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Application News

LCMS-8060NX Liquid Chromatograph Mass Spectrometer

Quantitation of Extractable Per- and Polyfluorinated Alkyl Substances (PFAS) in Consumer Products on the Shimadzu Triple Quadrupole Mass Spectrometer LCMS-8060NX

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User Benefits

- A newly developed and cost-effective full workflow for the analysis of extractable PFAS in consumer products without the need of labor-intensive solid phase extraction.
- Meet or exceed the proposed reporting limits of the new ASTM method for 46 PFAS and 25 surrogates using the highly sensitive Shimadzu LCMS-8060NX.
- Reduce pressure failures with the new Shimadzu Nexcol PFAS Delay column (backpressure tolerance of 15,000 psi) while minimizing PFAS background levels.

■ Introduction

Per-and Polyfluorinated alkyl substances (PFAS) are a broad class of thousands of chemicals with a varied global definition that include carbon-fluorine bonds. Since the carbon-fluorine bond is the strongest in organic chemistry, PFAS was manufactured for desirable water-resistant, oil-resistant, and heat-resistant properties. Since the 1940's, industries have integrated PFAS in products such as food packaging, textiles, and household products due to their unique properties. However, a characteristic of PFAS of concern is their slow breakdown rate, leading to potential accumulation in people, animals, and the environment. Studies have shown widespread exposure to PFAS among the United States population.[1] In response to growing concerns regarding consumer exposure to PFAS, many states have initiated bans of PFAS uses in various consumer products and food packaging materials.[2] These bans have led to a need for analytical testing to determine the amount of PFAS in consumer products, however, the lack of standardized methods presents challenges in ensuring reliability and reproducibility between labs.

RJ Lee Group

This application news provides a foundation for the collaborative efforts between Shimadzu Scientific Instruments, Inc. and RJ Lee Group Inc., along with ASTM, towards establishing the first standardized method for the quantitation of extractable PFAS in various consumer product categories. The tested products in this method include sand, non-stick foil, and HDPE plastic with the potential applicability to other sample types such as other polymers, paper, fabric, and coatings.

Using a Shimadzu LCMS-8060NX liquid chromatography triple quadrupole mass spectrometer (**Fig. 1**), the minimum reporting limits on products are 100 ng/kg with an analytical range of 100-4000 ng/kg for most analytes. The reporting limit for the test method is defined as an integer value that is equal to the concentration of the lowest calibration standard. The target analytes and reporting ranges are summarized in **Table 1**. This new method utilizes a simple co-solvation of the solid matrices to extract the PFAS and eliminates the tedious step of solid phase extraction (SPE). The co-solvation along with a larger volume injection provides a robust and streamlined approach to achieve the low 100 ng/kg reporting limit. The use of the co-injection technique further aids the typical drawback of early eluter peak distortion when using larger injection volumes with high organic composition. Additionally, the system demonstrates exceptional robustness with the incorporation of Shimadzu's new Nexcol PFAS delay column which affords high pressure tolerances to elongate delay column longevity.

Fig. 1: Shimadzu LCMS-8060NX

Table 1: Analyte List with Reporting Range

Analyte Name	Acronym	CAS Number	Range (ng/kg)
Perfluorotetradecanoic acid	PFTreA	376-06-7	100-4000
Perfluorotridecanoic acid	PFTriA	72629-94-8	100-4000
Perfluorododecanoic acid	PFDoA	307-55-1	100-4000
Perfluoroundecanoic acid	PFUnA	2058-94-8	100-4000
Perfluorodecanoic acid	PFDA	335-76-2	100-4000
Perfluorononanoic acid	PFNA	375-95-1	100-4000
Perfluorooctanoic acid	PFOA	335-67-1	100-4000
Perfluoroheptanoic acid	PFHpA	375-85-9	100-4000
Perfluorohexanoic acid	PFHxA	307-24-4	100-4000
Perfluoropentanoic acid	PFPeA	2706-90-3	500-20000
Perfluorobutanoic acid	PFBA	375-22-4	1000-20000
Perfluorodecanesulfonic acid	PFDS	335-77-3	100-4000
Perfluorononanesulfonic acid	PFNS	68259-12-1	100-4000
Perfluorooctanesulfonic acid	PFOS	1763-23-1	100-4000
Perfluoroheptanesulfonic acid	PFHpS	375-92-8	100-4000
Perfluorohexanesulfonic acid	PFHxS	355-46-4	100-4000
Perfluoropentanesulfonic acid	PFPeS	2706-91-4	100-4000
Perfluorobutanesulfonic acid	PFBS	375-73-5	100-4000
Perfluorooctanesulfonamide	PFOSA	754-91-6	100-4000
8:2 Fluorotelomer sulfonic acid	8:2 FTS	39108-34-4	200-4000
6:2 Fluorotelomer sulfonic acid	6:2 FTS	27619-97-2	100-4000
4:2 Fluorotelomer sulfonic acid	4:2 FTS	757124-72-4	200-4000
N-Ethylperfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6	100-4000
N-Methylperfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9	100-4000
Perfluorododecanesulfonic acid	PFDoS	79780-39-5	100-4000
N-Methylperfluorooctanesulfonamide	NMeFOSA	31506-32-8	100-4000
N-Ethylperfluorooctanesulfonamide	NEtFOSA	4151-50-2	100-4000
N-Methylperfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7	100-4000
N-Ethylperfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2	100-4000
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	100-4000
4,8-dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4	100-4000
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1	100-4000
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9	100-4000
Pentafluorpropanoic acid	PFPrA	422-64-0	1000-20000
Perfluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6	100-4000
Perfluoro(2-ethoxyethane) sulfonic acid	PFEESA	113507-82-7	100-4000
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1	100-4000
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5	100-4000
2H,2H,3H,3H-Perfluorohexanoic Acid	3:3 FTCA	356-02-05	400-4000
2H,2H,3H,3H-Perfluorooctanoic Acid	5:3 FTCA	914637-49-3	100-4000
2H,2H,3H,3H-Perfluorodecanoic acid	7:3 FTCA	812-70-4	100-4000
2H-perfluoro-2-octenoic acid	FHUEA	70887-88-6	100-4000
2H-perfluoro-2-decenoic acid	FOUEA	70887-84-2	100-4000
Lithium Bis(trifluoromethane)sulfonimide	HQ-115	90076-65-6	100-4000
Bis[2-(perfluorohexyl)ethyl]phosphate	6:2-diPAP	57677-95-9	500-20000
Bis[2-(perfluorooctyl)ethyl]phosphate	8:2-diPAP	678-41-1	500-20000
Surrogates	**Acronym**	**CAS Number**	**Range (ng/kg)**
Perfluoro-n-[13C4] butanoic acid	MPFBA	1017281-29-6	1000-20000
Perfluoro-n-[13C5] pentanoic acid	M5PFPeA	2283397-79-3	500-20000
Perfluoro-n-[1,2,3,4,6-13C5] hexanoic acid	M5PFHxA	2328024-54-8	100-4000
Perfluoro-n-[1,2,3,4-13C4] heptanoic acid	M4PFHpA	2328024-55-9	100-4000
Perfluoro-n-[13C8] octanoic acid	M8PFOA	1350614-84-4	100-4000
Perfluoro-n-[13C9] nonanoic acid	M9PFNA	2283397-80-6	100-4000
Perfluoro-n-[1,2,3,4,5,6-13C6] decanoic acid	M6PFDA	2328024-56-0	100-4000
Perfluoro-n-[1,2,3,4,5,6,7-13C7] undecanoic acid	M7PFUnA	N/A	100-4000
Perfluoro-n-[1,2-13C2] dodecanoic acid	MPFDoA	960315-52-0	100-4000
Perfluoro-n-[1,2-13C2] tetradecanoic acid	M2PFTreA	N/A	100-4000
Perfluoro-1-[13C8] octanesulfonamide	M8FOSA	N/A	100-4000
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	D3-N-MeFOSAA	N/A	100-4000
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	D5-N-EtFOSAA	N/A	100-4000
N-methyl-d3-perfluoro-1-octanesulfanamide	D3-N-MeFOSA	N/A	100-4000

Surrogates	Acronym	CAS Number	Range (ng/kg)
N-ethyl-d5-perfluoro-1-octanesulfanamide	D5-N-EtFOSA	1265205-97-7	100-4000
2-(N-methyl-d3-perfluoro-1-octanesulfonamido) ethan-d4-ol	d7-N-MeFOSE	1265205-95-5	100-4000
2-(N-ethyl-d5-perfluoro-1-octanesulfonamido) ethan-d4-ol	D9-N-EtFOSE	1265205-96-6	100-4000
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy-13C3-propanoic acid	MHFPO-DA	N/A	100-4000
1H,1H,2H,2H-perfluoro-1-[1,2-13C2] hexane sulfonate	M4:2FTS	2708218-88-4	200-4000
1H,1H,2H,2H-perfluoro-1-[1,2-13C2]-octane sulfonate	M6:2FTS	2708218-89-5	100-4000
1H,1H,2H,2H-perfluoro-1-[1,2-13C2]-decane sulfonate	M8:2FTS	2708218-90-8	100-4000
Perfluoro-1-[13C8] octanesulfonate	M8PFOS	2522762-16-7	100-4000
Perfluoro-1-[2,3,4-13C3] butanesulfonate	M3PFBS	2708218-84-0	100-4000
Perfluoro-1-[1,2,3-13C3] hexanesulfonate	M3PFHxS	2708218-86-2	100-4000
(13C2)2 Bis[2-(perfluorohexyl)ethyl]phosphate	M4-6:2-diPAP	N/A	500-20000

■ Materials and Methods

Calibration Curve

Stock standard solutions containing native analytes and labeled isotopes (surrogates) were diluted from commercially available mixed or single stock standards using a 95:5 (vol:vol) methanol:water mixture. The native analytes were purchased from AccuStandard and the isotopically labeled surrogates were purchased from Wellington Laboratories. The stocks were prepared in high-density polyethylene (HDPE) bottles and stored at 4°C. Prior to preparing the calibration curve, the stock solution was brought to room temperature and thoroughly vortexed to allow adequate homogenization for calibrator preparation. The stock solutions were not filtered, and a 7-9 point external calibration curve was prepared in 50:50 (vol:vol) methanol:water with a 0.1% acetic acid mixture at the levels shown in **Table 2**. The curve was established at low ng/L concentrations, considering that the described sample preparation process effectively dilutes the original consumer product by approximately 20 times (**Equation 1**).

Equation 1: Conversion from in-vial (ng/L) to on-product (ng/kg)

$$C_s\left(\frac{ng}{kg}\right) = \frac{\left[C_i\left(\frac{ng}{L}\right)\right] \times [V_s(L)]}{[W_d(kg)]}$$

where:

C_s = concentration of target analyte in sample,

C_i = concentration of target analyte in sample from instrument,

V_s = volume of sample, and

W_d = dry weight of sample.

Sample Preparation

Samples (0.5 grams +/- 0.01 g) were weighed into a 50-ml polypropylene centrifuge tube and spiked with 40 µl of surrogate spiking solutions (SSS). The mixture was briefly mixed and allowed to equilibrate at room temperature for 15 minutes. A 50:50 (vol:vol) methanol:water solution was added to the sample and vortexed for approximately 1 minute. The sample's pH was then adjusted to approximately 9-10 using 20 µL of ammonium hydroxide, and briefly vortexed again. The centrifuge tubes were then tumbled on a rotator for 2 hours. After tumbling, the entire sample was filtered through a 0.2 µm polypropylene syringe filter (preconditioned with two 10 mL aliquots of methanol), into a 15-mL polypropylene centrifuge tube, to remove particulates while retaining solids behind.

Post filtration, the sample's pH was adjusted to approximately 3-4 using 5 µL acetic acid per 1 mL recovered filtered sample volume. The samples were then briefly vortexed and centrifuged at 8°C for 15 minutes at 3000 rpm. Following centrifugation, the sample was aliquoted into a silanized glass LC vial with a polyethylene/silicone septa, verified to be free of PFAS analytes contained within this method. The sample preparation is outlined in **Fig. 2**.

Table 2: In-vial Native and Surrogate External Calibration Curve Concentrations (ng/L)

Analyte/Surrogate	Cal 1	Cal 2	Cal 3	Cal 4	Cal 5	Cal 6	Cal 7	Cal 8	Cal 9
All Analytes Not Specified Below	5	10	20	40	60	80	100	150	200
3:3 FTCA	--	--	20	40	60	80	100	150	200
8:2 FTS, 4:2 FTS	--	10	20	40	60	80	100	150	200
PFPeA, 6:2-diPAP, 8:2-diPAP	25	50	100	200	300	400	500	750	1000
PFPrA, PFBA	--	50	100	200	300	400	500	750	1000

Fig. 2: Sample preparation procedure for consumer products

Analytical Conditions

Standard analysis was performed using a Shimadzu Nexera UHPLC consisting of 2 pumps (LC-40D X3, 130 MPa), autosampler (SIL-40C X3), system controller (SCL-40), and column oven (CTO-40C). The LC was coupled to a Shimadzu LCMS-8060NX with IonFocus ESI source. The source design allows for high sensitivity analysis while maintaining long-term robustness by implementing electrostatic focusing of analytes while selectively removing neutrals and matrix from the MS inlet.

Compound parameters including quantitation ion, confirmation ion, and collision energies, were optimized using LabSolutions LCMS software. At least two MRM transitions, if available, were used. Data analysis was performed using Insight version 4.0. Analytical conditions are shown in **Table 3**.

Table 3: Analytical Conditions for Consumer Product PFAS Assay using Shimadzu LCMS-8060NX

[LC] Nexera	
Mobile Phase (LCMS Grades)	A: 2 mmol/L Ammonium Acetate in H_2O/ Acetonitrile = 95/5
	B: Acetonitrile
Delay Column	Shimadzu Nexcol PFAS Delay
	3.0 mm x 50 mm, 5 µm (P/N: 220-91394-09)
Analytical Column	Shim-pack Scepter C18-120
	2.1 mm x 100 mm, 3 µm (P/N: 227-31014-05)
Gradient (%B)	10% (0.5 min) ⇒22% (2.3-3.0 min)⇒45% (6.0 min) ⇒ 75% (12.0 min)⇒95% (12.1-14.0 min) ⇒10% (14.1-17.0 min)
Interface	IonFocus ESI (-)
Column Oven Temp.	45°C
Flow Rate	0.45 mL/min
Injection Volume	40 µL
Multiple Draw Injection Program	Co-injection 20 µL Sample → 25 µL 0.1% Acetic acid in H_2O → Co-injection 20 µL Sample → 25 µL 0.1% Acetic acid in H_2O
Autosampler Rinsing	60/40 Acetonitrile/2-propanol, Before/After Aspiration 5 seconds
[MS] LCMS-8060NX	
Interface Temp.	170°C
Probe Position	+3 mm
Nebulizer Gas Flow	3 L/min
Heating Gas Flow	15 L/min
Interface Voltage	-0.5 kV (same value for all compounds)
DL Temp.	200°C
Heatblock Temp.	300°C
Drying Gas Flow	8 L/min
Focus Bias	-2 kV (same value for all compounds)
CID Cell Pressure	270 kPa; 350 kPa 6.6 - 7.6 min and 11.6 - 12.6 min

Autosampler Pretreatment Programming

The Nexera series (SIL-40 series) offers a variety of standard pretreatment functions ranging from sample dilution, reagent addition, and co-injection. The co-injection function provides the ability to inject samples in higher elution strength solvents than the initial mobile phase conditions. Injecting a weaker strength solvent, such as water, lowers the overall elution strength of the injection, improving early eluter peak shapes and the reliability of quantitative results. More details on the effect of co-solvent injections are previously described.[3]

Fig. 3 illustrates the user-friendly co-injection graphical interface within LabSolutions software to build the pretreatment program automatically.

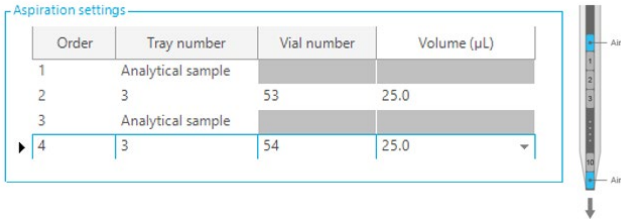

Fig. 3: Co-Injection graphical user interface for pretreatment

■ Results and Discussion

Co-injection Pretreatment Programming

Applying the co-injection technique to the method allowed high organic composition samples (50% methanol) to be injected onto a reverse phase analytical column, which improved the analytical peak shapes for the early eluting analytes. **Fig. 4** highlights with (right panels) and without (left panels) co-injection for PFPrA, PFBA, and PFPeA. The addition of 0.1% acetic acid in water reduced the organic strength of the sample injection (50% methanol) which allowed for better retention and peak shape of the early eluting compounds on the reverse phase column.

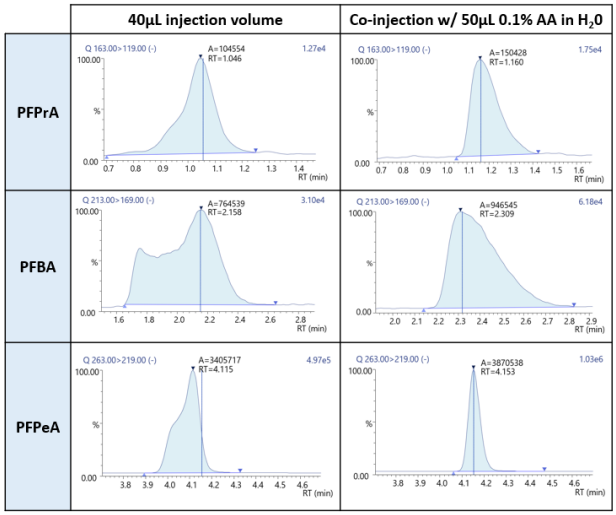

Fig. 4: Comparison of peak shapes of PFPrA (top), PFBA (middle) and PFPeA (bottom) with and without Co-injection

Linearity Study

Calibration curves for each analyte were found by Shimadzu LabSolutions Insight data processing software to have a % RSD Response Factor (RF) of less than 20%. These results meet the criteria for many ASTM LCMS analytical methods that require the % RSD RF to be 30% or less.[4] **Table 4** shows the quantitation and confirmation ions, if available, analyte retention time, and curve % RSD RF.

Example chromatograms and their respective external calibration curves are shown in **Fig. 5** and **Fig. 6** for HQ-115 and PFOS, respectively. HQ-115, an electrolyte used in battery production, is an emerging PFAS target now included in targeted methodologies due to its frequent detection in environmental samples. Analytes that contained linear and branched isomers, as seen with PFOS, had both linear and branched isomers integrated for quantitation.

Table 4: Summary of Calibration Data

Compound	Quantitation Ion	Confirmation Ion	Retention Time (min)	% RSD RF
PFTreA	712.95>668.95	712.95>169.00	10.84	9.3
PFTriA	662.95>618.95	662.95>169.00	10.19	8.0
PFDoA	612.95>568.95	612.95>319.00	9.55	9.7
PFUnA	562.95>518.95	562.95>269.00	8.91	6.2
PFDA	512.95>468.95	512.95>219.00	8.29	14.5
PFNA	462.95>418.95	462.95>219.00	7.72	12.7
PFOA	412.95>369.00	412.95>169.00	7.16	4.8
PFHpA	362.95>319.00	362.95>169.00	6.51	7.9
PFHxA	312.95>269.00	312.95>119.00	5.64	2.9
PFPeA	263.00>219.00	263.00>69.00	4.15	1.6
PFBA	213.00>169.00	----	2.32	7.6
PFDS	598.90>79.95	598.90>98.95	9.91	4.3
PFNS	548.95>79.95	548.95>98.95	9.24	2.8
PFOS	498.95>79.95	498.95>98.95	8.59	7.9
PFHpS	448.95>79.95	448.95>98.95	7.96	12.5
PFHxS	398.95>79.95	398.95>98.95	7.35	7.9
PFPeS	348.95>79.95	348.95>98.95	6.64	6.8
PFBS	298.95>79.95	298.95>98.95	5.63	9.6
PFOSA	497.95>77.95	497.95>477.95	10.45	3.7
8:2FTS	526.95>506.95	526.95>80.90	8.00	19.0
6:2FTS	426.95>406.95	426.95>80.90	6.90	12.9
4:2FTS	326.95>306.95	326.95>80.90	5.23	19.8
NEtFOSAA	584.00>418.95	584.00>526.00	8.55	9.4
NMeFOSAA	569.95>418.95	569.95>482.95	8.28	4.7
PFDoS	698.90>79.95	698.90>98.95	11.21	7.6
NMeFOSA	511.95>219.00	511.95>169.00	12.75	4.8
NEtFOSA	526.00>219.00	526.00>169.00	13.36	4.9
NMeFOSE	616.00>59.00	----	12.45	4.9
NEtFOSE	630.00>59.00	----	13.07	14.5
HFPO-DA	285.00>169.00	285.00>185.00	6.01	3.7
ADONA	376.95>251.00	376.95>85.00	6.77	6.2
9Cl-PF3ONS	530.90>350.95	532.90>352.95	9.07	3.4
11Cl-PF3OUdS	630.90>450.95	632.90>452.95	10.40	4.1
PFPrA	163.00>119.00	----	1.16	6.8
NFDHA	294.95>201.00	294.95>85.00	5.51	5.5
PFEESA	314.95>135.00	314.95>82.95	6.16	6.2
PFMPA	228.95>85.00	----	3.21	8.1
PFMBA	278.95>85.00	----	4.61	6.3
3:3 FTCA	241.00>177.00	241.00>117.00	3.55	14.6
5:3 FTCA	341.00>237.00	341.00>217.00	6.16	13.3
7:3 FTCA	441.00>317.00	441.00>337.00	7.57	9.5
FHUEA	357.00>293.00	----	6.23	2.4
FOUEA	456.95>393.00	----	7.44	3.5
HQ-115	279.90>146.95	279.90>210.90	6.57	3.6
6:2-diPAP	789.00>442.90	789.00>97.00	9.99	8.3
8:2-diPAP	989.00>543.00	989.00>97.00	12.07	9.8
13C4-PFBA_Surr	217.00>172.00	----	2.32	19.8
13C5-PFPeA_Surr	268.00>223.00	----	4.15	6.1
13C5-PFHxA_Surr	318.00>273.00	318.00>120.00	5.64	3.7

Compound	Quantitation Ion	Confirmation Ion	Retention Time (min)	% RSD RF
13C4-PFHpA_Surr	367.00>322.00	----	6.51	18.4
13C8-PFOA_Surr	421.00>376.00	----	7.16	2.5
13C9-PFNA_Surr	472.00>427.00	----	7.72	7.8
13C6-PFDA_Surr	519.00>474.00	----	8.89	6.3
13C7-PFUnA_Surr	570.00>525.00	----	8.91	2.1
13C2-PFDoA_Surr	614.95>569.95	----	9.55	5.5
13C2-PFTreA_Surr	714.95>669.95	----	10.84	16.5
13C8-PFOSA_Surr	505.95>77.95	----	10.45	5.0
D3-NMeFOSAA_Surr	573.00>418.95	----	8.27	6.5
D5-NEtFOSAA_Surr	589.00>418.95	----	8.54	4.6
D3-NMeFOSA_Surr	515.00>219.00	515.00>168.90	12.74	10.6
D5-NEtFOSA_Surr	531.00>219.00	531.00>168.90	13.34	11.3
D7-NMeFOSE_Surr	623.05>59.00	----	12.42	4.5
D9-NEtFOSE_Surr	639.10>59.00	----	13.02	11.0
13C3-HFPO-DA_Surr	287.00>169.00	284.90>185.00	6.01	5.6
13C2-4:2FTS_Surr	329.00>308.95	329.00>80.90	5.23	13.4
13C2-6:2FTS_Surr	428.95>408.95	428.95>80.90	6.90	11.5
13C2-8:2FTS_Surr	528.95>508.95	528.95>80.90	8.00	12.5
13C8-PFOS_Surr	506.95>79.95	506.95>98.95	8.58	6.2
13C3-PFBS_Surr	301.95>79.95	301.95>98.95	5.63	5.9
13C3-PFHxS_Surr	401.95>79.95	401.95>98.95	7.35	6.6
M4-6:2-diPAP_Surr	793.00>445.00	793.00>97.00	9.99	9.2

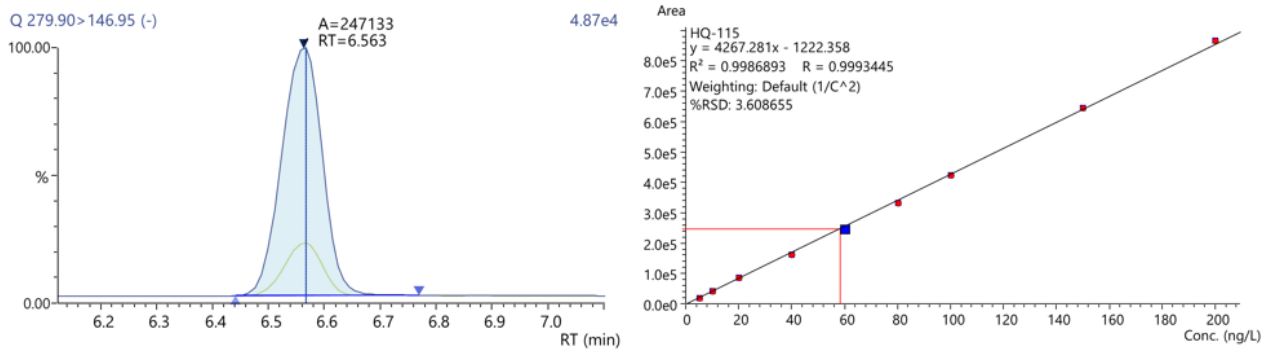

Fig. 5: HQ-115 mid-point chromatogram and calibration curve

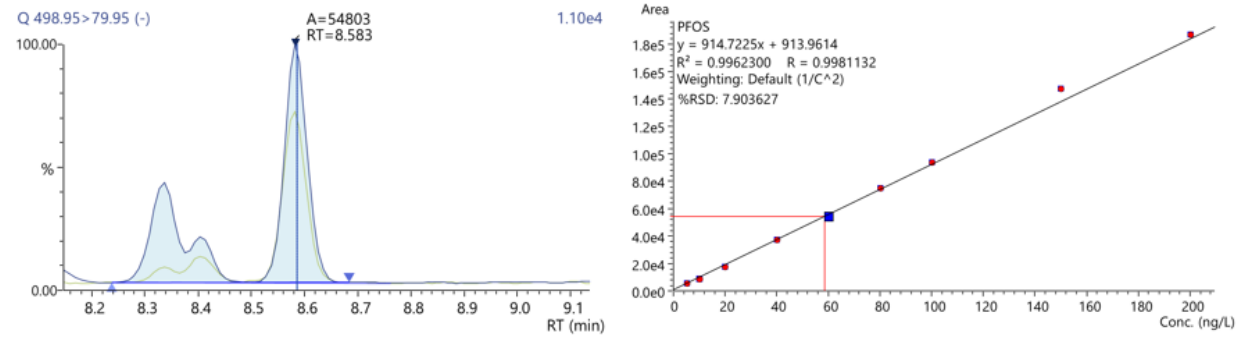

Fig. 6: PFOS mid-point chromatogram (branched and linear isomers integrated) and calibration curve

Recovery and Repeatability Study

Recovery and repeatability were evaluated for the surrogates in PFAS free Ottawa sand, a plastic product, and nonstick aluminum foil. Each matrix was spiked with surrogate spiking solution at the concentration specified in **Table 5**, based on a 0.5 g sample.

Three replicates of each matrix were extracted and analyzed with the above method.

Table 5: Blank Sand Matrix, Plastic Product, and Non-stick Foil Surrogate Spiking Recovery (n=3)

Compound	Spike Conc. ng/kg	Sand Matrix Recovery (%)	Sand %RSD	Plastic Product Recovery (%)	Plastic Product %RSD	Non-stick Foil Recovery (%)	Non-stick Foil %RSD
13C4-PFBA_Surr	8000	104.6	9.7	102.6	2.4	100.5	1.4
13C5-PFPeA_Surr	8000	99.8	6.3	103.4	0.5	101.5	0.6
13C5-PFHxA_Surr	1600	95.7	5.6	98.2	1.4	99.3	0.4
13C4-PFHpA_Surr	1600	96.2	2.1	100.4	2.9	105.3	2.5
13C8-PFOA_Surr	1600	96.4	1.9	96.7	0.8	102.9	3.3
13C9-PFNA_Surr	1600	95.9	2.1	98.1	3.6	95.3	7.1
13C6-PFDA_Surr	1600	95.9	1.6	102.1	2.4	101.4	4.3
13C7-PFUnA_Surr	1600	95.0	4.6	96.7	1.0	103.7	0.5
13C2-PFDoA_Surr	1600	97.0	3.8	119.2	1.6	118.4	2.9
13C2-PFTreA_Surr	1600	92.0	8.6	116.4	0.9	104.1	4.7
13C8-PFOSA_Surr	1600	96.0	7.0	90.8	3.1	93.2	2.0
D3-NMeFOSAA_Surr	1600	97.7	2.5	105.3	1.7	112.0	12.5
D5-NEtFOSAA_Surr	1600	102.2	4.1	98.5	4.1	88.2	12.7
D3-NMeFOSA_Surr	1600	91.2	13.2	97.3	2.5	93.5	4.1
D5-NEtFOSA_Surr	1600	93.4	5.4	88.1	3.5	90.8	3.5
D7-NMeFOSE_Surr	1600	91.5	0.8	99.0	1.5	93.9	1.7
D9-NEtFOSE_Surr	1600	85.1	5.0	91.0	0.5	90.1	3.3
13C3-HFPO-DA_Surr	1600	95.1	3.6	104.5	1.0	103.1	2.9
13C2-4:2FTS_Surr	1600	97.1	6.7	102.7	8.2	104.4	8.2
13C2-6:2FTS_Surr	1600	96.0	14.8	93.4	2.5	102.7	0.2
13C2-8:2FTS_Surr	1600	94.7	12.9	102.5	15.4	101.9	9.6
13C8-PFOS_Surr	1600	99.0	10.3	102.6	1.5	102.4	4.4
13C3-PFBS_Surr	1600	103.0	3.1	100.1	3.4	98.4	1.0
13C3-PFHxS_Surr	1600	95.3	2.1	97.7	8.4	102.1	4.5
M4-6:2-diPAP_Surr	8000	128.7	3.2	117.8	3.2	102.2	6.4

■ Conclusion

This application news demonstrates the analysis of 46 PFAS and 25 surrogate compounds in solid matrices, such as plastic and foil consumer products, using a newly developed method employing a Shimadzu LCMS-8060NX LC-MS/MS system. PFAS-free Ottawa sand was used to confirm surrogate recoveries within a blank solid sample matrix. The extraction procedure, chromatography, and mass spectrometry conditions were optimized to ensure optimal sensitivity for the co-solvation sample preparation procedure. These conditions resulted in a method that eliminates the need for solid phase extraction, therefore significantly reducing cost and time associated with SPE sample preparation.

Target analytes were quantitated using a 7-9-point calibration curve with a reporting range between 100-4,000 ng/kg (dependent on analyte, **Table 1**). Excellent surrogate recoveries were obtained on the plastic and non-stick foil samples, with recovery values within 70-130%. Triplicate extractions resulted in %RSD less than 15%. This method provides a foundation for ongoing efforts to develop a new standardized method with ASTM on PFAS analysis in consumer products.

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