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Chromatography Comparison of Ultra-Short-Chain PFAS in Complex Food Matrix Extracts

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Introduction

Ultra-short-chain PFAS (USC PFAS) compounds such as trifluoroacetic acid (TFA), have gained increasing attention in food safety analysis, yet their determination remains analytically challenging. Their high polarity and very low molecular weight result in poor retention, elevated background noise, significant suppression or interfering from polar matrix co-extractives when analyzed using conventional reversed-phase chromatography. In this study, the chromatographic performance of several commercially available mixed-mode LC columns designed for USC PFAS analysis was systematically evaluated using extracts from diverse and complex food matrices. Performance was assessed based on analyte retention and separation efficiency on columns, mitigation of solvent effects from high-organic injections, and susceptibility to matrix effects and interferences.

Target	Abbreviation	Precursor Ion (m/z)	Product Ion (m/z)
Trifluoroacetic acid	TFA	113	68.9
Trifluoromethane sulfonic acid	TFMS	149	98.9 79.9
Perfluoropropionic acid	PFPrA	163	118.9
Pentafluoroethanesulfonic acid	PFEtS	199	98.9 79.8
Perfluorobutanoic acid	PFBA	213	169
Perfluoropropane sulfonic acid	PFPrS	248.9	98.9 79.8
Perfluoropentanoic acid	PFPeA	263	219
Perfluorobutanesulfonic acid	PFBS	293.9	99 80

Table 1. USC PFAS analytes and MRM transitions.

Experimental

Sample preparation for food matrix extract

Four food matrices were included in this study: two types of baby food, whey protein powder, pet food and shrimp. Samples were extracted following previously established protocols using QuEChERS extraction followed by EMR mixed-mode passthrough cleanup. In this study, the method was used solely to prepare matrix-blank extracts and matrix-matched post spike.

After collection of the EMR-cleaned extracts, a portion of each was spiked with a USC/SC standard spiking solution at 1 ng/mL and 0.1 ng/mL. Both matrix blanks and fortified QC samples were used to evaluate chromatographic performance.

Experimental

LC/MS/MS Detection

LC conditions (Agilent 1290 Infinity II)				
Columns	Agilent Altura Poroshell 120 PFAS HPLC Column, 2.1 x 50 mm, 2.7 µm (p/n 227205-007) with Poroshell 120 PFAS Delay Column, 4.6 x 30 mm, (p/n 027403-007)			
Flow Rate	0.5 mL/min			
Inj. Volume	10 µL (sandwich or feed)		Column Temp.	40 °C
Mobile Phase	A: 5 mM ammonium acetate in water w/ 0.05% acetic acid. B: acetonitrile:water = 95:5 (v/v)			
Gradient	Grad A (Altura & Column 1)		Grad B (Columns 2 & 3)	
	Time (min)	% B	Time (min)	% B
	0.0	10	0.0	2
	0.5	10	2.0	2
	9.0	75	9.0	75
	10.0	100	10.0	100
Sample Solv.	90:10 ACN:water		50:50 ACN:water	
Stop Time	12 min		Post time	3 min

1290 Infinity III Hybrid Multisampler Settings				
Feed Injection	Injection Mode: Feed		Injection volume: 10 µL	
	Feed Speed: Adaptive, 10% of pump flow			
	Flush out mode: automatic			
Injection Path Cleaning	Inner wash mod: Extended		Outer wash mod: Extended	
	Step	Task	Solution	Duration/Volume
	Draw sample			
	1	Outer wash	S1 = ACN	10 sec.
	2	Outer wash	S3 = 1:1 ACN/IPA	10 sec.
	Injection			
	1	Inner wash	S2 = 90:10 MPA/MPB	150 µL
	2	Inner wash	S2 = 90:10 MPA/MPB	150 µL
	3	Seat wash	S1 = ACN	150 µL
	4	Seat wash	S3 = 1:1 ACN/IPA	150 µL
	5	Reconditioning	S2 = 90:10 MPA/MPB	

TQ Conditions (Agilent 6495D LC/TQ)			
Stop Time	200 °C, 18 L/min	Sheath Gas	300 °C, 11 L/min
Nebulizer Gas	15 psi	Polarity	Negative
Capillary Volt.	2500 V	Nozzle Volt.	0 V

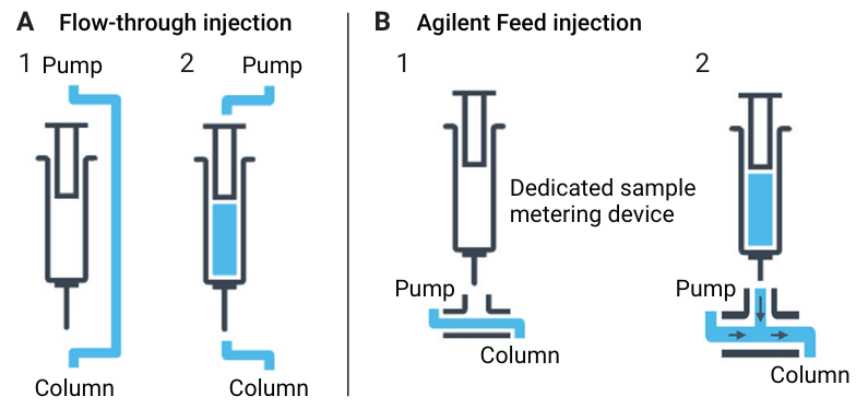

Figure 1. Classic flow-through injection (A) versus feed injection (B).

Results and Discussion

Improved USC PFAS Chromatographic Performance

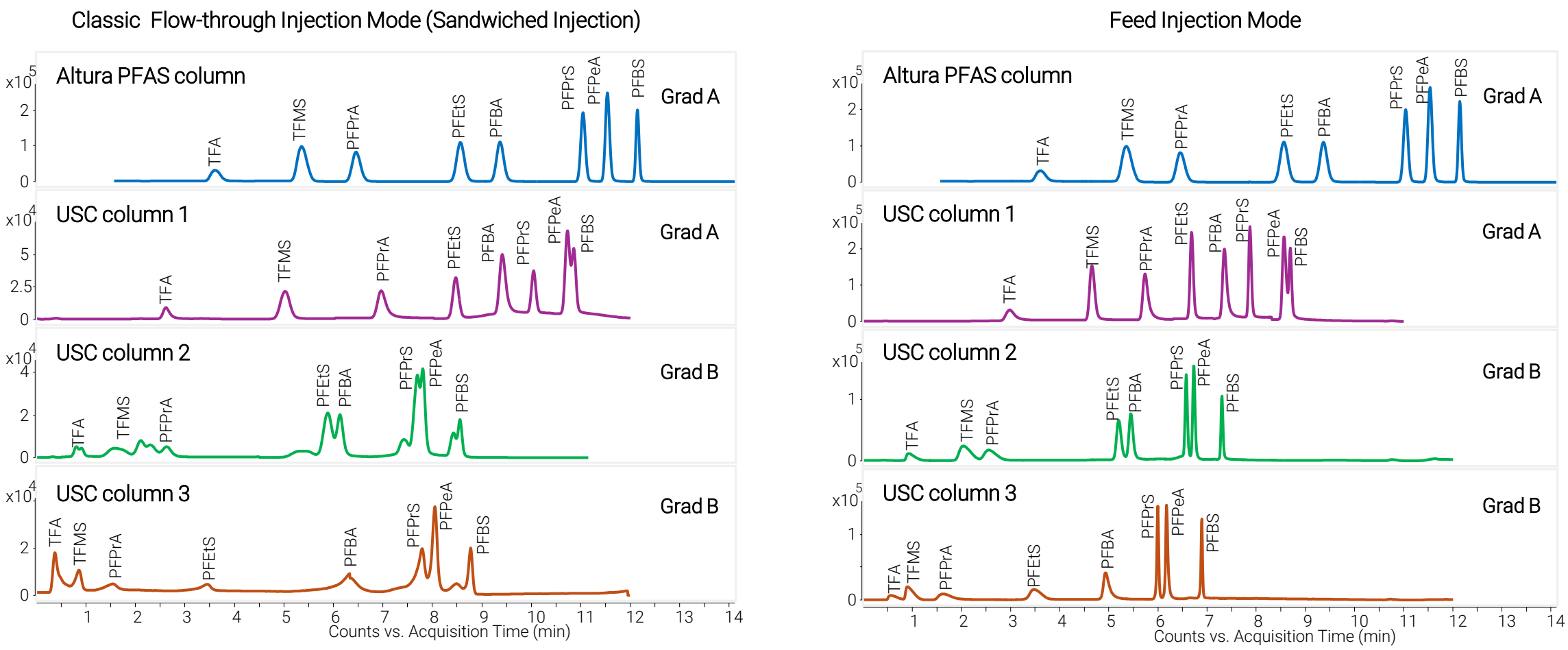

Figure 2. Chromatograms of neat standards analyzed on four mixed-mode LC columns on classic flow-through injection mode (left) versus feed injection mode (right).

- Feed injection improved the peak shape and retention for USC PFAS, especially for USC columns 2&3.
- Altura PFAS column provided the strongest analyte retention, and excellent symmetry for all analyte peaks. This column enables the injection of sample in high organic and more tolerance on LC gradient adjustment.
- Column 1 also demonstrated decent performance, with slightly shorter retention and several pronounced tailing analytes (TFA, PFPrA, and PFBA).
- Columns 2 & 3 barely achieved acceptable peak integrity even with multiple strategies for solvent effect mitigation. These two columns require samples in weak solvent in high aqueous.

Reduced Matrix Effects on USC PFAS

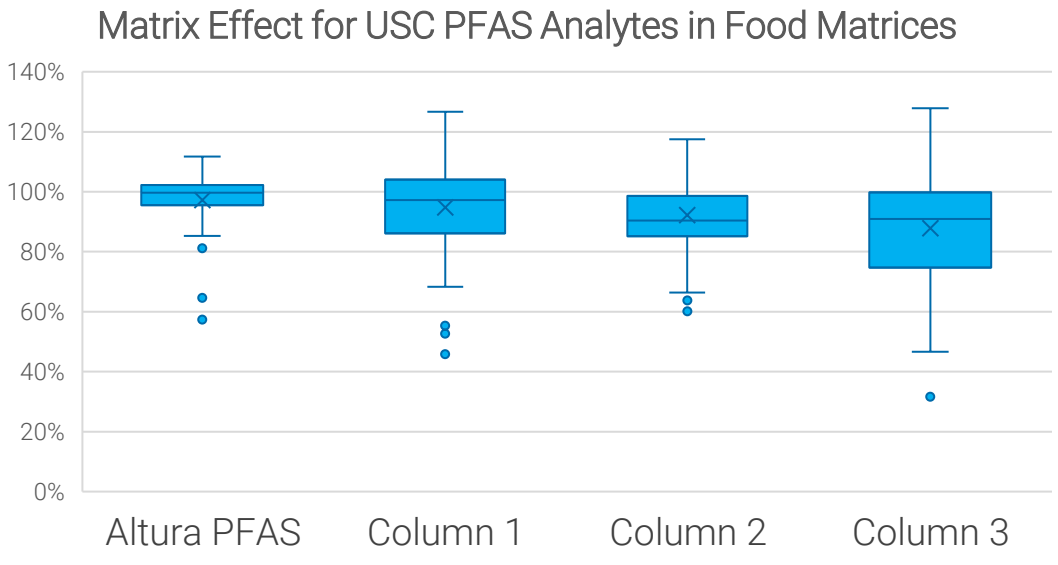

Figure 3. Matrix effects for USC analytes in food matrix extracts using four mixed-mode LC columns. Matrix-matched samples were compared with neat standard at corresponding 1 ng/mL level.

- To evaluate the matrix-effect mitigation on each column, matrix effects for eight USC PFAS analytes were calculated across five food matrix extracts.
- The better reduction in matrix effects, the less ion suppression in the MS source caused by co-eluting polar analytes.
- The Altura PFAS column yielded matrix effects within 90–110% for all analytes except TFA, indicating minimal matrix-induced analyte response suppression or enhancement.
- Column 1 exhibited the next highest retention for TFA and consequently produced the second strongest response.
- Columns 2 and 3 showed the poorest retention for TFA, resulting in either the lowest response (column 3) or co-elution with isobaric interferences (column 2).

Results and Discussion

Baseline separation of PFBA with Isobaric Matrix Interference on the Altura PFAS Column

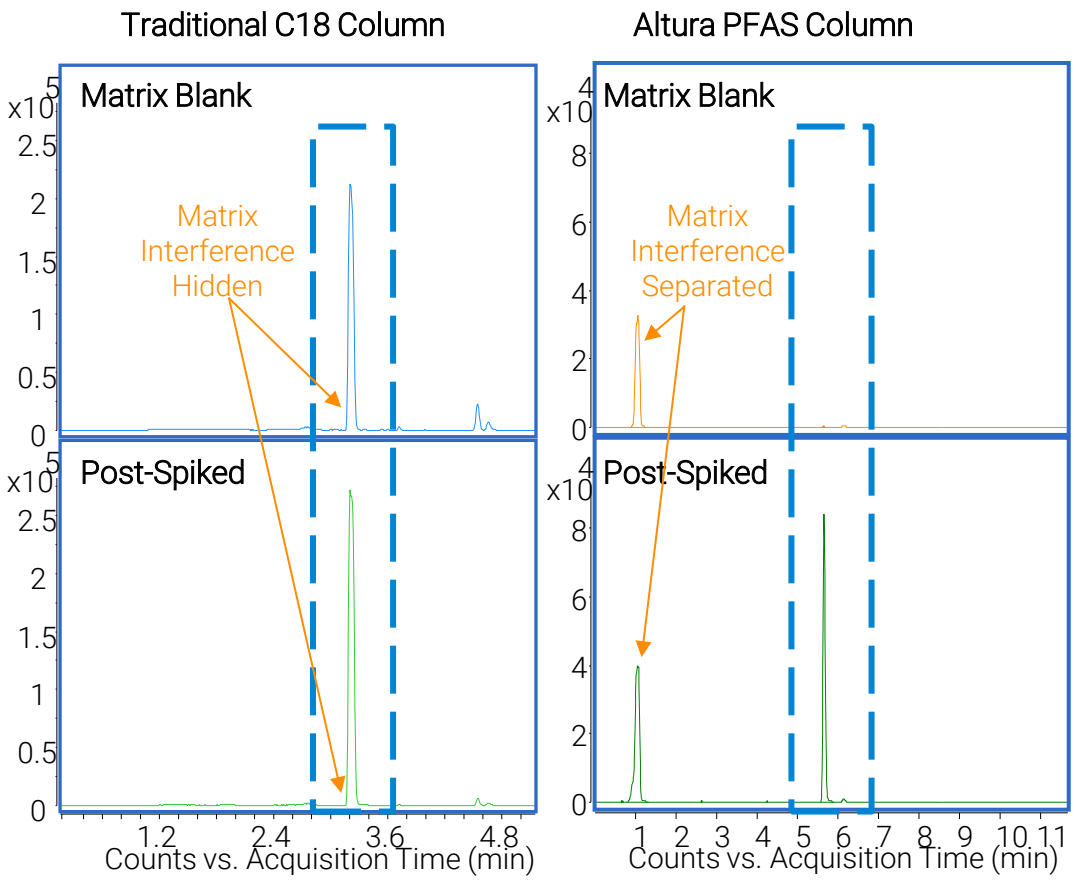

Figure 4. Comparison of performance in separating PFBA from isobaric interference on traditional C18 column (left) vs the Altura PFAS column (right) in baby food extract.

- A strong isobaric interference from plant-based food matrices often co-elutes with PFBA on traditional C18 column.
- PFBA and PFPeA only have one MRM transition, resulting in lack of qualifier for these two analytes.
- Polar isobaric interference coeluted with PFBA on the traditional C18 column, increasing the risk of false-positive PFBA detection and driving a higher method LOQ.
- The enhanced retention and selectivity of USC PFAS on the Altura PFAS column offer great potential to surpass limitations in current guidance.
- An excellent 2nd confirmatory approach for PFBA and PFPeA analysis in food.

Products and Publications

Captiva EMR PFAS Food cartridges

Altura Poroshell 120 PFAS column

Conclusions

- Feed Injection function equipped on Agilent Hybrid Multisampler successfully mitigates the significant solvent effects observed on columns 2&3.
- The Altura PFAS column effectively mitigates solvent-related injection effects, permitting direct analysis of high-organic food extracts, and exhibits stronger retention and improved chromatographic resolution of USC PFAS analytes relative to other mixed-mode USC PFAS columns.
- Through consistently high-quality chromatograms and strong analyte-matrix separation, the Altura PFAS column achieves effective matrix-effect mitigation through minimized ion suppression and improved resolution of matrix-derived isobaric interferences.

References

1. Kamuf, M. More Sensitive Quantification of PFAS by LC/MS with the Agilent 1260 Infinity II Hybrid Multisampler, *Agilent Technologies application note*, publication number 5994-6994EN.
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