Analysis of PFOS and interference chemicals in food samples using the YMC Accura Triart C18 column

Per- and polyfluoroalkyl substances (PFAS) are a large class of synthetic fluorinated chemicals characterised by highly stable carbon–fluorine bonds that confer exceptional resistance to heat, chemical degradation, and environmental breakdown. PFAS are used in a wide range of industrial and consumer products due to their surfactant properties and chemical durability. However, their persistence enables bioaccumulation in the environment, ultimately entering the food chain. Exposure has been linked to various adverse health effects. As a result, PFAS, including perfluorooctanesulfonic acid (PFOS) are now regulated worldwide, making reliable monitoring essential.

Accurate PFOS determination by liquid chromatography–mass spectrometry (LC-MS) particularly in food can be challenging due to matrix-derived interferences. Cholic acids are naturally occurring biological compounds involved in lipid digestion and metabolism and are therefore present in certain food types, particularly animal-derived products such as eggs, meat, and fish. Cholic acid–related compounds present in food can generate the same selected reaction monitoring (SRM) transition as PFOS in ESI negative mode, resulting in potential false positives. This makes them indistinguishable without chromatographic separation (Figure 1).

Table 1: Final method conditions [1].

Column:	YMC Accura Triart C18 (3 µm, 12 nm) 150 x 2.1 mm ID
Part No.:	TA12S03-15Q1PTC
Eluents*:	A) 5 mM ammonium acetate in water B) acetonitrile
Gradient:	30%B (0-20 min), 80%B (20-20.1 min), 98%B (20.1-25 min), 30% B (25-25.1 min), 30%B (25.1-30 min)
Flow rate:	0.2 mL/min
Temperature:	40 °C
Injection:	10 µl
Detection:	ESI-MS/MS (negative mode)
Sample:	Standards of PFOS (Wellington Laboratories), PFAS (PFAC-MXC, Wellington Laboratories), TUDCA (Nacalai Tesque), TCDCA (Nacalai Tesque) and TDCA (Cayman Chemical)

**a PFAS delay column, 3.0 × 30 mm, was installed after mobile phase supply to eliminate background contamination*

This Application Note, based on the work of Takayama et al. [1], demonstrates the effective separation of PFOS from these interfering compounds using the YMC Accura Triart C18 column in LC-MS/MS analysis. The column's hybrid silica particles are not only highly robust but also provide an increased surface area compared to other columns, making it very tolerant to complex food matrices. Further, the used (bio)inert coated stainless steel YMC Accura column hardware prevents any kind of possible unwanted interaction of sample substances with metal ions.

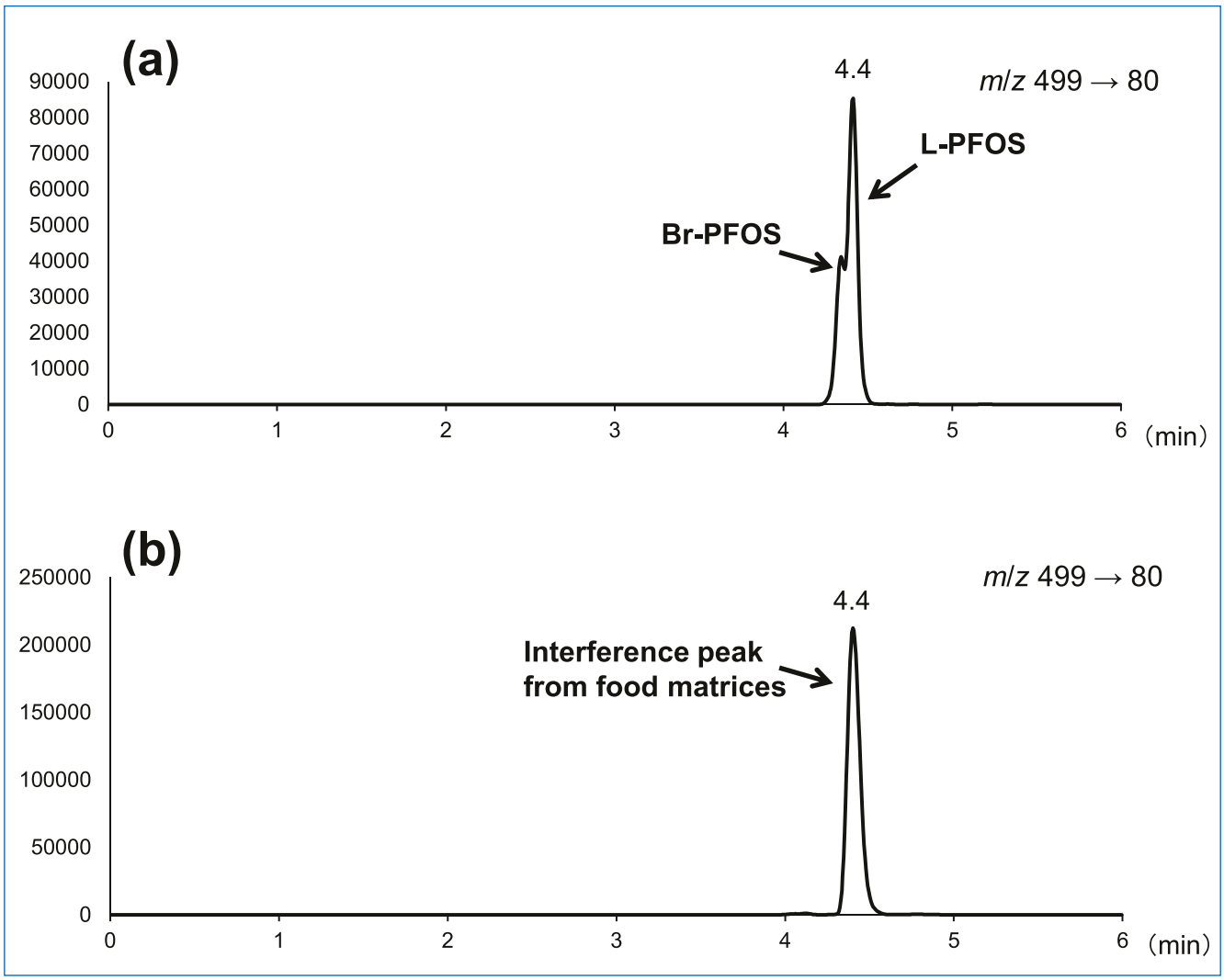

Figure 1: SRM chromatograms of the m/z 499 → 80 transition acquired from a PFOS standard solution (a) and an unspiked egg sample (b) under methanol-based LC-MS/MS conditions [1].

Methanol-free mobile phase as a key differentiator in PFOS and taurocholic acid isomer separation

PFOS and several isomers of taurocholic acid including tauroursodeoxycholic (TUDCA), taurochenodeoxycholic acid (TCDCA) and taurodeoxycholic acid (TDCA), share the same mass transition (m/z 499 → 80). In PFAS analysis, methanol is commonly employed as the organic solvent because it generally provides strong retention and good ionisation efficiency. However, under these conditions, PFOS co-elutes with the taurocholic acid isomers, making selective detection impossible. To overcome this limitation, acetonitrile was evaluated as alternative organic solvent, leading to significantly improved separation (Figure 2).

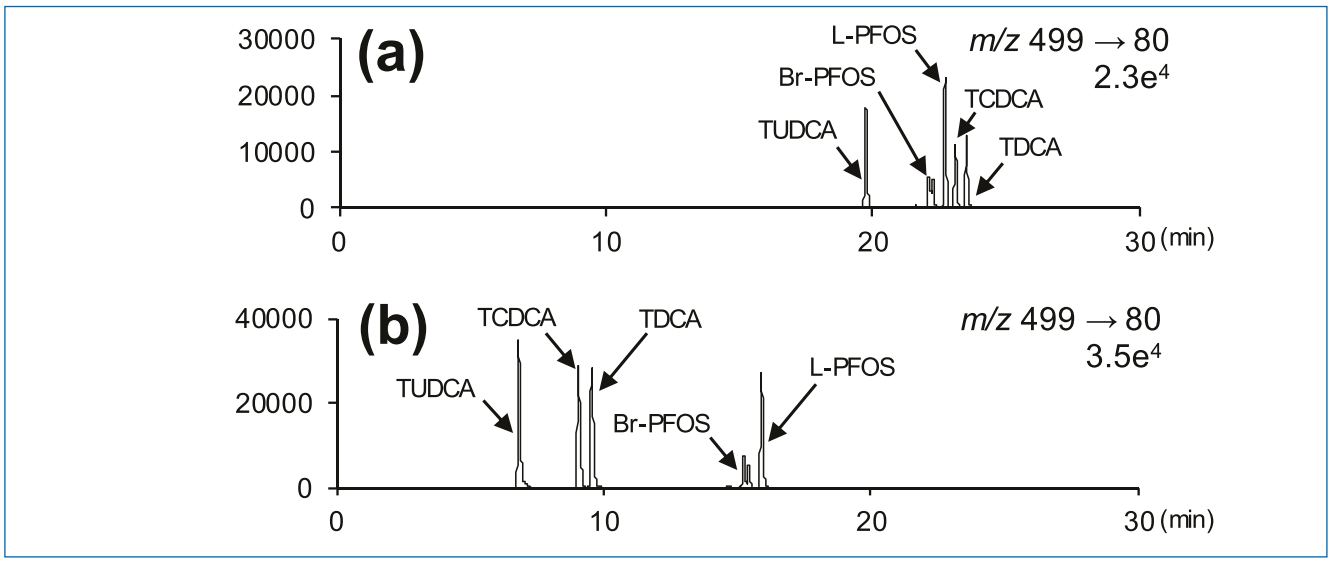

Figure 2: SRM chromatograms of linear (L-PFOS) and branched (Br-PFOS) PFOS and taurocholic acids isomers including TUDCA, TCDCA, TDCA using methanol (a) and acetonitrile (b) as organic solvent [1].

When acetonitrile was used as the organic solvent, PFOS and the taurocholic acid isomers were completely separated (Figure 2). These results demonstrate that full separation can only be achieved using a methanol-free method. The YMC Accura Triart C18 column was selected from a panel of seven evaluated columns, as it provided the best overall performance, including superior resolution, peak shape, and recovery. For this reason, it was chosen for all subsequent analyses.

Reliable PFAS profiling under optimised LC-MS/MS conditions

Figure 3 shows the SRM chromatograms of the 21-component PFAS mixture (PFAC-MXC, Table 2) analysed under the optimised LC-MS/MS conditions using an acetonitrile-based mobile phase. Under these conditions, all target PFAS were successfully separated across the chromatographic run, with distinct and well-resolved peaks for both carboxylate- and sulfonate-type species. Short- and mid-chain PFAS (C4–C9) eluted with sharp peak shapes and strong signal intensities, supporting sensitive and quantitative detection. Although the four long-chain PFAS (PFTeDA, PFHxDA, PFDoS, PFODA) eluted only in the final gradient step (98% acetonitrile) and showed lower response due to their strong hydrophobic retention, they were still clearly identifiable within the chromatograms.

These results show that the optimised method, in combination with the YMC Accura Triart C18 column, offers stable retention and effective selectivity for a broad range of PFAS.

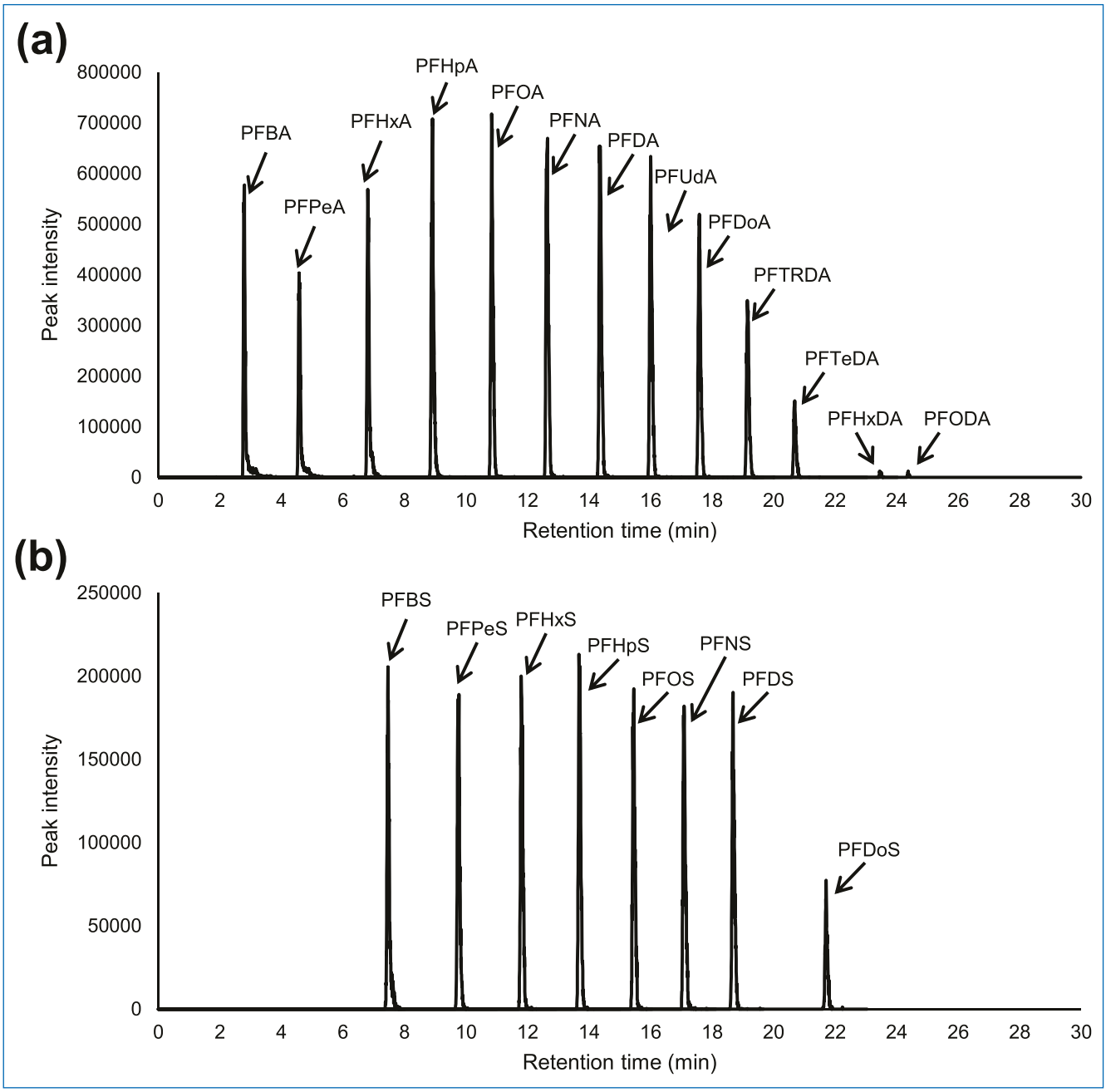

Figure 3: SRM chromatograms of the 21-component PFAS mixed standard (PFAC-MXC) acquired under the optimised LC-MS/MS conditions, shown as overlaid traces for carboxylate-type PFAS (a) and sulfonate-type PFAS (b) [1].

Table 2: Analytes and internal standards list for PFAC-MXC and MPFAC-C-ES [1].

Analytes of PFAS (Abbreviation)	Monitoring ions of analytes	I.S.	Monitoring ions of I.S.
Perfluoro-n-butanoic acid (PFBA)	213 → 169	$^{13}C_4$-PFBA	217 → 172
Perfluoro-n-pentanoic acid (PFPeA)	263 → 219	$^{13}C_5$-PFPeA	268 → 232
Perfluoro-n-hexanoic acid (PFHxA)	313 → 269	$^{13}C_5$-PFHxA	318 → 273
Perfluoro-n-heptanoic acid (PFHpA)	363 → 319	$^{13}C_4$-PFHpA	367 → 322
Perfluoro-n-octanoic acid (PFOA)	413 → 369	$^{13}C_8$-PFOA	421 → 376
Perfluoro-n-nonanoic acid (PFNA)	463 → 419	$^{13}C_9$-PFNA	472 → 427
Perfluoro-n-decanoic acid (PFDA)	513 → 469	$^{13}C_6$-PFDA	519 → 474
Perfluoro-n-undecanoic acid (PFUdA)	563 → 519	$^{13}C_7$-PFUdA	570 → 525
Perfluoro-n-dodecanoic acid (PFDoA)	613 → 569	$^{13}C_2$-PFDoA	615 → 570
Perfluoro-n-tridecanoic acid (PFTrDA)	663 → 619	$^{13}C_{12}$-PFDoA	615 → 570
Perfluoro-n-tetradecanoic acid (PFTeDA)	713 → 669	$^{13}C_2$-PFTeDA	715 → 670
Perfluoro-n-hexadecanoic acid (PFHxDA)	813 → 769	$^{13}C_2$-PFTeDA	715 → 670
Perfluoro-n-octadecanoic acid (PFODA)	913 → 869	$^{13}C_2$-PFTeDA	715 → 670
Potassium perfluoro-1-butanesulfonate (PFBS)	299 → 80	$^{13}C_3$-PFBS	302 → 80
Sodium perfluoro-1-pentanesulfonate (PFPeS)	349 → 80	$^{13}C_3$-PFBS	302 → 80
Sodium perfluoro-1-hexanesulfonate (PFHxS)	399 → 80	$^{13}C_3$-PFHxS	402 → 80
Sodium perfluoro-1-heptanesulfonate (PFHpS)	449 → 80	$^{13}C_9$-PFNA	472 → 427
Sodium perfluoro-1-octanesulfonate (PFOS)	499 → 80	$^{13}C_8$-PFOS	507 → 80
Sodium perfluoro-1-nonanesulfonate (PFNS)	549 → 80	$^{13}C_7$-PFUdA	570 → 525
Sodium perfluoro-1-decanesulfonate (PFDS)	599 → 80	$^{13}C_8$-PFOS	507 → 80
Sodium perfluoro-1-dodecanesulfonate (PFDoS)	699 → 80	$^{13}C_8$-PFOS	507 → 80

Recovery of PFAS in real food samples

To determine the recovery of PFAS in real food, various food matrices (meat (beef), vegetable (cabbage), fish (sea bream), and egg (chicken)) were spiked with final concentrations of 1 ng/g as well as 0.1 ng/g using standard PFAS mixtures (PFAC-MXC and MPFAC-C). Chromatograms are shown as representative examples for meat samples (Figures 4 and 5). Across four representative food types and two low-level spike concentrations, the method consistently delivered high recoveries within the desirable 80–120% range for all C4–C9 PFAS, confirming accurate quantification even at trace levels (Table 3). The precision was demonstrated by the fact that the vast majority of relative standard deviation (RSD) values remained well below 15%. These results highlight the excellent performance of the YMC Accura Triart C18 column for PFAS analysis even in complex food matrices. The column's outstanding reproducibility, along with its ability to maintain stable retention, sharp peak shapes, and reliable performance across diverse matrices such as meat, fish, vegetables, and eggs, further supports its suitability for routine PFAS analysis.

Table 3: Recovery test of 12 PFAS in food samples [1].

Foods	Concentration (0.1 ng g^{-1})	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS
Meat	Accuracy (%)	103.3	96.0	100.1	92.4	102.2	98.2	96.3	95.0	96.8	98.7	102.1	104.7
	RSD (%)	2.0	7.9	6.8	4.8	3.7	5.1	7.7	3.6	5.6	12.3	8.6	6.9
Vegetable	Accuracy (%)	78.4	125.0	94.4	102.3	99.2	97.8	92.7	96.0	95.1	96.3	98.5	97.1
	RSD (%)	8.2	1.6	5.8	2.0	2.3	5.5	15.2	1.8	14.6	12.5	8.6	4.0
Fish	Accuracy (%)	100.3	100.3	98.7	94.6	105.3	101.6	100.3	92.3	103.7	93.5	96.1	80.8
	RSD (%)	3.8	2.1	6.2	1.6	3.6	3.1	14.6	7.0	3.4	11.4	9.8	8.2

Foods	Concentration (1.0 ng g^{-1})	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS
Meat	Accuracy (%)	103.3	101.6	97.5	98.2	103.4	96.0	93.2	99.2	98.7	95.2	93.5	95.7
	RSD (%)	1.1	2.7	0.4	3.6	3.5	2.1	15.0	1.6	1.0	9.3	9.8	12.1
Vegetable	Accuracy (%)	98.6	99.1	96.5	94.9	100.7	98.0	100.7	99.2	101.2	94.4	97.8	117.3
	RSD (%)	0.7	3.5	3.3	4.8	3.4	2.0	3.0	1.8	3.9	6.0	7.4	3.9
Fish	Accuracy (%)	99.7	97.9	95.9	93.1	99.0	99.9	113.0	95.4	93.7	84.0	100.9	13.7
	RSD (%)	2.1	1.7	0.4	0.7	1.7	4.6	9.4	3.4	4.1	4.8	4.4	3.4

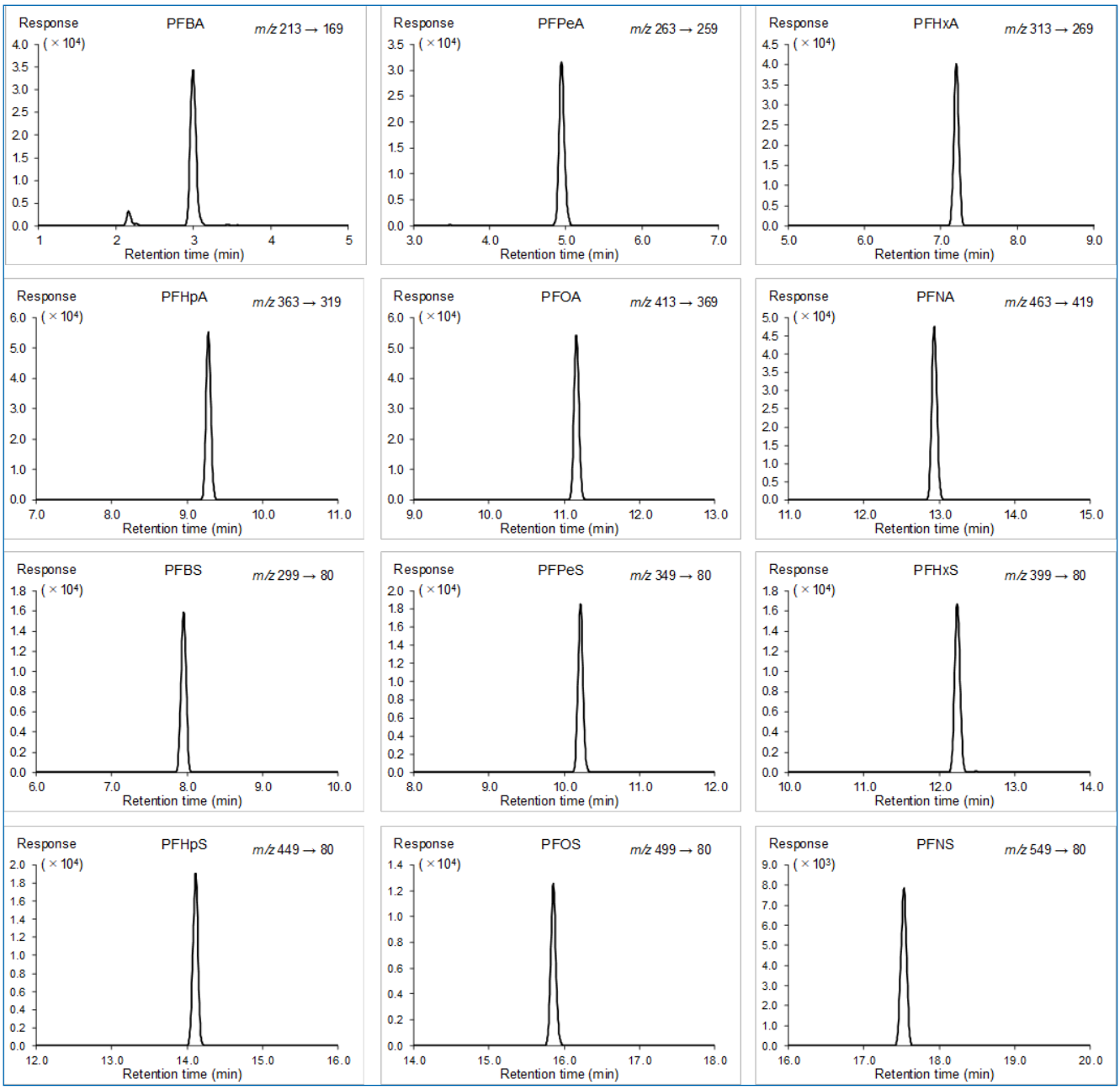

Figure 4: SRM chromatograms of 12 PFAS spiked at a final concentration of 1 ng/g into meat (beef), acquired under the optimised LC-MS/MS conditions [1].

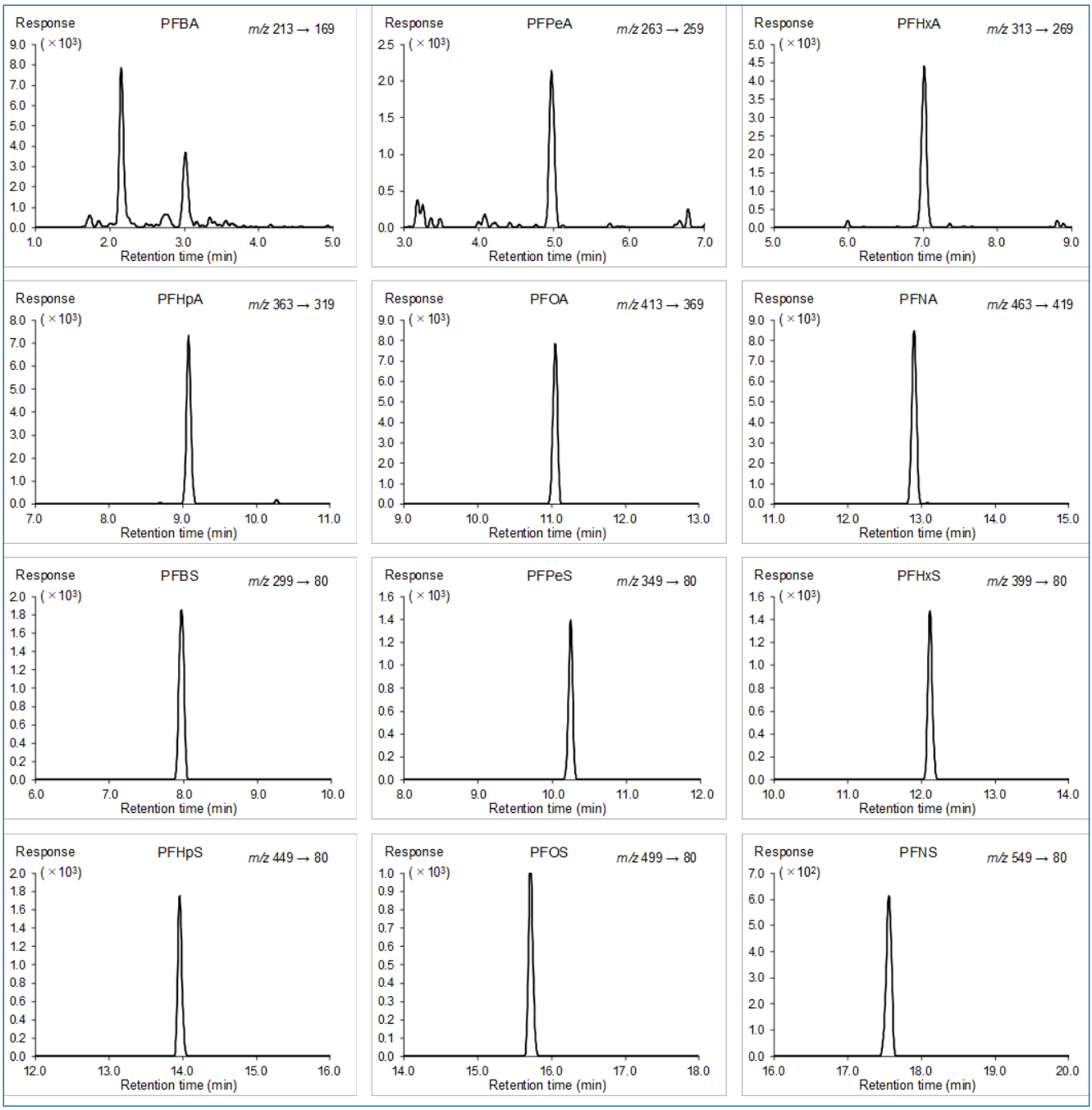

Figure 5: SRM chromatograms of 12 PFAS spiked at a final concentration of 0.1 ng/g into meat (beef) samples, acquired under the optimised LC-MS/MS conditions [1].

In the next step, endogenous matrix components in meat and egg samples were evaluated for potential interference in the PFOS transition. Figure 6 shows SRM chromatograms from unspiked samples analysed under the optimised LC-MS/MS conditions. In both matrices, interference peaks attributed to taurocholic acid–related compounds are observed at earlier retention times, while PFOS would appear much later at approximately 15–16 minutes. This demonstrates that the chosen chromatographic conditions are able to fully separate PFOS from matrix-derived interferences.

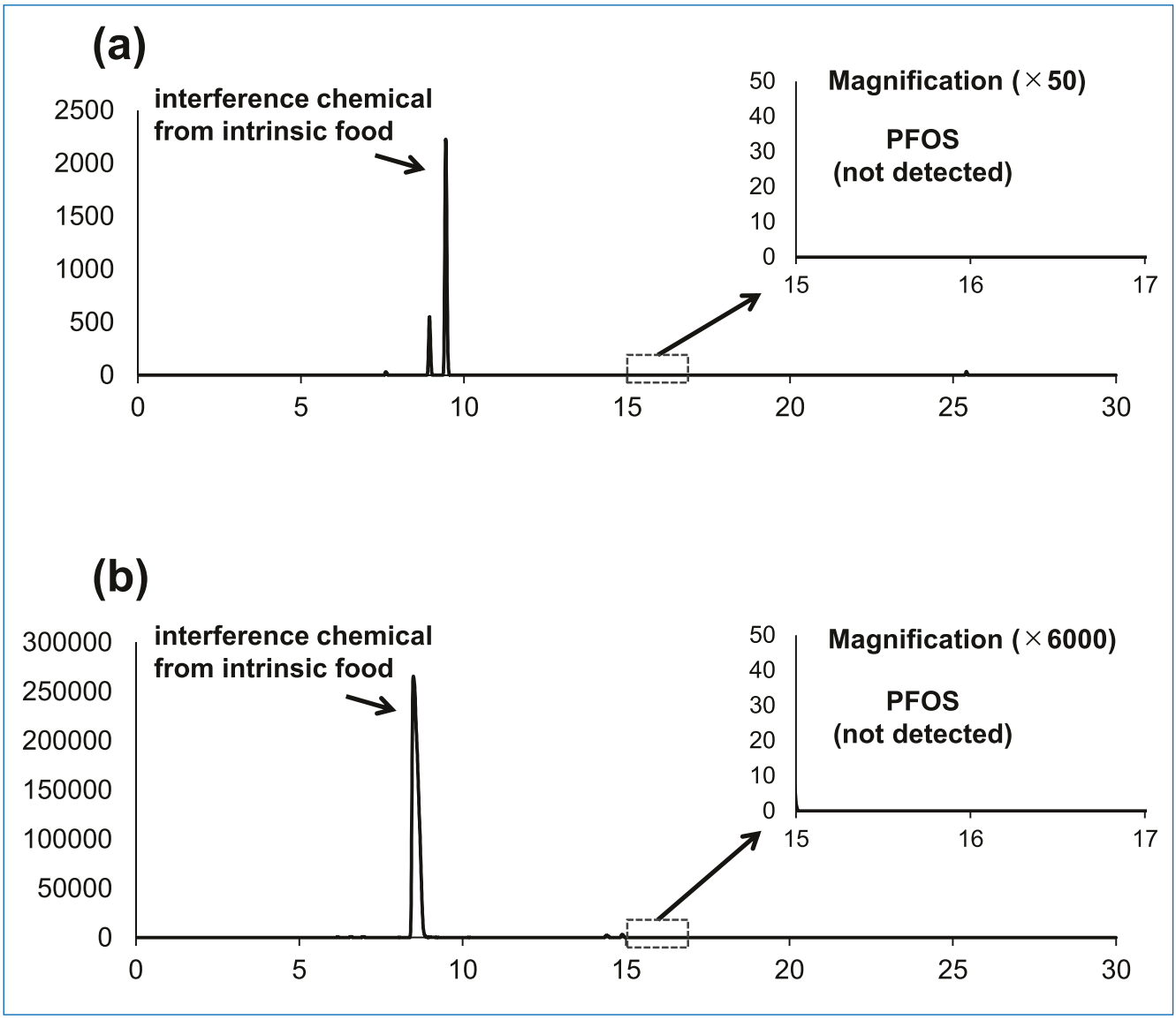

Figure 6: SRM chromatograms of unspiked meat (a) and egg (b) samples obtained under the optimised LC-MS/MS conditions [1].

Conclusions

The developed method using the YMC Accura Triart C18 column demonstrates:

- **Clear separation** between PFOS and taurocholic acid interferences only under methanol-free conditions.
- **Stable retention** and **sharp peak shapes** for a broad range of PFAS, including C4–C9 analytes.
- **High recoveries** (80–120%) and **RSD values** (<15%) across multiple food matrices demonstrating suitability for **trace-level PFAS analysis**.
- **Accurate quantification** of PFAS in a certified reference material.

Overall, the YMC Accura Triart C18 column offers a reliable platform for high-quality, interference-free PFAS determination in LC-MS/MS workflows.

References

[1] T. Takayama, A. Shingu, S. Kato, R. Nagatomo, T. Tsutsumi, K. Inoue, Countermeasure for interfered monitoring ion of perfluorooctanesulfonic acid (PFOS) from intrinsic food samples based on LC-MS/MS analysis of per- and polyfluoroalkyl substances, J. Food Compos. Anal., 2024, 133, 106436

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