응용 자료

Enhanced Reliability for Long-Term PFAS Analysis with the Xevo TQ Absolute XR Mass Spectrometer

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Abstract

Accurate and reliable LC-MS/MS measurement is critical for monitoring per- and polyfluoroalkyl substances (PFAS) across environmental matrices using the United States Environmental Protection Agency (EPA) Method 1633. Despite SPE clean-up, co-extractive interferents from complex matrices like river water, biosolid, and landfill leachate can degrade system performance over time. In this study, the novel StepWave™ XR Ion Guide within the Xevo™ TQ Absolute XR Mass Spectrometer was evaluated for long-term reliability across 10 weeks and more than 7000 injections and with no unscheduled downtime. PFAS concentrations were consistently quantified in matrix with trueness in the range 70–130% and %RSDs <8.4%, demonstrating excellent system uptime and measurement robustness.

Benefits

- Xevo TQ Absolute XR provided maximal system uptime for PFAS analysis across 10 weeks and more than 7000 injections with no unscheduled downtime.
- Reliable calculated concentrations were maintained for PFAS at relevant levels in extracts of river water, biosolid, and landfill leachate with %RSDs of <8.4%.
- Seamless batch review and long-term calibration management using waters_connect™ for Quantitation Software accelerated data validation workflows.

Introduction

As awareness grows around the environmental and health impacts of PFAS, regulatory pressure continues to intensify globally. The EPA Method 1633A[1] has become a critical protocol for the quantitation of 40 PFAS compounds in diverse environmental matrices, including non-potable water, soils, biosolids, and tissues. Although the method describes a solid phase extraction (SPE) clean-up step for all sample types, these matrices still contain co-extractive interferents when introduced into an LC-MS/MS system, which can contaminate the system and degrade method performance over time. This application note describes how the StepWave XR Ion Guide, a novel slotted bandpass ion guide within the Xevo TQ Absolute XR Mass Spectrometer, can be integrated into a typical PFAS workflow to enhance signal robustness while preserving sensitivity and speed, thereby boosting method reliability.

Experimental

Sample Preparation

Samples included river water, biosolid, and landfill leachate. River water was collected in Scotland, UK from the River Tay. Biosolid (domestic sludge National Institute of Standards and Technology (NIST) 2781) and landfill leachate (LGC 6177) were both purchased as reference materials (landfill leachate contained no certified PFAS values).

Extraction and SPE clean-up were carried out as outlined in previously published work[2,3] with 250 mL river water, 0.5 g biosolid or 25 mL landfill leachate taken as the relevant sample sizes. The river water was spiked with native and internal standards after a screening analysis identified that the 40 PFAS in EPA Method 1633 were not detected. The biosolid and landfill leachate both had PFAS present and were only spiked with internal standards. For the biosolid, 2 replicates were extracted, combined and then diluted 1 in 10 with methanol before analysis because the certified values were greater than the calibrated range. For both the landfill leachate and spiked river water samples, 5 replicates of each were extracted and combined before analysis.

Analysis Sequence

The analytical sequence of a single batch is shown in Figure 1 and shows the order and number of injections over a 25-hour period. Linear, 1/x weighted calibration curves were plotted weekly with all data from the following 7 days measured against these plots. Multiple reaction monitoring (MRM) dwell times were calculated automatically by waters_connect for Quantitation ensuring sufficient data points (at least 12) were acquired per peak. Data acquisition, processing and review were performed using waters_connect for Quantitation. The data quality guidelines outlined in EPA 1633A were used to demonstrate capability. Pre-emptive routine maintenance was performed with the cone assembly cleaned and isolator and analytical columns changed monthly (approx. every 3,000 injections). Other maintenance on the Xevo TQ Absolute XR was not performed, and the system was not vented for the entirety of the study apart from an unscheduled electrical power cut after 6,278 injections.

1 x	**8 x Solvent calibration standards**
3 x	Methanol solvent
	5 x Biosolid sample extract, 1/10 dilution
	Methanol solvent
	5 x Leachate sample extract
	Methanol solvent
	Solvent calibration QC
	5 x River water spiked sample extract
	Methanol solvent
	5 x Biosolid sample extract, 1/10 dilution
	Methanol solvent
	Solvent calibration QC
	5 x Leachate sample extract
	Methanol solvent
	5 x River water spiked sample extract
	Methanol solvent
	Solvent calibration QC

Figure 1. Analytical sequence of a single batch (lasting 128 injections and 25 hours) used during the robustness study.

LC Conditions

UPLC system:	ACQUITY™ Premier System with Binary Solvent Manager and Flow-Through Needle
Vials:	700 µL polypropylene screw cap vials, p/n: 186005219
Analytical column:	ACQUITY Premier BEH™ C_{18} Column 2.1 x 50 mm,

	1.7 µm. p/n: 186009452
Isolator column:	Atlantis™ Premier BEH C_{18} AX Column 2.1 x 50 mm, 5 µm. p/n: 186009407
Column temperature:	35 °C
Sample temperature:	10 °C
Injection volume:	2 µL
Flow rate:	0.3 mL/min
Mobile phase A:	2 mM ammonium acetate in water
Mobile phase B:	2 mM ammonium acetate in acetonitrile

Gradient Table

Time (min)	%A	%B	Curve
0	95	5	Initial
0.5	75	25	6
3	50	50	6
6.5	15	85	6
7	5	95	6
8.5	5	95	6
9	95	5	6
11	95	5	6

MS Conditions

MS system:	Xevo TQ Absolute XR Mass Spectrometer
Ionization mode:	ESI-
Capillary voltage:	0.5 kV
Source temperature:	100 °C
Desolvation temperature:	350 °C
Desolvation gas flow:	900 L/hr
Cone gas flow:	150 L/hr
MRM method:	See Waters Application Note 720008143

Data Management

Software:	waters_connect for Quantitation

Results and Discussion

QC Performance

Method performance was evaluated every 10 sample extract injections to ensure that the weekly calibration of the method was stable. Figure 2 shows the QC plots for GenX, L-PFHxS, L-PFOS, PFBS, PFNA, and PFOA with a tolerance of ±30% set from the assigned concentration level (0.1–0.2 ng/mL) over the course of the study. Table 1 lists the %RSDs for the ion ratios for the same compounds. QC measured concentrations and ion ratios were within the set tolerances across 10 weeks and more than 7,000 injections with no unscheduled downtime. This illustrates the reliability of the Xevo TQ Absolute XR for PFAS analysis using a weekly calibration routine.

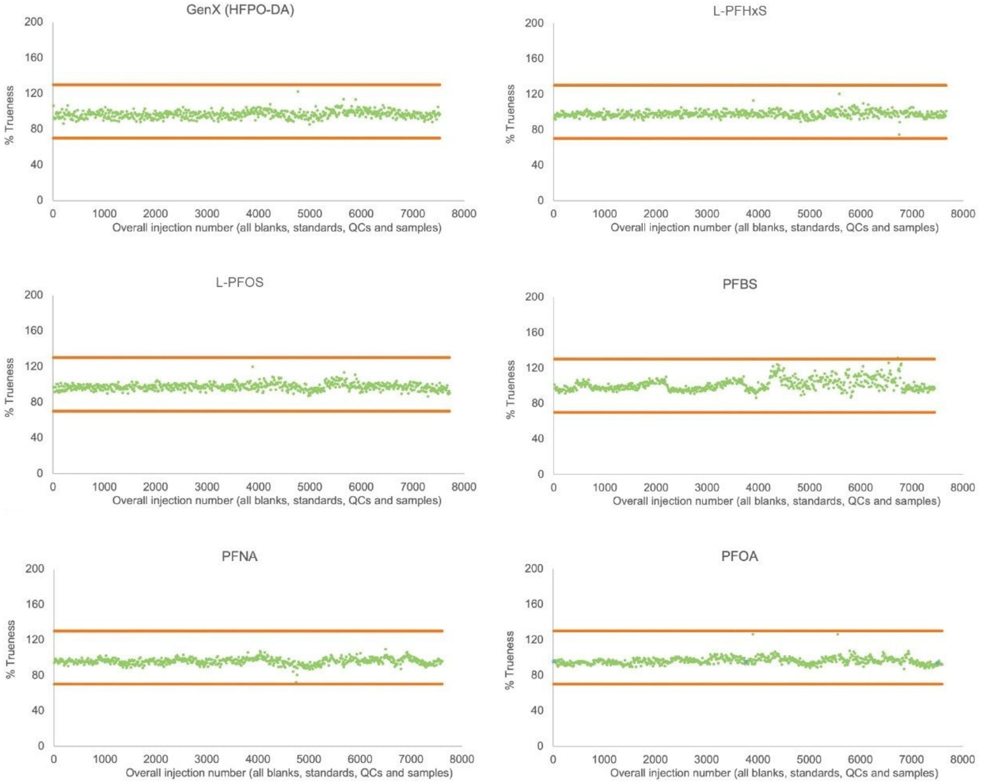

Figure 2. QC Plots from weekly calibrations for PFAS chemicals GenX, L-PFHxS, L-PFOS, PFBS, PFNA, and PFOA over the robustness study (Orange = ±30% from assigned concentration level).

PFAS	QC concentration (ng/mL)	Mean ion ratio	%RSD
GenX	0.2	0.31	7.1
L-PFHxS	0.1	0.56	4.4
L-PFOS	0.1	0.60	5.0
PFBS	0.1	0.38	4.0
PFNA	0.1	0.36	6.4
PFOA	0.1	0.60	5.4

Table 1. %RSDs of ion ratios for GenX, L-PFHxS, L-PFOS, PFBS, PFNA, and PFOA over the robustness study.

Spiked River Water Results

River water spiked samples were repeatedly measured across the robustness study. Figure 3 shows the trueness plots for PFAS chemicals GenX, L-PFHxS, L-PFOS, PFDoDS, NFDHA, and PFNA with a tolerance of ±30% set from the spiked concentration level over the course of the study. Trueness was within the set tolerance across 10 weeks and more than 7000 injections with no unscheduled downtime. This demonstrates the overall measurement reliability of the Xevo TQ Absolute XR for PFAS analysis in aqueous matrices.

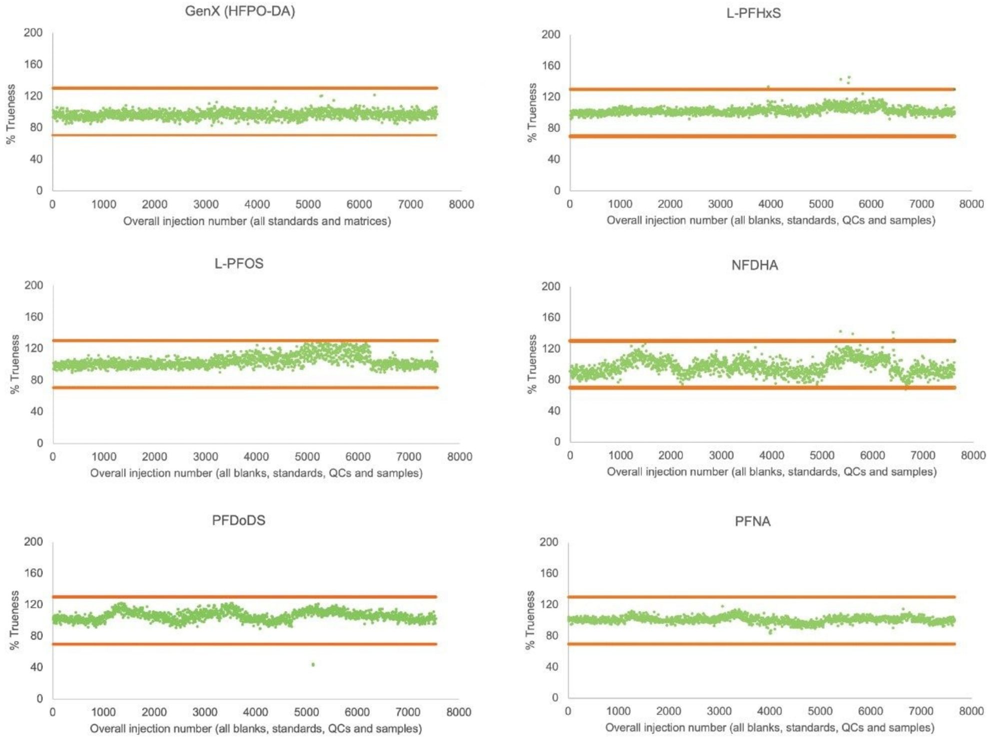

Figure 3. Trueness plots for GenX, L-PFHxS, L-PFOS, NFDHA, PFDoDS, and PFNA in spiked river water over the robustness study (Orange = ±30% from spiked concentration level).

Biosolid Results

The certificate of analysis for the biosolid sample stated reference values for different PFAS. One example was for L-PFHxS, where the measured values in the biosolid extracts over the course of the study are plotted in Figure 4. The mean measured value was 8.82 µg/kg with a %RSD of 7.3% across 10 weeks and more than 7,000 injections with no unscheduled downtime. This compared very favorably (-6.1% error) with the certified value of 9.39 µg/kg. Table 2 lists the mean measured values and the %RSDs for all the PFAS compared to the certified value and range. This demonstrates the overall measurement reliability of the Xevo TQ Absolute XR for PFAS analysis in biosolids.

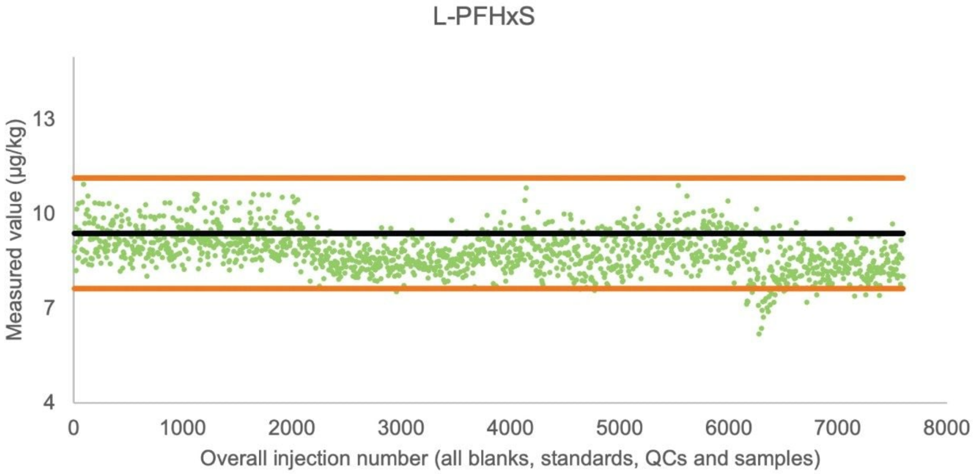

Figure 4. Measured value of L-PFHxS in the biosolid extracts over the robustness study (Black = assigned value, Orange = upper, and lower uncertainty from the certificate of analysis).

PFAS	Mean measured value (µg/kg)	%RSD	Certified value (µg/kg)	Certified value range (µg/kg)
FOSA	5.61	3.5	6.31	5.34–7.28
L-PFHxS	8.82	7.3	9.39	7.63–11.15
L-PFOS	232	8.0	225	184–266
PFHpA	8.67	7.2	7.96	6.46–9.46
PFOA	26.6	3.6	28.5	25.2–31.8

Table 2. Mean measured values and %RSDs of PFAS in the biosolid extracts over the robustness study and how they compare to the certified values.

Landfill Leachate Results

Several incurred residues were detected in the landfill leachate sample. Figure 5 shows one example, PFOA, where the mean calculated concentration was 2.52 ng/mL with a %RSD of 2.7% across 10 weeks and more than 7,000 injections with no unscheduled downtime. Table 3 lists the mean calculated concentrations and the

%RSDs for the PFAS in the landfill leachate sample. This demonstrates the overall measurement reliability of the Xevo TQ Absolute XR for PFAS analysis in other complex environmental matrices.

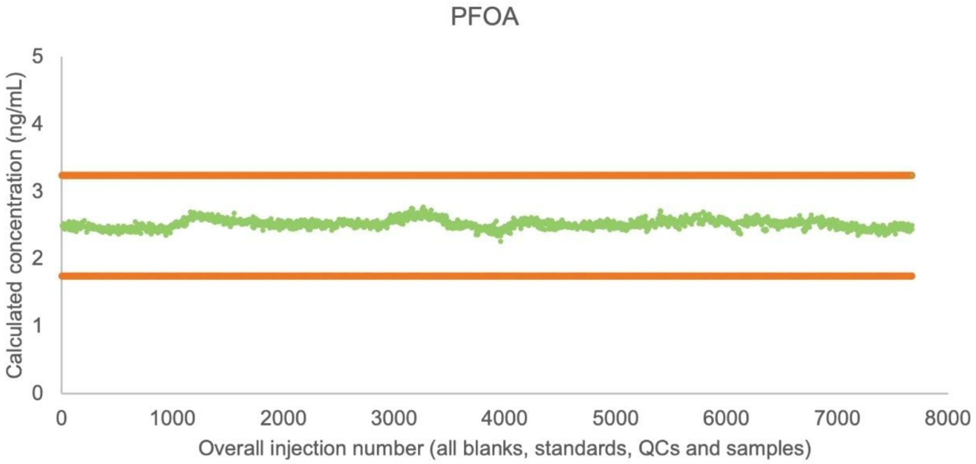

Figure 5. Calculated concentration of PFOA in landfill leachate extracts over the robustness study.

PFAS	Mean calculated concentration (ng/mL)	Calculated concentration %RSD	Mean ion ratio	Ion ratio %RSD
L-PFHxS	0.76	2.3	0.59	3.4
L-PFOS	0.29	3.6	0.63	7.2
PFBS	7.39	3.8	0.26	4.9
PFHpA	1.37	3.3	0.49	3.6
PFHxA	5.05	5.2	0.11	7.4
PFOA	2.52	2.7	0.62	3.0
PFPeA	1.53	3.7	0.03	8.4

Table 3. Mean calculated concentrations, mean ion ratios and %RSDs for the PFAS reported in the landfill leachates extract.

Conclusion

The StepWave XR Ion Guide within the Xevo TQ Absolute XR Mass Spectrometer was introduced into a typical PFAS workflow and shown to maintain method reliability for extended periods of time. The Xevo TQ Absolute XR provided maximal system uptime for PFAS analysis across 10 weeks and more than 7,000 injections with no unscheduled downtime. Concentrations for PFAS at relevant levels were reliably calculated in extracts of river water, biosolids, and landfill leachate with trueness in the range 70–130% and %RSDs of <8.4%. These findings support the Xevo TQ Absolute XR as a powerful platform for laboratories seeking to meet regulatory requirements for PFAS monitoring while minimizing system downtime and maintenance.

References

1. US Environmental Protection Agency. EPA Method 1633A, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. Dec 2024.
2. Organtini KL, Rosnack KJ, Plummer C, Hancock PM, Burt O. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Accordance with EPA 1633 Part 2: Analysis of Aqueous Matrices. Waters Application Note 720008143. Dec 2023.
3. Organtini KL, Rosnack KJ, Plummer C, Hancock PM, Burt O. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Accordance with EPA 1633 Part 3: Analysis of Soil and Tissue. Waters Application Note 720008230. Feb 2024.

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Xevo TQ Absolute Triple Quadrupole Mass Spectrometer < https://www.waters.com/nextgen/global/products/mass-spectrometry-systems/xevo-tq-absolute.html>

Oasis WAX for PFAS Analysis <https://www.waters.com/nextgen/global/products/sample-preparation/oasis-wax-for-pfas-analysis.html>

Atlantis Premier Columns <https://www.waters.com/nextgen/global/products/columns/atlantis-premier-columns.html>

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