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Liquid Chromatography Mass Spectrometry LCMS-8050

Application News

Determination of PFASs in Food Contact Material by LCMS-8050

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User Benefits

- Compared with the original standard, the number of PFCs tested was increased to 17, which improved the applicability of the method.
- Run time of 15 minutes while ensuring good chromatographic separation and peak shape.

■ Introduction

Due to the unique physical and chemical properties of per- and polyfluoroalkyl substances (PFASs), such as reducing surface tension, good stability, hydrophobicity, and hydrophilic, so it is very widely used in the field of food contact materials. It is mainly used for the production of plastic film, PVC food packaging, waterproof, and oil-proof coating on the surface of food packaging (such as waterproof oil paper products) and non-stick cooker coating, etc. Research has shown that PFASs will migrate from food contact materials to food, and then contaminate the food that comes into contact with it, entering the human body. PFASs are very stable, difficult to be metabolized and biodegraded in the body, and exhibit the characteristics of persistence, accumulation, and long-distance migration. It can be accumulated and amplified in the food chain and enriched in the organisms, endangering human health.

Refer to the sample preparation method in GB 31604.35-2016, determination of both Perfluorooctane Sulfonates (PFOS) and Perfluorooctanoic Acid (PFOA), an analytical method for the determination of 17 perfluorinated compounds in food contact materials was built.

■ Sample Preparation

The samples (1.0 g) were packed into extraction cell. The extraction was conducted by means of the ASE instrument with the following program: heat 5 min, static time 5 min, temperature 110 ˚C, cycles 2, and use MeOH as a solvent. The aliquot of supernatant was pipetted to a clean test tube and evaporated to nearly dryness at 40 ˚C under a stream of nitrogen gas. The residues were dissolved in 10 mL of water and further cleaned up by SPE.

A WCX cartridge was previously activated with 4 mL of (0.1%, v/v) ammoniacal methanol, 4 mL of methanol, and 4 mL of water, successively. The sample solution obtained after extraction was transferred to the cartridge which was rinsed with 4 mL of 25 mM ammonium acetate in water after the extract had passed through. The cartridge was dried by giving a low positive pressure and the analytes were eluted with 4 mL of (0.1%, v/v) ammoniacal methanol. The eluate was dried under a stream of nitrogen gas at 40 ˚C and the resulting sample residue was dissolved in 1 mL of methanol for LC-MS/MS.

■ Analysis Conditions

The analytical conditions for HPLC and MS are shown in Table 1. The MRM transitions are shown in Table 2.

Table 1 Analysis Conditions of Nexera™ and LCMS-8050

System	:	Nexera XS
Column	:	Shim-pack GIST C18-AQ HP (2.1 mm I.D.×100 mm, 1.9 µm)*1
Temperature	:	30 ˚C
Injection volume	:	5 µL
Mobile phases	:	A-5 mM ammonium acetate in Water B-ACN
Flow rate	:	0.3 mL/min
Elution mode	:	Gradient elution
Time program (%B)	:	15% (0 min) → 15% (2.5 min) →98% (10-12 min) → 15% (12.1-15 min)
System	:	LCMS-8050 (ESI Negative)
Nebulizing gas	:	3 L/min
Drying gas	:	10 L/min
Heating gas	:	10 L/min
DL temp.	:	250 ˚C
Heat block temp.	:	400 ˚C
Interface temp.	:	300 ˚C

*1 P/N：227-30807-02

■ Chromatographic separation

The use of the Shim-pack GIST C18-AQ HP column, combined with optimized gradient conditions, resulted in the effective retention of analytes and enabled baseline separation of seventeen PFASs within 15 min, as shown in Figure 1.

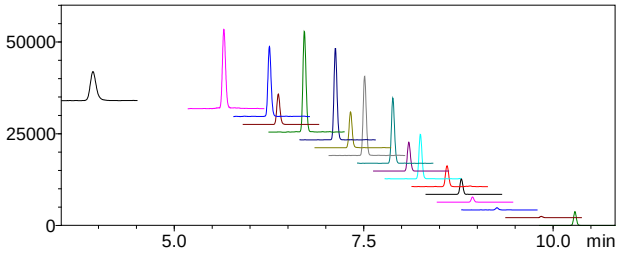

Fig. 1. Chromatograms of seventeen PFASs (0.05 ng/mL)

Table 2 MRM Transition

NO.	Compound	Precursor Ion (*m/z*)	Product Ion (*m/z*)	Q1 (V)	Collision Energy (V)	Q3 (V)	Retention Time (min)
1	PFBA	213.0	169.0	14	10	15	4.014
2	PFPeA	263.0	219.0	18	8	21	5.684
			69.0	20	44	20	
3	PFHxA	313.0	269.0	11	9	10	6.285
			119.0	11	21	17	
4	PFHpA	299.0	80.0	10	33	23	6.748
			99.0	10	27	30	
5	PFOA	363.0	319.0	13	10	13	7.157
			169.0	12	18	15	
6	PFNA	413.0	369.0	11	10	15	7.542
			169.0	14	19	15	
7	PFDA	399.0	80.0	14	45	23	7.916
			99.0	14	35	30	
8	PFUnDA	463.0	419.0	10	10	18	8.282
			219.0	10	17	12	
9	PFDoDA	513.0	469.0	20	11	14	8.636
			219.0	24	17	12	
10	PFTrDA	499.0	80.0	13	55	23	8.969
			99.0	11	39	30	
11	PFTeDA	563.0	519.0	20	13	22	9.291
			269.0	20	18	10	
12	PFHxDA	613.0	569.0	22	12	26	9.875
			269.0	22	19	10	
13	PFODA	599.0	80.0	20	55	26	10.321
			99.0	20	47	10	
14	PFBS	663.0	619.0	22	14	20	6.409
			269.0	22	21	10	
15	PFHxS	713.0	669.0	20	13	22	7.357
			369.0	26	22	15	
16	PFOS	813.0	769.0	22	14	20	8.126
			369.0	22	23	15	
17	PFDS	913.0	869.0	20	16	22	8.822
			369.0	20	26	15	

■ Calibration Curve

The calibration curve (external standard method) prepared using the standard sample showed good linearity over a wide dynamic range from 0.05 to 5 µg/L with R^2 of at least 0.99. The exemplary calibration curves are shown in Figure 2.

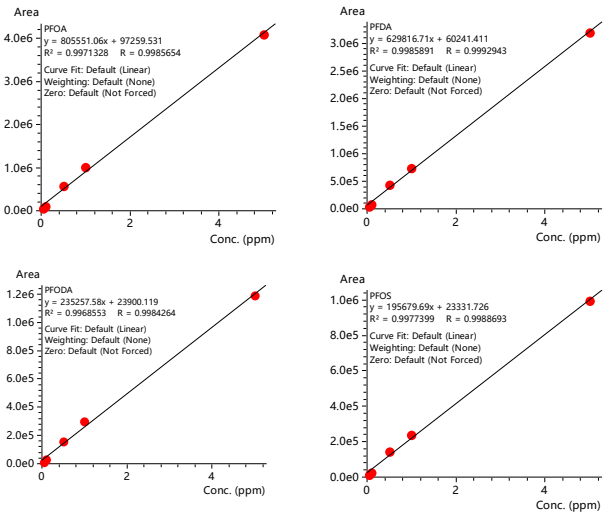

Fig. 2. Calibration Curves

■ Reproducibility recovery

Table 3 shows the reproducibility for the PFASs standard solution which concentration of 0.05, 0.5, and 5 µg/L (n=6).

Table 3 RSD% of R.T. and Area (n=6)

NO.	Compound	0.05 µg/L		0.5 µg/L		5 µg/L	
		R.T	Area	R.T	Area	R.T	Area
1	PFBA	0.51	2.52	0.17	1.38	0.29	0.27
2	PFPeA	0.18	1.75	0.05	1.11	0.08	0.47
3	PFHxA	0.17	1.39	0.06	1.20	0.06	0.60
4	PFHpA	0.17	1.42	0.04	1.13	0.05	0.94
5	PFOA	0.15	3.97	0.04	0.70	0.05	0.64
6	PFNA	0.13	3.43	0.04	1.57	0.04	0.59
7	PFDA	0.12	2.18	0.05	1.97	0.04	1.23
8	PFUnDA	0.14	6.08	0.04	1.61	0.05	1.31
9	PFDoDA	0.12	7.31	0.03	2.17	0.04	3.10
10	PFTrDA	0.11	6.31	0.02	3.25	0.04	2.06
11	PFTeDA	0.14	11.92	0.02	3.10	0.04	2.25
12	PFHxDA	0.11	12.83	0.02	1.87	0.04	1.70
13	PFODA	0.10	6.64	0.01	2.07	0.05	1.14
14	PFBS	0.19	3.07	0.05	1.18	0.06	0.43
15	PFHxS	0.13	0.92	0.04	0.86	0.05	0.83
16	PFOS	0.14	6.55	0.04	0.91	0.04	0.33
17	PFDS	0.12	12.63	0.03	2.71	0.04	1.83

■ Conclusion

Utilizing the LCMS-8050 system for quantitative analysis of PFASs in Food Contact Material revealed that the method can accurately determine PFASs levels within a broad concentration range of 0.05 to 5 ng/mL. This approach exhibits several notable advantages, including excellent repeatability, stability, and reliability.

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