Advancements in Analytical Techniques for PFAS Detection in Environmental Samples

RemPlex 2025 Summit

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Sarah Choyke
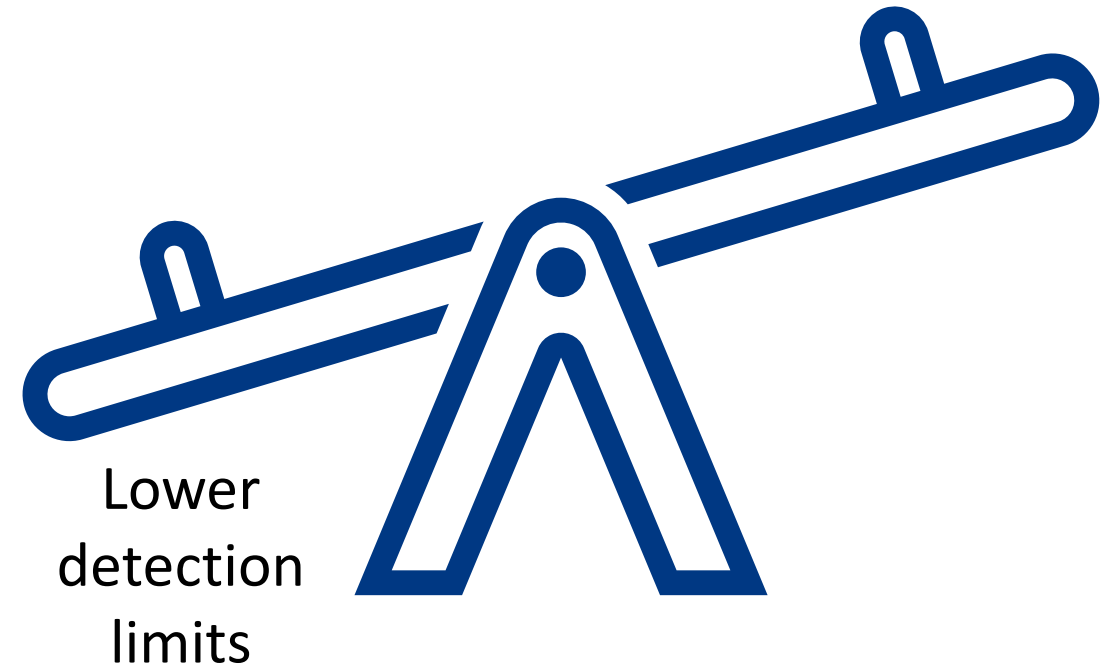*Technical Director, Eurofins Environment Testing*

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Topics for Discussion

- PFAS Introduction

- Pre-Planning & Method Selection

- PFAS Methodology

- Advanced Analytical Tools

More analytes
More methods
More data

Lower detection limits

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What are PFAS?

- A group of synthetic compounds formed from **Carbon**-**Fluorine** chain.

- Known for water- and grease-resistant properties

- Persistent in the environment and long half life in organisms

- Linked to Health outcomes
  - high cholesterol, thyroid disease, liver damage, decrease immune response, certain types of cancer
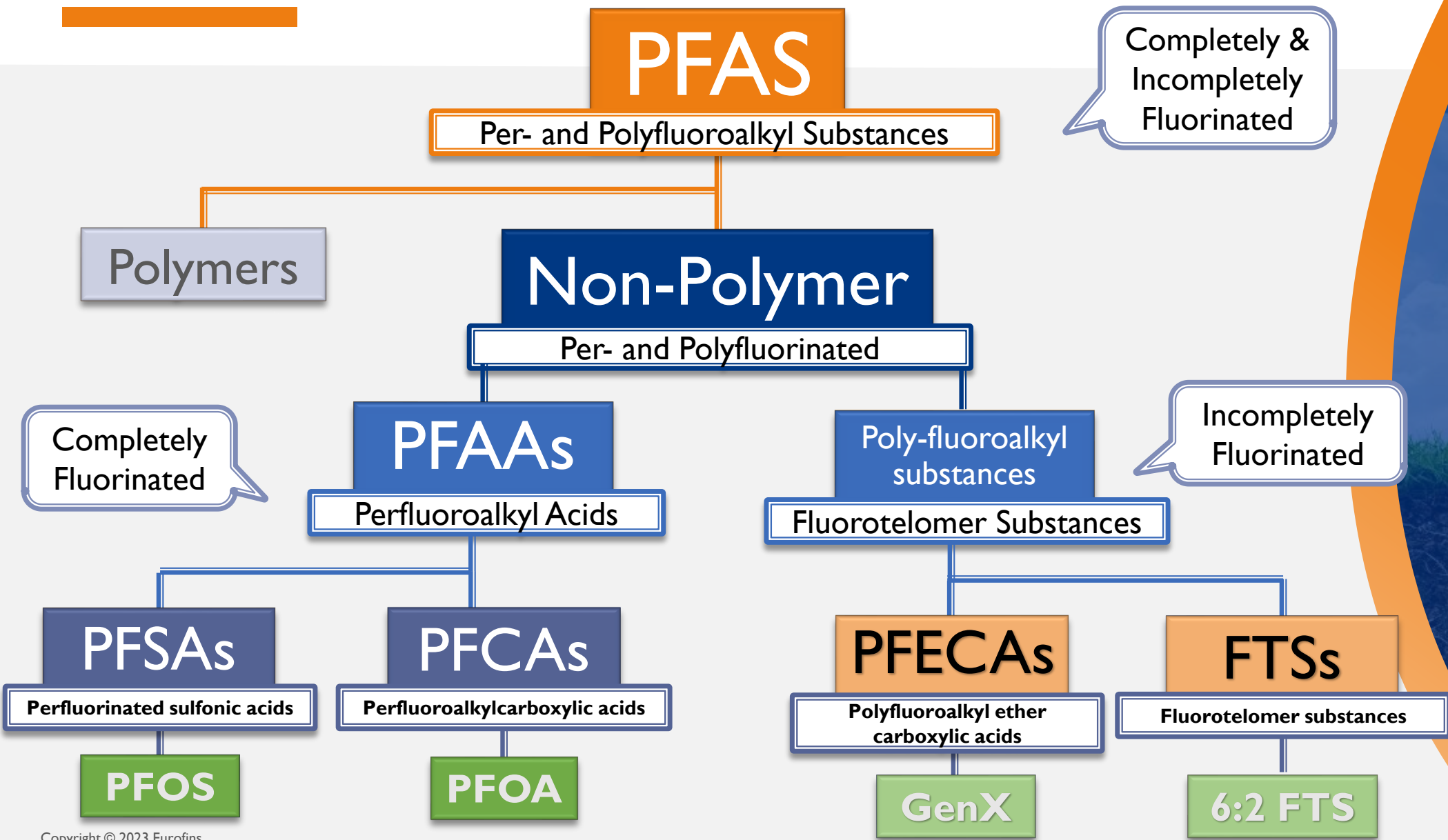
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Analyzing PFAS? You've got options

Drinking Water

EPA Method 533
25 PFAS

EPA Method 537.1
18 PFAS
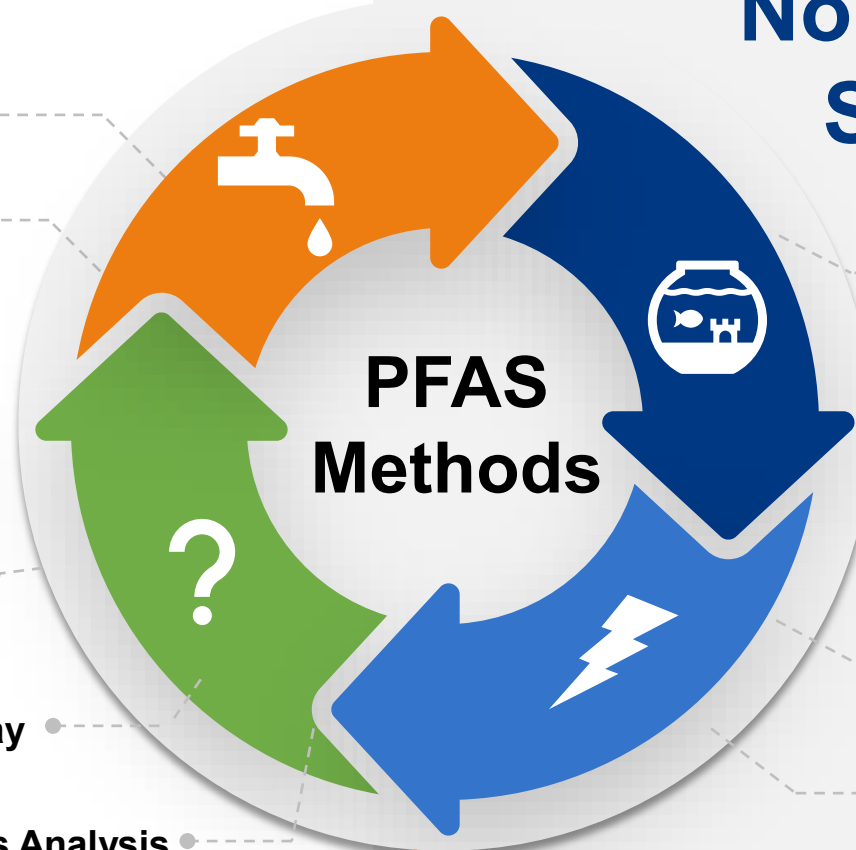
Unknowns

Combustion Ion Chromatography
EPA 1621 Adsorbable Organic Fluorine
Extractable Organic Fluorine

Total Oxidizable Precursor (TOP) Assay

Forensics Analysis
Branched & Linear
Non-Targeted Analysis & Suspect Screening

PFAS
Methods

Non-Potable Water, Solid, & Tissue

EPA Method 1633A
40 PFAS

Rapid Screening

ASTM D8421 / EPA 8327 (Aq)

D7968 (Solids)

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Isotope Dilution: Quantitation method uses ^{13}C- or deuterated stable isotopes for internal standardization

Matrix + Target

+ ^{13}C or d-labelled analogue

+ =

- Most accurate & precise quantitation method
- Broader range of matrices
- Reduces potential of false positives & error

Isotope Dilution Analysis: Wastewater Matrix

Analyte	CAS No.	External Standard		1633A OPR Limits	
		MS	MSD		
		% Rec	% Rec	Lower	Upper
PFBA	375-22-4	0	0	70	140
PFHxA	307-24-4	63	86	70	145
PFOA	335-67-1	142	139	70	150
PFNA	375-95-1	129	129	70	150
PFDA	335-76-2	61	65	70	140
PFBS	375-73-5	84	99	60	145
PFHxS	355-46-4	100	108	65	145
PFOS	1763-23-1	117	111	55	150
6:2FTS	27619-97-2	297	295	65	155
HFPO-DA	13252-13-6	39	40	70	140

Signal suppression: PFBA/$^{13}C_4$-PFBA
Signal enhancement: 6:2FTS/$^{13}C_2$-6:2-FTS

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PFAS Methodology

Non-Potable Water, Solids and Tissues

EPA Method 1633A

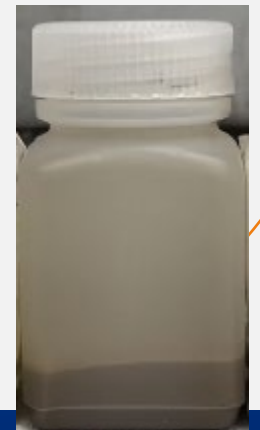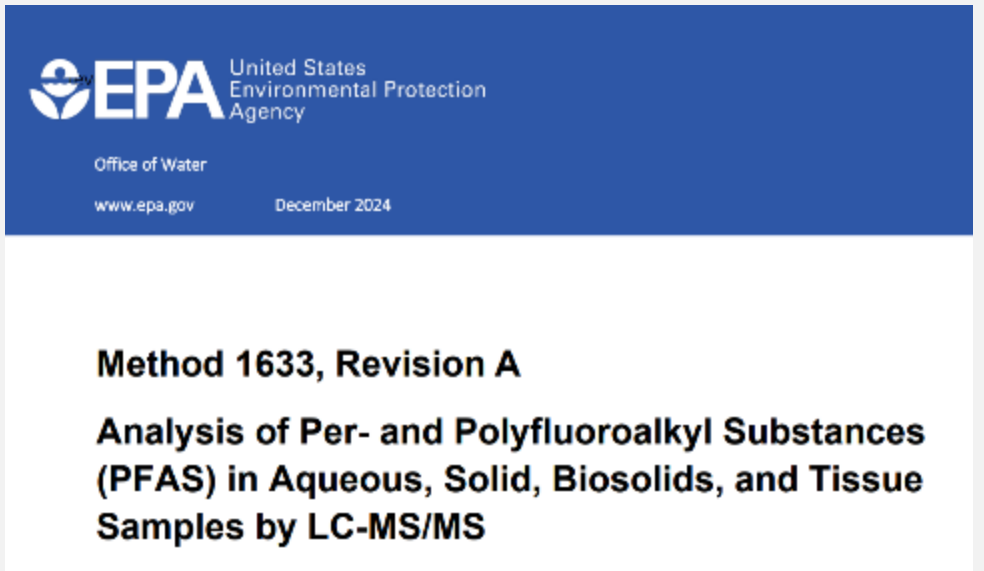| |
|---|
| NPW, Solids, Biosolids & Tissues |
| 40 PFAS (including branched) |
| Solid Phase Extraction Preparation |
| TSS in bottle < 50 mg |
| Hold Time: 28 days for aqueous 90 days for solids & tissues |
| LC-MS/MS with confirmation ion |
| Extracted internal standard (EIS) quantification |
| RL: 2-10 ppt |

United States Environmental Protection Agency

Office of Water

www.epa.gov   December 2024

Method 1633, Revision A

Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS
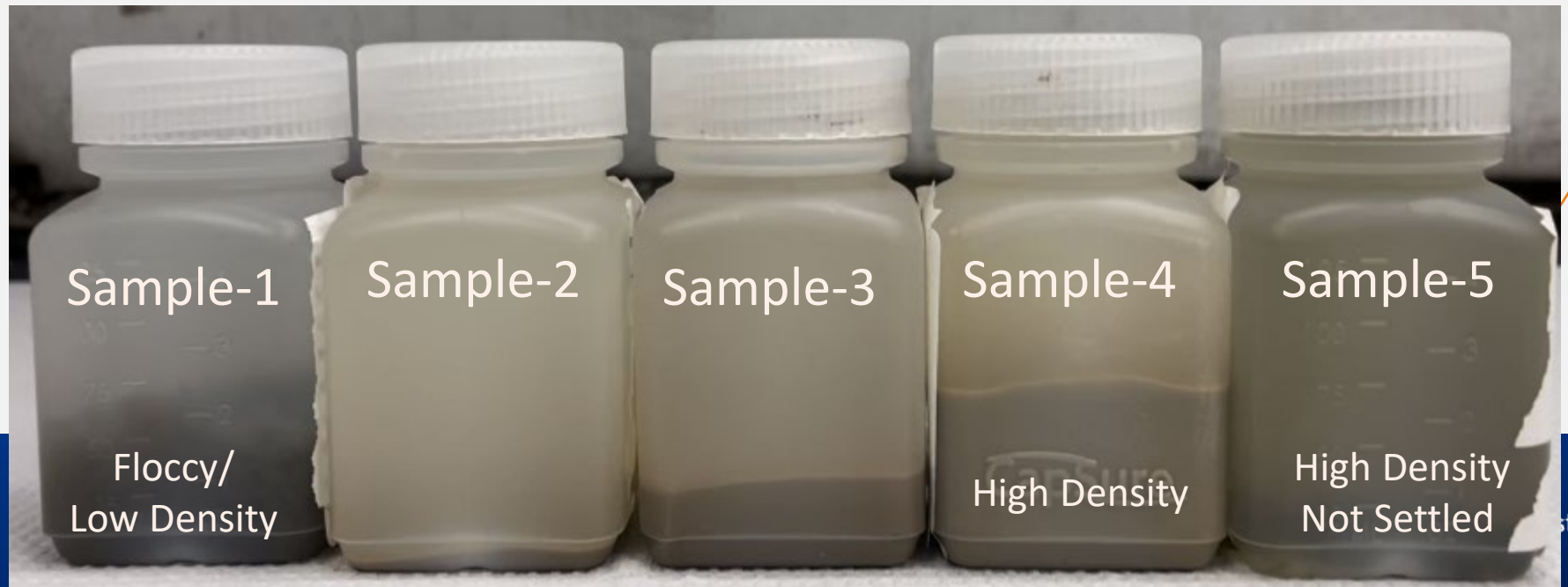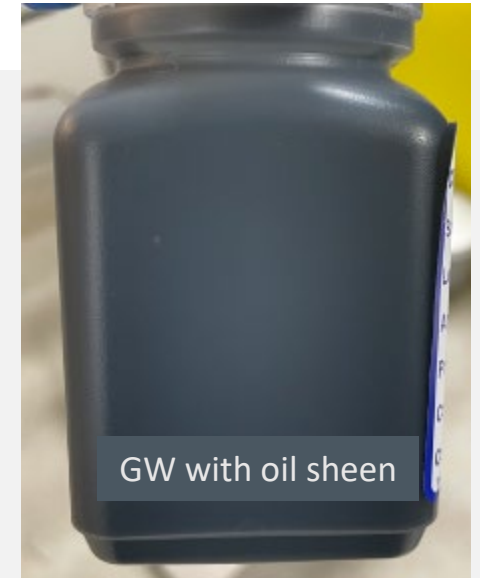
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Aqueous Sample:

- EPA 1633A Section 11.2
- Aqueous (not solvent)
- Low TSS (<50mg/bottle)
  - Must not be filtered
- Does not foam
- Not multiphasic
- Homogenous

Foam

Chalky

1 - WW

2 - Biosolid

GW with oil sheen

Sample-1

Floccy/ Low Density

Sample-2

Sample-3

Sample-4

High Density

Sample-5

High Density Not Settled

Advances and Lessons in EPA Methods 1633A

TSS Matters (>50 mg TSS/sample bottle)
- Density, Amount, Type, etc.

Procedural Options:

- Multiple SPE Cartridges
  - Unknown bias for all analytes

- Subsample
  - Whole bottle represented
  - Elevated RL all analytes

- Spike > Equilibrate* > Centrifuge > Decant
  - Only Water Fraction analyzed
  - Potential data bias for longer chain PFAS due to sorption

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What happens to analytes during equilibration?

- EIS may sorb to particulates

- **Long-Chain EIS** may have significant losses

- Let isotopes (EIS) quantitate

Analyte	LCS	LCSD	Waste Water	Ground Water	1633A %R Limit
13C4 PFBA	95	96	56	55	5-130
13C5 PFPeA	92	97	45	61	40-130
M2-4:2 FTS	92	84	62	96	40-200
13C3 PFBS	88	94	51	71	40-135
13C5 PFHxA	94	96	60	67	40-130
13C3 HFPO-DA	94	95	75	74	40-130
13C4 PFHpA	94	96	66	74	40-130
M2-6:2 FTS	89	95	57	108	40-200
13C8 PFOA	95	96	45	72	40-130
13C3 PFHxS	91	95	34	70	40-130
13C9 PFNA	95	97	23	69	40-130
M2-8:2 FTS	96	95	10	93	40-130
13C6 PFDA	94	97	8	65	40-130
d3-NMeFOSAA	86	83	3	46	40-170
13C8 PFOS	89	90	4	62	40-130
d5-NEtFOSAA	83	81	3	46	25-135
13C7 PFUnA	91	92	2	46	30-130
13C2 PFDoA	88	93	1	36	10-130
13C8 FOSA	91	96	4	70	40-130
13C2 PFTeDA	68	73	1	24	10-130
d7-N-MeFOSE-M	81	87	0	20	10-130
d3-NMePFOSA	66	66	NR	46	10-130
d9-N-EtFOSE-M	79	83	NR	12	10-130
d5-NEtPFOSA	55	60	NR	37	10-130

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What have we learned?

- Screen every sample for high concentration

- Unknown & known biases introduced by:
  - Improper sample collection
  - Increased sampling handling
    - Potential biases (high and low)
    - Increased potential for contamination
  - Complex matrices

- Communication:
  - Field Staff <> Lab <> DOE Project Managers
  - Tell the lab about:
    - Known high concentrations or heavy matrix samples
  - Understand the bias of sampling & sample handling

1633A Project Directives

High TSS

Preserve lowest possible RLs and accept potentially compromised data quality from poor recoveries of EIS or Preserve data quality and accept elevated RLs up to 5x.

Report Over-Cal Results

Over-Calibration results are acceptable as addressed by method 1633A.
"Projects may accept such an estimated value if the result is so high that is it well above the project criteria or action level, especially if additional samples is planned for the site."

Report Best Data

If multiple analyses are performed due to failing QC, report on the data with passing QC.

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Emerging Technologies

PFASsive
TOP Assay
Total Organic Fluorine (TOF)
Non-Targeted Analysis (NTA)

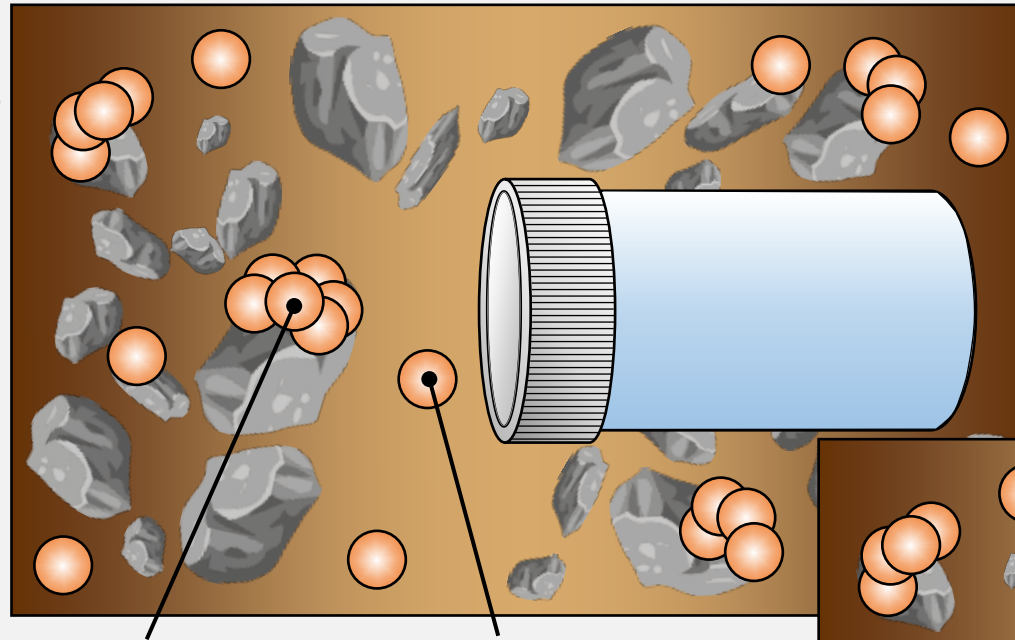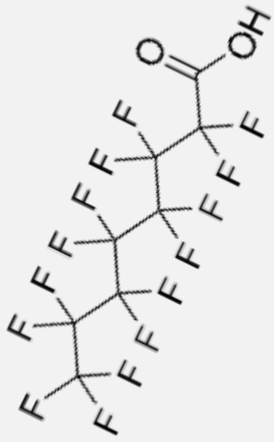eurofins | **Environment Testing**

PFASsive™ – The Solution for PFAS Monitoring

- Provide Critical Data for:
  - Fate and risk assessment
  - Toxicity identification
  - Remediation Design
  - Long Term Monitoring

- SiREM & Eurofins partnered to provide a passive solution for dissolved PFAS monitoring.
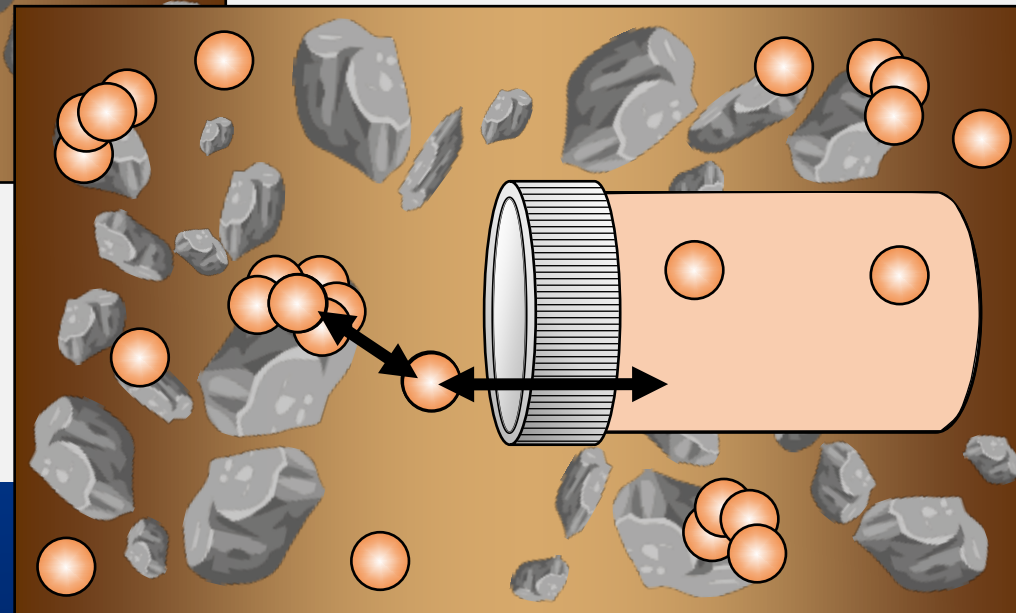
PFASsive™ – The Solution for PFAS Monitoring

- Solution in sampler equilibrates with freely-dissolved species in sediment (days-weeks)
- Sampler removed from sediment, solution transferred and preserved, measured for target analytes using standard methods for water (e.g., EPA 1633A)
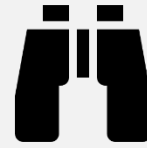
Unavailable Analyte

Freely-dissolved Analyte

Results in ng/L

Screening Methods for PFAS

Rapid Screening

EPA Method 8327

ASTM D8421 (aq.)

ASTM D7968 (solid)

Non-Target Screening

TOP Assay

AOF/EOF

Non-Targeted Analysis

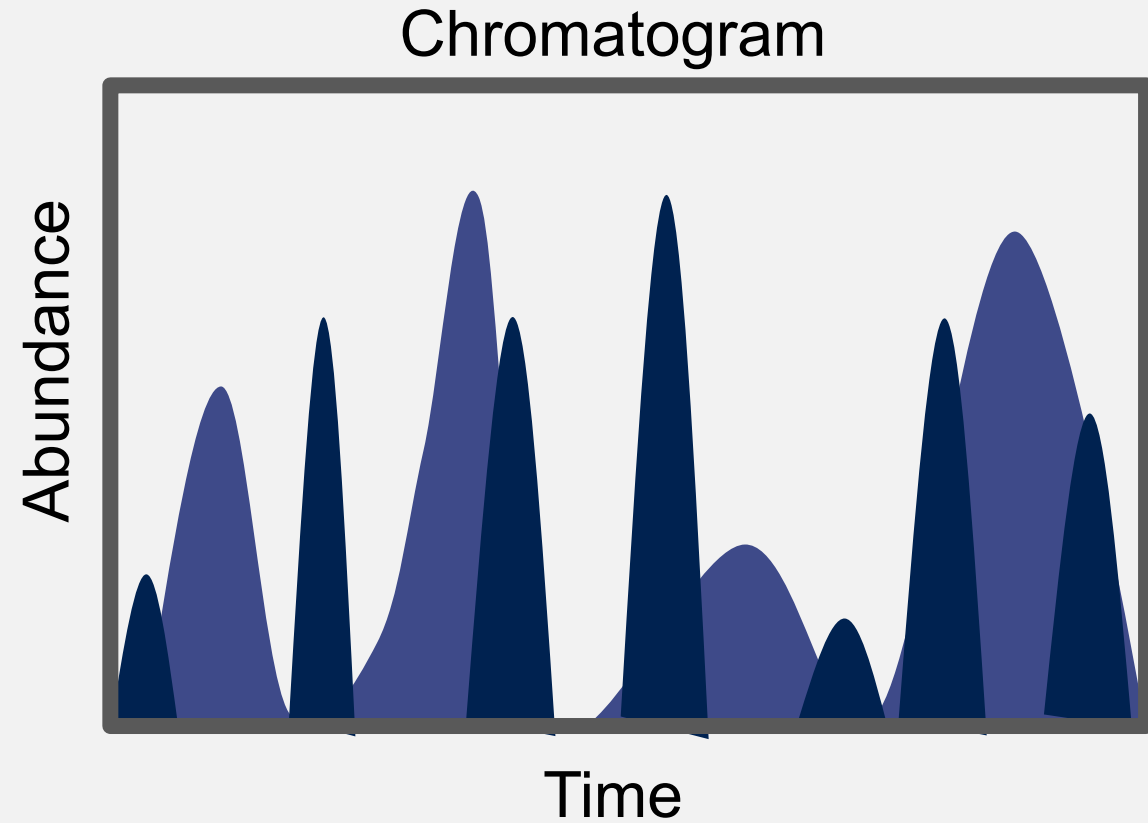**Rapid Screening**

Unknown Screening

- Select compounds
- Specific matrix
- Analytical Standards
- Quantitative
- Closed Analysis

PFBA ☑
PFPeA ☑
PFHxA ☑
PFOA ☐

Targeted Methods:

How much PFOA is in the sample?

Chromatogram

Abundance

Time

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Why explore non-targeted analysis?

Target analysis of priority pollutants

Non-targeted analysis

1-5% identified

95-99% unidentified

- TSCA inventory >86,000 chemicals in commerce (2019)

    - 100s-1000s of suspect PFASs

- >219 million entries in the CAS registry

- Current standard methods include a very limited number of chemicals

We will only find what we are looking for!

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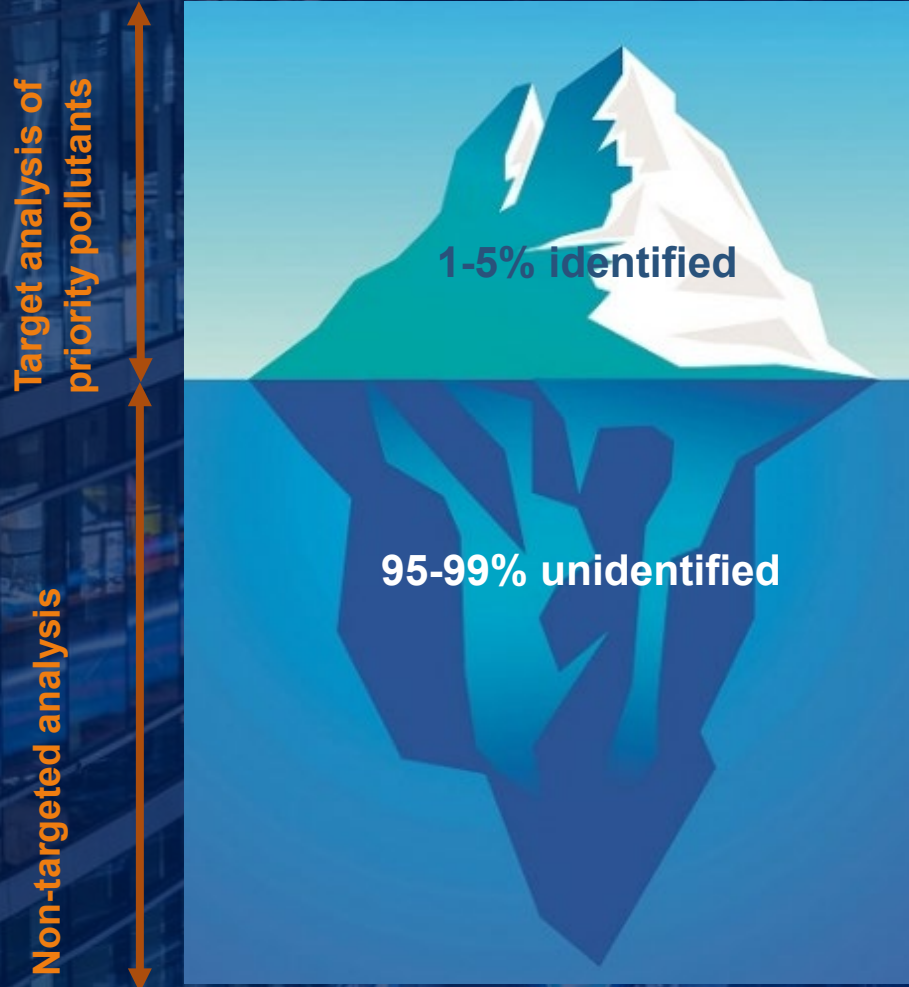
How much transformable PFAS?

TOP Assay

PFAA Precursors **+** OH• OH• OH• 85°C **=** PFCAs
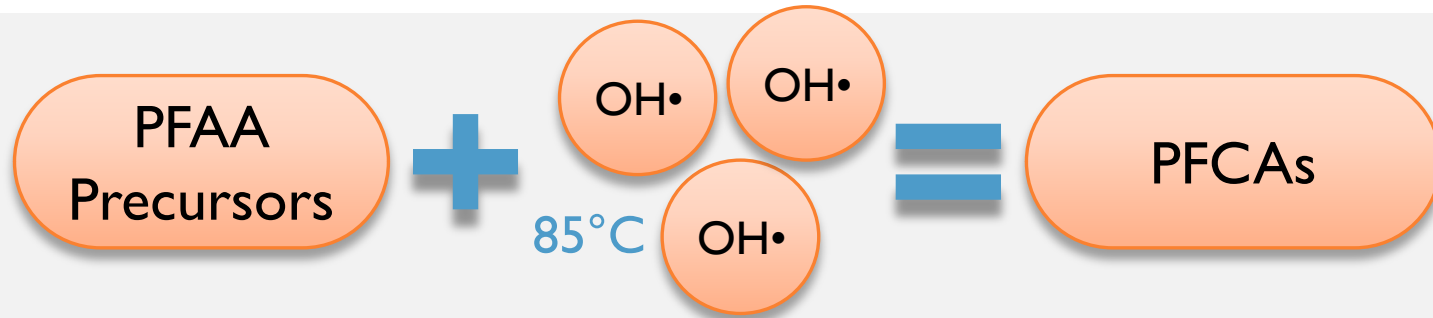
	Pre-Treatment (ng/L)	Post-Treatment (ng/L)
PFBA	ND	46
PFPeA	15	15
PFHxA	11	42
PFHpA	ND	6.3
PFOA	13	14
Total PFCAs	**39**	**148.3**
PFCA Difference:	**109.3 ng/L**	

Cons:
- Matrix effects quench rxn
- Un-measureable transformation products
- Not robust

Pros:
- Quantify oxidizable precursors with some chain length information

AOF/EOF

How much organic fluorine?

Cons:

- Not PFAS specific

- High reporting limits

Pros:

- EPA Method 1621 - AOF

- Robust analysis

- Single result

CIC: Combustion Ion Chromatography
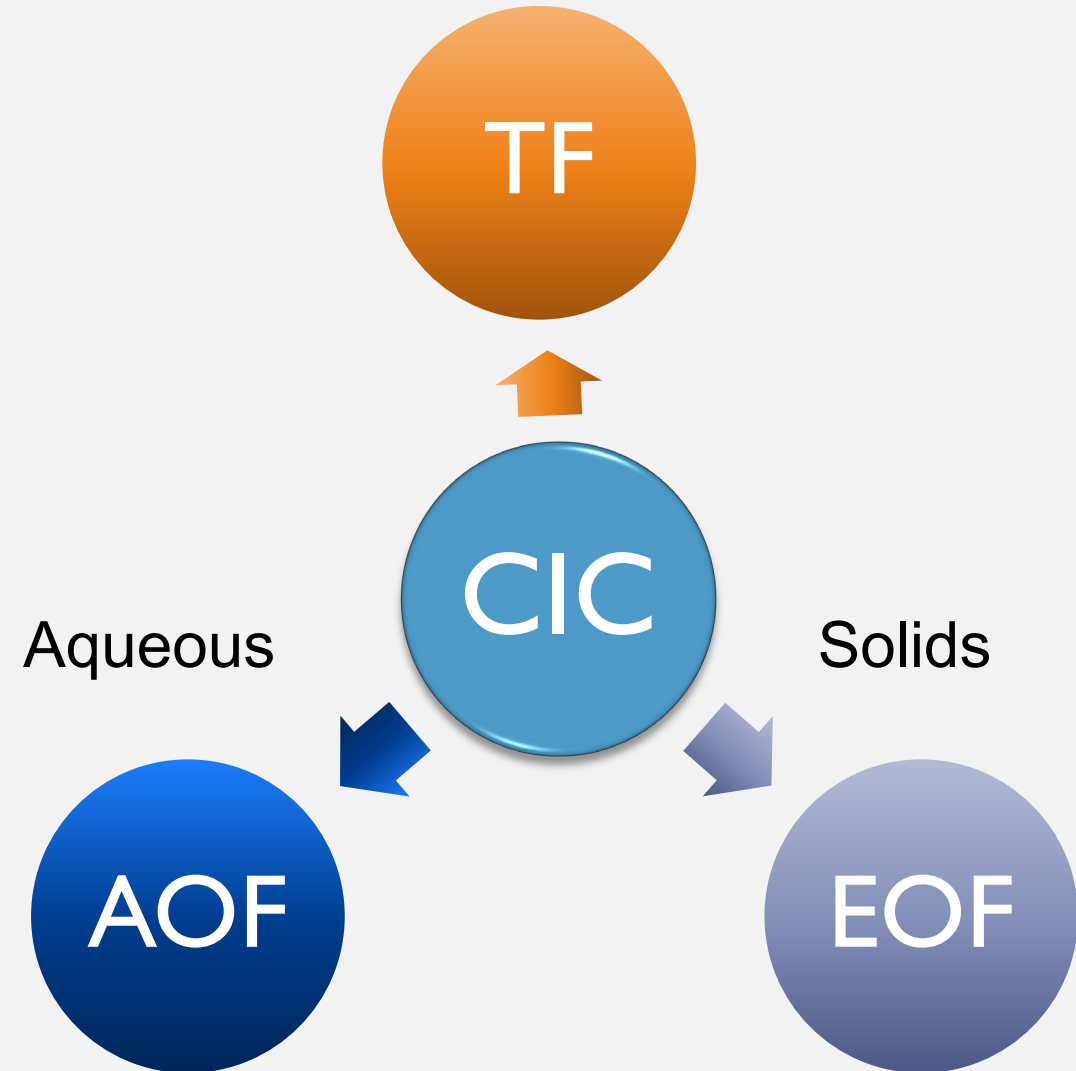
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NTA

Cons:

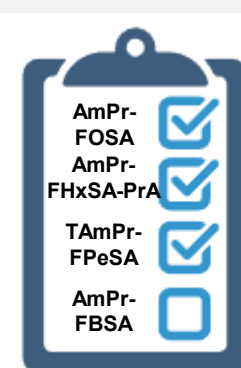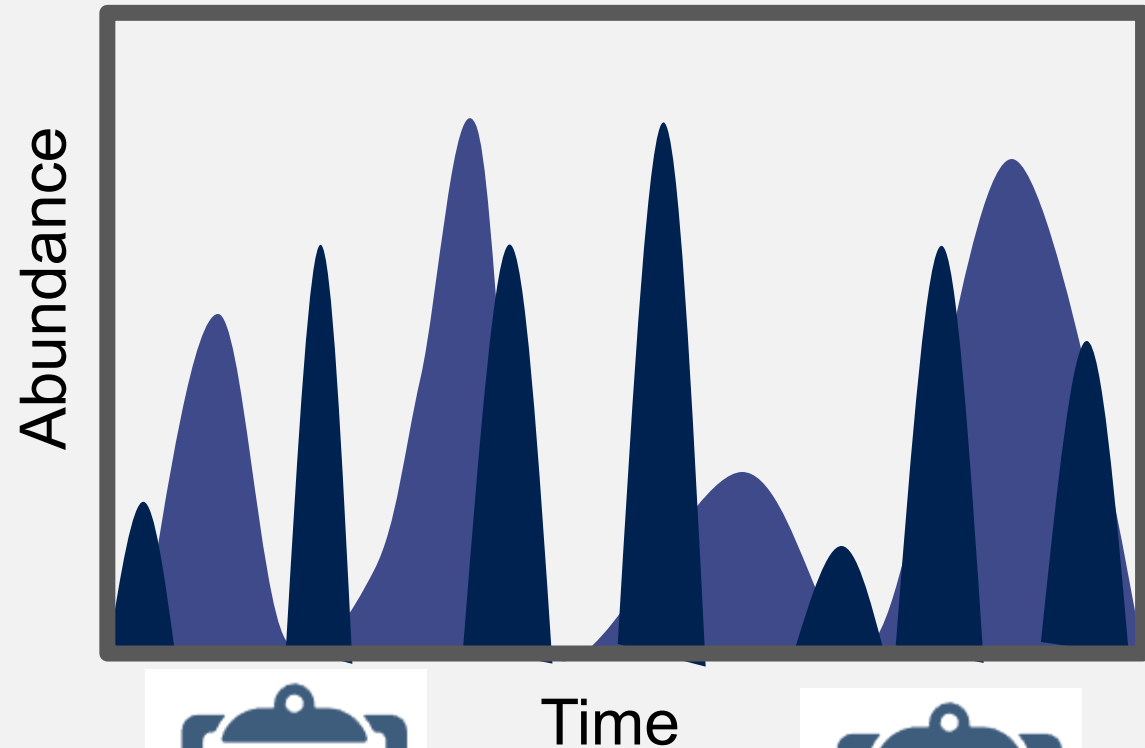- High Resolution Mass Spectrometry

- Semi-Quantitative

Pros:

- User Defined Compound Lists

- No Standards required
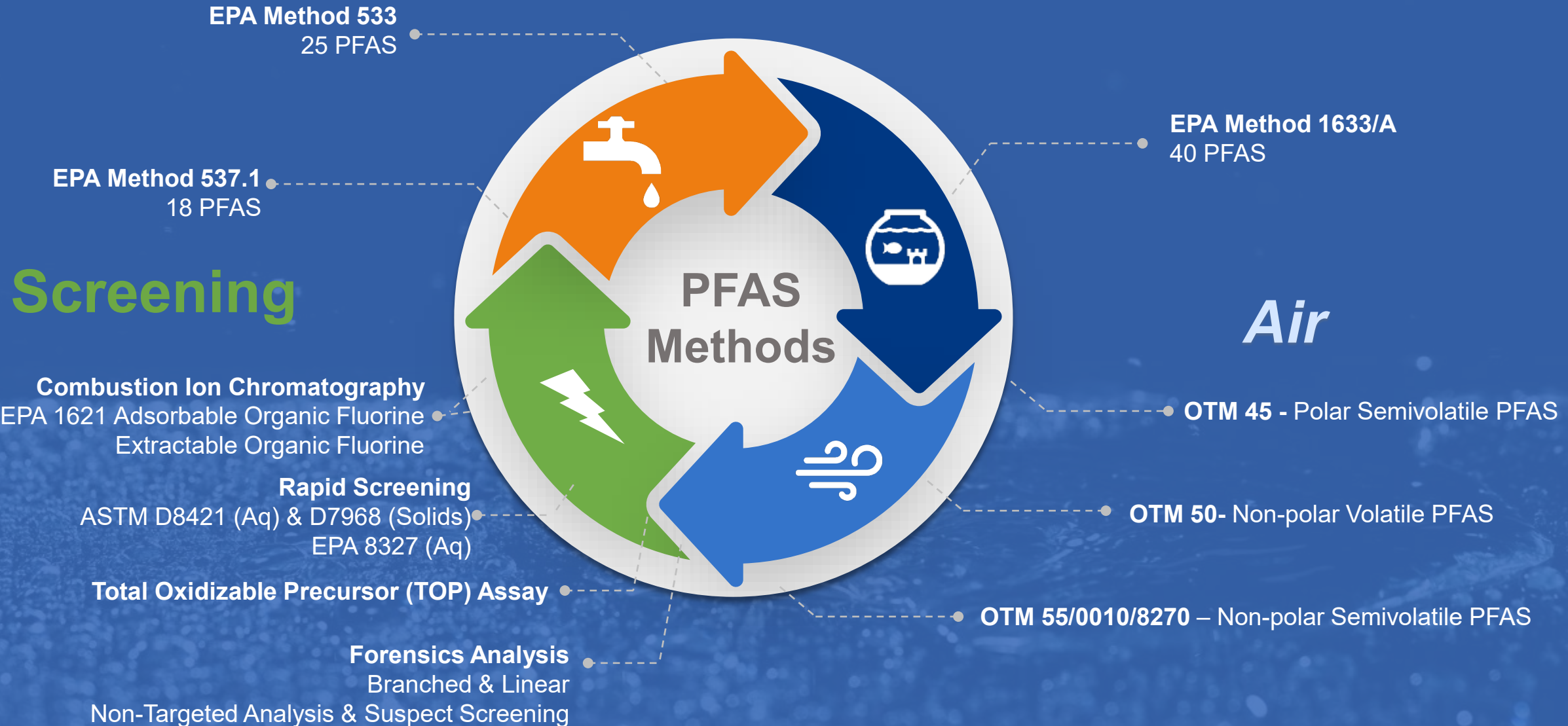
- Open Ended Analysis

Non-Targeted & Suspect Screening
What is in my sample?

Chromatogram

Abundance

Time

PFBA ☑
PFPeA ☑
PFHxA ☑
PFOA ☐

AmPr-FOSA ☑
AmPr-FHxSA-PrA ☑
TAmPr-FPeSA ☑
AmPr-FBSA ☐

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Screening

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EPA 1621 Adsorbable Organic Fluorine
Extractable Organic Fluorine

Rapid Screening
ASTM D8421 (Aq) & D7968 (Solids)
EPA 8327 (Aq)

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Non-Potable Water, Solid, & Tissue

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40 PFAS

PFAS Methods

Air

OTM 45 - Polar Semivolatile PFAS

OTM 50- Non-polar Volatile PFAS

OTM 55/0010/8270 – Non-polar Semivolatile PFAS

https://www.epa.gov/water-research/pfas-analytical-methods-development-and-sampling-research

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QUESTIONS?

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