

Ultra Low-Level Detection of Perfluoroalkyl Substances (PFASs) Using the PFC Analysis Kit

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GOAL

To demonstrate the use of the PFC Analysis Kit on the ACQUITY UPLC® I-Class System and the limits of detection afforded for PFASs analyzed on the Xevo® TQ-S.

BACKGROUND

Perfluoroalkyl substances (PFASs) encompass a range of fully fluorinated alkyl compounds, typically with an anionic end group. Previously described as perfluoroalkyl compounds (PFCs), they have been implemented in a range of consumer goods and industrial processes due to their hydro- and lipophobic properties. As a result of their widespread use and subsequent leaching from materials, they have been found in various environmental and biological samples. PFASs are also ubiquitous in the laboratory environment, namely in Teflon components used in analytical equipment. Waters® PFC Analysis Kit ([part no. 176001744](#)) specifically addresses this challenge. The kit is comprised of PFAS-free components to ensure a clean system, a sample preparation protocol, along with standards, columns, vials and caps.

The PFC Analysis Kit can be implemented on the ACQUITY UPLC I-Class System and Xevo TQ-S to achieve ultra-sensitive detection of routinely monitored PFASs.

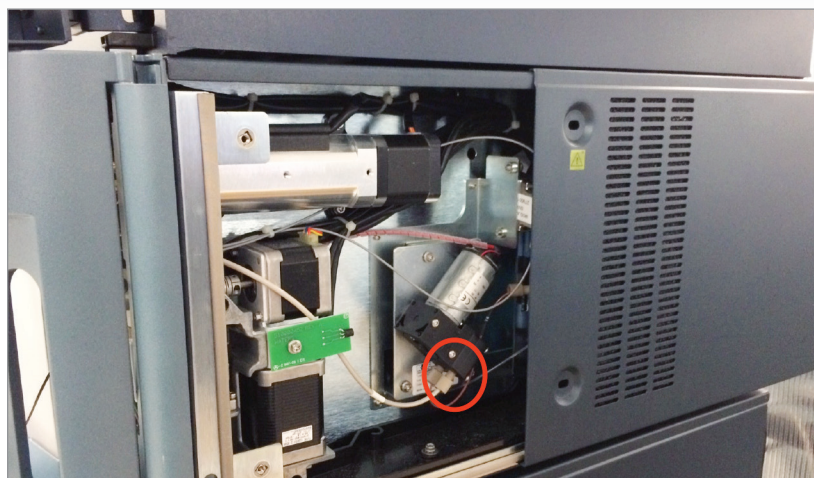


Figure 1. Side panel removal for sample manager wash tubing removal (fitting circled in red).

Alterations to existing ACQUITY UPLC systems can easily be performed. Although minor variations exist in the solvent line configurations across the ACQUITY UPLC models, the PFC Analysis Kit can be easily implemented across all models. In this technology brief, we describe the detection of various routinely monitored PFASs that was performed on the ACQUITY UPLC I-Class System and Xevo TQ-S. Special considerations for the I-Class with flow through needle (FTN) hardware are also detailed.

SOLUTION

The LC analysis was performed on an ACQUITY UPLC I-Class System. Pre-cut PEEK tubing from the PFC Analysis Kit were used to replace all of the Teflon solvent lines, per the kit instructions. The side panel of the I-Class sample manager was removed in order to replace the sample manager wash line, as shown in Figure 1; all of the other solvent lines changed are affixed in the BSM. The use of the ACQUITY® BEH C₁₈ Isolator Column and pre-cut stainless steel tubing provided in the kit further ensured that PFAS contamination was retained prior to injection of the sample. The LC method employed an ACQUITY BEH C₁₈ (50 x 2.1 mm, 1.7 μm) Column, with the gradient described in Table 1a. Standards provided in the PFC Kit were used to create the dilution series, covering a range of three orders of magnitude. The Xevo TQ-S showed exceptional sensitivity for the analytes, with their respective MRMs and optimized instrumental parameters summarized in Table 1b.

Measured limits of detection (based on peak-to-peak, signal-to-noise measurements of 1:3) for the analytes were below 0.07 ng/mL in solvent standards. In the case of the most commonly monitored PFAS analyte, perfluorooctanesulfonate (PFOS), the limit of detection was 0.0125 ng/mL (Figure 2). These low limits of detection highlight the notable sensitivity of this platform for PFASs analysis.

Min	Flow rate (mL/min.)	%A	%B
Initial	0.65	90	10
0.5	0.65	90	10
5.1	0.65	0	100
6.6	0.65	0	100
6.7	0.65	90	10
8.5	0.65	90	10

Table 1a. ACQUITY UPLC I-Class gradient utilized for the analysis. Mobile phase A was 98:2 water:methanol 2 mM ammonium acetate, and mobile phase B was methanol 2 mM ammonium acetate.

Compound	Formula	MRM*	RT (min)	CE (V)	Cone Voltage (V)
PFBuA	C ₄ HF ₇ O ₂	212.9 > 168.9	0.76	15	30
		212.9 > 212.9		2	30
PFPA	C ₅ HF ₉ O ₂	262.9 > 262.9	1.94	2	30
		262.9 > 218.9		10	30
PFBuS	C ₄ HF ₉ O ₃ S	298.9 > 79.9	2.25	35	20
		298.9 > 98.9		25	20
PFHxA	C ₆ HF ₁₁ O ₂	312.9 > 268.9	2.84	5	30
		312.9 > 118.9		20	30
PFHpA	C ₇ HF ₁₃ O ₂	362.9 > 318.9	3.42	10	30
		362.9 > 168.9		15	30
PFHxS	C ₆ HF ₁₃ O ₃ S	398.9 > 79.9	3.49	40	30
		398.9 > 98.9		40	30
		398.9 > 118.9		30	30
PFOA	C ₈ HF ₁₅ O ₂	412.9 > 368.9	3.84	10	30
		412.9 > 168.9		20	30
		412.9 > 218.9		15	30
PFNA	C ₉ HF ₁₇ O ₂	462.9 > 418.9	4.18	10	30
		462.9 > 218.9		10	30
PFOS	C ₈ HF ₁₇ O ₃ S	498.9 > 79.9	4.20	45	30
		498.9 > 98.9		45	30
		498.9 > 229.9		35	30
PFDA	C ₁₀ HF ₁₉ O ₂	512.9 > 218.9	4.46	20	30
		512.9 > 268.9		15	30
		512.9 > 468.9		10	30
PFUnDA	C ₁₁ HF ₂₁ O ₂	562.9 > 518.9	4.69	15	30
		562.9 > 268.9		20	30
		562.9 > 318.9		20	30
PFDoDA	C ₁₂ HF ₂₃ O ₂	612.9 > 568.9	4.90	15	35
		612.9 > 168.9		30	35
PFTiA	C ₁₃ HF ₂₅ O ₂	662.9 > 618.9	5.07	15	35
		662.9 > 318.9		20	35
		662.9 > 368.9		20	35
PFTeTA	C ₁₄ HF ₂₇ O ₂	712.9 > 668.9	5.22	15	35
		712.9 > 218.9		25	35
13C PFOS		502.9 > 79.9	4.20	45	30
		502.9 > 98.9		35	30
13C PFOA		416.9 > 168.9	3.84	18	30
		416.9 > 171.9		16	30
13C PFDA		514.9 > 269.9	4.46	15	30
		514.9 > 219.9		20	30

Table 1b. PFASs surveyed with optimized MRMs, RT, collision energies (CE), and cone voltages. Bold MRM transitions indicate the quantitative trace. Italicized compounds are isotopically labeled standards provided in the PFC Analysis Kit.

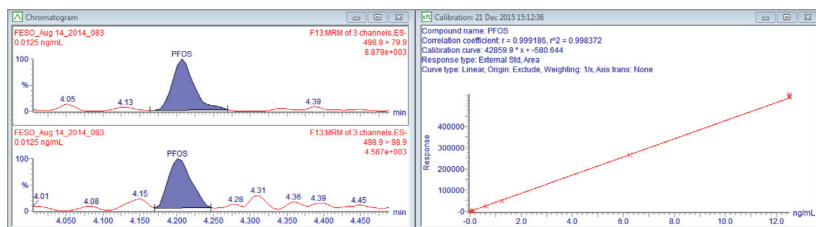


Figure 2. MRM at limit of detection for PFOS, as linearity shown for calibration curve over three orders of magnitude.

SUMMARY

Accurate and highly sensitive PFASs analysis can be achieved using the PFC Analysis Kit, which is adaptable to any ACQUITY UPLC system. A complete analytical solution for the PFASs is presented, particularly when used in combination with the Xevo TQ-S for ultra low-level detection.

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