

Sediment Management Work Group

**The Forgotten Matrix, PFAS in Sediments:
Regulatory Landscape and Analytical Methods
Beyond EPA 1633**

October 28th, 2025

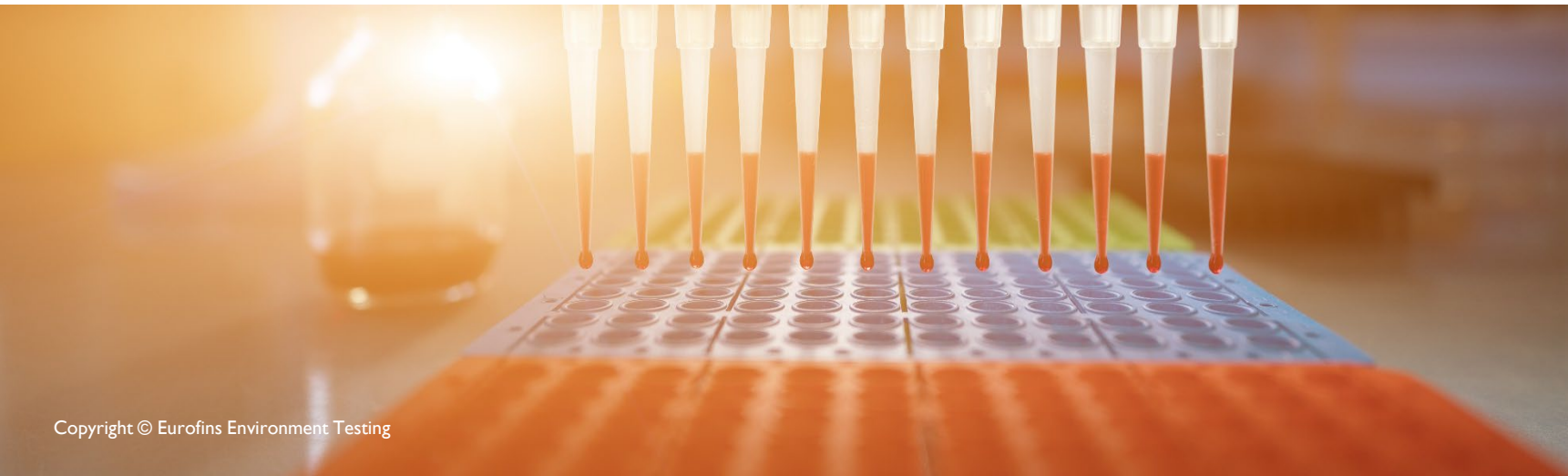
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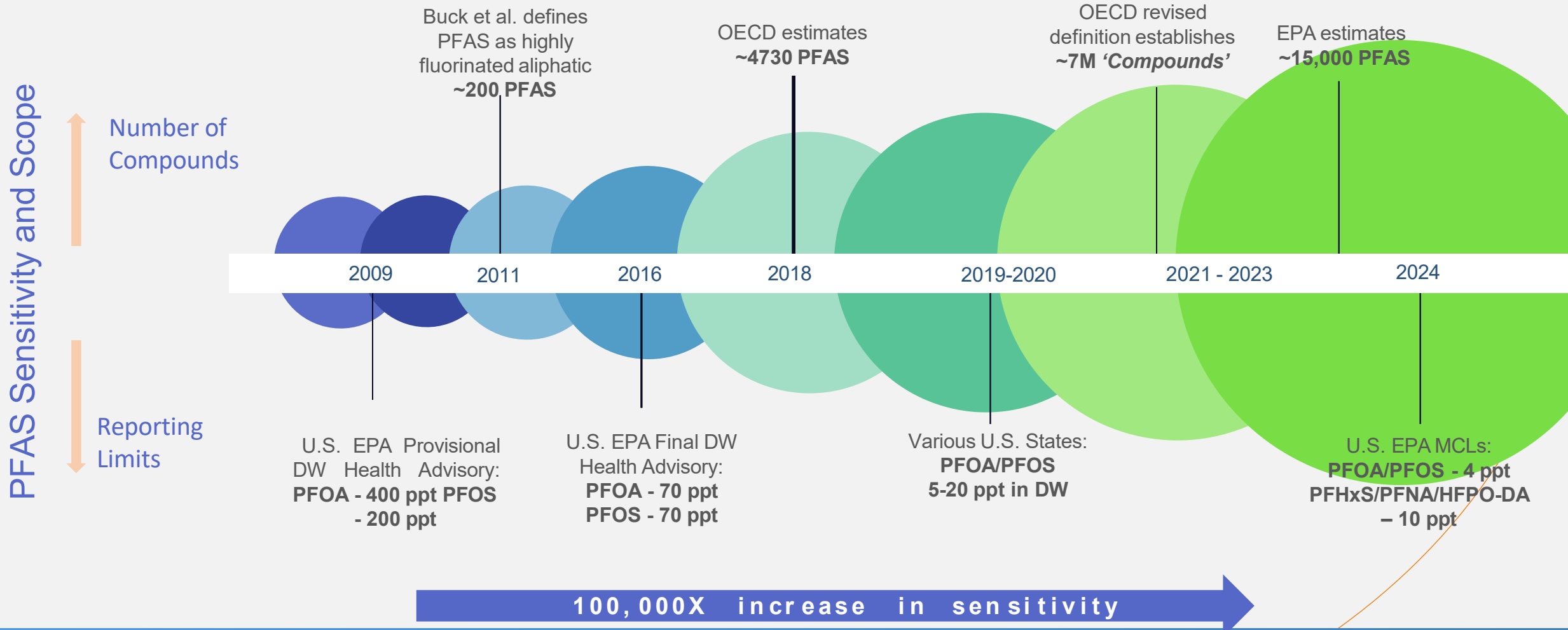
Environment Testing

Presentation Topics / Outline

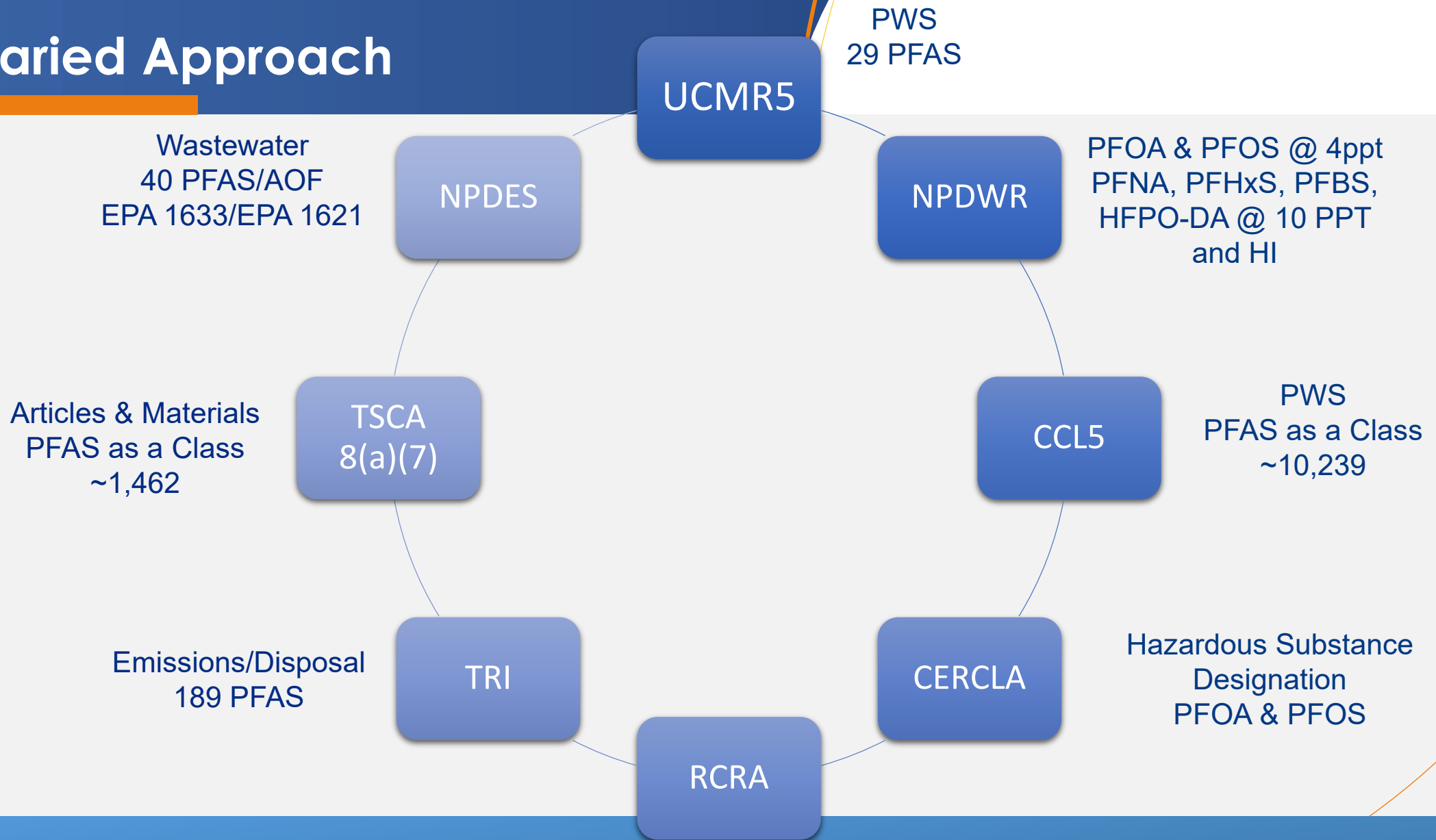
- Regulatory landscape
- EPA 1633A
- 1633 expanded...
- Proxy Methods
- Analytical Results



Evolving Definition of "PFAS"



Varied Approach



Regulatory Landscape



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Legislative Actions and Regulations

- No federal or state regulatory standards specific to PFAS in sediment.
- EPA RSLs
- USACE dredge material projects
- Canada – Federal Contaminated Sites Inventory
- EU – no regulatory standards
- Australia – NEMP 3.0

The Published Method: EPA 1633A



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EPA 1633A (December 2024)

- CWA method
- Multi-lab validated for surface water, groundwater, wastewater, landfill leachate, soil, sediment, biosolids, fish, and shellfish tissue
- Solid Phase Extraction - aqueous
- Solvent extraction followed by SPE – all other matrices
- Carbon cleanup
- LC/MS/MS
- Isotope Dilution quantification
- 40 target analytes

EPA 1633A Multi-Lab Validation Study

- 8 Commercial / 2 state laboratories
- Tested in real world matrices
 - No fortifications
 - Low-level fortification
 - High-level fortification
 - Each in triplicate
- Four method drafts, two final versions
- Announced for promulgation December 2024
- MUR 22 posted 1/21/2025

Links to MLV Study Reports:

- [Wastewater, Surface Water, and Groundwater](#)
- [Soil and Sediment](#)
- [Biosolids and Landfill Leachate](#)
- [Tissue](#)

EPA 1633A – Target Analytes

| PFCA | PFSA | FTS | PFOSA | PFECA |
|---|---|---|--|--|
| <ul style="list-style-type: none">•PFBA•PFPeA•PFHxA•PFHpA•PFOA•PFNA•PFDA•PFUnA•PFDoA•PFTTrDA•PFTeDA | <ul style="list-style-type: none">•PFBS•PFPeS•PFHxS•PFHpS•PFOS•PFNS•PFDS•PFDoS | <ul style="list-style-type: none">•4:2 FTS•6:2 FTS•8:2 FTS | <ul style="list-style-type: none">•PFOSA•NMeFOSA•NEtFOSA | <ul style="list-style-type: none">•HFPO-DA•ADONA•PFMPA•PFMBA•NFDHA |
| | | FTCA | FOSAA | PFESA |
| | | <ul style="list-style-type: none">•3:3 FTCA•5:3 FTCA•7:3 FTCA | <ul style="list-style-type: none">•NMeFOSAA•NEtFOSAA | <ul style="list-style-type: none">•9Cl-PF3ONS•11Cl-PF3OUdS•PFEEESA |
| | | | FOSE | |
| | | | <ul style="list-style-type: none">•NMeFOSE•NEtFOSE | |

EPA 1633A – Section 9.1.2

In recognition of advances that are occurring in analytical technology, and to overcome matrix interferences, the laboratory is permitted certain options to improve separations or lower the costs of measurements.

These options include alternative extraction, concentration, and cleanup procedures, and changes in sample volumes, columns, and detectors.

Alternative determinative techniques and changes that degrade method performance are not allowed without prior review and approval (see 40 CFR 136.4 and 136.5).

1633A Expanded...



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1633A EXP... What is that??

- Section 1.6: In addition, under the allowance for flexibility and method modifications described at 40 CFR 136.6, laboratories may include additional PFAS compounds as target analytes and/or alternative labeled PFAS compounds as authentic standards become commercially available without the need for the EPA review and approval, provided that they complete the requirements in Section 9 for those new analytes.
- 1633A EXP – 59 additional analytes, 15 new EIS

1633A EXP - Analytes

| | EPA 1633 | 1633 EXP | Combined |
|--|----------|----------|----------|
| Perfluoroalkyl carboxylic acids (PFCA) | 11 | 3 | 14 |
| Perfluoroalkyl sulfonic acids (PFSA) | 8 | 4 | 12 |
| Fluorotelomer sulfonic acids (FTSA) | 3 | 1 | 4 |
| Perfluorooctane sulfonamides (PFOSA) | 3 | 6 | 9 |
| Perfluorooctane sulfonamidoacetic acids (FOSAA) | 2 | 0 | 2 |
| Perfluorooctane sulfonamide ethanols (FOSE) | 2 | 0 | 2 |
| Per- and Polyfluoroether carboxylic acids (PFECA) | 5 | 15 | 20 |
| Perfluoroalkyl ether sulfonic acids (PFESA) | 3 | 6 | 9 |
| Fluorotelomer carboxylic acids (FTCA) | 3 | 3 | 6 |
| Perfluoroalkyl Phosphinates (PFPIA) | 0 | 4 | 4 |
| Polyfluoroalkyl Phosphate di-esters (diPAP) | 0 | 4 | 4 |
| Fluorotelomer unsaturated carboxylic acids (FTUCA) | 0 | 3 | 3 |
| Diprotic | 0 | 3 | 3 |
| perfluorinated sulfonylimides (PFSI) | 0 | 2 | 2 |

Total PFAS and Proxy Methods



Combustion Ion Chromatography (CIC)

- Option 1 – Total Fluorine (TF)
 - Direct combustion of a sample
 - RL ~ 300ppb
 - Soluble and Insoluble Inorganic Fluoride
 - All Organic Fluorine
 - Polymeric and non-polymeric PFAS
 - Other non-PFAS organic fluorine



Combustion Ion Chromatography (CIC)

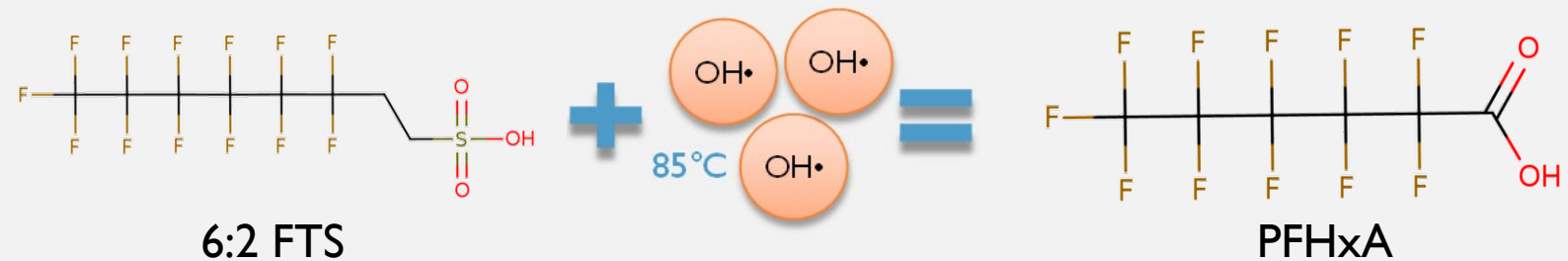
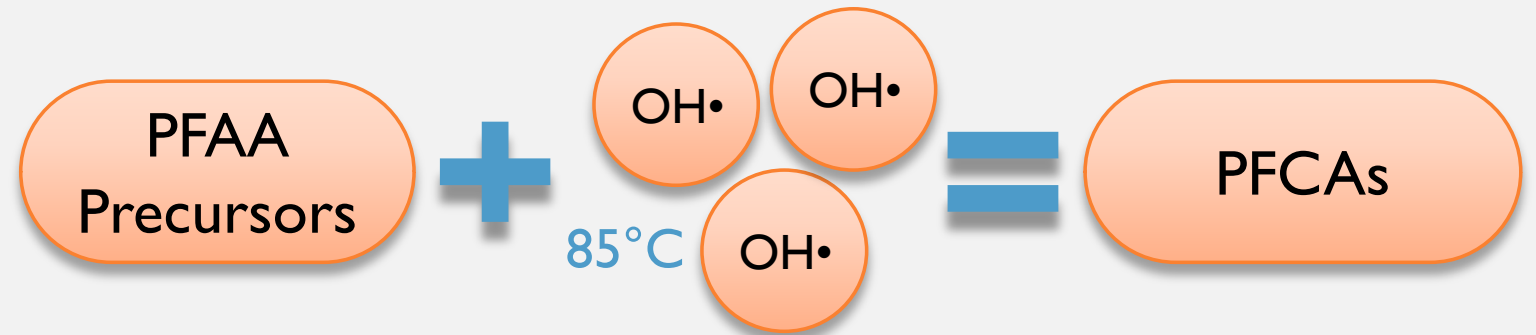
- Option 2 – Extractable Organic Fluorine (EOF)
 - RL ~ 300ppb
 - Solvent extraction followed by combustion
 - Organic Fluorine amenable to the solvent used
 - non-polymeric PFAS
 - Other non-PFAS organic fluorine



EPA 300 – Inorganic Fluoride

- What about the Inorganic?
 - Only includes soluble Inorganic Fluoride
 - Does not include insoluble
 - Reporting Limits much higher (low ppm)
- All samples were non-detect for soluble inorganic fluoride
- Sources may include:
 - Insoluble inorganic fluoride (CaF_2)
 - Poor solubility of organic fluorine in the solvent
 - Organic fluorine bound to the matrix
 - Polymeric PFAS

Total Oxidizable Precursor (TOP) Assay



Under-oxidation = 6:2 FTS untransformed

Over-oxidation = PFHxA, PFPeA, PFBA, PFPrA, PFEtA, TFA

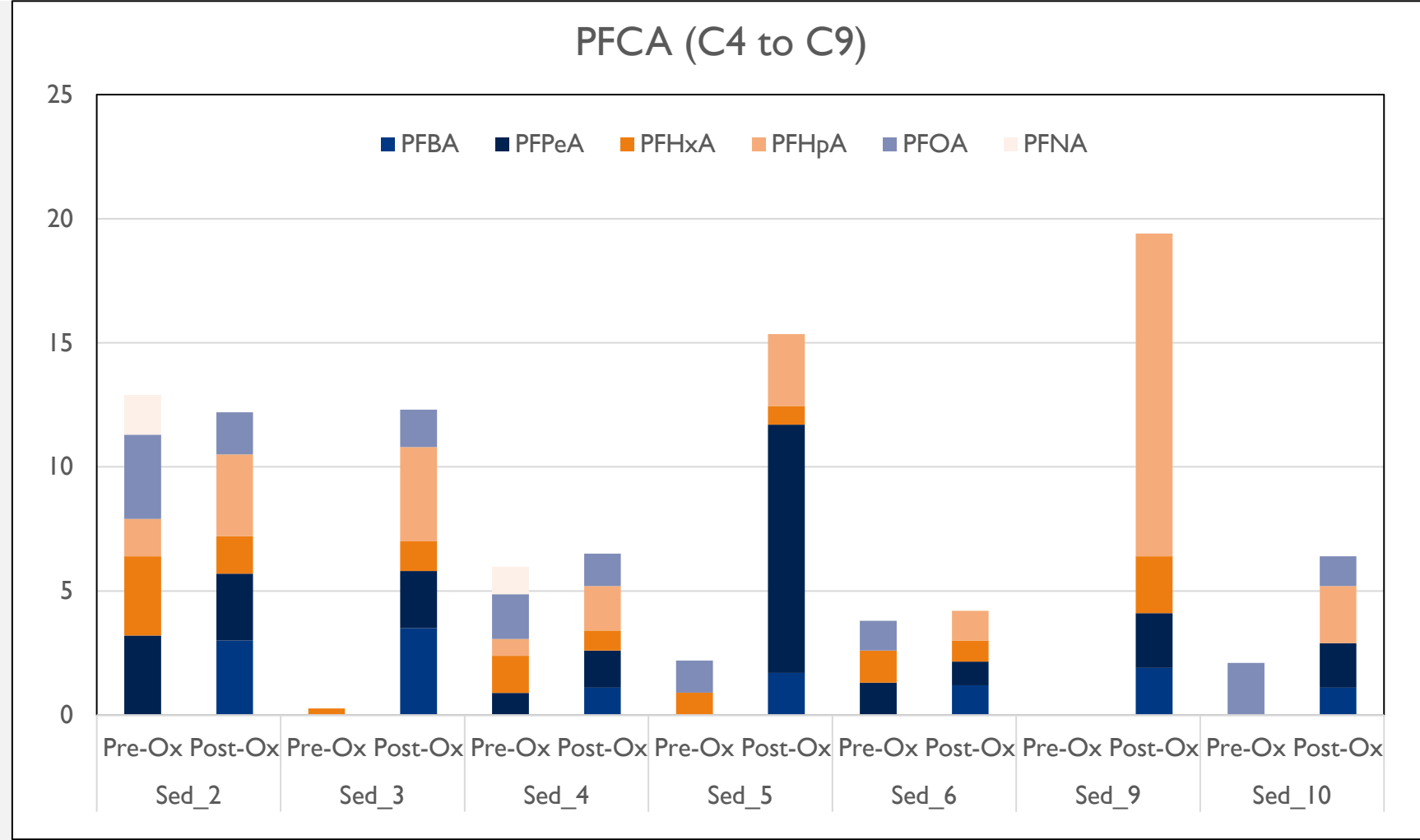
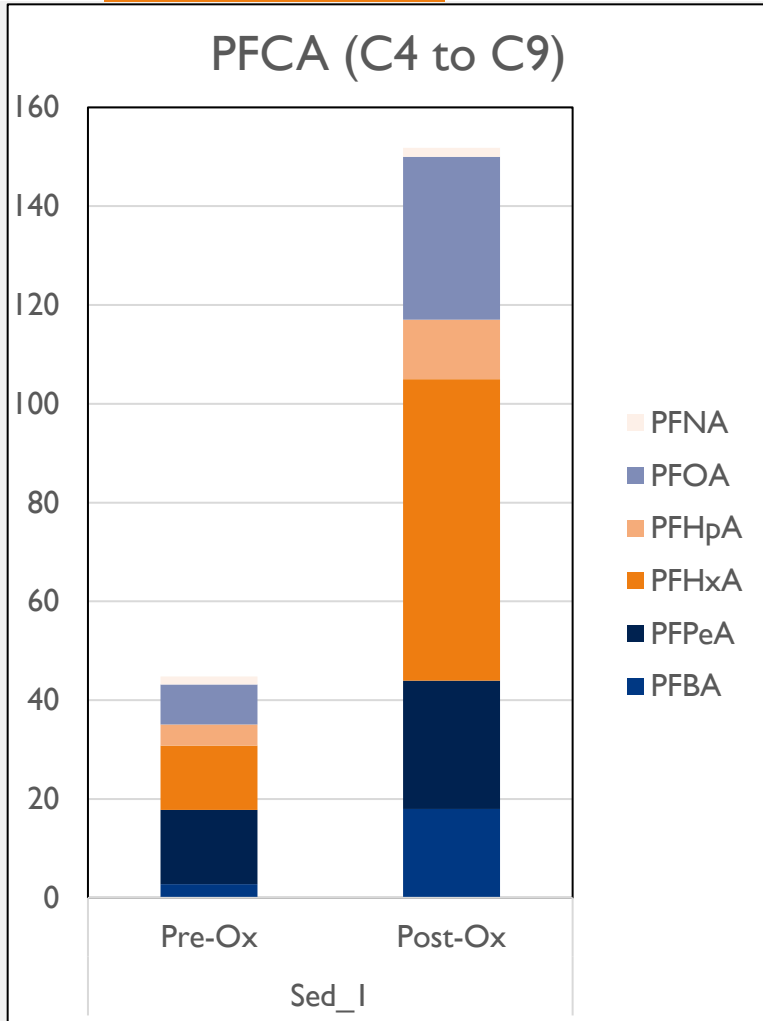
- Cons
 - Matrix interference
 - Forms non-measurable transformation products
 - Not robust
 - Over & Under oxidation
- Pros
 - Quantify precursor PFAS with Chain length

Analytical Results

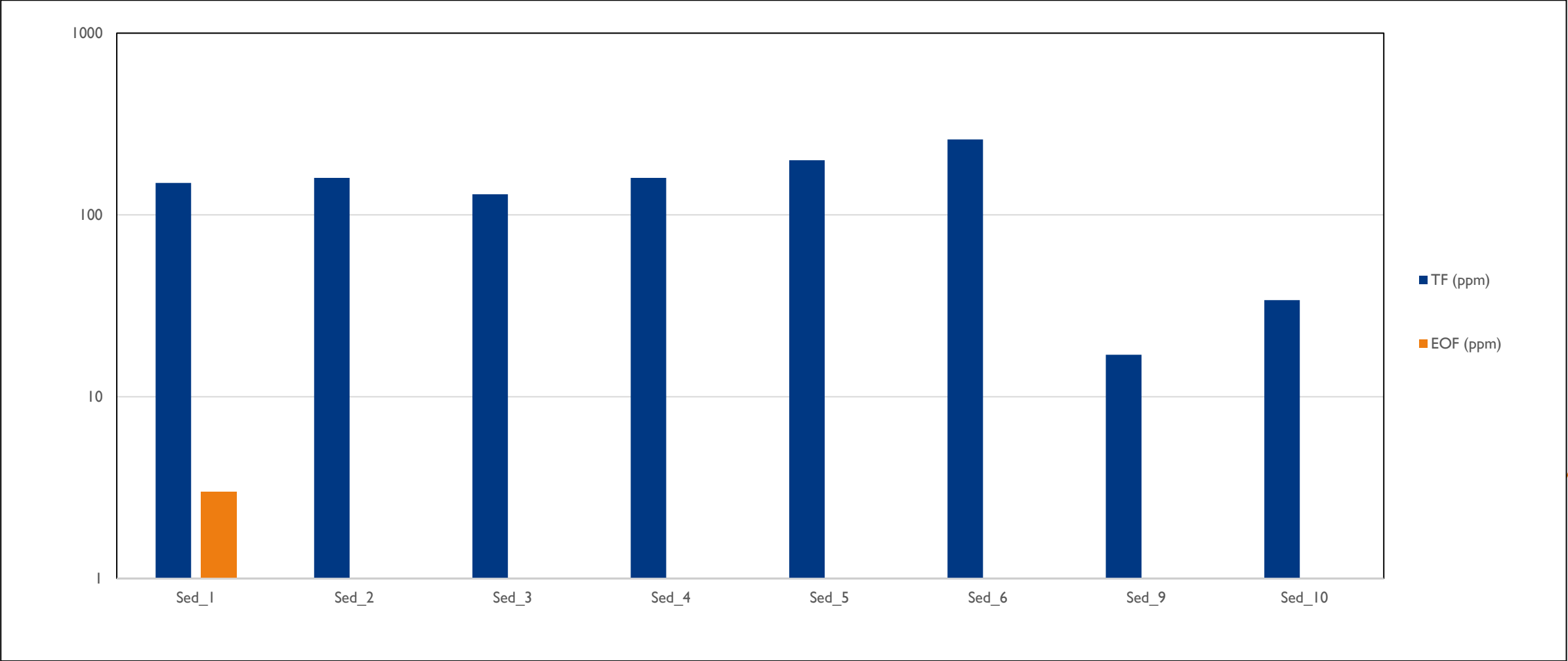


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Total Oxidizable Precursors (TOP)



Combustion Ion Chromatography (CIC)



Conclusions

- EPA 1633A
- Sediments are a receptor for PFAS
- Precursors are present in sediment samples
- Hopefully... we will see suggested limits on sediments soon??

THANK YOU



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