after acquisition. The retention times of the internal standards must be within ±30 seconds of the internal standards from the mid point standard of the initial calibration or the calibration verification standard. Any blank, sample, or QC sample that fails to meet these criteria must be re-analyzed.

9.2 Instrument QC

9.2.1 GC/MS Instrument Performance Check (BFB): The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection or purging of 50ng of 4-Bromofluorobenzene (BFB) meets the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all BFB key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. For method 8260D tune checks are only required prior to initial calibration. (**NOTE**: see Method Modifications in Section 16.0).

BFB Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
50	15.0-40.0 percent of the base peak
75	30.0-60.0 percent of the base peak
95	Base peak, 100% relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	Greater than 50% of the base peak
175	5.0-9.0 percent of mass 174
176	Greater than 95.0% but less than 101% of mass174
177	5.0-9.0 percent of mass 176

9.2.1.1. The BFB mass spectrum may be evaluated using one of the procedures listed below. The spectrum may be background subtracted using a single peak no more than 20 scans before the peak apex. The BFB spectrum must meet the technical acceptance criteria listed in the table above:

- A single scan on the peak;
- An average of the peak;
- Use of three scan averaging and background subtraction techniques. Select the scan at the BFB peak apex, add +1 scan from the apex and -1 scans from the apex;

9.2.1.2. BFB parameter settings are stored in a tune file, which ill be used in all subsequent analysis of standards and samples.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. **Initial Calibration:** The initial calibration range consists of a five-point concentrations (six points for second order regression) of analytical standards prepared as described in Tables 3, 3A and 3B as applicable (attached). The initial calibration range must be analyzed only after the BFB instrument performance check has met the criteria in Section 9.2.1. A separate initial calibration range is analyzed for each sample introduction technique. The last initial calibration standard may be used to be the start of the 12 hour clock for samples analyzed after initial calibration. Verify closely eluting isomers resolution in the mid-point concentration of the ICAL. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is less than or equal to 50% of the average of the two peak heights. This should also be checked in the daily CC's.

9.2.2.2. If analysis by the SIM technique is required, prepare calibration standards for, Vinyl Chloride, Chloroform, Benzene 1,2-dibromoethane, 1,2,3-Trichloropropane and 1,2-dibromo-3-chloropropane at concentrations of 0.02, 0.04, .05, 0.10, 0.20, 0.50, 1.0 and 2.0 ppb; 1,4-Dioxane at 0.4, 1, 5, 10, 20, 30, 40 and 50ppb. See Table 5 that summarizes the preparation information.

9.2.2.3. **Initial Calibration Verification (ICV):** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.1.3 and Tables 4 and 4a (full scan) and Table 6 (SIM) (attached). The ICV must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3 **Continuing Calibration Verification (CCV):** A approximately mid-point (20ug/ml and 0.050/5ug/ml for SIM) Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the BFB instrument performance check. BFB is not a requirement for 8260D CCV verification. The CCV is prepared as detailed in Section 7.2.1.1 and Table 3 (attached).

9.2.4 **Calibration Acceptance Summary**

9.2.4.1. **Retention Time:** The relative retention times of each compound in the five calibration standards must agree within 0.06 relative retention time units.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. After the initial calibration range has been analyzed as detailed in Section 10.3.3 the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

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

Where:
A_x = Area characteristic ion for the compound (see attached Table 7)
Ais = Area characteristic ion of internal standard (see attached Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound using the five or six RFs from the initial calibration range.

9.2.4.2.2. The average RFs of the target analytes listed in the table below must meet the indicated minimum RF criteria:

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone *	0.050
Carbon disulfide	0.100
Methyl Acetate *	0.005
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone *	0.050
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone *	0.050
Toluene	0.400

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone*	0.050
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Note: Alternate ions chosen for the analytes in the table above may result in lower than recommended value

* These values are lower than method recommended values.

9.2.4.2.3. Any individual analyte that fails the minimum response factor above must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. The low level CCV would normally be analyzed immediately after the mid-level CCV

9.2.4.2.4. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

The % RSD of the common target compounds listed above must be ≤20% for average RF in order for the calibration range to be acceptable. If more than 10% of the compounds exceed the 20%RSD limit and do not meet the minimum correlation coefficient (0.99) or Relative Standard Error (RSE) of ≤20% for alternative curve fits, appropriate instrument maintenance like source cleaning should be performed. Any compound that do not meet the 20%

RSD or 0.99 correlation coefficient/RSE criteria must be flagged as estimated for detects.

9.2.4.2.5. For all compounds (including those analyzed by SIM): in order to assume linearity, the % RSD of the RRF's for each target analyte must be ≤20%.

9.2.4.2.6. If the above listed criteria is met, the system can be assumed to be linear, sample analysis may begin and the average RF from the initial calibration range may be used to quantitate all samples.

9.2.4.2.7. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.4 Linear regression: Calculate the first order linear regression for any compound which did not meet the 20% RSD criteria the r^2 (Correlation Coefficient) value must be $\geq$ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.4 Quadratic (or second order) regression: may be used if the linear regression correlation coefficient exceeds criteria. Quadratic regression requires the use of a minimum six calibration points. If second order regression calibration is used, , the r^2 (Correlation Coefficient) value must be $\geq$ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.8. If neither of the alternative calibration techniques meets acceptance criteria i.e for more than 10% of the analytes fail both 20%RSE and 0.990 the calibration is not valid. Corrective action must be taken and the initial calibration range reanalyzed.

9.2.4.2.9. Non-detect results for any analyte that fails both 20%RSD/RSE and 0.990 correlation coefficient may be reported without flagging if (and only if) there has been a successful analysis of a LLCCV (CCV at the reporting limit) in the same analytical batch. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative criteria in the method must be met. Flagging of detected analytes results as estimated is discouraged when the 20%RSD/RSE and 0.990 criteria fails. In general no more than one or two of the poorest performing analytes should fail both criteria.

9.2.4.2.10. Due to significant bias to the lower portion of a calibration curve using the linear regression fit model a quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve as if it were an unknown sample (rename the lower point calibration file as a separate data file before re-processing). The results should be within ±30% of the standard's true concentration. This is not required for average RF or quadratic fits. Additionally forcing a linear regression through zero will meet the requirement of not re-fitting. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered 'out of control'. Report those target analyte outliers as estimated when the concentration is at or near the lowest calibration point and/or report to the next reporting level (i.e., the next higher calibration point for the analyte).

9.2.4.2.11. For additional detail refer to Eurofins Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, latest revision.

9.2.4.3. Calibration Point Read-back Criteria: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be < 50%. Relative standard error (RSE) can also be used and must be ≤ 20% for each calibration point. See section 11.10 for the Calculation of the %Error.

9.2.4.4. Initial Calibration Verification (ICV): Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 9.2.2.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed

this criteria as long as their recoveries are within 65-135%. For the poor performers the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.5. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a BFB instrument performance check, and analysis of a calibration verification standard.

9.2.4.4.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of BFB. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For method 8260D, tune verification is not required for daily CCV.

9.2.4.4.1.1 Calibration Verification: Analyze the calibration verification standard immediately after a BFB that meets criteria. For method 8260D, BFB is not needed. Use the mid point calibration standard (20ug/L). **NOTE:** The same sample introduction technique employed for the initial six-point calibration must be used for the calibration verification.

9.2.4.4.1.2 Calculate response factors (RF) for each compound using the internal standard method.

9.2.4.4.1.3 The RFs must meet the minimum RF criteria listed in the table in Section 9.2.4.2.2.

9.2.4.4.1.4 Calculate the % Difference for each response factor in the calibration check standard vs. the response factors from the initial calibration.

9.2.4.4.1.5 If the percent difference/drift (%D) for the compounds listed in the table in Section 9.2.4.2.2 is ≤20%, the initial calibration is assumed to be valid. If the ≤20% D criteria is not met for more than 20% of the compounds in the initial calibration, corrective action/ investigation may be taken. After corrective action, another calibration verification standard may be injected. If the response for the analyte is still not ≤20%, a new initial calibration range must be generated.

9.2.4.4.1.6 For the poor performing compounds listed below that fail the 20%D or 50%D criteria adequate sensitivity may be demonstrated by including a low level standard (LLCCV) in the analytical batch.

Poor Performers	
Acetone	Acrolein
Carbon disulfide	1,4-Dioxane
2-Butanone	Cyclohexane
2-Hexanone	Methyl cyclohexane
4-Methyl-2-pentanone	Benzyl chloride
Chlorodibromomethane	Naphthalene
1,2-Dibromo-3-chloropropane	Cis-Dichloropropene
Bromomethane	Trans-Dichloropropene
Chloroethane	All Alcohols

When samples have non-detects for an analyte that fails the SOP criteria with low recovery a low level CCV must be analyzed in the batch as a demonstration of adequate sensitivity. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. Any sample detects for an analyte that fails the SOP criteria must be flagged as estimated, or detailed in the case narrative. In all cases every effort should be made to re-analyze on an instrument with a passing CCV.

9.2.4.4.1.7 Percent drift is used instead of percent difference in calibrations employing either the linear or second order regression modes.

9.2.4.4.1.8 For the compounds not listed in the table in Section 9.2.4.2.2: No one individual compound of interest may exceed 50%D. For SIM analysis the %D is 20%.

9.2.4.4.1.9 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.4.1.10 Internal standard area response is also evaluated immediately after acquisition. The response (area count) of each internal standard in the calibration verification standard must be within 50% - 100% of its corresponding internal standard in the mid-level calibration standard of the initial calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The instrument operating parameters are set as follows at the beginning of a method of analysis and remain constant throughout the entire analytical procedure

10.1.1.1 Full Scan Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C

Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

10.1.1.2 SIM Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

SIM Parameters:

Group 1
Plot 1 Ion: 51.0/96

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	51.0	100	58.0	100	65.0	100
	67.0	100	70.0	100	88.0	100
	96.0	100	78.0	100	83.0	100
	85.0	100	62.0	100	64.0	100

Group 2
Group Start Time: 6.20
Plot 1 Ion: 82/117

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	82.0	100	107.0	100	109.0	100
	117.0	100				

Group 3
Group Start Time: 8.50
Plot 1 Ion: 75/157

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	75.0	100	95.0	100	150.0	100
	152.0	100	152.0	100	157.0	100
	174.0	100				

10.2. Sample Preparation

10.2.1. Screening: All soil samples extracts must be screened by GC/FID static headspace or MS analysis to provide the analyst with appropriate initial dilution factors. Aqueous samples without historical results are also screened. For additional details see Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

10.2.2. Aqueous Samples: Unopened 40 mls vials with aqueous samples are placed in an Archon autosampler. 1 uL of Internal Standard/Surrogate Mix (see Section 7.2.4) is added by the Archon as the 5 mL of the sample passes through the sample loop.

10.2.3. **Medium or high level soils:** Medium or high level extracts that will be run on an Archon autosampler are prepared in 50mL volumetric flasks. The Archon can be set up to add 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) to each sample as the 5mL portion passes through the sample loop.

10.2.4. **Low level soils:** Low level soils must be run on an Archon autosampler. 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) and 5mL reagent water is added to each sample vial by the Archon immediately before the sample is purged.

10.2.5. **SIM analysis:** Aliquot 10ml of sample and manually add 2ul of 2.5/50ppm of internal standard/surrogate mix. Load to soil section of the autosampler for heated purge.

10.3. Instrument Performance and Calibration Sequence

10.3.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.3.2. Analyze the Instrument Performance Check Standard (BFB) as discussed in Section 9.2.1.

10.3.3. A unique initial calibration is then prepared for each sample introduction technique.:

10.3.3.1 **40 ml VOA Vial (Aqueous/Medium-High Level Soils):** Prepare aqueous calibration standards at six concentration levels for each parameter by adding the volumes of working standards listed in Table 3 to a 50mL volumetric flask of reagent water. Pour the calibration standards into 40mL VOA vials and load into the autosampler tray. If the internal standard is to be added by the Archon/OI autosamplers the addition of internal standard into the 50ml volumetric flaks may be omitted.

10.3.3.2 **40 ml VOA Vial (Low Level Soils):** If the calibration is for low-level soils prepared according to Method 5035AA, the calibration standards must be prepared by adding the volumes of working standards listed in Table 3 into a 5 mL syringe filled with reagent water and pouring the prepared standards into 40 mL VOA vials containing a magnetic stir bar.

10.3.4. Purge the standard for 11 minutes.

10.3.5. After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C for VOCARB, 190°C for #10 and backflushing it with helium.

10.3.6. Begin the GC temperature program and data acquisition.

10.3.7. Re-condition the trap by baking for 12 minutes at 260°C for VOCARB, 210°C for #10.

10.3.8. Cool the trap to (<31°C). The trap is now ready for the next sample.

10.3.9. Transfer data to network, and process using CHROM software.

10.4. Sample Analysis Sequence

10.4.1. Once the initial calibration has been verified by successful analysis of an ICV and Method Blank, analysis of samples may begin.

10.4.2. Samples must be analyzed under the same instrument conditions and using the same injection volume as the calibration standards.

10.4.3. Equilibrate all samples to room temperature prior to analysis.

10.4.4. If the sample concentration exceeds that of the range, the sample must be diluted and re-analyzed.

10.4.5. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.5. Data Processing

10.5.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled in the Chrom system. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.5.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.5.3. Data is transferred from the acquisition PC to the network for auto-processing with CHROM software.

10.5.4. Each data file is checked for correct information including sample number, job number, QA batch, dilution factor, initial volume, final volume, and % moisture.

10.5.5. The data processing service from Chrom queries LIMS for the sample processing parameters.

10.5.6. Each data file is processed using calibration factors from the most recent initial calibration, quantitation from the daily calibration verification standard is not permitted.

10.5.7. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8260D are listed in Table 7.

10.6. Interpretation and Qualitative Identification:

10.6.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.6.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.6.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.6.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.6.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.6.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.6.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.6.1.7. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Otherwise, structural isomers are identified as isomeric pairs.

10.6.1.8. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.6.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.6.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.6.2.2 The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.6.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

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

10.6.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6.2.6 If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc..).

10.7. Data Reporting

10.7.1. Final Report. LIMS TALS system automatically produces a data report consisting of key, hardcopy reports corresponding to specific data reporting requirements.

10.7.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.7.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.7.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.7.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.7.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.7.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

11.0. Calculations / Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method.

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3.. See Section 9.2.4.2 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.1.4. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RRF)(Vs)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Vs = Volume of sample purged (ml)

11.1.5. Low Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)}{(Ais)(RRF)(Ws)\,(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Ws = Weight of sample purged (g)

11.1.6. Medium Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)(Vt)(1000)(D)}{(Ais)(RRF)(Va)(Ws)(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1

DW = Dry wt correction = $\frac{100 - \% \text{ moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Va = Volume of the aliquot of sample methanol extract added to reagent water for purging in ul

Vt = Total volume of methanol extract in milliliters

Ws = Weight of sample purged (g)

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method . For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Relative Response Factors

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

Where:

A_x = Area characteristic ion for the compound (see Table 7)
Ais = Area characteristic ion of associated internal standard (See Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.4. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.2. (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.5. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% \text{ D} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.6. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.7. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

11.8. Accuracy:

$$\text{ICV , CCV and LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.9. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.10. Calculation of Percent (%) Error:

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units
x_i = True amount

11.11. Relative Standard Error (RSE):

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the Eurofins corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-

up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure. The annual LLOQ verification is completed and documented with the required annual MDL evaluation

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The LLOQ shall be verified annually on each instrument used for client sample analysis.

12.3.4 Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0 Pollution Control

13.1. It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision. The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples/Returned Methanol Preservative - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.2. .United States Environmental Protection Agency, “Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Waste, SW846, Update VI, Revision 4, June 2018.

15.3. United States Environmental Protection Agency, “Method SW8000D: Determinative Chromatographic Separations”, Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012.

15.4. U.S. EPA. 2003. "Method 5030C (SW-846): Purge-and-Trap for Aqueous Samples," Revision 3. Washington, DC.

15.5. U.S. EPA. 2002. "Method 5035A (SW-846): Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Draft Revision 1. Washington, DC.

15.6. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.7. Eurofins Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.8. Eurofins Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision.

15.9. Eurofins Edison ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

15.10. Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

15.11. Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.12. Eurofins Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.13. Eurofins Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision

15.14. Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision

15.15. Eurofins Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, current revision.

15.16. Eurofins Edison SOP No. ED-GEN-022, *Training*, current revision.

15.17. Eurofins Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision

15.18. Eurofins Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.19. Eurofins Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.20. Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision

16.0 Method Modifications:

16.1 Method 8260D requires the BFB tune standard to be analyzed once prior to an ICAL and not daily after that prior to sample analysis. The laboratory will analyze the BFB tune daily, prior to QC and sample analysis. The laboratory will use the

tighter criteria from Methods 8260B/8260C for tune evaluation, rather than the criteria suggested in Table 3 of Method 8260D.

17.0 Attachments

N/A

18.0 Revision History

- Revision 10, dated 08/09/2023
    - Throughout: added analyte 2-ethylhexyl acrylate
    - Sections 2.3 and 10.2.1: Screening clarified to indicate which samples are screened and samples that derive their dilutions from historical data.

- Revision 9, dated 10/17/2022
    - Updated throughout to Eurofins branding throughout the document.
    - Removed all references to 8260C
    - Section 8.3.1: added requirements for handing aqueous samples from chlorinated sources.
    - Section 9.2.2: updated throughout to detail requirements for Relative Standard Error (RSE) evaluation if ICAL.
    - Section 11.11: added formula for calculation of RSE.

- Revision 8, dated 07/16//2020
    - Updated throughout to include requirements of SW 8260D and 8000D.
    - Updated Table 1 with full current analyte list.
    - Add text to Section 1.1.6 detailing procedures for documenting method variations via NCMs.
    - Section 2.6: clarified text regrading lower than standard RLs.
    - Section 4.4: clarified text regarding trip blanks.
    - Section 7.1.1: revised source and details of organic free water.
    - Section 8.0: added handling and preservation details for various soil and aqueous sample types.
    - Section 9.1.1.4: Added that concentrations allowed in blanks (one half of RL), how blank concentration relates to sample concentration (<1/10) and some guidance on re-analysis when concentration exceeds criteria.
    - Section 9.1.3: added that CCV/LCS can be the same run.
    - Tune verifications as not required for daily CCV updated throughout.
    - Section 9.2.2.1.: Allowance for last calibration standard to be the start of 12-hour clock for samples analyzed after initial calibration. Calculations for verifying peak resolution
    - New Section 9.2.4.2 added: Calibration Point Read Back Criteria
    - Section 11.10: added formula for calculation of Percent Error.

- Section 12 (Method Performance) updated to include new MDL procedure and annual LLOQ procedure.
- Updated references in Section 15 as necessary.

- Revision 7, dated 04/01/2020:

  - Updated formatting and branding to Eurofins
  - Sec 7.2.8: Revised Expiration dates based on concentration level and corrected storage requirements.
  - Sec 8.1: Changed Storage Blanks storage period from 1 week to 1-2 weeks.

- Revision 6, dated 01/17/2018:

  - Revised Table 5 and 6: revised to add additional levels plus Benzene and chloroform and to updated concentration level of ICV to current level.

  - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

  - Section 7.2.5: SIM IS/SS mix corrected to reflect lower concentration of IS mix .

  - Section 9.1.2.1.4: SIM MS/MSD preparation revised.

  - Section 9.1.3.2: SIM LCS/LCSD preparation revised.

  - Section 9.2.2.2: additional SIM levels added.

  - Section 9.2.3: SIM CCV level concentration revised to reflect lower concentration.

  - Section 10.2.5: New SIM analysis preparation narrative added

  - Section: 6.1: New instrumentation added.

  - Section: Section: 9.1.4.3: Revised to have any one surrogate out without the need for corrective action. This corrects previous narrative of one surrogate out of two.

- Revision 5, dated 12/11//2015:

  - Revised Table 5: new concentration of low standard (1,4-dioxane only).

- Revision 4, dated 12/08//2014:

  - Section 9.2.4.2.2: Table revised to reflect minimum RF of 0.050 for following compounds: acetone, 2-butanone, 4-methyl-2-pentanone, 2-hexanone.

  - Section 9.2.4.3: added statement ‘for poor performers the range is 50-150%’.

- Revision 3, dated 11/10/2014:

- Tables 1 and 7: added 1,2,4,5-Trimethylbenzene, 1,4-Diethylbenzene, Butadiene, 1,4-Difluorobenzene, 1-Chlorohexane, Freon 114, Freon 123a, Isooctane, 4-Ethyltoluene, t-Amyl Alcohol, Chlorofluoroethylene to list of target compounds and list of standard sources.

- Section 2.5: added chloroform, vinyl chloride and benzene to the list of SIM analytes addressed in this section.

- Section 2.6: revised the concentration of the low ketone standard to 2.5 ug/l.

- 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items. All standards prep tables revised to reflect current standard prep instructions.

- Section 8. Preservation by TSP and holding time is added.

- Section 9.1.2.1: updated source of standards used in various spiking solutions.

- Section 9.1.3: LCS/MS/MSD. Preparation tables now indicate using calibration mix and not the second source mix.

- Sections 9.1.4.3 and 9.1.1 : Revised to indicate that we are now spiking with 4 surrogates instead of the method required 3. One surrogate is now allowed to be out of limit criteria for either 1,2-Dichloroethane-d4 and Dibromofluoromethane.

- Section 9.2.2: Chloroform, Vinyl Chloride and Benzene added as SIM compounds.

- Section 9.2.4.2.3.1. A list of 'poor performing compounds' is added with a ICAL RSD criteria of 50%.

- Section 9.2.4.3: now specifies that up to 10% of the compounds are allowed to exceed the 70-130% ICV recovery criteria as long as their recoveries are within 65-135%..

- Section 9.2.4.4.1.6: Added the following to the first sentence: '…or 50%D for the poor performing compounds'.

- Section 10.1.1.2: updated masses/dwell time for Group 1 under SIM Parameters.

- Throughout document as appropriate: Replaced references to Target with references to CHROM

- Added Section 10.5.5: "The data processing service from Chrom queries LIMS for the sample processing parameters."

- Revision 2, dated 11/04/2013:

- Tables 1 and 7: added methyl acrylate, 1-methylnaphthalene and 2-methylnapthalene.

- Revision 1, dated 09/16/2011:

  - Tables 1 and 7: added cyclopentene, 2-chloro-1,3-butadiene, methacrylonitrile, propionitrile, ethyl methacrylate, 2-nitropropane, indan and isobutyl alcohol to list of target compounds and list of standards sources.

  - Section 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

  - Table 3: Initial Calibration Standards Preparation: is now split into three tables to include aqueous low level analysis.

  - Table 5: added following footnote:
    Levels 1 and 2 respectively are prepared in 500ml and 100ml final volumes
    [1]This level is also used as the Continuing Calibration Verification.

- Revision 0, dated 02/15/2011: New

Table 2: Working Standards Preparation							
Target Compound Standard Name	**Lab Name**	**Vendor**	**Cat. #**	**Vol. Std. Added**	**Conc. of Stock Std.**	**Concentration of Standard**	**Final Vol/ Total vol of MeOH**
Gas Mix Hi	Gas (Hi)	Restek	567645	5ml mL	2000 ppm	500 ppm	20mL 15mL TV/M
Gas Mix Li	Gas (Li)	Restek	567645	500 uL	2000 ppm	50 ppm	20mL 19.5mL TV/M
8260Mix 1	Mix 1 (Hi)	Restek	567641 567646 567642 568022	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10ml
8260 combined	Mix 1 (Li)	Restek	567641 567646 567642 568022 567643 568018 568713 568722 568723	1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml	2000 ppm	50 ppm	40ml 31ml TV/M
Acrolein	AC	Restek	82402	1.0ml	20000 ppm	500 ppm	40ml 39ml TV/M
8260 Mix 2	Mix 2 (Hi)	Restek	567643 568722 568019-fl 568713-fl	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10mL
8260 Mix 3	Mix 3 (Hi)	Restek	568723 568021-fl	2.5ml 2.5ml	2000 ppm	500 ppm	10ml 5ml TV/M
1,4-Dioxane	1,4-Dioxane	Supelco	360481	483.6ul	Neat	50000 ppm	10ml/9.52TVM
1,4-Dioxane	1,4-Dioxane	Supelco	NA	100ul	50000 ppm	500 ppm	10ml/9.90TVM
Propenes*	Propenes	Supelco	21240202	NA	1000/2000 ppm	NA	NA
Propenes*	Propenes	Supelco	21240202	1ml	1000/2000 ppm	50 ppm (varied)	20ml/ 19ml
Gas SS	Gas SS	Restek	567645.sec	1ml	2000ppm	50 ppm	40ml 39ml/TV/M
8260 Mix 1 SIM	8260 Mix 1 SIM	Supelco	5-02111	50 ul	2000ppm	10 ppm	10ml 9.95 TV/M
1,4-Dioxane SIM	1,4-Dioxane	Supelco	NA	100 ul	50000 ppm	500 ppm	10ml/9.90TVM

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
8260 SS	8260 SS	Restek	567641.sec 567646.sec 567642.sec 568022- sl 567643.sec 568019- sl 568713- sl 568722.sec 568723.sec 568021- sl	1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml	2000 ppm	50 ppm	40 ml 30 ml TV/M
Acrolein SS	AC SS	Restek	568720.sec	1 ml	20000 ppm	500 ppm	40 ml 39 ml TV/M
Propenes SS	Propenes SS	Supelco		1 ml	1000/2000 ppm	50/100 ppm	40 ml 39 ml TV/M
8260Mix 1 SIM SS	SIM MIX1 SS	Supelco	5S-02111	50ul	2000 ppm	10 ppm	10ml 9.95 TV/M
Benzene/ Chloroform	Ben/chl	Absolute	70025/ 70076	100ul each	1000ppm	10ppm	10ml 9.90 TV/M
1,4-Dioxane (SS)	1,4-Dioxane	Absolute	70373	1ml	1000 ppm	500 ppm	2ml/1ml TV/M

Asterisk (*) indicates a custom standard mix.

Table 3: Initial Calibration Standards Preparation, Low Level Soil

Standard Solution	Final Volume Reagent Water (ml)	Volume of Standard Added to Reagent Water (ul)					
		1ppb *	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Gas Mix (500ppm)	5	-	-	-		2.0	5.0
		-	-	-			
Mix 1 (combined) (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Mix 1 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-	-	-
Freon Mix							
AC (500ppm)	5	-	-	3.0	4.0	5.0	6.0
	50	10	20			-	-
Mix 2 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-		
Mix 3 (500ppm)	5					2.0	5
Propenes (50ppm)	-	-	-	-	-	-	-
	50	10.0	20.0		-	-	-
Propenes (Hi)(500ppm)	5	-	-	2.0	5.0	20	50
	-	-	-	-	-	-	-

[1]**This level is also used as the Continuing Calibration Verification.**

Table 3a: Initial Calibration Standards Preparation, Aqueous (LOW LEVEL)

Standard Solution	Volume of Standard Added to Reagent Water (ul)						
	0.5ppb*	1ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	0.5	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 3 (varied)	0.5	1	1	2	5	20	50
AC (500ppm)	2	4	4	4	10	20	40
1,4-Dioxane (500ppm)	15	30	-	-	-	-	-
Freons mix	0.5	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.5	0.5	0.5	1	2.5	10	25
Methanol Compensate	3000	2800	610	300	280	190	0
Final vol. (reagent water)	500ml	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 3b: Initial Calibration Standards Preparation, Aqueous

Standard Solution	Volume of Standard Added to Reagent Water (ul)					
	1.0ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	1	1	2	5	20	50
Mix 3 (varied)	1	1	2	5	20	50
AC (500ppm)	4	4	4	10	20	40
1,4-Dioxane (500ppm)	30	-	-	-	-	-
Freons Mix	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.25	0.5	1	2.5	10	25
Methanol Compensate	2800	610	300	280	190	0
Final vol. (reagent water)	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 4 : ICV Standard Preparation, Low Level Soil

Standard Solution	Concentration	Volume of Standard Added to 5.0 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	2	20
8260 SS (Separate lot)	50ppm (+varied)	2	20
AC SS (separate lot)	500ppm	3	300
Freon SS (Separate lot)	50ppm	2	20
Propenes SS(separate lot)	50ppm (varied)	2	20 (varied)

Table 4a: ICV Standard Preparation, Aqueous

Standard Solution	Concentration	Volume of Standard Added to 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	20	20
8260 SS (Separate lot)	5000ppm (varied)	20	20
AC SS (separate lot)	500ppm	4	400
Freons SS (Separate lot)	50ppm	20	20
Propenes (second source)	50ppm (varied)	20	20 (varied)

Table 5: SIM Initial Calibration Standards Preparation

Standard Solutions	Volume Standard Solution Added to Reagent Water (Final Concentration)							
8260 Mix 1 SIM (10ppm)	1 ul (0.02 ppb)	2 ul (0.04 ppb)	1 ul (0.05 ppb)	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1 .0 ppb)	10 ul (2.0 ppb)
1,4-Dioxane (500ppm)	4 ul (0.4 ppb)	2 ul (1.0 ppb)	1 ul (5.0 ppb)	1 ul (10 ppb)	1 ul (20 ppb)	2.5 ul (30 ppb)	5 ul (40 ppb)	10 ul (50 ppb)
SIM (ben/chl) 10ppm	1 ul (0.02 ppb)	1 ul (0.02 ppb)	1 ul (0.05 ppb	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1.0 ppb)	10 ul (2.0 ppb)
Final Vol. (reagent water)	**500ml**	**500ml**	**200ml**	**100ml**	**50ml**	**50ml**	**50ml**	**50ml**

levels 1 and 2 are respectively prepared in 500ml and 100ml final volumes
[1]**This level is also used as the Continuing Calibration Verification.**

Table 6 : SIM ICV Standard Preparation

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
SIM MIX1 SS (Second source)	10ppm	1	0.05
1,4-Dioxane SS	50ppm	20	5

TABLE 7
Characteristic Ions of Volatile Organic Compounds

Parameter	Primary ion	Secondary ion
1,1,1-Trichloroethane	97	99,117,119
1,1,2,2-Tetrachloroethane	83	85,131,133,166
1,1,2-Trichloroethane	97	83,85,99,132,134
1,1-Dichloroethane	63	65,83,85,98,100
1,1-Dichloroethene	96	61,98
1,1-Dichloropropene	75	110. 77
1,2,3-Trichlorobenzene	180	182
1,2,3-Trichloropropane	110	75
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-Chloropropane	75	155, 157
1,2-Dibromomethane	107	109
1,2-Dichloroethane	62	64,100,98
1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	65,114
1,2-Dichlorotrifluoroethene	67	117
1,2-Difluorotetrachloroethene	101	103, 167
1,3,5-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
1,4-Dioxane	88	58
1-Chloropropane	63	78
1-Methylnaphthalene	142	141
1-Propene	41	42
2,2-Dichloropropane	77	97

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
2,4,4-trimethyl-1-pentene	41	57, 97
2-Butanone	72	57
2-Chloroethyl vinyl ether	63	65, 106
2-Chloropropane	78	63
2-Chlorotoluene	91	126
2-Chloro-1,3-butadiene	88	53
2-Hexanone	43	58,100
2-Methylnaphthalene	142	141, 115
2-Nitropropane	39	42, 44
2-Octane	43	58
2-Octanol	45	55
4-Chlorotoluene	91	126
4-Methyl-2-Pentanone	43	58,100
Methacrylonitrile	67	41
Acetone	43	58
Acetonitrile	39	40, 41
Acrolein	56	55
Acrylonitrile	53	52
Allyl Alcohol	57	40, 39
Allyl Chloride	76	41
Amyl Acetate	43	70, 61
Benzene	78	--
Benzyl Chloride	91	126, 65
Bromobenzene	156	77, 158
Bromochloromethane	129	49, 130
Bromodichloromethane	83	85
Bromoform	173	171,175,
Bromomethane	94	96
Butyl Acetate	73	56, 43
Butyl Acrylate	73	56, 55
Butyl methacrylate	87	69
Camphene	93	121
Camphor	95	81
Carbon disulfide	76	78
Carbon tetrachloride	117	119,121
Chlorobenzene	112	114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
Chlortrifluoroethene	116	118
cis-1,3-Dichloropropene	75	77

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
Cyclohexane	56	84, 69
Cyclopentene	67	68, 68, 53
Dibromochloromethane	129	208,206
Dibromomethane	93	95, 174
Dichlorodifluoromethane	85	87
Dimethylnaphthalene (total)	141	156, 155
Epichlorohydrin	57	62, 49
Ethanol	46	45
Ethyl Acetate	70	61, 43
Ethyl Acrylate	55	56
Ethyl Ether	59	74, 75
Ethylbenzene	106	91,
Ethyl methacrylate	69	41, 99
Freon TF	101	103, 151, 85
Hexachlorobutadiene	225	223
Hexane	56	57, 86
Indan	117	118, 58
Iodomethane (methyl iodide)	142	127
Isobutyl Alcohol (Isobutanol)	43	41, 42
Isoprene	67	53, 59
Isopropanol	45	59
Isopropyl Acetate	43	61, 87
Isopropyl Ether (DIPE)	45	87
Isopropylbenzene	105	120
Methyl Acetate	43	74
Methyl Acrylate	55	85, 42
Methyl cyclohexane	83	55, 98
Methyl Methacrylate	100	69
Methyl tert-butyl ether (MTBE)	73	57
Methylene chloride	84	49,51,86
Methylnaphthalene (total)	142	141, 115
Naphthalene	128	--
n-Butanol	56	41, 43
n-Butylbenzene	91	92, 134
n-Heptane	57	43, 71
n-Pentane	72	57
N-Propanol	60	59
n-Propylbenzene	91	120
P-Isopropyltoluene`	119	134, 91
Propyl Acetate	43	61, 73
Propionitrile	54	52, 54

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
sec-Butylbenzene	105	134
Styrene	104	78,103
Tert-Amyl Methyl Ether	73	55, 87
Tert-butyl Alcohol	59	--
Tert-Butyl Ethyl Ether	59	87
Tert-Butylbenzene	119	91, 134
Tetrachloroethene	164	129,131,166
Tetrahydrofuran	42	72, 71
Toluene	92	91
Total Xylenes	106	91
trans,-1,3-Dichloropropene	75	77
Trans-1,4-dichloro-2-butene	53	75
Trichloroethene	130	95,97,132
Trichlorofluoromethane	101	103
Vinyl acetate	43	86
Dichlorofluoromethane	67	69
Chlorotrifluoroethene	116	118
1,2-tetrachlorodifluoroethane	101	103,167
1,2-Dichlorotrifluoroethane	67	117
Vinyl chloride	62	64
Isooctane	57	41, 56
1- Chlorohexane	91	93, 55, 56
1,2,4,5-Tetramethylbenzene	119	134, 91
4-EthylToluene	105	120, 77
Chlorotrifluoroethylene	66	116,118,85
Freon 114	85	87,135,137
t-Amyl Alcohol	59	55, 73, 43
1,4-Difluorobenzene	114	63
1,4-Diethylbenzene	119	105,134
Freon 123a	67	69, 117, 119
Butadiene	54	53, 39
4-Bromofluorobenzene (sur)	95	174,176
1,2-Dichloroethane-d4 (sur)	65	102, 104
Toluene-d8 (sur)	98	70,100
Fluorobenzene (istd)	96	77
Chlorobenzene-d5 (istd)	117	82,119
1,4-Dichlorobenzene-d4 (istd)	152	115,150