

High Throughput Automated Solid Phase Extraction of UCMR5 PFAS Compounds



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Presentation Outline



SPE-03 8-Channel Automated SPE System

199939

UCMR 5 Overview

1. Scope

- EPA Methods 537.1 + 533 (29 PFAS)
- >10,000 PWSs
- 2. Participation
 - 34 labs currently approved
 - Sampling period 2023-2025



EPA Method 533 and 537.1

• EPA Method 533

- 25 compounds
- Weak Anion Exchange
- Isotope Dilution

• EPA Method 537.1

- 18 compounds
- Reverse Phase SPE

Both	EPA 533	EPA 537.1
PFBS	PFBA	NMeFOSAA
PFHxA	PFMPA	NEtFOSAA
PFHpA	PFPeA	PFTA
PFHxS	PFMBA	PFTrDA
PFOA	PFEESA	
PFOS	NFDHA	
PFNA	PFPeS	
PFDA	FPHpS	
PFUnA	4:2 FTS	
PFDoA	6:2 FTS	
9 CI-PF3ONS	8:2 FTS	
11 Cl-PF3OUdS		
HFPO-DA (GenX)		
ADONA		

EPA Method 533

EPA Method 537.1



EPA Method 533

Conditioning

10mL MeOH 10 mL of phosphate buffer 2-3 mL of phosphate buffer Fill with water

EPA Method 537.1

15 mL MeOH 15-18 mL of water Fill with water

*Do not allow cartridge to go dry

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Sample loading

250 mL sample at 5 mL/min

250mL sample at 10-15 mL/min





Bottle rinsing and cartridge wash

1 mL of MeOH

7.5 mL of water







Addressing Challenges of Manual SPE





Manual SPE	SPE-03 (8-Channel)
Flow control	Positive pressure pumps with constant flow
Bottle rinsing	Automated rinse with bottle shaking
Cartridge clogging	Much higher pressure than vacuum Cartridge clogging detection

- 1. Size and Efficiency
 - Compact footprint
 - 8 samples in parallel

Valves in conventional design

Patented multi-channel valve



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2. PFAS Background

- Replace PTFE lines
- Replace PTFE valve rotor



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3. Bottle Rinsing

• Integrated rack



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- Built-in resonators



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4. Flow control

Vacuum Manifold

- One shared vacuum source
- Non-uniform flow
- Constant supervision
- Clogs easily



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4. Flow control

SPE-03 Pumps

- Positive pressure
- Uniform flow across all samples
- Sorbent does not go dry
- Resistant to clogging



Method 533 - Data Collection

- Field extraction data from **Alpha Analytical**, Massachusetts, March to April 2021
- Including both **SPE-03** and **manual extraction**



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• Materials:

- PromoChrom SPE-03 with MOD-004 (sample bottle rinsing) and MOD-005 (minimal-Teflon option)
- o Phenomenex Strata[™]-X-AW 33 µm Polymeric Weak Anion, 500 mg/6 mL SPE cartridge
- o SCIEX 4500 LC/MS/MS using ExionLC UHPLC







 Used for background check









Method 533 - SPE-03 Method

Action Inlet Flow Volume Elute W2 10 mL/min Solvent 1 10 mL Elute W1 Solvent 3 10 mL 10 mL/min Conditioning Elute W1 Solvent 3 3 mL 10 mL/min Elute W1 Solvent 5 10 mL/min 3 mL Add Samp W1 Sample Sample loading 5 mL/min 270 mL Rinse W1 Solvent 4 5 mL/min 10 mL Shake Time based 30 s Bottle rinsing and cartridge wash Clean Solvent 1 5 mL/min 1 mL Add Samp W2 Sample 5 mL/min 9 mL Nitrogen drying Blow N2 Time based 5 min Rinse 1 Solvent 5 2 mL/min 5 mL 2 mL/min 5 mL Rinse 1 Solvent 5 Bottle rinsing and Shake Time based 10 s elution Collect 1 Sample 2 mL/min 4.5 mL

Compound	Measured Concentration [ng/L]							Std	MDL	
	#1	#2	#3	#4	#5	#6	#7	#8	Dev	[ng/L]
PFBA	1.00	1.00	0.97	1.16	1.00	0.98	0.83	1.00	0.10	0.29
PFMpA	1.00	1.06	0.98	1.12	1.13	1.05	1.00	0.97	0.07	0.20
PFPeA	1.00	1.44	1.37	1.44	1.32	1.26	1.42	1.36	0.07	0.20
PFBS	0.88	0.97	0.92	1.32	0.91	0.94	0.83	0.93	0.16	0.48
PFMBA	1.00	1.09	0.98	1.10	0.99	0.87	1.04	1.17	0.09	0.27
PFEESA	0.88	0.90	0.82	0.89	0.95	0.88	0.81	0.86	0.05	0.14
NFDHA	1.00	1.11	1.08	1.22	1.03	0.94	0.96	1.08	0.09	0.26
4:2FtS	0.92	0.96	0.83	0.98	1.00	0.75	1.03	0.98	0.11	0.34
PFHxA	1.00	1.05	0.95	1.27	1.07	1.21	0.98	1.04	0.13	0.39
PFPeS	0.96	0.74	1.01	0.92	1.27	0.96	0.81	1.06	0.19	0.56
HFPO-DA	1.00	1.18	1.04	0.89	0.90	0.72	0.95	1.12	0.15	0.44
PFHpA	1.00	1.05	1.06	1.14	0.86	1.14	0.94	0.90	0.11	0.34
PFHxS	0.92	1.03	0.75	0.96	1.01	0.84	0.72	0.90	0.15	0.44
ADONA	0.96	1.05	1.14	1.13	1.14	0.84	0.93	0.96	0.11	0.33
6:2FtS	0.96	0.80	1.88	1.56	1.12	1.30	1.43	1.20	0.32	0.95
PFOA	1.00	0.98	1.14	1.12	0.96	1.00	0.89	1.27	0.12	0.37
PFHpS	0.96	1.04	0.84	0.78	1.09	0.97	0.79	0.75	0.14	0.41
PFNA	1.00	1.14	1.12	0.81	1.07	1.04	1.02	1.23	0.14	0.43
PFOS	0.92	0.95	0.84	1.04	1.14	0.92	0.91	0.70	0.14	0.42
9CI-PF3ONS	0.92	0.95	0.83	0.81	0.85	0.81	1.00	0.79	0.08	0.23
8:2FtS	0.96	0.99	0.88	1.09	1.34	1.18	0.97	1.00	0.15	0.44
PFDA	1.00	1.18	1.04	1.14	1.09	1.02	0.97	1.11	0.10	0.30
PFUnA	1.00	1.09	1.10	1.22	1.17	0.98	1.04	1.04	0.07	0.22
11Cl-PF3OUdS	0.96	0.75	0.73	0.74	0.98	0.76	0.80	0.72	0.09	0.26
PFDoA	1.00	1.22	1.09	1.12	1.11	1.05	1.00	1.07	0.07	0.22

MDL

- 8 x 1 ng/L spikes
- All MDL < 1 ng/L
- MRL set at 2 ng/L

Method 533 - Results – Background



Manual LRB Levels

- N = 7 LRBs
- MRL = 2 ng/L
- < 1/3 MRL, 0.67 ng/L



Manual and SPE-03 LRB Levels

- Similar analyte traces on both
- Contamination likely outside of extraction system



Average Analyte Recovery

Manual LFB Recovery

- $N = 8 \times LFBs$ •
 - 2 low (2 ng/L)
 - 4 mid (40 ng/L) Ο
 - 2 high (160 ng/L) 0
- Method requires 70% to • 130%
- All recoveries within 90% to 120%



Manual and SPE-03 LFB Recovery

- All SPE-03 recoveries within 95% to 110%
- Well within method limits

Method 533 - Results – Reproducibility



SPE-03 %RSD of LFBs

- Across different:
 - o sample batches
 - o dates
 - o concentrations
 - o extractor positions
 - o lab personnel
- < ~15%

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- Including both **SPE-03** and **manual extraction**, and results using inline filter



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• Materials:

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- o Phenomenex Strata[®] SDBL 100 μm Styrene-divinylbenzene, 500 mg/6 mL SPE cartridge
- SCIEX 6500+ LC/MS/MS using Agilent® 1260 Infinity HPLC



Method 537.1 - SPE-03 Method



Method 537.1 - Results – MRL



SPE-03 MRL

- 7 x 2 ng/L spikes

 Span of 3 days
 3 lab personnel
- Method requires:
 50% to150% PIR
- SPE-03
 - 92% to 112% recovery
 - 70% to 128% PIR

Method 537.1 - Results – Background



SPE-03 vs Manual LRB

- N = 19 blanks
- 1/3 MRL = 0.667 ng/L
- No detected contamination

Method 537.1 - Results – 2 ppt



Manual vs SPE-03 LFB

- N = 19 at 2 ppt
- SPE-03: 88% to 105% recovery
- Manual: 90% to 108% recovery
- Similar recoveries and RSD

Method 537.1 - Results – 50 ppt



Manual vs SPE-03 LFB

- N = 19 at 50 ppt
- SPE-03: 88% to 105% recovery
- Manual: 87% to 100% recovery
- Similar recoveries and RSD

Method 537.1 – Using Inline Filter

- Using **inline filters** for **dirty samples**
- $N = 4 \times 20 \text{ ng/L spikes}$







Summary of Automation

1. Efficiency

- Fully automated
- 2 hours for 8 samples following EPA Method 533
- 75 mins for 8 samples following EPA Method 537.1
- <15 min clean cycle



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- Range of PFAS methods on single unit
- Open bench or fume hood

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3. Performance

- Excellent recoveries and consistency
- Clean background
- Ability to handle sample particulates

Acknowledgements







- ian_wan@promochrom.com
- www.promochrom.com/pfas-extractions

