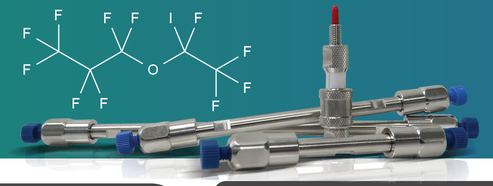
Analysis of Per-and Polyfluoroalkyl Substances (PFAS) in Aqueous Matrices Using SelectraCore® C18 HPLC Column and LC-MS/MS Analysis According to EPA Method 1633

Arielle Cocozza, Abderrahim Abdelkaoui | UCT, Inc. | 2731 Bartram Rd, Bristol, PA 19007 | (800)-385-3153 | info@unitedchem.com



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SUMMARY

The list of Per- and polyfluoroalkyl substances (PFAS) is continually expanding such that the EPA released a new draft method 1633 for monitoring these compounds in aqueous, solid, and tissue samples. These analytes are very resistant to degradation and must be identified and quantified in various media. The diversity of these analytes, which stretches from very hydrophobic long chain molecules to very short chain polar analytes, presents a challenge to calibrate in one method. This poster demonstrates a method on UCT's SelectraCore® HPLC Column with adequate sensitivity, chromatography, and linearity for the 40 compounds in the EPA 1633 Draft Method.

HPLC Conditions

HPLC system	SCIEX Exion LC
Delay column	UCT Selectra® C18, 50 × 4.6 mm, 5 µm (p/n: **SLC-1850ID46-5UM**)
HPLC column	UCT SelectraCore® C18, 50 × 2.1 mm, 2.7 µm (p/n: **SCS27-C18521**)
Guard column	UCT SelectraCore® C18, 5 × 2.1 mm, 2.7 µm (p/n: **SCS27-C18GDC21**) Guard column holder p/n: **SLGRDHLDR**
Column temp.	40°C
Flow rate	0.400 µL/min
Injection volume	2 µL

GRADIENT PROGRAM

Time (min)	Mobile Phase A (%) 20 mM Ammonium Acetate in Water	Mobile Phase B (%) Acetonitrile
0.0	98	2
7.5	0	100
8.5	0	100
8.51	98	2
11.5	98	2

MS Conditions

MS/MS system	ABSCIEX QTrap 6500+
Ionization Mode	Electrospray Ionization in negative mode (-ESI-)
Ion Spray Voltage	(IS) -4500.00
Temperature	(TEM) 300°C
Curtain Gas	(CUR) 40
Ion Source Gas 1	(GS1) 50
Ion Source Gas 2	(GS2) 50

CHROMATOGRAMS

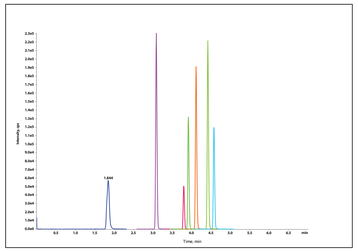

Figure 1: Non Extracted Internal Standards (in the range of 1.25 - 5.0 ng/mL)

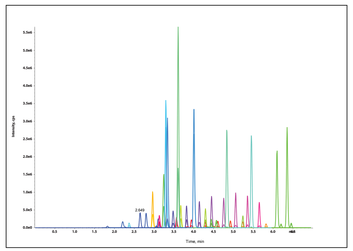

Figure 3: Target Analytes at low concentration (In the range of 0.2 - 5.0 ng/mL)

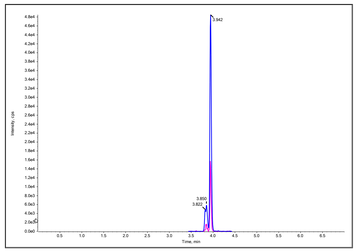

Figure 5: Isomer Separation PFHxS (at 2.5 ng/mL)

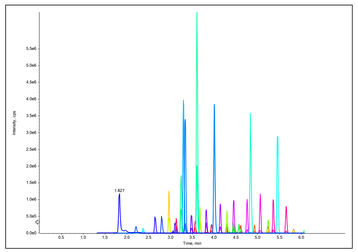

Figure 2: Extracted Internal Standards (In the range of 1.25 - 25 ng/mL)

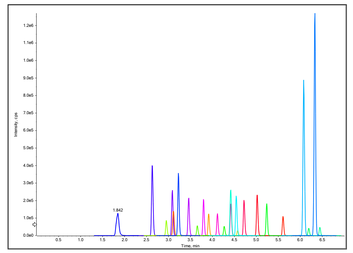

Figure 4: Target Analytes at high concentration (In the range of 12.5 - 312 ng/mL)

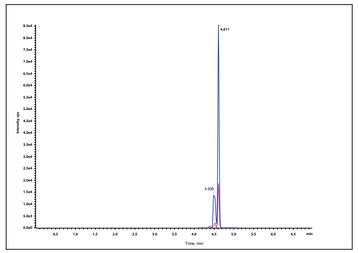

Figure 6: Isomer Separation PFOS (at 1.25 ng/mL)

CALIBRATION CURVES

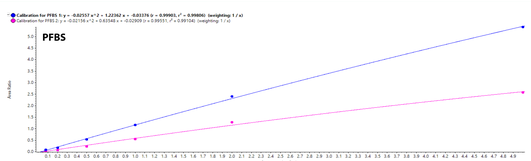

Figure 7: PFBS Calibration Plot (0.2, 0.5, 1.25, 2.5, 5.0, 12.5 ng/mL)

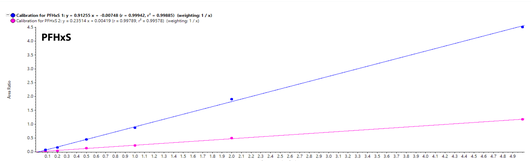

Figure 9: PFHxS Calibration Plot (0.2, 0.5, 1.25, 2.5, 5.0, 12.5 ng/mL)

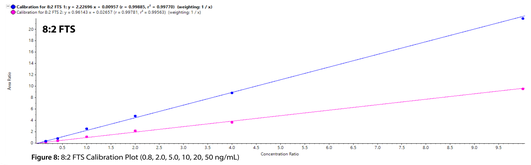

Figure 8: 8:2 FTS Calibration Plot (0.8, 2.0, 5.0, 10, 20, 50 ng/mL)

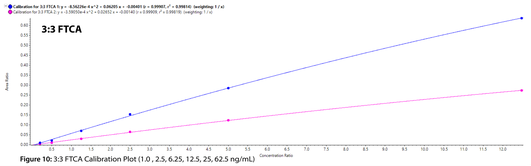

Figure 10: 3:3 FTCA Calibration Plot (1.0 , 2.5, 6.25, 12.5, 25, 62.5 ng/mL)

CALIBRATION CURVE LINEARITY TABLE

The EPA states that the correlation coefficient r and r^2 are no longer acceptable criteria for linearity in this method. Shown below are RSD (relative standard deviation) and/or RSE (relative standard error) calculations, all within 20% accuracy per method requirements.

Analyte	RSD (%)	Linear Range (ng/mL)	
PFBA	10.4	0.8	50
PFPeA 1	11.9	0.4	25
PFHxA 1	12.9	0.2	12.5
PFHpA 1	12.9	0.2	12.5
PFOA 1*	14.7	0.2	12.5
PFNA 1	10.8	0.2	12.5
PFDA 1	7.6	0.2	12.5
PFUnA 1	10.5	0.2	12.5
PFDoA 1	9.3	0.2	12.5
PFTrDA 1	10.5	0.2	12.5
PFTeDA 1	10.2	0.2	12.5
PFBS 1	10.7	0.2	12.5
PFPeS 1	9.2	0.2	12.5
PFHxS 1	5.0	0.2	12.5
PFHpS 1	5.8	0.2	12.5
PFOS 1	10.7	0.2	12.5
PFNS 1	11.0	0.2	12.5
PFDS 1	11.9	0.2	12.5
PFDoS 1	9.9	0.2	12.5
4:2 FTS 1	10.3	0.8	50
6:2 FTS 1	10.9	0.8	50
8:2 FTS 1	8.1	0.8	50
PFOSA 1	11.2	0.2	12.5
NMeFOSA 1	14.0	0.2	12.5
NEtFOSA 1	8.4	0.2	12.5
NMeFOSAA 1	11.1	0.2	12.5
NEtFOSAA 1	12.5	0.2	12.5
NMeFOSE 1	8.0	2	125
NEtFOSE 1	9.3	2	125
HFPO-DA 1	9.3	0.8	50
ADONA 1	12.2	0.8	50
9Cl-PF3ONS 1	10.3	0.8	50
11Cl-PF3OUdS 1	11.8	0.8	50
3:3 FTCA 1	11.6	1	62.4
5:3 FTCA 1	11.8	5	312
7:3 FTCA 1	11.7	5	312
PFEESA 1	13.3	0.4	25
PFMPA 1	13.5	0.4	25
NFDHA 1	14.4	0.4	25

* Linear regression, RSE presented in this table