PFAS Accredited Laboratories – Hurdles and Other Challenges

DOE/ASP Annual Training Workshop

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QUALITY DRIVERS

What Are The Data Being Used For?

NON-POTABLE WATER, SOLIDS, & AIR

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User-Defined Methods: PUT TO THE TEST!

Complex Matrices

- Biphasic
- Biosolids
- Tissues
- Dispersions
- Activated Carbon
- Cosmetics
- Concrete

Audits & PTs

- NELAC
- DoD ELAP
- Client/Program Specific Audits
- Semiannual PT
- NMI International Round Robin
- DOW Study

3rd Party Validation

- >85% of all PFAS data includes a validation package
- >400,000 sample data validated

Cornerstone of PFAS Methodology: Isotope Dilution

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Intention of isotope dilution:

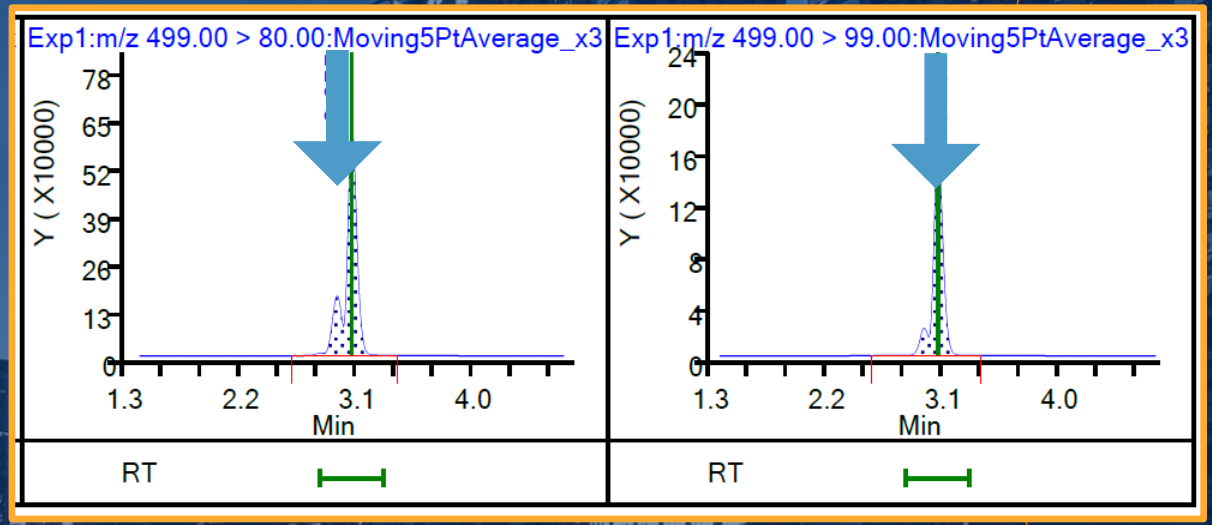
- allow for recovery correction
- normalize performance across matrices

EIS recoveries of 50 – 200% are arbitrary and limiting of the application

Can result in unnecessary qualification of data

Native = 100 ppt

EIS Rec = 25%



Legacy isotope dilution methods practiced for over 30 years (D/F by 1613) allow recovery of isotopes as low as 10% and 15%, with no deleterious impact on performance

EPA Draft Method In Progress

EPA Draft 1633

- Targeted Analysis of 40 PFAS
- Non-Potable Water, Soil & Tissue
- LCMSMS, WAX SPE, Isotope Dilution
- Multi-Lab Validation Underway

EPA METHODS

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https://www.epa.gov/cwa-methods/cwa-analytical-methods-and-polyfluorinated-alkyl-substances-pfas

Department of Defense

"PFAS by LCMSMS Compliant with Table B-15 QSM 5.1 or latest version"

PFAS by Draft 1633 Table B-24 of QSM 5.4

www.denix.osd.mil/edqw/documents/manuals/qsm-version-5-3-final/

EPA *Draft* 1633 for Non-Potable Water & Solids

SIMILARITIES

- Applicable to a variety of solids and aqueous matrices
- Solid Phase Extraction using WAX
- Isotope Dilution Quantitation using all available isotopes
- Ion Transitions, monitoring ratios
- *Using non-Extracted Internal Standards (NEIS) for quantitation of extracted internal standards (EIS)
- **Use of carbon cleanup

*QSM 5.3 dropped it, but they are bringing it back with B-24

**User-defined methods use stacked carbon vs. loose carbon

Compared to:
User-Defined Methods
and
DoD QSM Table B-15

DIFFERENCES

- Soil/Tissue Prep: concentration step
- S/N Ratio
- Waters Oasis WAX SPE Cartridge with loose carbon cleanup
- TDCA Check: 60 sec window specification
- Includes frozen storage option
- Complex dilution scheme with 10X dilution limitation
- Mass transitions vary for some

Cost, Capacity, and Quality Implications

REQUIREMENT

1. 3:1 S/N Ratio
2. WAX SPE Cartridge with loose carbon cleanup
3. TDCA Check: 60 second window specification
4. Analytical Run Time
5. Additional NIS

IMPLICATIONS

	COST	CAPACITY	QUALITY
1.			Criteria going from 10:1 down to 3:1
2.	Stacked cartridge is cheaper	More sample handling impacts throughput	More sample handling & Time of exposure on loose carbon impacts recovery of the long chain acids
3.	Cost of standards & Preparing/integrating into the calibration	RT requirement adds to run time, impacting throughput	Improvement from requirement for chromatographic resolution
4.	To avoid the 2X run time for waters and soils would have to dedicate instruments to tissues only, added instrumentation costs	Run time is now 2X with the 60 sec TDCA check window, unless you dedicate an instrument to tissues only, huge throughput implications either way	
5.	Cost of additional standards	Additional compounds that must be within the acceptance limits may lead to more re-analyses	Potential for improved data quality

Cost, Capacity, and Quality Implications

REQUIREMENT

6. Holding time/preservation options
7. 500mL sample volume
8. EIS criteria and corrective action
9. Use of glass wool
10. Additional filtration step

IMPLICATIONS

	COST	CAPACITY	QUALITY
6.	If frozen storage is required there are significant cost implications to purchase and build out frozen storage	To thaw out a frozen sample is ~8hr process, implications on interim storage and throughput	Quality implication is unclear
7.	Cost of shipping, storage, and processing larger volumes	Processing larger volumes impacts throughput	
8.	Potential for multiple re-analyses and re-extractions	Potential for multiple re-analyses and re-extractions	Potential for raised RLs to be reported
9.	Cost of manual process and materials	Manual process impacts throughput	Manual process can lead to variability
10.	Cost of manual process and materials	Manual process impacts throughput	More sample handling

Cost, Capacity, and Quality Implications

REQUIREMENT

11. Soil/Tissue Extraction Procedures

12. TSS measurement

13. Reporting Limits for Waters and Biosolids

14. Final Volume

IMPLICATIONS

	COST	CAPACITY	QUALITY
11.	Requires two 8hr work shifts so potentially a two-day process, adds labor costs	Requires two 8hr work shifts so potentially a two-day process, limits rush capabilities and impacts throughput significantly	More sample handling
12.	Depending upon interpretation of the method, added cost from separate analysis	Depending upon interpretation of the method, diminished throughput from separate analysis	
13.			Potential impacts on achieving DQOs with elevated limits from previous method used.
14.	Cost of standards		

Industry-Wide Capacity Implications

Instrument configuration is so unique it requires dedicated instruments (uses a different mobile phase solvent and modifier)

Without statewide adoption of a DRAFT method, we must juggle capacity for existing accredited methods and the Draft 1633 method.

Has not been put through the rigor with wide range of real-world samples yet. Efficiencies that result in increased throughput are yet to be realized.

CAPACITY

Industry Capacity Implications

The Key To Success….

will be implementation of the method as the performance-based method it was written to be

This method is "performance-based," which means that modifications may be made without additional EPA review to improve performance (e.g., overcome interferences, or improve the sensitivity, accuracy, or precision of the results) provided that all performance criteria in this method are met.

Draft Method 1633, August 2021

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SUSTAINABILITY

Doing Our Part

Advanced Technology
Reducing shipping volumes
Reducing solvent use
Reducing waste

LOW VOLUME Initiative

50mLs or less

- Collaboration with Government and Industry
- Validate Draft 1633 using smaller sample volumes

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THANK YOU

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