Application Note

Food & Beverage Testing

Determination of 30 Per- and Polyfluoroalkyl Substances in Fruits, Vegetables, and Juices

Using the Agilent Captiva EMR PFAS Food I passthrough cleanup and LC/MS/MS detection

Authors

Limian Zhao and
Matthew Giardina
Agilent Technologies, Inc.

Abstract

This application note presents the development and validation of a multiresidue method for the analysis of per- and polyfluoroalkyl substances (PFAS) in fruits, vegetables, and juices. The method uses QuEChERS extraction, followed by Enhanced Matrix Removal (EMR) mixed-mode passthrough cleanup using the Agilent Captiva EMR PFAS Food I cartridge, then LC/MS/MS detection. The method features simplified and efficient sample preparation, sensitive LC/MS/MS detection, and reliable quantitation using neat standard calibration curves. The method was demonstrated to meet the required limits of quantitation (LOQs), recovery, and repeatability for four core PFAS targets—perfluorooctane sulfonic acid (PFOS), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), and perfluorohexane sulfonic acid (PFHxS)—and the remaining 26 PFAS targets in produce and juices.

Introduction

Determination of PFAS residues in food has become a topic of rising concern, gaining more attention over the last several years. In April 2023, the European Commission enforced regulations for four core PFAS compounds—PFOS, PFOA, PFNA, and PFHxS—in eggs, fish, seafood, meat, and offal.[1] In November 2023, the AOAC released the SMPR 2023.003 for the analysis of 30 PFAS in produce, beverages, dairy products, eggs, seafood, meat products, and feed.[2]

Agilent Captiva EMR PFAS Food cartridges were developed and optimized specifically for PFAS analysis in foods. Two types of cartridges (I and II) were designed to cover the large variety of food matrices. The objectives of this study were to develop and validate a complete workflow for the determination of 30 PFAS in fresh fruits and vegetables, and juices, which uses QuEChERS extraction followed by EMR mixed-mode passthrough cleanup with the Captiva EMR PFAS Food I cartridge and detection with the Agilent 6470B triple quadrupole LC/MS. Six representative food matrices were used in the study, including grape, lettuce, mushroom, carrot, tomato, and orange juice.

Experimental

Chemicals and reagents

Native and isotopically labeled PFAS certified standard solutions were purchased from Wellington Laboratories (Guelph, Ontario, Canada). Methanol (MeOH), acetonitrile (ACN), and isopropyl alcohol (IPA) were from VWR (Radnor, PA, U.S.). Acetic acid and ammonium acetate were procured from MilliporeSigma (Burlington, MA, U.S.).

Solutions and standards

Three native PFAS stock solutions were prepared by diluting the certified standards with MeOH. The final concentrations were 2, 20, and 200 ng/mL. However, for PFBA and PFPeA, the concentrations were adjusted to be 10 times and 5 times higher, respectively. They were also used for matrix prespiked quality control (QC) samples. The isotopically labeled PFAS solution at a concentration of 100 ng/mL was prepared by diluting the certified standard in MeOH and was used as an internal standard (ISTD). All standards were stored at 4 °C and used for no more than two weeks.

The native PFAS and ISTD spiking solutions were used for preparing neat calibration standards at 10, 20, 50, 100, 200, 500, 1,000, 2,000, and 5,000 ng/L for native PFAS targets and ISTD concentration of 1,000 ng/L in MeOH.

The ACN with 1% acetic acid (AA) extraction solvent was prepared by adding 10 mL glacial acetic acid into 990 mL of ACN and stored at room temperature. LC mobile phase A was 5 mM NH_4OAc in water, and mobile phase B was MeOH.

Equipment and materials

The study was performed using an Agilent 1290 Infinity II LC system consisting of a 1290 Infinity II high-speed pump (G7120A), a 1290 Infinity II multisampler (G7167B), and a 1290 Infinity II multicolumn thermostat (G7116A). The LC system was coupled to an Agilent 6470B LC/TQ system. Data were acquired using MassHunter Workstation software version 10.1. For data analysis, MassHunter Quantitative Analysis software version 10.0 was used.

Other equipment used for sample preparation included:

- Centra CL3R centrifuge (Thermo IEC, MA, U.S.)
- Geno/Grinder (Metuchen, NJ, U.S.)
- Multi Reax test tube shaker (Heidolph, Schwabach, Germany)
- Pipettes and repeater (Eppendorf, NY, U.S.)
- Agilent positive pressure manifold 48 processor (PPM-48; part number 5191-4101)
- CentriVap and CentriVap Cold Trap (Labconco, MO, U.S.)
- Ultrasonic cleaning bath (VWR, PA, U.S.).

The 1290 Infinity II LC system was modified using an Agilent InfinityLab PFC-free HPLC conversion kit (part number 5004-0006), including an Agilent InfinityLab PFC delay column, 4.6 × 30 mm (part number 5062-8100). Chromatographic separation was performed using an Agilent ZORBAX RRHD Eclipse Plus C18 column, 95 Å, 2.1 × 100 mm, 1.8 µm (part number 959758-902) and an Agilent ZORBAX RRHD Eclipse Plus C18 column, 2.1 mm, 1.8 µm, 1,200-bar pressure limit, UHPLC guard (part number 821725-901).

Other Agilent consumables used included:

- Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (part number 5982-5650CH)
- Captiva EMR PFAS Food I cartridges, 6 mL cartridges, 340 mg (part number 5610-2230)
- Polypropylene (PP) snap caps and vials, 1 mL (part numbers 5182-0567 and 5182-0542)
- PP screw cap style vials and caps, 2 mL (part numbers 5191-8150 and 5191-8151)
- Tubes and caps, 50 mL, 50/pk (part number 5610-2049)
- Tubes and caps, 15 mL, 100/pk (part number 5610-2039)

All the consumables used in the study were tested and verified for acceptable PFAS cleanliness.

LC/MS/MS instrument conditions

The LC binary pump conditions are listed in Table 1, and the multisampler program is listed in Table 2. The column temperature was set at 55 ± 0.8 °C. Mass spectrometer data were acquired in negative ion mode with a constant fragmentor setting of 166 V. The ESI source settings were drying gas at 150 °C, 18 L/min; sheath gas at 390 °C, 12 L/min; nebulizer gas at 15 psi; capillary voltage at 2,500 V; and nozzle voltage at 0 V. The 6470B LC/TQ dMRM acquisition settings are listed in Table 3.

Table 1. LC pump conditions for LC/MS/MS.

Parameter	Setting			
Mobile phase A	5 mM NH_4OAc in water			
Mobile phase B	MeOH			
Gradient	Time (min)	A%	B%	Flow (mL/min)
	0.00	98.00	2.00	0.400
	2.00	98.00	2.00	0.400
	2.50	45.00	55.00	0.400
	6.50	30.00	70.00	0.400
	8.00	20.00	80.00	0.460
	14.20	0.00	100.00	0.460
	17.00	0.00	100.00	0.400
	17.10	98.00	2.00	–
Post-time	3.0 min			

Table 2. LC multisampler program for LC/MS/MS.

Parameter	Setting				
Injection Program	– Draw 10.00 µL of water – Draw 20.00 µL of sample – Wash needle – Draw 50.00 µL of water – Mix 10.00 µL from air five times – Inject				
Multiwash	Step	Solvent	Time (s)	Seat Backflush	Needle Wash
	1	IPA	10	Enabled	Enabled
	2	ACN	10	Enabled	Enabled
	3	Water	10	Enabled	Enabled
	Start cond.	Water	NA	Enabled	Enabled

Table 3. LC/MS/MS acquisition settings.

Compound	Retention Time (min)	Precursor Ion (*m/z*)	Product Ion (*m/z*)	Fragmentor (V)	Collision Energy (V)	Collision Cell Accelerator Voltage (V)
PFBA	4.8	213	169	72	8	2
PFPeA	5.3	263	219	72	4	2
PFHxA	5.9	313	269 119	72	8 24	2
PFHpA	6.7	363	319 169	72	8 16	2
PFOA	7.6	413	369 219	72	8 16	2
PFNA	8.5	463	419 219 169	72	8 16 20	2
PFDA	9.3	513	469 269 219	72	12 16 20	2
PFUnDA	9.9	563	519 319 269	100	12 20 20	2
PFDoDA	10.3	613	569 319 269	100	8 20 24	2
PFTrDA	10.8	663	619 319 169	100	12 20 32	2
PFTeDA	11.2	713	669 219 169	100	12 28 32	2
PFBS	5.4	299	99 80	154	34 36	2
PFPeS	6.0	349	99 80	144	40 44	2
PFHxS	6.8	399	99 80	156	40 56	2
PFHpS	7.6	449	99 80	148	42 50	2
PFOS	8.5	499	99 80	148	50 54	2
PFNS	9.3	549	99 80	148	52 56	2
PFDS	9.9	599	99 80	148	56 60	2
PFUnDS	10.3	649	99 80	132	56 76	2
PFDoS	10.7	699	99 80	156	62 67	2
PFTrDS	11.1	749	99 80	185	64 80	4
PFOSA	10.0	498	169 78 48	150	36 36 110	3
9Cl-PF3ONS	9.0	531	351 83	150	28 32	3
11Cl-PF3OUdS	10.1	631	451 83	150	36 32	2
HFPO-DA	6.1	285	185 169 119	50	20 4 32	5
DONA	6.8	377	251 85	50	8 32	5
4:2 FTS	5.9	327	307 81 80	150	20 36 42	2
6:2 FTS	7.5	427	407 81 80	150	30 32 58	2
8:2 FTS	9.3	527	507 81 80	200	30 46 50	4
10:2 FTS	10.4	627	607 81 80	208	34 42 54	4

Table 3. LC/MS/MS acquisition settings (continued from previous page).

Compound	Retention Time (min)	Precursor Ion (*m/z*)	Product Ion (*m/z*)	Fragmentor (V)	Collision Energy (V)	Collision Cell Accelerator Voltage (V)
$^{13}C_2$-4:2 FTS	5.87	329	309	150	24	2
$^{13}C_2$-6:2 FTS	7.55	429	409	150	28	2
$^{13}C_2$-8:2 FTS	9.29	529	509	200	28	4
$^{13}C_2$-PFDoA	10.35	615	570	90	12	2
$^{13}C_2$-PFTeDA	11.17	715	670	90	12	2
$^{13}C_3$-HFPO-DA	6.15	287	169	64	4	5
$^{13}C_3$-PFBS	5.39	302	80	130	44	2
$^{13}C_3$-PFHxS	6.76	402	80	156	48	2
$^{13}C_4$-PFBA	4.78	217	172	72	8	2
$^{13}C_4$-PFHpA	6.72	367	322	72	8	2
$^{13}C_5$-PFHxA	5.93	318	273	72	8	2
$^{13}C_5$-PFPeA	5.29	268	223	72	4	2
$^{13}C_6$-PFDA	9.3	519	474	72	8	2
$^{13}C_7$-PFUnDA	9.88	570	525	100	8	2
$^{13}C_8$-PFOS	7.6	421	376	72	8	2
$^{13}C_8$-PFOA	8.52	507	80	148	54	2
$^{13}C_8$-PFOSA	10	506	78	150	36	3
$^{13}C_9$-PFNA	8.51	472	427	72	8	2
TUDCA	6.8	498	124 80	146 280	53 80	4
TCDCA	8.6	498	124 80	114 280	65 80	4
TDCA	9.0	498	124 80	146 280	69 80	4

Sample preparation

Organic fruits and vegetables, and orange juice (with pulp) were purchased from local grocery stores. The fruits and vegetables were washed by water and cut into small pieces, then frozen at –20 °C at least overnight. The frozen pieces were blended into fine powder using a mechanical blender. This fine powder was either used for direct sample preparation or stored short term at –20 °C. Orange juice was sampled directly, without processing.

Both produce and juice sample preparation used a 10 g sample for extraction. The native PFAS and ISTD spiking solutions were added to the QC samples appropriately, and only the ISTD spiking solution was added to the matrix blanks. The samples were vortexed for 10 to 15 seconds after spiking. The samples were then ready for the preparation procedure, which is described in Figure 1.

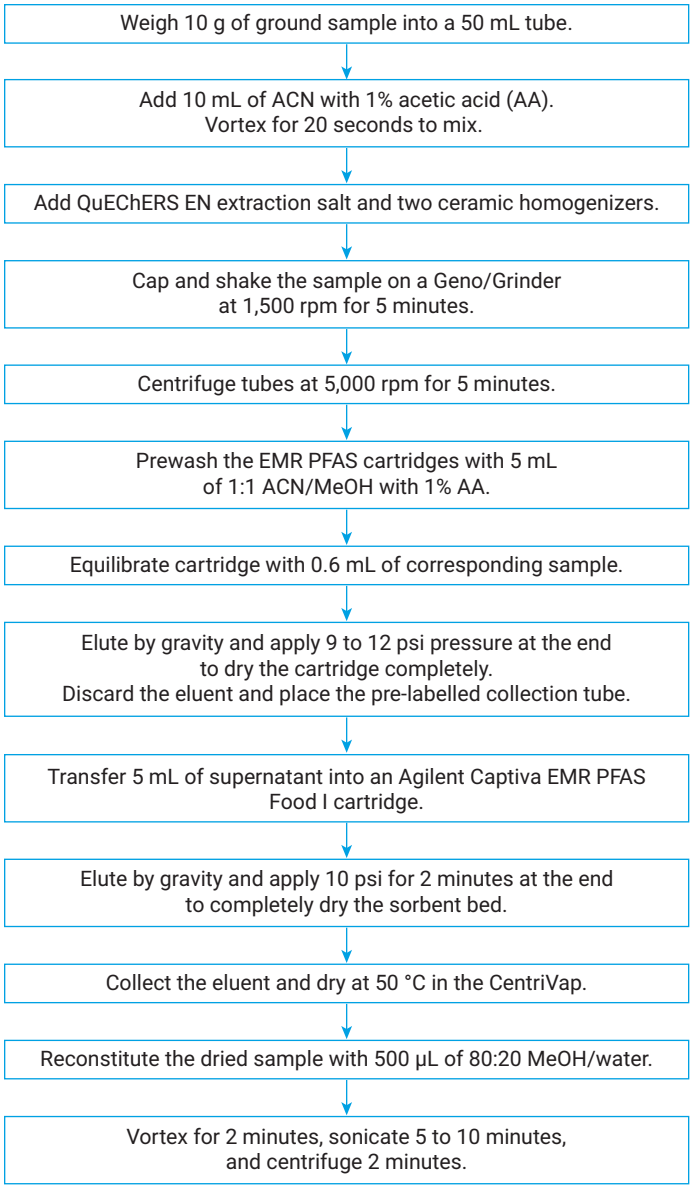


Figure 1. Sample preparation procedure for PFAS analysis in fruits, vegetables, and juice.

Method validation

The method was validated based on the evaluation of a calibration study, method LOQ determination, and recovery accuracy and precision. Due to the different requirements of the target LOQs and the ultralow LOQ requirements for PFOA, PFOS, PFNA, and PFHxS in produce[1], seven prespiked QC-level samples were prepared in replicates of four to five at each level. In addition, the matrix blanks were prepared in replicates of five to seven for quantitation of the targets in the matrix control sample. This is important for accuracy evaluation, as the contribution from the matrix for some PFAS is unavoidable. The PFAS spiking levels for prespiked QC samples were 0.001, 0.002, 0.004, 0.01, 0.02, 0.1, and 0.2 µg/kg for 28 PFAS with 10x for PFBA concentration and 5x for PFPeA concentration in baby food. The ISTD spiking level in all of the prespiked QC samples and matrix blanks was 0.1 µg/kg.

Results and discussion

EMR mixed-mode passthrough cleanup

The Captiva EMR PFAS Food cartridges provide comprehensive matrix removal after traditional QuEChERS extraction through a mixed-mode passthrough cleanup. Passthrough cleanup is a simplified yet efficient procedure to remove matrix interferences including carbohydrates, organic acids, pigments, fats and lipids, and other hydrophobic and hydrophilic matrix co-extractives, while allowing targets to flow through. The Captiva EMR PFAS Food I cartridges contain less sorbent with a simpler formula and are recommended for fresh and processed fresh foods of plant origin, such as fruits and vegetables, baby food, and juices. The Captiva EMR PFAS Food II cartridges contain more sorbent with a more complex formulation and are recommended for fresh and processed foods of animal origin, such as milk, eggs, meat, fish, and infant formula; dry seed feed and food of plant origin; and oils.

Fruits and vegetables are considered to be less complex food matrices, where sample preparation based on QuEChERS extraction followed with dispersive SPE (dSPE) cleanup has been used widely for pesticide testing of many fruits and vegetables. The dSPE cleanup was used in PFAS analysis in food matrices; however, it resulted in the loss of many PFAS.[3] This was confirmed in our study of PFAS recovery in grape extract recovery using different cleanup methods. Figure 2 shows the comparison of recovery for PFAS in grape extract using different cleanup methods. The EMR mixed-mode passthrough cleanup provided the best PFAS target recovery in the range of 89 to 114% with RSD of 5%, while dSPE cleanup generated lower recoveries for many PFAS targets, resulting in the wide range of 47 to 105% with RSD of 20% when using dSPE 1 cleanup, and 63 to 109% recovery and 14% RSD when using dSPE 2 cleanup.

EMR mixed-mode passthrough cleanup also demonstrated the efficient matrix removal for produce matrices. For plant-origin fresh produce, matrix pigment can be significant and thus requires efficient pigment removal. Figure 3 demonstrates high efficiency of produce sample matrix pigment removal provided by EMR mixed-mode passthrough cleanup using Captiva EMR PFAS Food I cartridges.

Besides the improvement of PFAS target recovery and matrix removal, another important feature provided by EMR mixed-mode passthrough cleanup is the higher sample volume recovery. Sample volume recovery usually is critical for PFAS analysis in food, since the required LOQs are in the low- to mid-ppt level, requiring the use of a postconcentration step to boost the method sensitivity. Comparing to the ~ 50% loss on sample volume when using traditional dSPE cleanup, the EMR passthrough cleanup provides > 90% volume recovery, which allows easy postconcentration and consistent sample reconstitution.

Entire method validation

The new method was validated for determination of 30 PFAS targets in five representative fruits and vegetables, and orange juice, by following the AOAC SMPR guidance. The method needed to meet the requirements for PFAS target LOQs, which were ≤ 0.01 µg/kg for the core PFAS targets; ≤ 1 µg/kg for PFBA and PFPeA; and ≤ 0.1 µg/kg for the remaining PFAS targets.

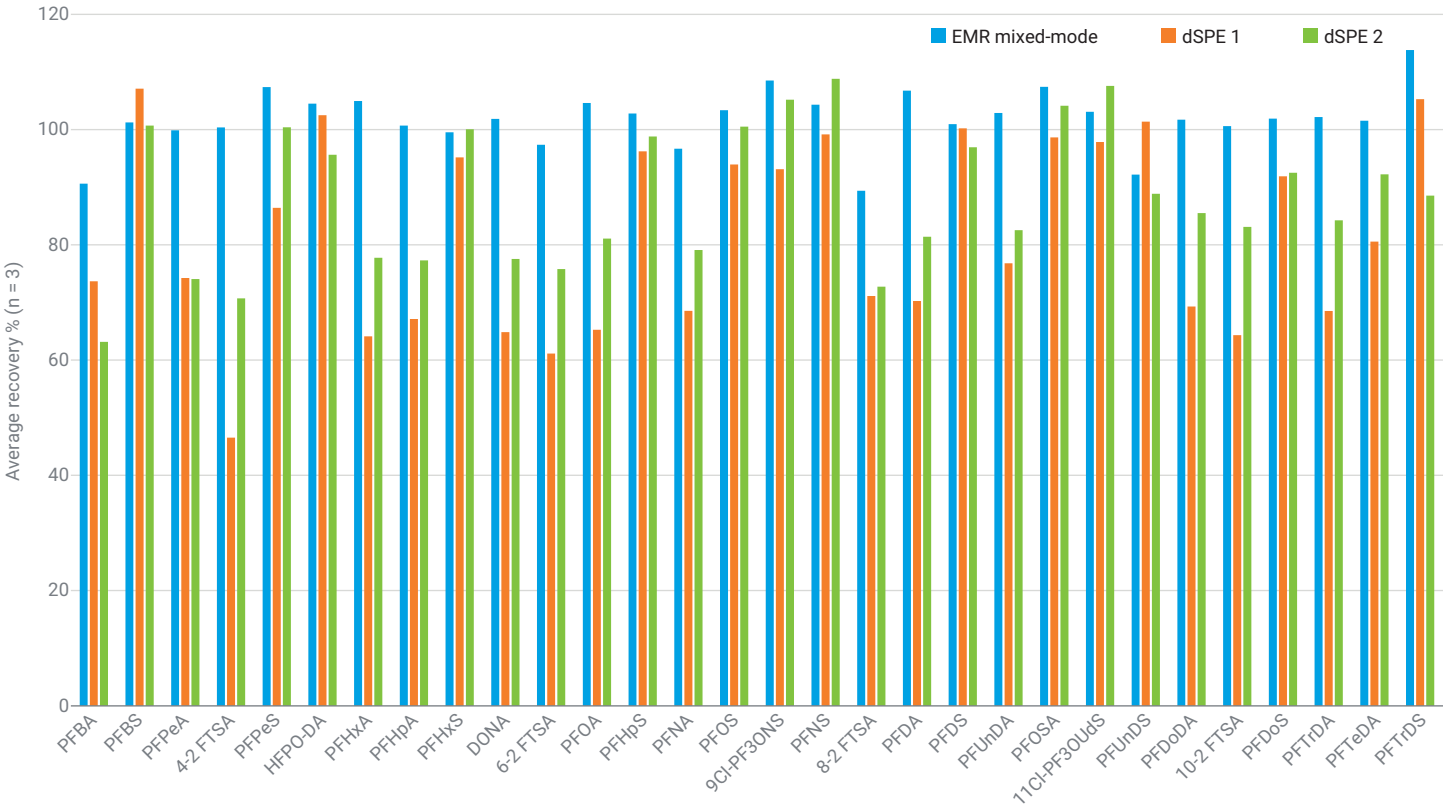
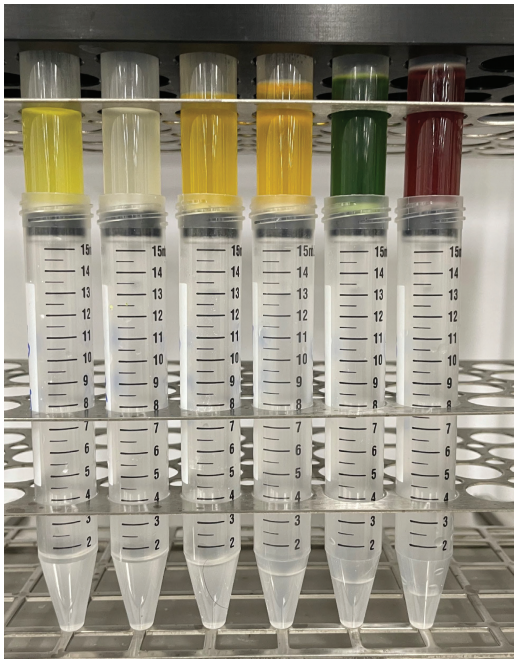
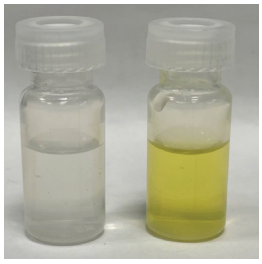


Figure 2. PFAS recovery in grape extract after sample cleanup procedures using either mixed-mode passthrough cleanup with Agilent Captiva EMR PFAS Food I cartridges or traditional dSPE cleanup.

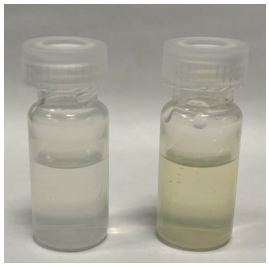
A Sample crude extract passthrough cleanup



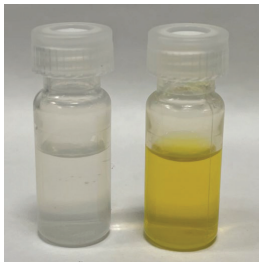
B Sample extract appearance, with (left) versus without (right) EMR mixed-mode passthrough cleanup



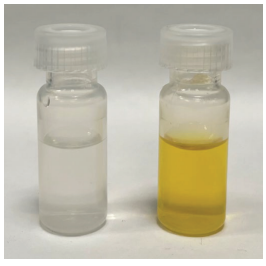
Orange juice



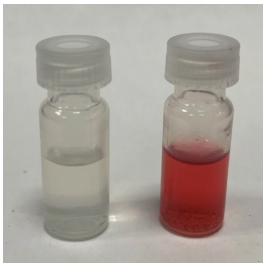
Mushroom



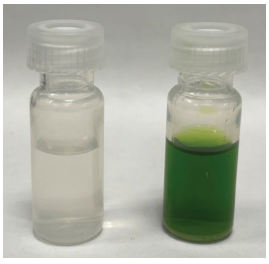
Carrot



Tomato



Grape



Lettuce

Figure 3. Fruits, vegetables, and juice extract passthrough cleanup on Captiva EMR PFAS Food I cartridges (A), and sample extract appearance with (left) versus without (right) EMR mixed-mode passthrough cleanup in six matrices (B).

Method LOQs and validation levels

The food matrices evaluated in this study all showed positive detection for few PFAS targets in matrix blanks. Matrix background correction was used for method validation for target recovery. Matrix blanks were prepared in five to seven replicates. The lowest method reportable LOQs were calculated based on the matrix blanks detection according to the following equation:

$LOQ_{lowest} = 10 \times SD_{MBs}$

Where:

- LOQ_{lowest} is the method's lowest reportable limit of quantitation
- SD_{MBs} is the standard deviation of detected targets from five to seven replicates of matrix blanks

The method LOQs were then decided based on the lowest validated QC spiking level that was equal to or above the lowest reportable LOQs. Table 4 shows the calculated lowest reportable LOQs (LOQ_{cal}) and validated method LOQs (LOQ_{val}) for each target in six replicates.

For the core PFAS targets, the validated method LOQs were demonstrated to be below or equal to the required LOQs in six matrices, with exceptions for PFNA in mushroom and PFOS in carrot. The higher validated LOQ of PFNA in mushroom (0.1 µg/kg) and PFOS in carrot (0.02 µg/kg) were due to the significant high positive occurrence in these two food matrices. For core PFAS targets, the LOQs below 0.01 µg/kg were also investigated. Overall, 50% of core PFAS targets in the tested produce matrices were validated with LOQs below or equal to 0.004 µg/kg in sample. The main reason for the rest of 50% core PFAS targets not being validated for LOQs below 0.01 µg/kg level was the positive occurrence of PFAS targets in sample matrix blank. LC/MS/MS instrument sensitivity also contributed slightly. Comparing to the validated method LOQs for core PFAS targets in baby food using the Agilent 6495D LC/TQ [5], less core PFAS targets with < 0.01 µg/kg LOQs in produce were validated on an Agilent 6470B LC/TQ when matrix background did not impact the LOQ determination. This confirms that the higher instrument sensitivity provided by the 6495D LC/TQ can support method validation at lower LOQ levels. For other PFAS targets, they all meet the required LOQs, except 4:2 FTS and 6:2 FTS in carrot, where method LOQs had to be lifted higher due to significant positive occurrence from carrot. Figure 4 shows the chromatograms of matrix blanks and validated method LOQs for the core targets in tomato, which were all below 0.01 µg/kg.

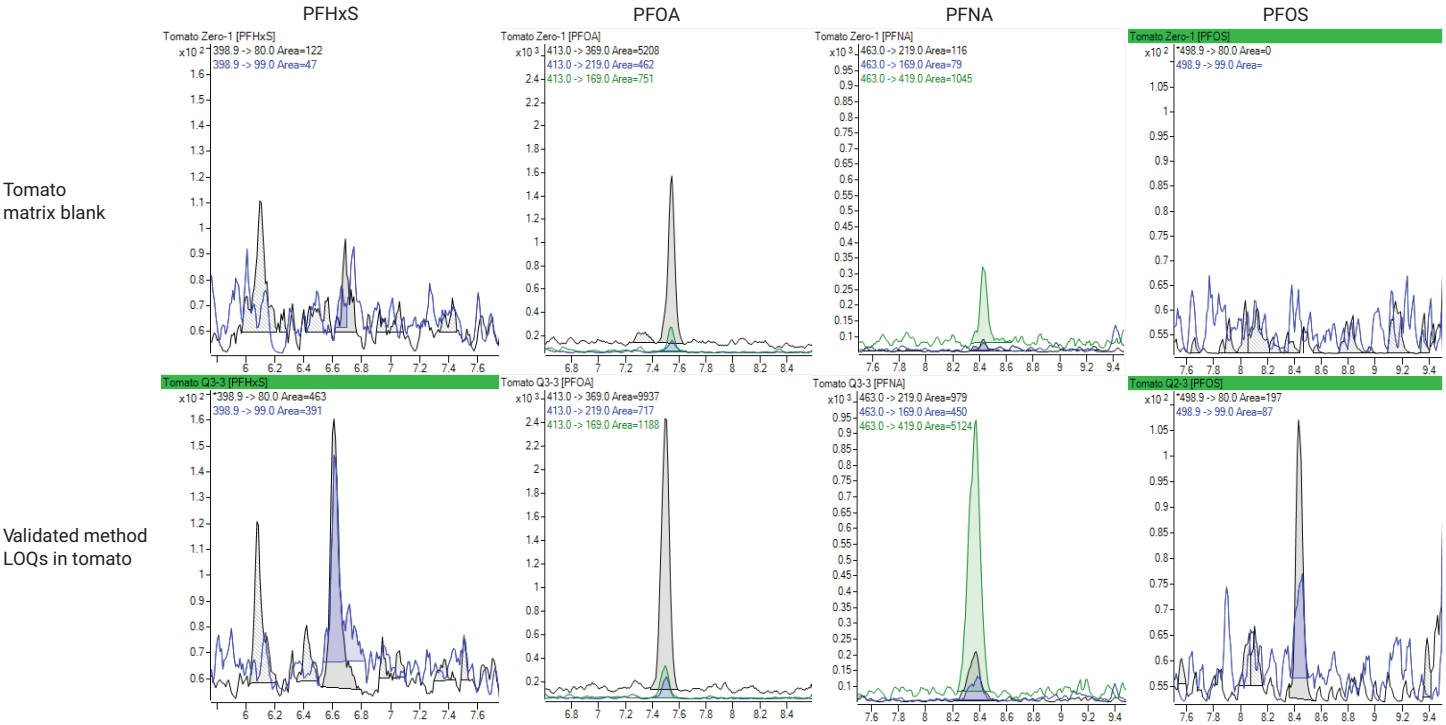


Figure 4. Tomato matrix blanks (top) and validated LOQ chromatograms (bottom) for the core PFAS targets, including PFHxS (0.004 µg/kg), PFOA (0.004 µg/kg), PFNA (0.004 µg/kg), and PFOS (0.002 µg/kg).

Table 4. Method lowest reportable LOQs (LOQ_{cal}) and validated LOQs (LOQ_{val}) for 30 targets in fresh produces and juice.

Target	Method LOQs in Fresh Produce and Juice (µg/kg)											
	Lettuce		Mushroom		Carrot		Grape		Orange Juice		Tomato	
	LOQ_{cal}	LOQ_{val}	LOQ_{cal}	LOQ_{val}	LOQ_{cal}	LOQ_{val}	LOQ_{cal}	LOQ_{val}	LOQ_{cal}	LOQ_{val}	LOQ_{cal}	LOQ_{val}
PFBA	0.142	0.2	0.143	0.2	0.138	0.2	NA	0.1	NA	0.2	0.273	1
PFPeA	NA	0.01	0.003	0.01	0.007	0.01	0.005	0.01	0.005	0.01	0.001	0.01
PFBS	0.001	0.01	0.004	0.01	0.008	0.01	0.006	0.01	0.002	0.01	0.005	0.01
4:2 FTS	0.038	0.1	NA	0.01	0.195	0.2	0.017	0.1	0.003	0.01	0.042	0.1
PFPeS	NA	0.01	0.004	0.01	NA	0.01	0.009	0.01	0.012	0.02	NA	0.01
PFHxA	NA	0.02	0.004	0.01	NA	0.02	NA	0.01	NA	0.01	NA	0.01
HFPO-DA	NA	0.1	NA	0.01	NA	0.01	NA	0.01	NA	0.01	NA	0.01
PFHpA	0.001	0.01	0.003	0.01	NA	0.01	0.004	0.01	0.003	0.01	0.002	0.01
PFHxS*	0.002	0.01	0.002	0.004	NA	0.01	0.003	0.01	NA	0.01	0.003	0.004
DONA	NA	0.01	NA	0.01	NA	0.1	NA	0.01	NA	0.01	NA	0.01
6:2 FTS	0.099	0.1	0.01	0.01	0.104	0.2	NA	0.01	0.013	0.02	NA	0.01
PFOA*	0.003	0.01	0.01	0.01	0.009	0.01	0.002	0.002	0.004	0.01	0.002	0.004
PFHpS	NA	0.01	NA	0.01	NA	0.01	NA	0.01	NA	0.01	NA	0.01
PFNA*	0.002	0.002	0.018	0.1	0.004	0.004	0.001	0.001	0.003	0.004	0.003	0.004
PFOS*	0.001	0.002	NA	0.002	0.015	0.02	0.001	0.002	0.001	0.004	NA	0.002
9Cl-PF3ONS	NA	0.01	NA	0.01	NA	0.01	0.001	0.01	NA	0.01	NA	0.01
8:2 FTS	NA	0.01	NA	0.01	0.002	0.01	NA	0.01	NA	0.01	NA	0.01
PFNS	0.001	0.01	NA	0.01	NA	0.01	0.001	0.01	NA	0.01	0.003	0.01
PFDA	NA	0.01	0.011	0.1	0.001	0.01	0.001	0.01	0.002	0.01	NA	0.01
PFDS	0.01	0.01	0.004	0.01	0.003	0.01	NA	0.01	0.000	0.01	NA	0.01
PFUnDA	0.002	0.01	0.012	0.1	0.003	0.01	0.001	0.01	0.002	0.01	NA	0.01
PFOSA	NA	0.01	0.1	0.01	0.003	0.02	0.002	0.01	NA	0.02	0.003	0.01
11Cl-PF3OUdS	NA	0.01	NA	0.01	NA	0.01	NA	0.01	0.008	0.02	NA	0.01
PFUnDS	0.004	0.01	NA	0.01	NA	0.02	0.001	0.01	NA	0.01	NA	0.01
PFDoDA	0.001	0.01	0.006	0.01	0.004	0.01	0.001	0.01	0.001	0.01	0.003	0.01
10:2 FTS	0.001	0.01	NA	0.01	0.001	0.01	0.001	0.01	NA	0.01	NA	0.01
PFDoS	NA	0.01	NA	0.01	NA	0.02	NA	0.01	NA	0.01	NA	0.01
PFTrDA	NA	0.01	0.004	0.01	0.001	0.01	NA	0.01	0.001	0.01	0.001	0.01
PFTrDS	NA	0.01	NA	0.01	NA	0.01	NA	0.01	0.002	0.01	NA	0.01
PFTeDA	0.001	0.01	0.003	0.01	0.002	0.01	0.001	0.01	0.001	0.01	0.001	0.01

* Core PFAS targets

Results marked in red indicate the method LOQ_{val} was higher than the AOAC SMPR requirement.

Method calibration

The use of 18 PFAS isotopically labeled ISTDs allows the same standard calibration curve to be used for PFAS quantitation in different food matrix samples. Therefore, a matrix-matched calibration curve is not needed for each food matrix. This significantly increases sample testing productivity, saving time and costs, and improving sample analysis consistency.

The calibration curve range was decided based on the required LOQs in the food matrices, the concentration factor introduced through sample preparation, and the instrument method sensitivity. Due to the lower LOQ levels required in produce, a calibration set range from 10 to 5,000 ng/L was used. The results confirmed a 500x calibration curve dynamic range with correlation coefficient $R^2 > 0.99$ for all 30 PFAS targets.

Method accuracy and precision

Method recovery and repeatability were validated. The acceptance criteria for produce are 65 to 135% recovery and ≤ 25% RSD for PFAS targets with corresponding isotopic ISTD, and 40 to 140% recovery and ≤ 30% for PFAS targets without corresponding isotopic ISTD.[2] The three levels of prespiked QCs were reported for method validation, including LOQ, and mid and high levels. There were several exceptions: PFBA and 4:2 FTS in tomato, 4:2 FTS, PFHxA, 6:2 FTS and PFOSA in lettuce, 4:2 FTS, DONA and 6:2 FTS in carrot, and 4:2 FTS in grape, where only two or one levels were reportable due to significantly high positive occurrence in sample matrix control.

Figure 5 shows the method validation recovery and repeatability (RSD) summary for PFAS analysis in six tested produce and juice matrices. Overall, the method delivered acceptable recovery and repeatability results for PFAS targets in food matrices that meet the acceptance criteria. Targets with corresponding isotopically labeled ISTD generated better quantitation results than targets without corresponding isotopically labeled ISTD, and all the outliers were from targets without corresponding isotopically labeled ISTDs, especially for 10:2 FTS, which generated significant higher unacceptable recovery in grape, lettuce, and mushroom due to matrix enhancement effect.

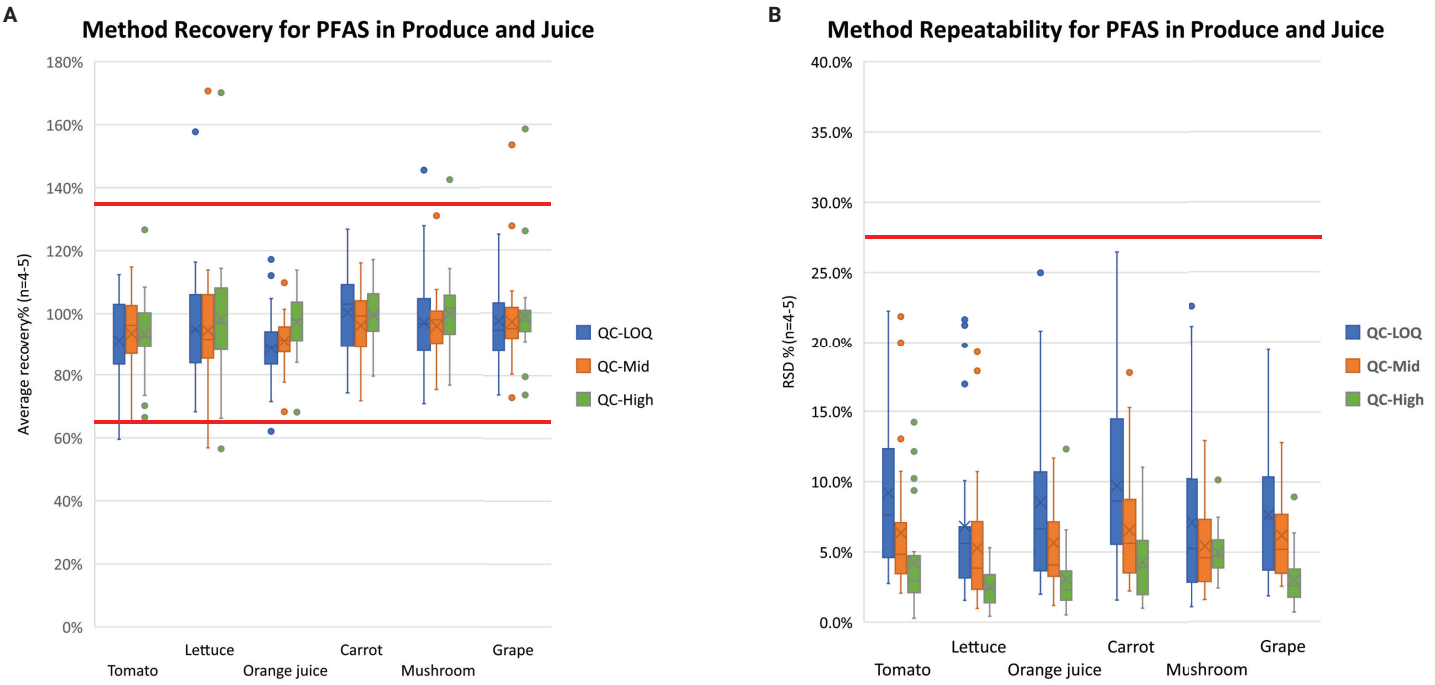


Figure 5. Method recovery (A) and repeatability (B) results summary for 30 PFAS in produce and juice.

Conclusion

A simplified, rapid, and reliable method using QuEChERS extraction followed by EMR mixed-mode passthrough cleanup with the Agilent Captiva EMR PFAS Food I cartridge and LC/MS/MS detection was developed and validated for 30 PFAS targets in six produce and juice matrices. The novel cleanup method demonstrated significant improvement in terms of matrix removal, PFAS recovery, and sample volume recovery over the traditional dSPE cleanup. It also features as a simplified sample cleanup method, saving time and effort, and thus improves overall lab productivity. The entire method was validated with acceptance criteria, and method performance was shown to meet the requirements described in AOAC SMPR 2023.003.

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