SHIMADZU
Excellence in Science

High Performance Liquid Chromatograph Mass Spectrometer LCMS™-8060RX

Application News

Robustness Evaluation of PFAS Analysis in Soil Using LCMS™-8060RX

Yui Higashi and Nami Iwasa

User Benefits

- The LCMS RX series, which improved stability and robustness with CoreSpray technology, showed excellent peak area repeatability when measuring PFAS at low concentrations 500 consecutive times in a soil matrix.
- Quality control samples measured for every 20 soil sample also showed good recovery rates of all PFAS remaining between 80 and 120 %.

■ Introduction

Perfluoroalkyl and polyfluoroalkyl substances (PFAS) are widely used in various field and indistries. But their tendency to accumulate in the environment due to their highly stable structures, which resist degradation, raises concerns about their effects on humans. Therefore, the Environment Protection Agency (EPA) in the USA and the European Chemicals Agency (ECHA) have recently strengthened regulations on PFAS. Research has shown they accumulate in the bodies of humans from drinking water and in livestock, agricultural, and marine products from water and soil and that they may have a potential adverse effect on health.[1)] This has created a need for highly accurate and reliable methods of measuring PFAS in not only water but also in soil and other samples with relatively complex matrixes.

This article presents a robustness study that added 30 PFAS (Table 1) to a soil matrix and used the LCMS-8060RX to measure these PFAS in the soil matrix 500 consecutive times. The LCMS-8060RX produced good results and provided a robust analytical platform for the measurement of low concentrations of PFAS in a soil matrix sample.

Table 1 List of Target Compounds

Compound	Type	Compound	Type
PFBA	Target	13C4-PFBA	ISTD
PFPeA	Target	13C5-PFPeA	ISTD
PFBS	Target	13C3-PFBS	ISTD
PFHxA	Target	13C5-PFHxA	ISTD
HFPO-DA	Target	13C3-HFPO-DA	ISTD
PFHpA	Target	13C4-PFHpA	ISTD
DONA	Target	13C2-6:2FTSA	ISTD
6:2FTSA	Target	13C8-PFOA	ISTD
PFOA	Target	13C3-PFHxS	ISTD
PFHxS	Target	13C2-8:2 FTUCA	ISTD
8:2 FTUCA	Target	13C9-PFNA	ISTD
PFNA	Target	13C2-8:2FTSA	ISTD
PFHpS	Target	D3-NMeFOSAA	ISTD
8:2FTSA	Target	13C6-PFDA	ISTD
NMeFOSAA	Target	D5-NEtFOSAA	ISTD
PFDA	Target	13C8-PFOS	ISTD
NEtFOSAA	Target	13C7-PFUnA	ISTD
PFOS	Target	13C2-PFDoA	ISTD
PFUnA	Target	13C8-FOSA	ISTD
9Cl-PF3ONS	Target	13C2-PFTeDA	ISTD
PFDoA	Target	D3-NMeFOSA	ISTD
FOSA	Target	13C4-8:2 diPAP	ISTD
PFDS	Target	13C2-PFHxDA	ISTD
PFTrDA	Target	D5-NEtFOSA	ISTD
PFTeDA	Target		
NMeFOSA	Target		
8:2 diPAP	Target		
PFHxDA	Target		
NEtFOSA	Target		
PFOcDA	Target		

■ LCMS-8060RX

The instrument used in this robustness study was the LCMS-8060RX triple-quadrupole mass spectrometer (Fig. 1). The LCMS-TQ RX series inherits the UF technology of previous models, preserving their high sensitivity and high speed, and is now equipped with CoreSpray technology, which improves the homogeneity of nebulizer flows and measurement stability.

Fig. 1 LCMS™- 8060RX

■ Analytical Conditions

The HPLC and MS conditions used are shown in Table 2. Caution is required when measuring PFAS as they can leach from the analytical system and the mobile phase. PFAS contamination from the system was minimized by attaching a delay column between the mixer and autosampler. Reagents intended for use in PFOS and PFOA analysis were used for the mobile phase.

Table 2 Analytical Conditions

UHPLC (Nexera™-X3 System)	
Analytical Column:	Shim-pack Scepter™ C18-120 (100 mm × 2.1 mm I.D., 1.9 µm, P/N: 227-31012-05)
Solvent Delay Column:	Delay column for PFAS (GL Science, P/N 5020-90005)
Mobile Phase A:	2 mM Ammonium Acetate in reagent water
Mobile Phase B:	Methanol
Gradient Program:	B 1 % – 50 % (2.0 min) – 100 % (11.0 – 15.0 min) – 1 % (15.1-20.0 min)
Flowrate:	0.3 mL/min
Column Temp.:	40 °C
Injection Volume:	5 µL
Run Time:	20 min

MS (LCMS-8060RX)	
Ionization:	ESI (Negative mode)
Mode:	MRM
Nebulizing Gas:	3 L/min
Drying Gas Flow:	5 L/min
Heating Gas Flow:	15 L/min
DL Temp.:	200 °C
Block Heater Temp.:	300 °C
Interface Temp.:	250 °C
Probe Position:	+3 mm
MRM Transition:	See Table 3

■ Analysis of Standards

Standards for calibration curves were prepared at concentrations of 0.01 to 10 µg/L in solution. Each concentration was measured three times. Fig. 2 shows calibration curves for PFOA and PFOS, and Fig. 3 shows MRM chromatograms for the target compounds at 0.05 µg/L. All compounds were separated at around 12 minutes, and the peak shapes were good. The quantitative ion, calibration curve range, and coefficient of correlation (R) used to measure each target compound are shown in Table 3. The coefficient of correlation (R) was above 0.996 for almost all the compounds in the correlation curve range of 0.01 to 10 µg/L, and the accuracy ranged from 70 to 130 %. Repeatability was good, with %RSD below 20 at all concentrations.

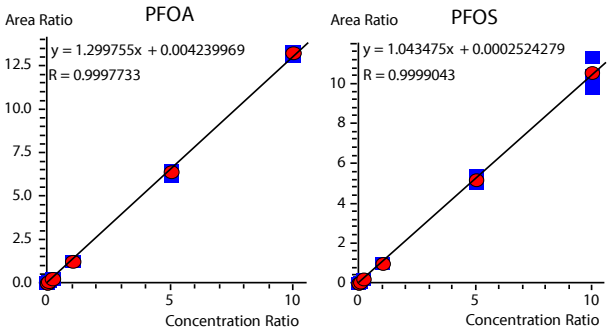

Fig. 2 Calibration Curves for PFOA and PFOS

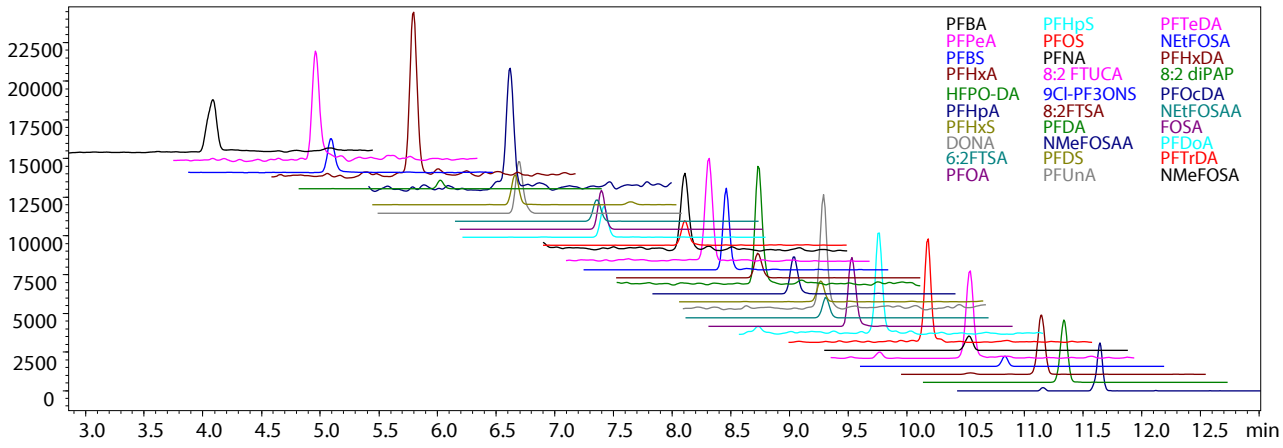

Fig. 3 MRM Chromatograms for 0.05 µg/L

Table 3 Quantitative Ion, Calibration Curve Range, and Coefficient of Correlation (R) for Each Target Compound

#	Compound	*m/z*	Calibration Curve Range (µg/L)	Coefficient of Correlation R
1	PFBA	213.00 > 169.00	0.01–10	0.99942
2	PFPeA	263.00 > 219.00	0.01–10	0.99947
3	PFBS	298.95 > 79.95	0.01–10	0.99951
4	PFHxA	312.95 > 269.00	0.01–10	0.99972
5	HFPO-DA	285.00 > 169.00	0.01–10	0.99997
6	PFHpA	362.95 > 319.00	0.01–10	0.99963
7	DONA	376.95 > 251.00	0.01–10	0.99961
8	6:2FTSA	426.95 > 406.95	0.05–5	0.99663
9	PFOA	412.95 > 369.00	0.01–10	0.99978
10	PFHxS	398.95 > 79.95	0.01–10	0.99949
11	8:2 FTUCA	456.95 > 393.00	0.01–10	0.99922
12	PFNA	462.95 > 418.95	0.01–10	0.99922
13	PFHpS	448.95 > 79.95	0.01–10	0.99950
14	8:2FTSA	526.95 > 506.95	0.01–5	0.99815
15	NMeFOSAA	569.95 > 418.95	0.01–10	0.99992
16	PFDA	512.95 > 468.95	0.01–10	0.99914
17	NEtFOSAA	584.00 > 418.95	0.01–10	0.99908
18	PFOS	498.95 > 79.95	0.01–10	0.99991
19	PFUnA	562.95 > 518.95	0.01–10	0.99923
20	9Cl-PF3ONS	530.90 > 350.95	0.01–10	0.99938
21	PFDoA	612.95 > 568.95	0.01–10	0.99993
22	FOSA	497.95 > 77.95	0.01–10	0.99951
23	PFDS	598.90 > 79.95	0.01–10	0.99984
24	PFTrDA	662.95 > 618.95	0.01–10	0.99982
25	PFTeDA	712.95 > 668.95	0.01–10	0.99973
26	NMeFOSA	511.95 > 169.00	0.05–10	0.99879
27	8:2 diPAP	989.00 > 97.00	0.01–10	0.99967
28	PFHxDA	813.00 > 768.80	0.01–10	0.99966
29	NEtFOSA	526.00 > 169.00	0.01–10	0.99960
30	PFOcDA	913.00 > 868.65	0.01–10	0.99956

■ Robustness Evaluation with a Soil Matrix

A soil sample was prepared using soil preparation procedures published by the National Agricultural and Food Research Organization.[2] Once the soil matrix was ready, the standard was added to create a soil sample with a target compound concentration of 0.1 µg/L in solution. The soil matrix content of the resulting soil sample was at least 90 %. This soil sample was then analyzed 500 times in succession. Fig. 4 shows the normalized peak areas of five major compounds (HFPO-DA, PFOA, PFHxS, PFNA, and PFOS) in the spiked soil sample. MRM chromatograms from the first and 500th analysis for these five compounds are shown in Fig. 5.

Good peak shapes were obtained at the start and end of the 500 analyses and peak area repeatability was good for PFAS in the soil matrix sample. Table 4 shows the %RSD and the detection limit in the soil matrix sample (based on the 500 consecutive analyses) for all 30 target PFAS. Peak area repeatability was good with %RSD below 8.5 for all target compounds. Quality control samples were also analyzed (n = 3) every 20 analyses. Recovery from the quality control samples was within 80 to 120 % for all target compounds for the duration of the 500 consecutive analyses (Fig. 6).

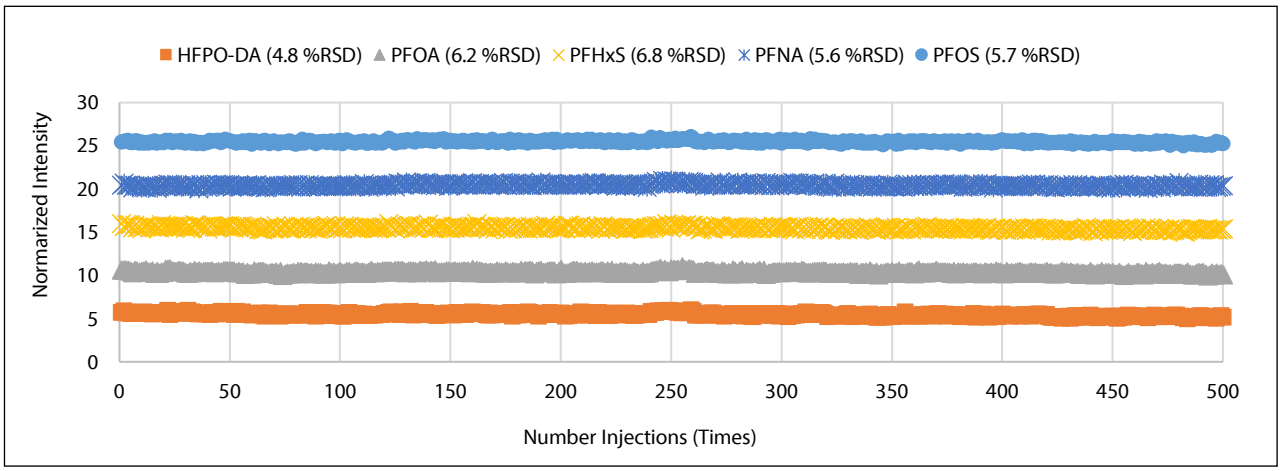

Fig. 4 Peak Area Repeatability (n = 500) for the Soil Sample Spiked to 0.1 µg/L (Concentration in Solution)

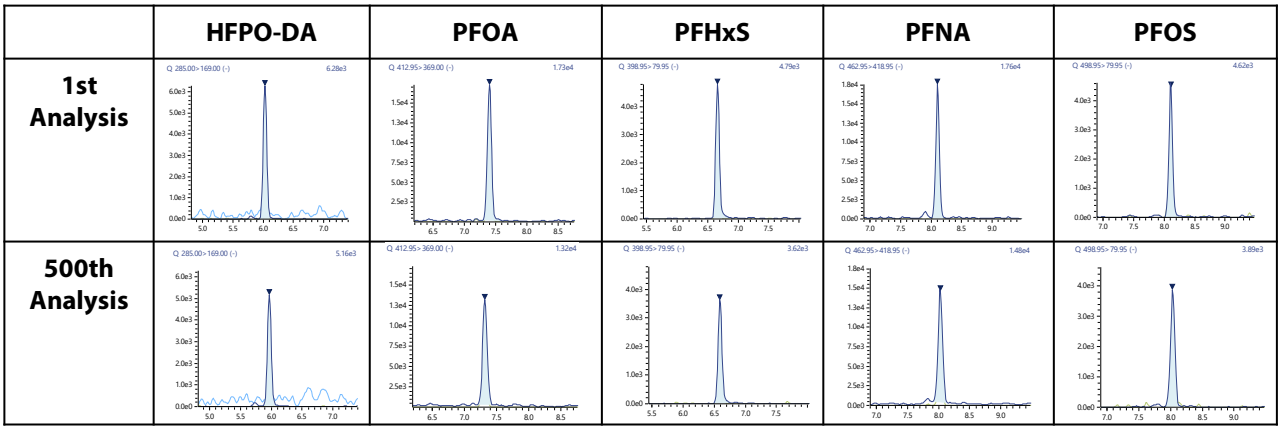

Fig. 5 MRM Chromatograms of the First and 500th Analysis of the Soil Sample Spiked to 0.1 µg/L (Concentration in Solution)

Table 4 Peak Area %RSD, Detection Limit , and Mean Recovery for the Soil Sample Spiked to 0.1 µg/L (Concentration in Solution)

#	Compound	Peak Area %RSD (n = 500)	Detection Limit in the Soil Matrix Sample (µg/L) (n = 500)	#	Compound	Peak Area %RSD (n = 500)	Detection Limit in the Soil Matrix Sample (µg/L) (n = 500)
1	PFBA	5.4 %	0.013	16	PFDA	7.0 %	0.016
2	PFPeA	5.2 %	0.012	17	NEtFOSAA	7.0 %	0.016
3	PFBS	5.0 %	0.012	18	PFOS	5.7 %	0.013
4	PFHxA	5.9 %	0.014	19	PFUnA	7.7 %	0.018
5	HFPO-DA	4.8 %	0.011	20	9Cl-PF3ONS	7.0 %	0.016
6	PFHpA	5.0 %	0.012	21	PFDoA	6.4 %	0.015
7	DONA	4.9 %	0.011	22	FOSA	6.9 %	0.016
8	6:2FTSA	6.8 %	0.016	23	PFDS	6.9 %	0.016
9	PFOA	6.2 %	0.014	24	PFTrDA	5.9 %	0.014
10	PFHxS	6.8 %	0.016	25	PFTeDA	5.9 %	0.014
11	8:2 FTUCA	6.0 %	0.014	26	NMeFOSA	6.7 %	0.016
12	PFNA	5.6 %	0.013	27	8:2 diPAP	7.3 %	0.017
13	PFHpS	7.7 %	0.018	28	PFHxDA	5.5 %	0.013
14	8:2FTSA	8.5 %	0.020	29	NEtFOSA	6.3 %	0.015
15	NMeFOSAA	5.7 %	0.013	30	PFOcDA	8.3 %	0.019

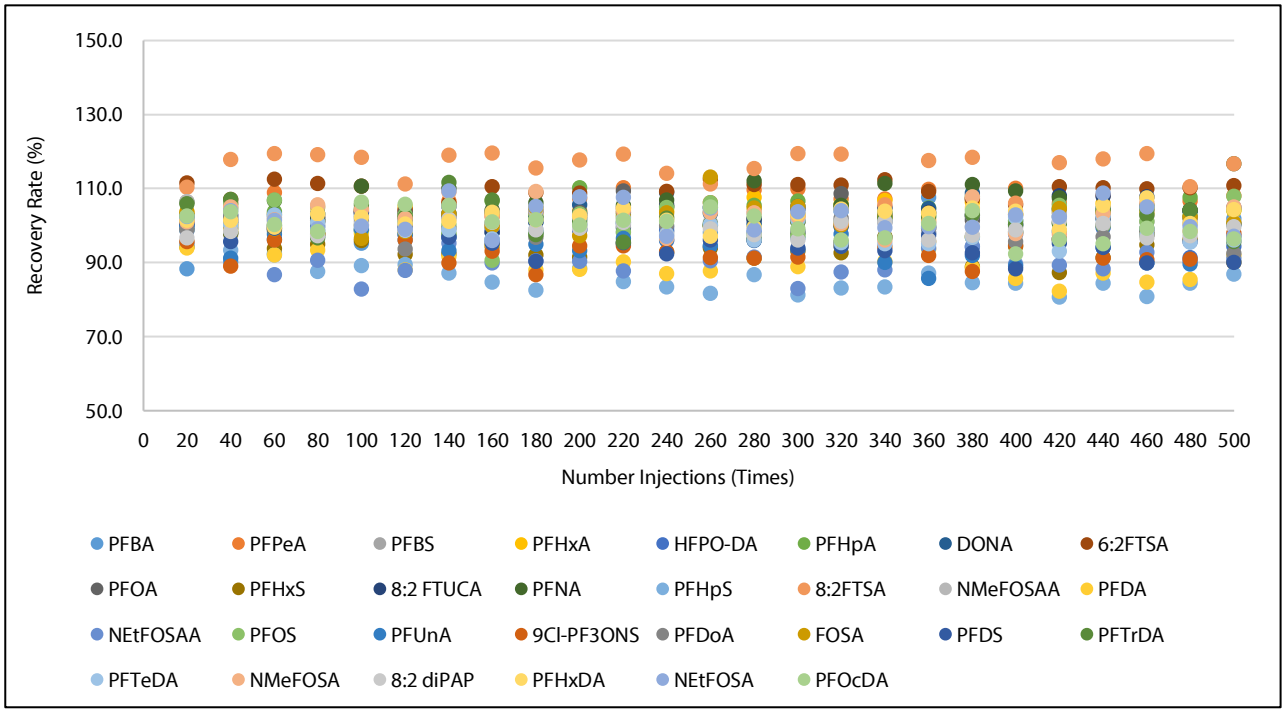

Fig. 6 Mean Recovery from QC Samples at 0.1 µg/L (Concentration in Solution) (n = 3)

■ Conclusion

The LCMS-8060RX performed 500 consecutive measurements of 30 PFAS added to a soil matrix sample. The results showed good peak area repeatability, good peak shapes, and good recovery. The LCMS-8060RX, which is now equipped with CoreSpray technology for improved stability and robustness, provides reliable analysis even with samples containing numerous impurities, such as soil matrix samples.

<References>

1) Basic research on the movement of PFOA, PFOS, and other PFAS from agricultural environments (water, soil, etc.) into agricultural products
https://www.maff.go.jp/j/syouan/seisaku/regulatory_science/shuryo_chem.html#pfas

1) DRAFT METHOD 202201 Determination of perfluoroalkyl and polyfluoroalkyl substances (PFAS) in soil
Determination of perfluoroalkyl and polyfluoroalkyl substances (PFAS) in soil | 農研機構

01-00851-EN First Edition: Apr. 2025

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/

› Please fill out the survey

Related Products Some products may be updated to newer models.

› LCMS-TQ RX Series Triple Quadrupole LC-MS/MS

Triple Quadrupole LC-MS/MS

Related Solutions

› Environment

› PFAS

› Soil

› Price Inquiry

› Product Inquiry

› Technical Service / Support Inquiry

› Other Inquiry