



An introduction to Analytical Testing for Per- and Polyfluoroalkyl Substances (PFAS)

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Our Canadian Presence

10

Laboratories
*including Halifax - 2022

20+

Support Locations

20+

Years

175K+ / yr.

Samples Processed @ WT Hub

Complimentary pick-ups within service areas

24/7 drop-off and pick-up access

Dedicated, responsive Client Service team

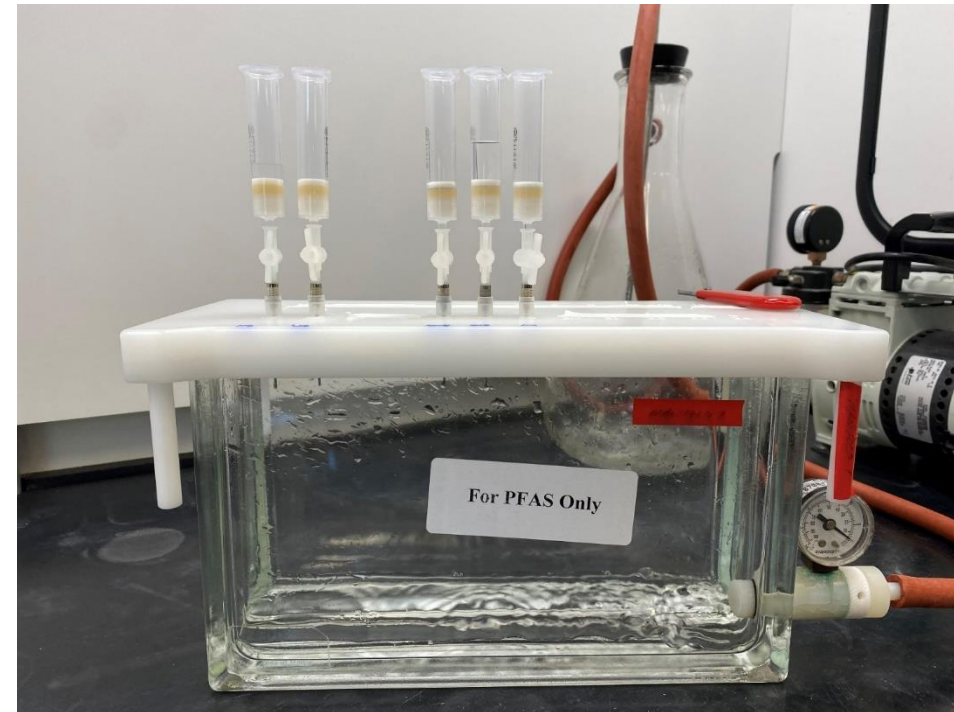


AVERAGE EXPERIENCE AT THE ALS ENVIRONMENTAL LABORATORIES IN CANADA			
	Managers: 16.7 years	Quality & Technical Services: 16 years	Laboratory: 7 years
		Customer Service: 9 years	

Outline



- General Principles and Method overview
- Targeted analysis
 - Drinking water
 - Non-Potable water
 - Soil/Solid
- Qualitative Techniques
- Sample Collection and Handling
- Quality Control





right solutions.
right partner.

General Principles and Method Overview

LC-MS/MS analysis of PFAS



- Selective and sensitive
- Fast and reliable
- Versatile
- Low reporting limits
 - ng/L
 - $\mu\text{g}/\text{kg}$

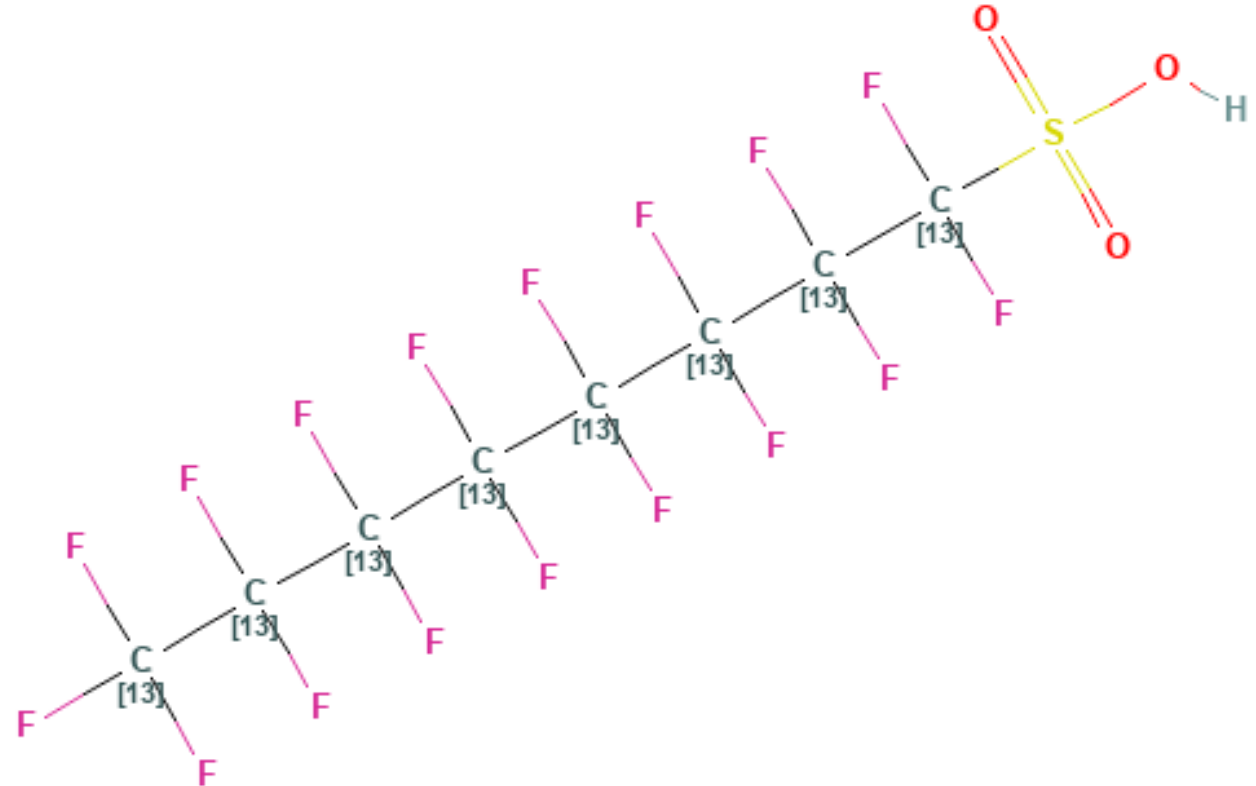
LC-MS/MS – Liquid chromatography coupled with tandem mass spectrometry



Isotope Dilution



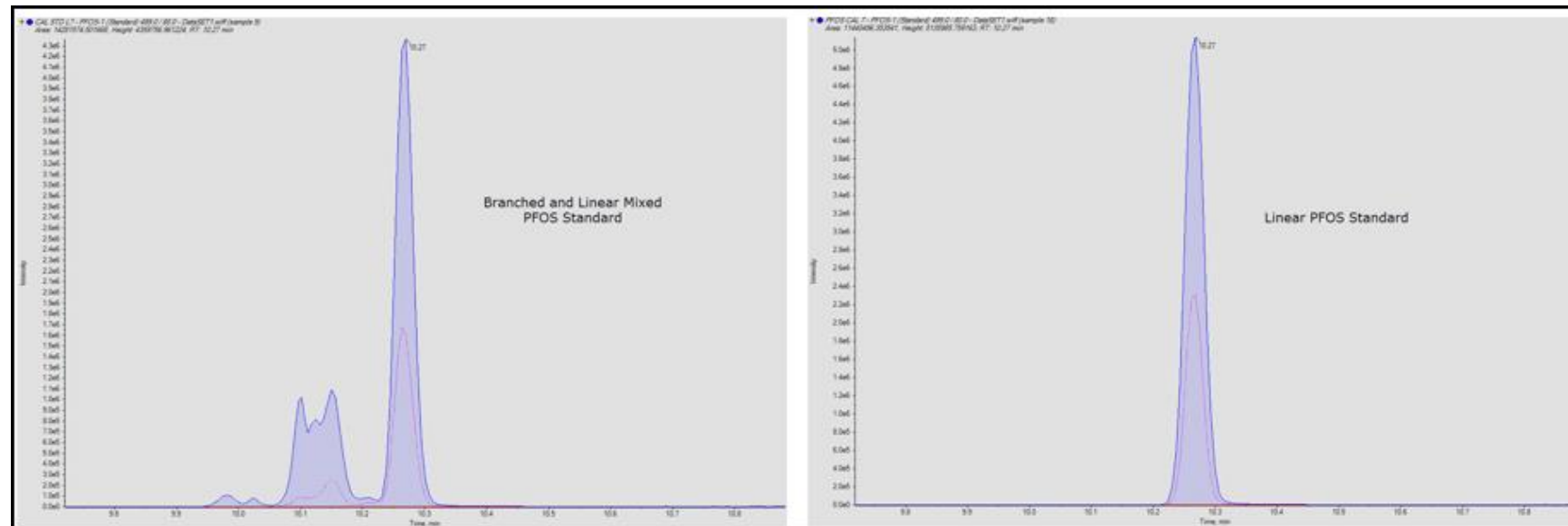
- Isotopically labeled standards
- Same physical and chemical properties as native compounds, but with different mass
- Used to track losses and matrix interference
- Great accuracy and precision



Branched and Linear Standards



- Many PFAS in the environment are found as a mixture of branched and linear isomers
- Quantitation using a mixed linear/branched standard will be more representative of environmental contamination and therefore more accurate
- Mixed standards are used where available (11)

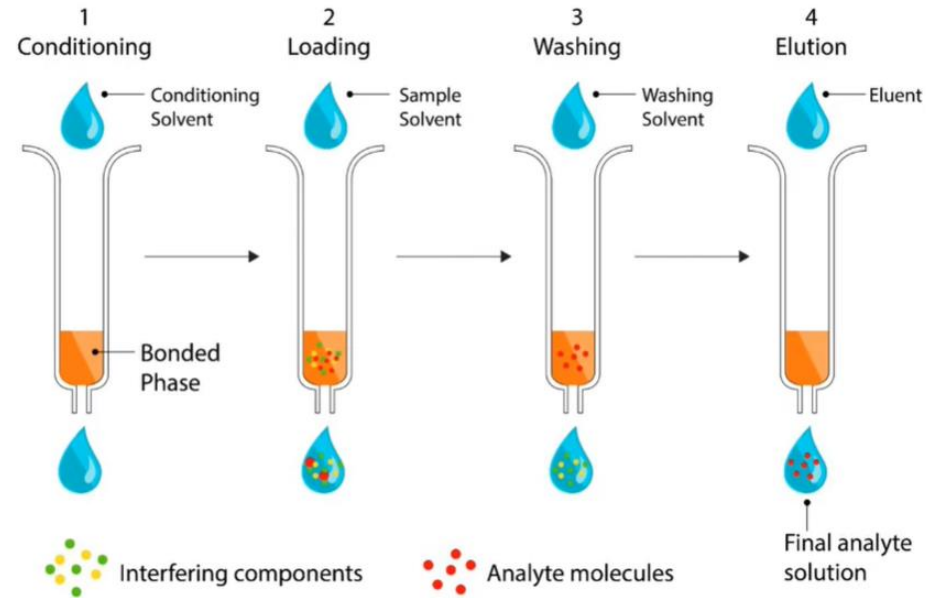


Sample preparation - SPE vs DI



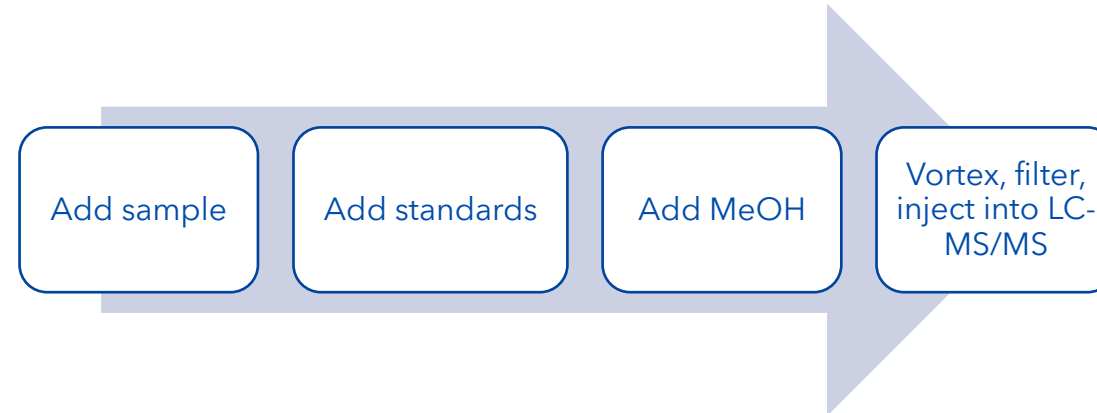
- Solid Phase Extraction (SPE)

- Used in most PFAS methods
- Cleanup sample / remove interfering components
- Complex and difficult matrices (including soil)
- Low level RLs (2 ng/L)



- Direct Injection (DI)

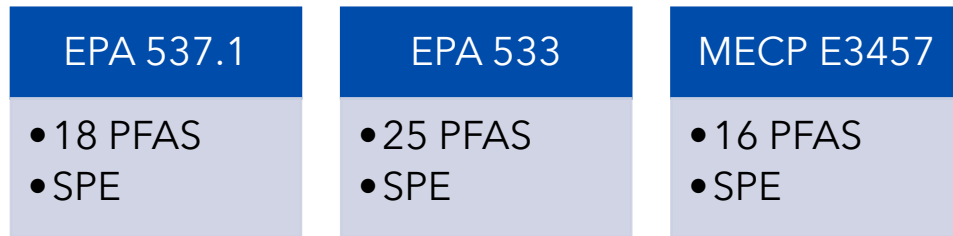
- Minimal sample preparation (no cleanup step)
- Only for aqueous samples (for now)
- Higher RLs (20 ng/L)



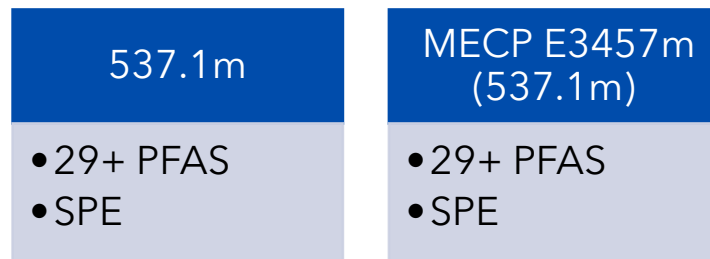
Method Overview - Water



Drinking Water

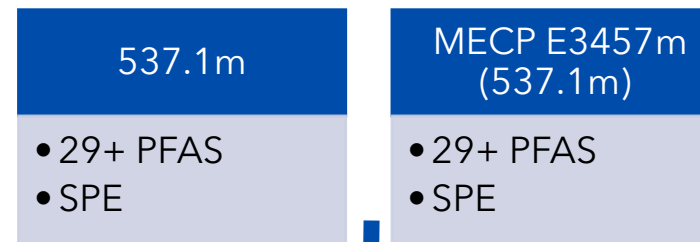


Modified Methods

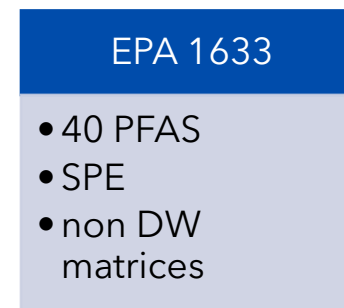
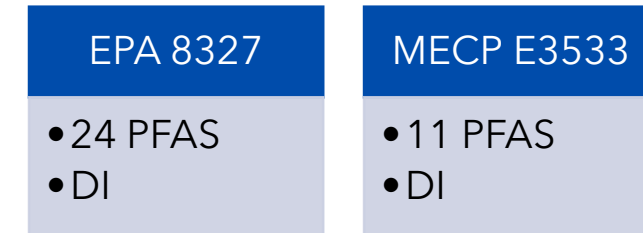


Non-Potable Water

Modified Methods

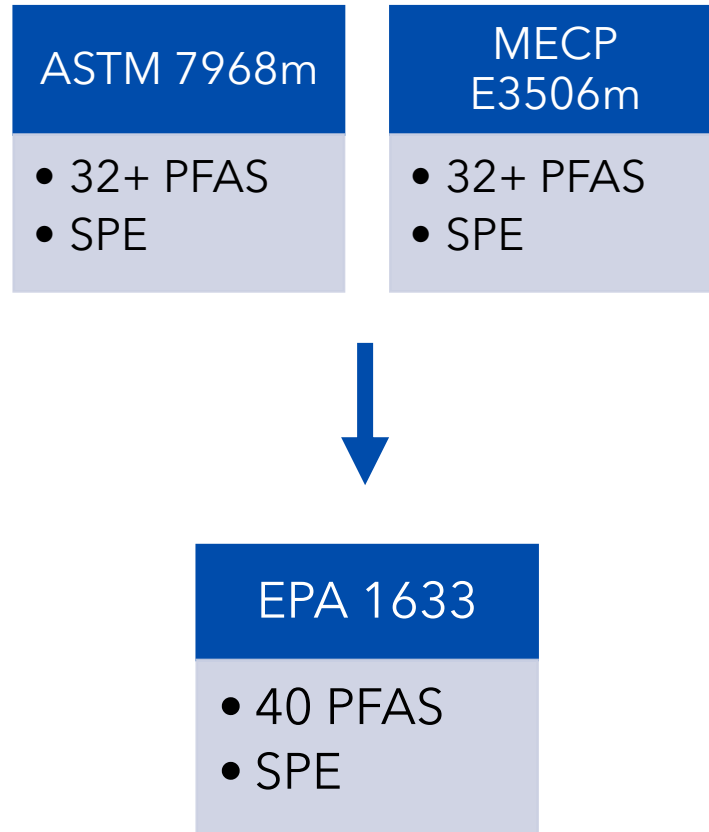


Direct Injection





Method Overview - Soil



ALS Methods



Drinking Water

E3457m
(537.1m)

- 29+ PFAS
- SPE
- Low level RLs
- **10 day TAT**

Non-Potable Water

E3533m

- 40+ PFAS
- Direct Injection
- Routine Level RLs
- **10 day TAT**

EPA 1633

- 40+ PFAS
- SPE
- Trace level RLs
- **15 day TAT**

Soil

EPA 1633

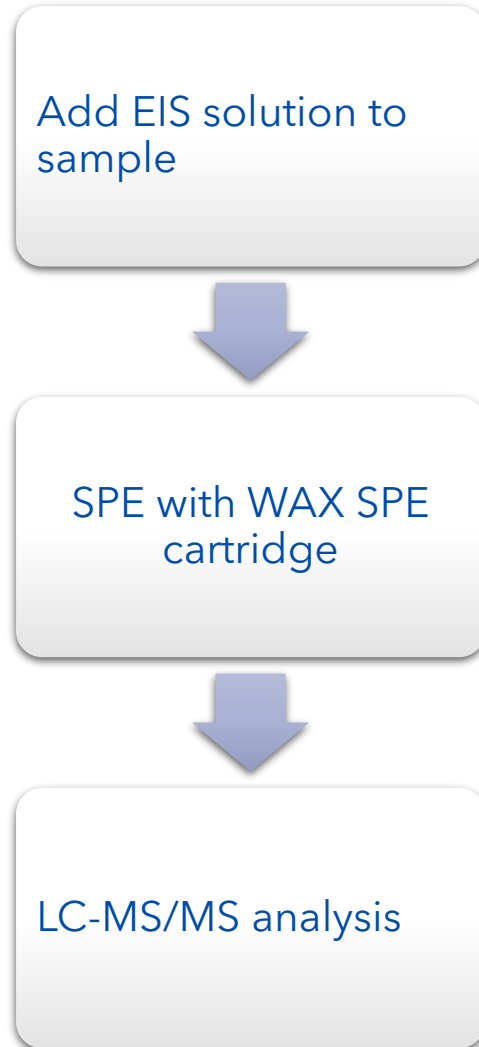
- 40 PFAS
- SPE
- Trace level RLs
- **15 day TAT**



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Targeted Analysis

Drinking Water



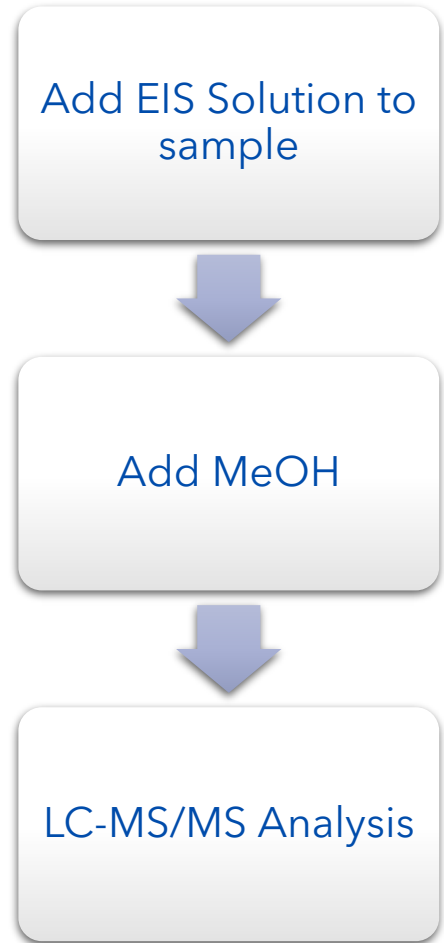
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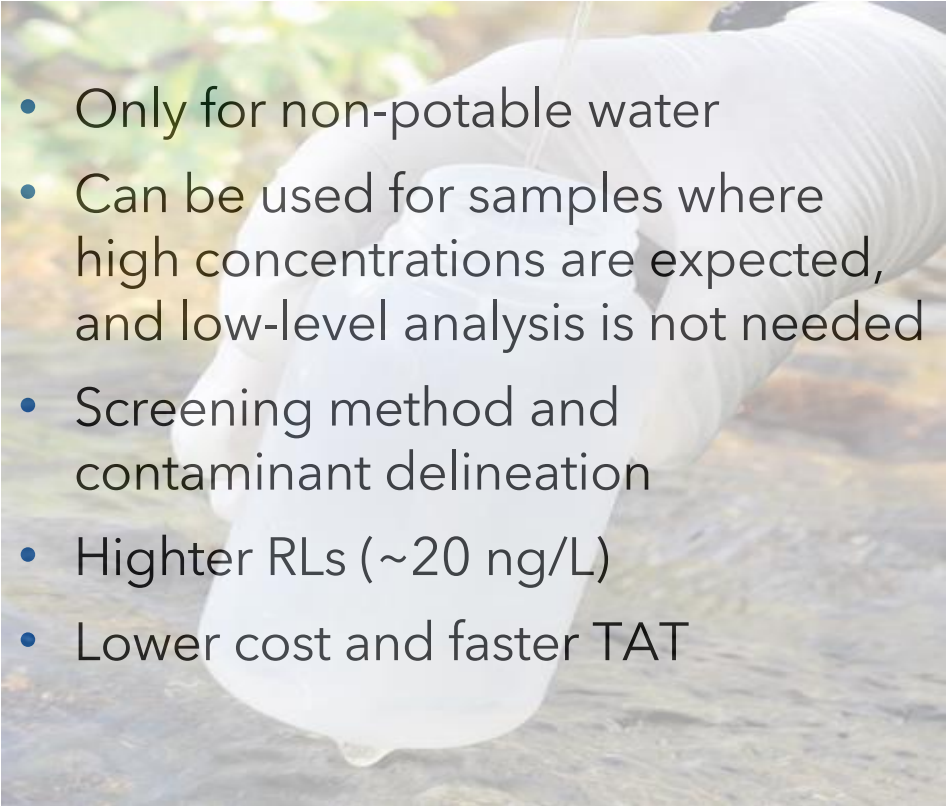


Water Samples – Routine Level

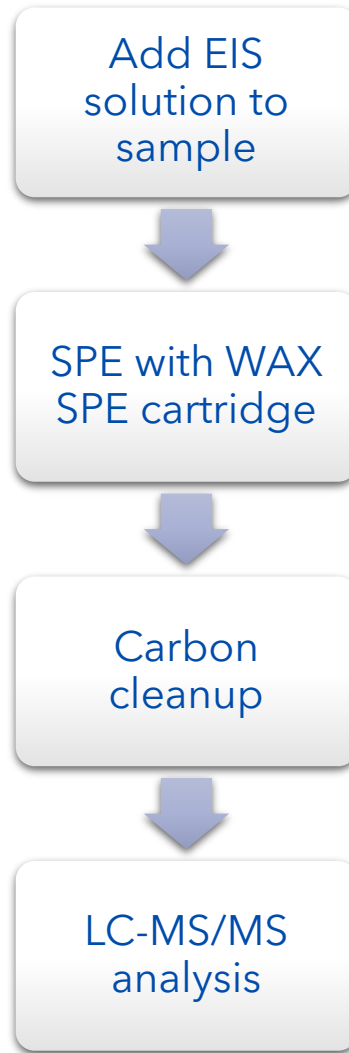


E3533m

- 40+ PFAS
- Direct Injection
- Routine Level RLs
- **10 day TAT**

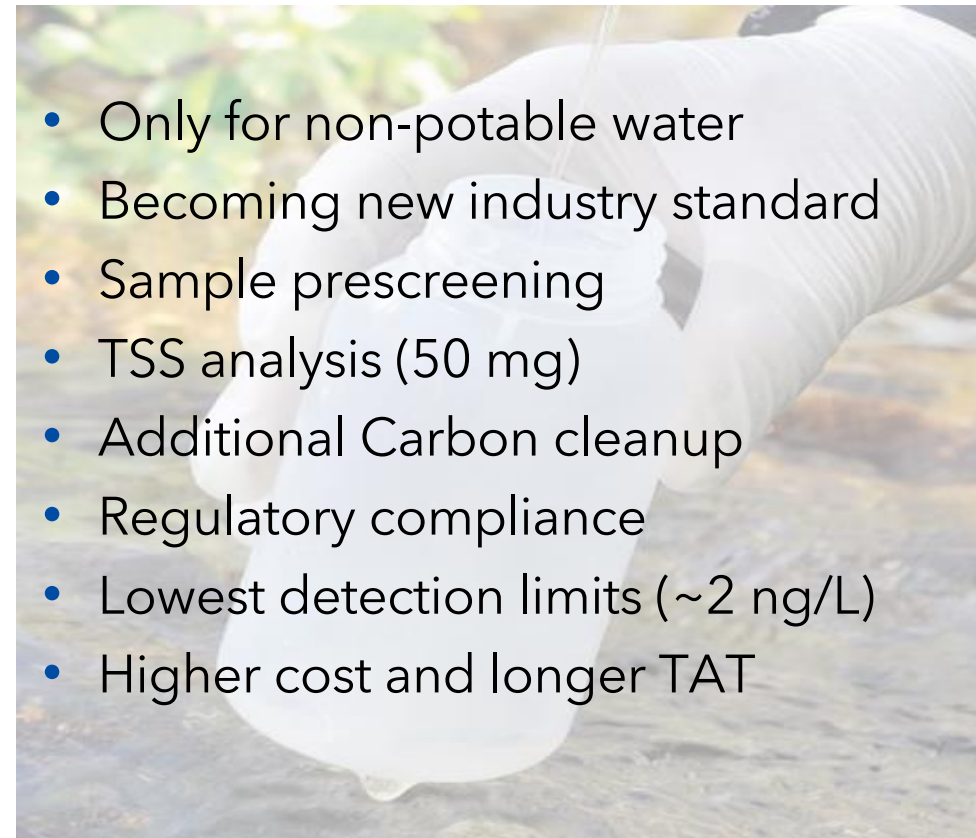


Water Samples - Trace Level Analysis

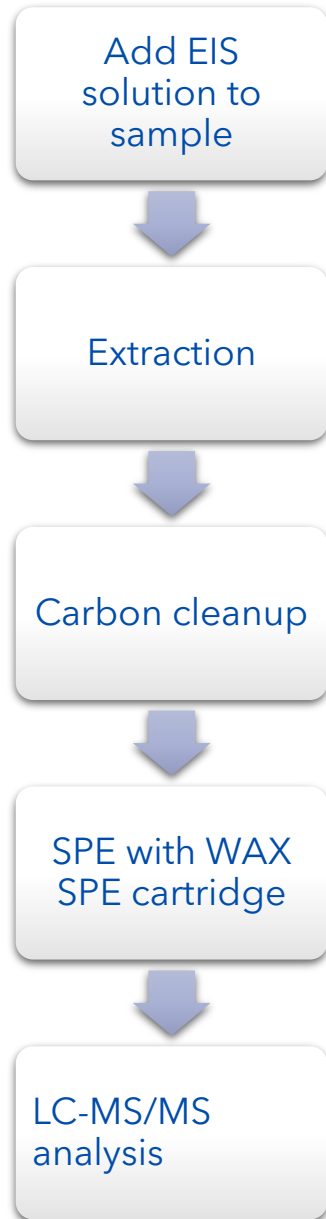


EPA 1633

- 40+ PFAS
- SPE
- Trace level RLs
- **15 day TAT**



Soil Samples



EPA 1633

- 40 PFAS
- SPE
- Trace level RLs
- **15 day TAT**





Changes with EPA 1633 from previous methods

- ✓ Increased number of target analytes
- ✓ Applicable to many matrices
- ✓ Improved sensitivity for complex samples (lower RLs)
- ✓ Sample pre-screening and Total solids
- ✓ Extensive QC acceptance criteria



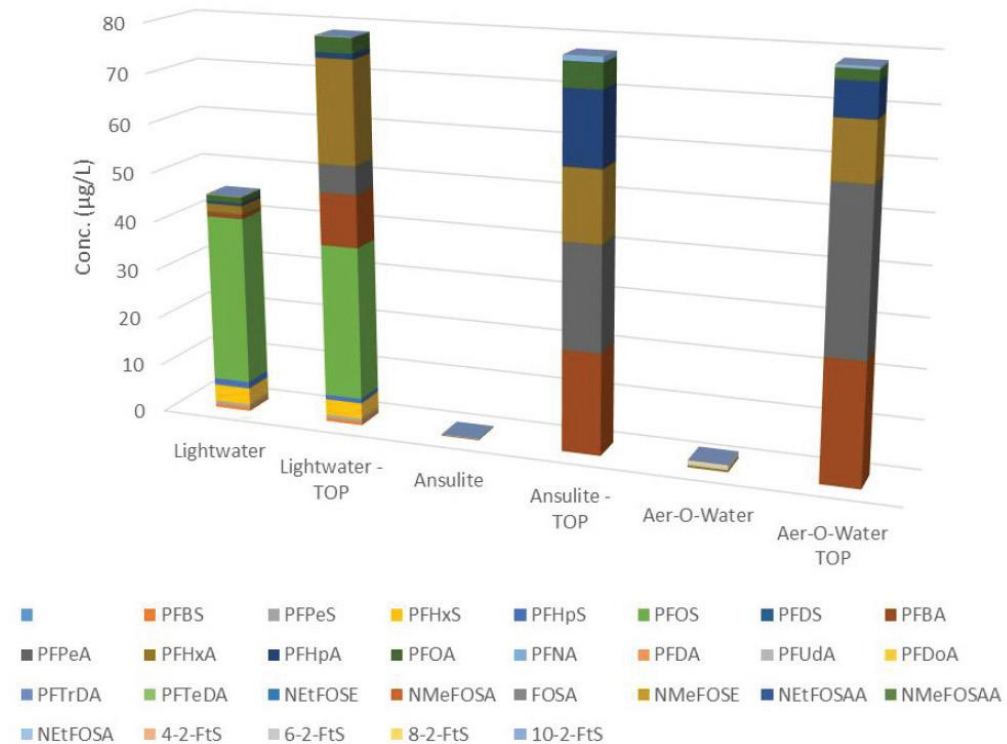
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Qualitative Techniques



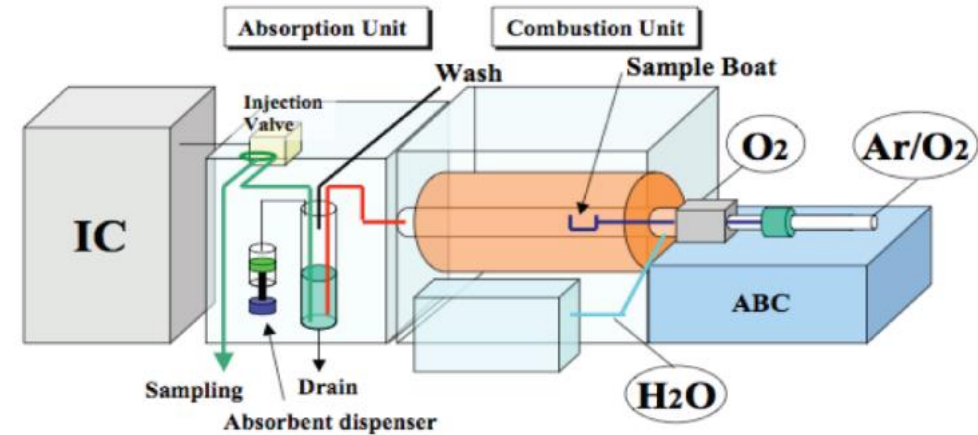
Total Oxidizable Precursor Assay (TOP Assay)

- TOP Assay is designed to expose underlying PFAS outside of the standard targets
- Samples is oxidized to transform any precursor compounds into PFAS end products
- Two data sets are provided: Pre- and Post-Oxidation
- Indication of precursors and potential transformation products
- Still limited to target analytes (cannot see all oxidation products)
- Higher cost (sample analyzed twice)



Total Organic Fluorine (TOF)

- Simple way to estimate the total mass of PFAS in a sample using the concentration of organic fluorine
- Using Combustion Ion Chromatography (CIC)
- No information on specific PFAS compounds
- Not selective for PFAS
- Higher RLs (1.5 ug/L = 1500 ng/L)
- Can be used for: screening, for confirmation of being "PFAS free", and in conjunction with TOP assay and standard analysis, mass balance for treatment processes
- Lower cost





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Sample collection and handling



Questions to ask before sampling

- What type of samples?
 - What will the data be used for?
 - Are there criteria you will be comparing too?
 - What detection limits are needed?
 - List of target PFAS?
-
- What method is best suited?
-
- Is there any existing information about what you expect to see in your samples?
 - Do the method RLs align?
 - Are there high concentrations?
 - Is there a defined source zone?

Consult with your lab!



Sample collection and handling



- Avoid materials where PFAS can adsorb to surface (glass)
- Avoid any potential sources of contamination including all sampling material
- Use of QC samples (blanks)
- Ensure you are taking a representative sample (ex. Limit solids in GW)
- Sample lowest to highest impacted area when possible
- Sample segregation from field to lab
- Submit to lab on ice (hold time 14-90 days)

Avoid	Acceptable alternative
Teflon™ pump or tubing	HDPE or Silicone tubing
Decon 90	Alconox®, Liquinox®, Citrinox®
LDPE or glass sample containers	HDPE or polypropylene containers ** ensure no Teflon™ liner
Chemical Blue Ice packs	Free ice
Waterproof field book	Metal clipboard / loose paper
Markers	Ball point pen or pencil
Water resistant or treated gloves / clothing	Powderless nitrile gloves / cotton clothing
Cosmetics, creams, sunscreen and related products	--
pre-packaged food, aluminum foil, fast food wrappers or containers	--
Plastic bags / packaging - screen before use	Polyethylene bags (Ziplock®)

Field QC samples



Trip Blanks. Analyte-free water prepared in a sealed sample container at the laboratory, transported from lab to sampling site and back to lab without exposure to sampling procedures.

Field Blanks. Analyte-free water poured into a sample container in the field, preserved and shipped with field samples.

Equipment Blanks. Analyte-free water poured over or passed through sampling equipment prior to collection of environmental samples.



Value



QA/QC In the Lab

- Extensive sample processing and laboratory cleaning procedures to ensure no background PFAS contamination
- Sample preparation equipment dedicated to PFAS
- Laboratory supplies and equipment selection to avoid PFAS
- Extensive QA/QC protocols
- Preparation and analysis of Laboratory Blanks

QC Parameter

Initial Calibration Curve

Instrument Sensitivity Check

Interference Check Standard

Calibration Verification Standard (CVS)

Continuing Calibration Verification (CCV)

Extracted Internal Standard (EIS)

Non-extracted Internal Standard (NIS)

Ion Abundance Ratio

Method Blank (MB)

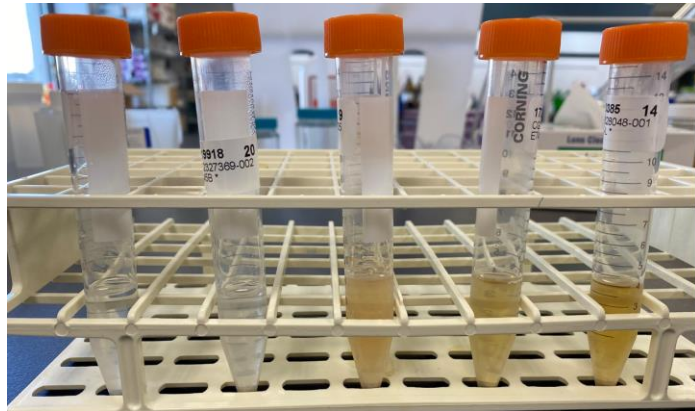
Laboratory Control Sample (LCS)

Laboratory Control Sample (LLLCS), Low-Level

Matrix Spike (MS)

Sample Duplicate

What causes detection limits to be raised?



- Matrix Interference
 - Other compounds in the sample that cause signal enhancement or suppression
- Dilution of Samples
 1. concentration exceeds the highest calibration level or
 2. matrix is suspected to be harmful to the instrument, or affect instrument QC
 - High concentration and matrix affects can cause instrument contamination (downtime for cleaning or parts replacement)
 - Dilution can impact other target analytes



What Else?

- PFAS in biosolids and tissue (EPA1633)
- PFAS in air (EPA OTM45 and EPA OTM50)
- PFAS in surface wipes
- HRMS analysis : determination of PFAS structures by comparison to library

Acknowledgments



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Thank you!
Questions?

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