

Poster Reprint

ASMS 2026
ThP 227

Streamlined EMR LC-MS/MS Approach for Sensitive PFAS Determination in Alcoholic Beverages

Limian Zhao and M. Lorna De Leoz

Agilent Technologies Inc., Wilmington, DE, 19808

Introduction

Per- and polyfluoroalkyl substances (PFAS) are persistent synthetic chemicals increasingly detected in the food chain, raising concerns about dietary exposure. Alcoholic beverages, produced through multi-step processes dependent on agricultural raw materials and water, may become contaminated when grapes or process water are exposed to PFAS-impacted soils, irrigation sources, or municipal supplies. Despite this potential exposure route, alcoholic beverages remain an underexamined matrix for PFAS monitoring.

To address this gap, we developed a streamlined method utilizing EMR (Enhanced Matrix Removal) and LC-MS/MS with feed injection, thereby enabling sensitive quantitation of 43 PFAS in alcoholic drinks. This streamlined workflow overcomes the analytical challenges posed by high-organic matrices while supporting the performance expectations of accurate and precise quantitative analysis in food safety laboratories.

Experimental

LC conditions (Agilent 1290 Infinity II)

Columns	RRHD Eclipse Plus C18, 1.8 µm, 2.1 x 100 mm (p/n 959758-902) Guard: Eclipse Plus C18, 1.8 µm, 2.1 x 5 mm (p/n 821725-901) Delay: Agilent InfinityLab PFC, 4.6 x 30 mm (p/n 5062-8100)		
Flow Rate	0.4 mL/min		
Mobile Phase	A: 5 mM ammonium acetate in water B: Acetonitrile		
Gradient	Time (min)	%B	Flow (mL/min)
	0	10	0.4
	2	30	0.4
	8.5	45	0.4
	11.5	75	0.4
	13.25	100	0.4
Stop Time	15.5 min	Post time	2 min

Agilent 1290 Infinity III Hybrid Multisampler

Feed Injection	Injection Mode: Feed Injection Volume: 12.50 µL Feed speed: Adaptive, 10% of pump flow Flush out mode: Automatic
Injection Path Cleaning	Outer wash: ACN & 1:1 ACN/IPA Inner wash: 90:10 A/B Seat wash: ACN & 1:1 ACN/IPA Reconditioning: 90:10 A/B

LC/TQ conditions (Agilent 6495D LC/MS system)

Drying Gas	200 °C, 18 L/min			Sheath Gas	300 °C, 11 L/min		
Nebulizer Gas	15 psi	Polarity	Neg	Acquisition	dMRM		
Capillary Voltage	2500 V			Nozzle Voltage	0 V		

Sample Preparation

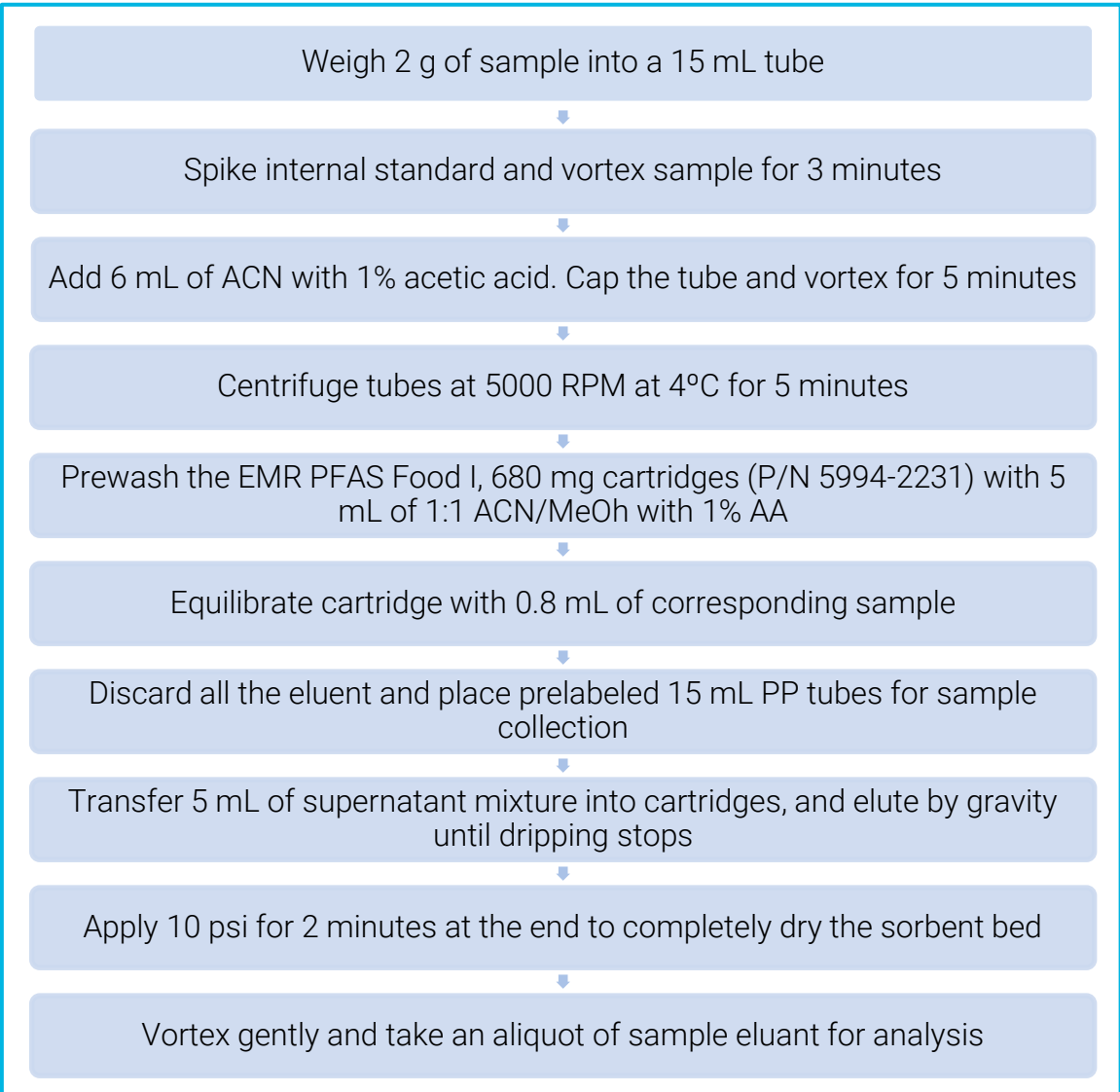


Figure 1. Sample preparation procedure chart for preparing alcoholic beverage samples.

LC/MS/MS chromatographic separation of target analytes and cholic acids



Figure 2. MRM chromatograms for all PFAS targets, EIS, and NIS compounds (top), and PFOS isomers and cholic acids interferences. (B)

Results and Discussion

Limit of Quantitation (LOQ), Quantitation Accuracy and Precision

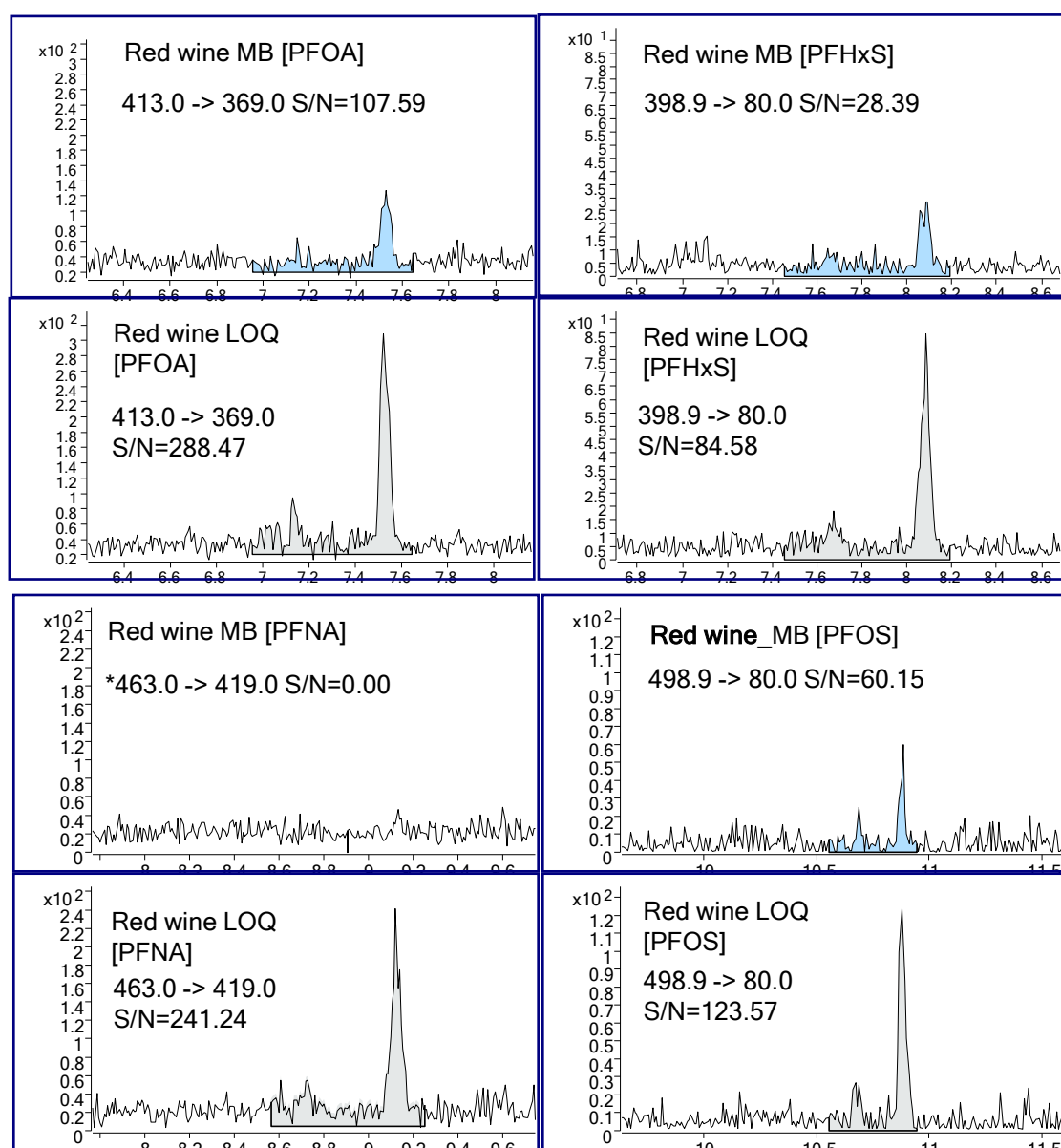


Figure 3. Four critical PFAS analytes chromatograms of red wine matrix blank and LOQ (0.01 ng/mL in red wine),

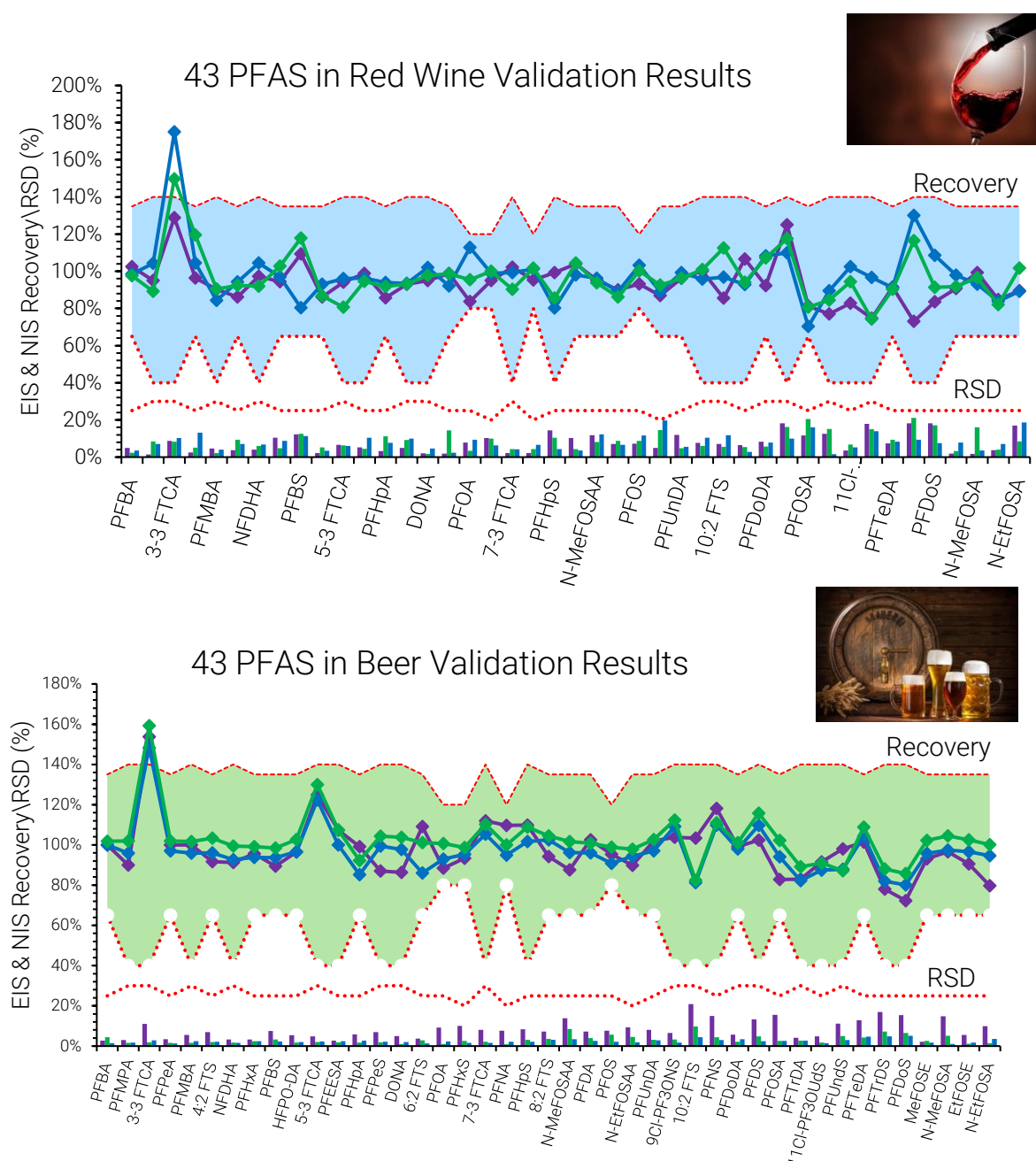


Figure 4. Method accuracy and precision for 43 PFAS analytes in red wine and beer. Three spiking levels.

Internal Standards (EIS and NIS) Recovery and Inter-batch Reproducibility

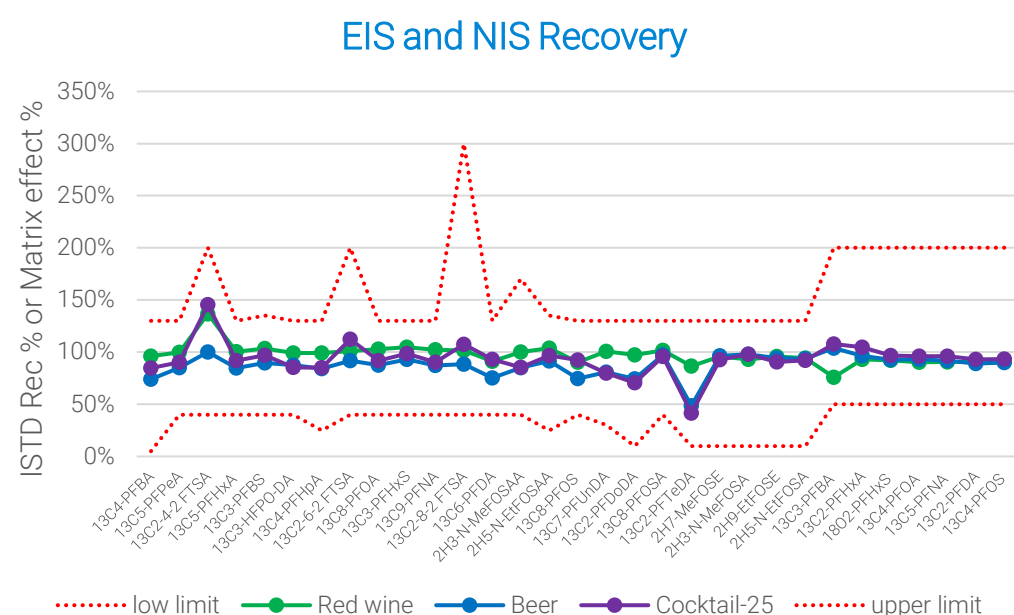


Figure 5. All EIS and NIS recoveries within acceptance criteria.

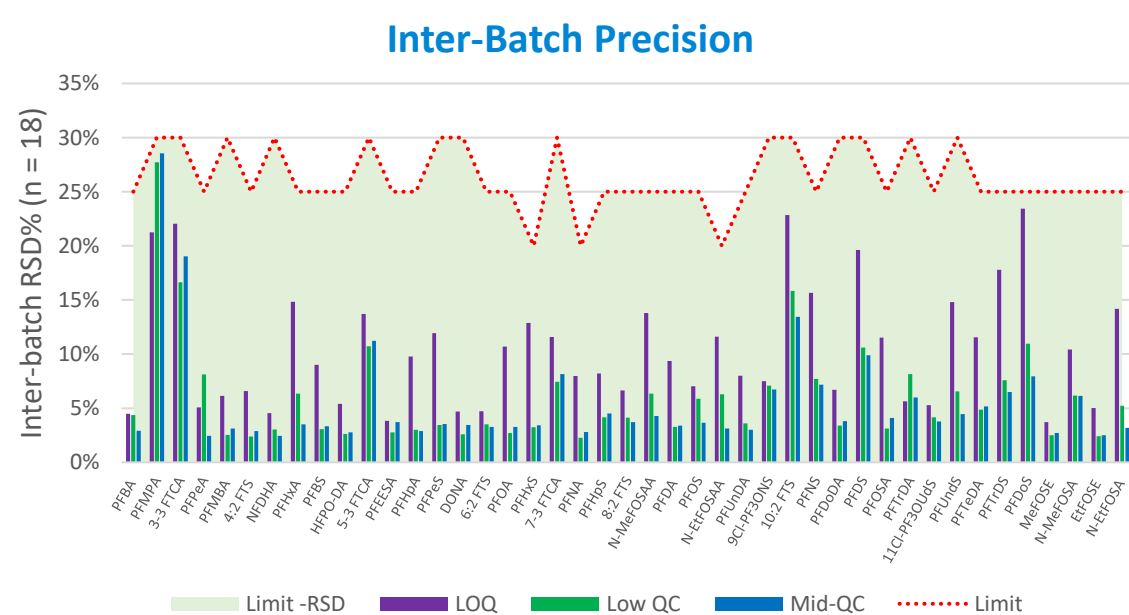


Figure 6. Inter-batch Reproducibility (RSD_R%).

Results and Discussion

Feed injection program improves chromatographic performance

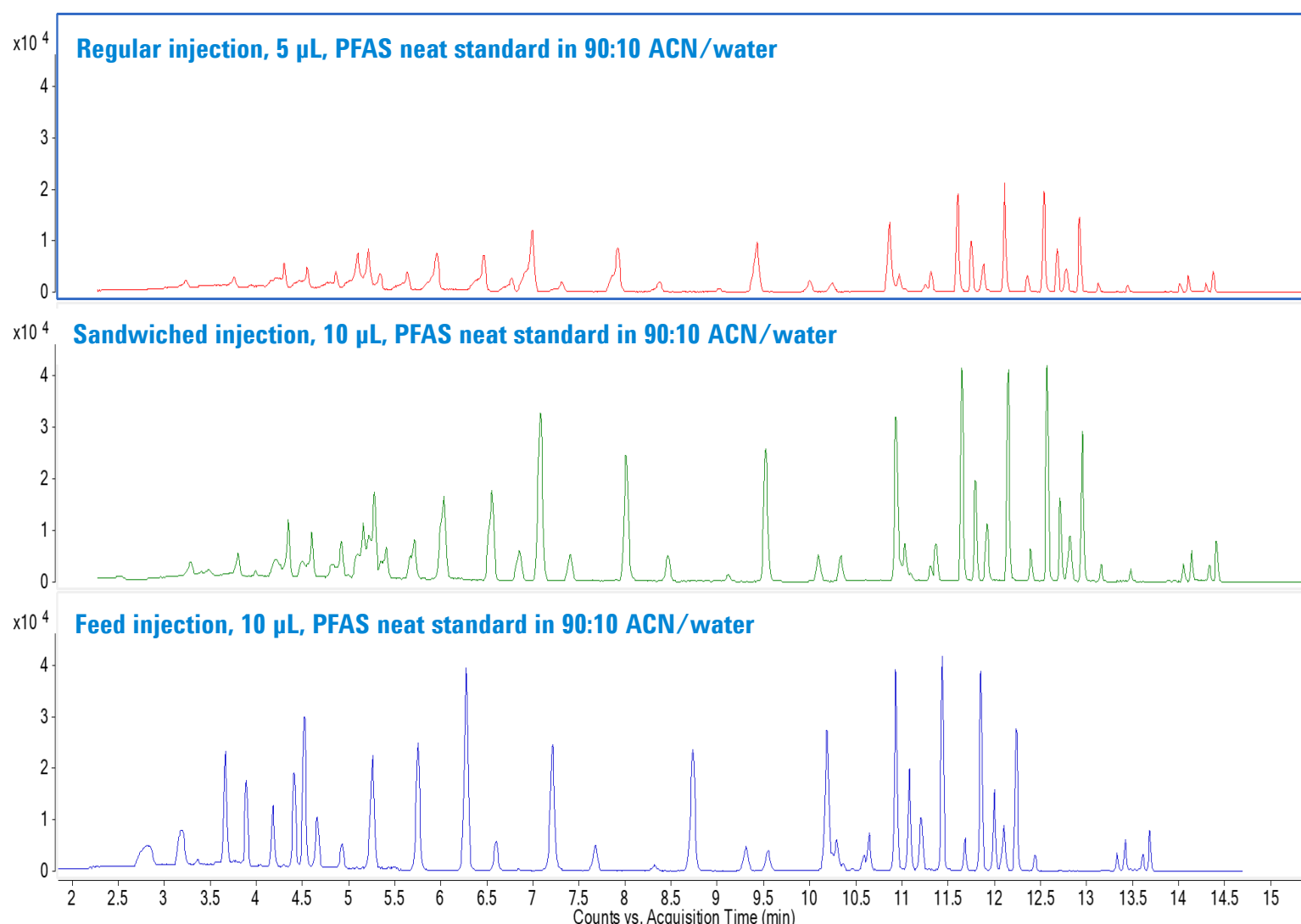


Figure 7. MRM chromatograms comparison with sandwiched injection program (A and B) versus feed injection program (C)

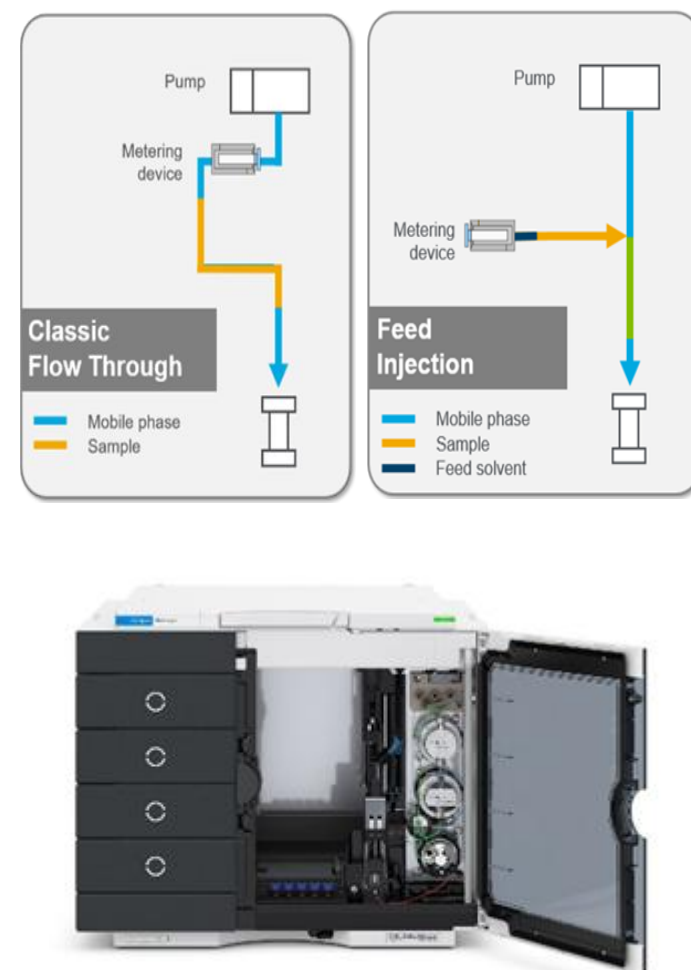


Figure 8. Schematic of flow through and feed injections, with Agilent 1290 Infinity III Multisampler

Conclusions

- A novel streamlined preparation method was developed for PFAS in alcoholic beverages.
- Recoveries and reproducibility consistent with AOAC SMPR performance criteria for PFAS in food.
- Processing time reduced by up to 50%.
- Hybrid Sampler feed injection program improved chromatographic integrity and peak shape consistency
- LC-MS/MS instrument method demonstrated excellent chromatography, sensitivity and selectivity.

Publications

- Determination of 43 PFAS in Beer and Wine. Using the Agilent Captiva EMR PFAS Food I passthrough cleanup and LC/MS/MS detection. Agilent app note [5994-8813EN](#),
[Captiva EMR PFAS Food cartridges](#)
[Sample Preparation for Food Analysis | Agilent](#)



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DE-014877

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Published in USA, June 30, 2026