

Pizza

Food and Food Packaging

Perfluoroalkyl Substances (PFAS) Testing Guide



Food and Food Packaging

It was recognized fairly early that PFAS compounds used in food packaging materials (such as pizza boxes and microwave popcorn bags) could migrate into consumable food products and contribute to increased PFAS body burden. In addition, as PFAS contamination has continued to spread throughout the environment it has been more recently recognized that these materials can also enter the human food supply chain through animal consumption of PFAS contaminated water and feed, thereby further increasing our PFAS body burden. Regardless of source, the analysis of PFAS in food and food packaging materials – and their myriad complex matrices - features additional difficult analytical challenges.



1. New Concerns about PFAS in Food

The Convergence of Environmental Contamination and Food Safety

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Abstract

Per- and Polyfluorinated Alkyl Substances (PFAS) are well known environmental contaminants that have a newly recognized potential to taint certain food products through agricultural consumption via environmental transport from contaminated industrial sites [1]. The analysis of PFAS in food products requires more extensive analytical preparation techniques, compared to PFAS testing of simple matrices such as drinking water, in order to reduce the impact of sample matrix interferences on the subsequent instrumental analysis. An example is provided of a PFAS method applicable to milk, butter, cheese and fish.

The Prequel

Per- and Polyfluorinated Alkyl Substances (PFAS) are an extensive family of synthetic, fluorochemicals with a unique set of physical and chemical properties. These properties have resulted in their widespread commercial use over the past 50 years in diverse applications ranging from fire fighting foams to stain resistant carpet to grease-proof pizza boxes. However, these same unique physical and chemical properties also have been found to bear serious environmental consequences: widespread dispersion ability, extreme environmental persistence and a high degree of bioaccumulation [2]. Although PFAS do not exhibit acute toxic properties, researchers have found that PFAS can demonstrate a large number of subtle, chronic health effects, primarily affecting the endocrine and reproductive systems. Consequently, health experts have long been concerned that low-level, cumulative exposure to PFAS over an extended period of time could have serious health consequences [3]. Therefore, chronic lifetime PFAS exposure pathways - such as through food or drinking water - are of particular concern to regulators and are receiving enhanced scrutiny.

Initial Concerns

In the US, the initial US Food and Drug Administration (FDA) concern about PFAS centered about the contamination of food products through contact with PFAS containing food packaging (and to a lesser extent with food processing equipment). The classic examples are those PFAS coated pizza boxes, fast-food hamburger wrappers and microwave popcorn bags that have done such a marvelous job of keeping grease off our clothes. That problem was summarily solved in late 2016 when FDA removed the approval for the use of PFAS in food packaging [4].

Likewise, the primary US Environmental Protection Agency (EPA) focus has been on drinking water as a primary source of lifetime PFAS exposure. EPA is continuing to conduct extensive nationwide testing for PFAS in drinking water under the Unregulated Contaminant Monitoring Rule (UCMR) program [5]. These efforts will very likely result in specific regulatory limits for the allowable concentration of certain PFAS in drinking water.

Concurrently, other government agencies, such as the US Department of Defense (DOD) have been extensively studying the widespread environmental contamination of military facilities owing to the extensive historical use of PFAS firefighting foams, principally at air bases [6].

Convergence

Initially, these three individual trains of concern seemed to be running on separate tracks. It was only more recently that they were seen to be converging toward a much larger, more complex problem requiring multimedia, multi agency examination and the use of more sophisticated analytical tools. The simplified pathway model shown in **Figure 1** illustrates the general scope of the problem. By the end of 2019, the FDA was fully on board with concerns about PFAS entering the general food supply through environmental sources, potentially leading to the contamination of dairy products, bottled water, seafood and other consumables [7].

Analytical Implications

This expanded concept of the PFAS problem is clearly a major step forward, but it has presented some analytical challenges. Much of the official PFAS methodology developed over the past decade has been focused on the analysis of drinking water and aimed at a very limited list of analytes. With little challenge from matrix interference, easily surmountable chromatography issues and straight forward mass spectrometry, these official drinking-water-only methods proved to be inadequate when applied to the analysis of PFAS in soil, sediment, sludge and wastewater. When applied to the analysis of foods - with a myriad of complex matrices, they are quite ineffective, resulting in a surge in PFAS analytical method development centered about complex matrices, with food testing occupying a prominent position. The following section features one such application as an illustration of the approaches now being pursued in pursuit of the expanded PFAS challenge.

Analysis of PFAS in Dairy Products, Eggs, and Fish by LC-MS/MS

Method Introduction

The following work was performed through a collaboration between Weck Laboratories, Inc., City of Industry, CA, USA and Phenomenex, Inc., Torrance, CA, USA, for the development of new sample preparation and analysis procedures for determining low levels of PFAS in food products. This particular application was directed at achieving sub-ppb sensitivity for 23 PFAS analytes in dairy products (milk, butter and cheese), eggs and fish as representative of difficult to analyze fatty matrices. The following discussion is a synopsis of the full work [8].

Sample Preparation

One gram of homogenized sample was spiked with internal standards and surrogates and an analyte mix of 23 PFAS compounds (**Table 1**) at the 1 ng/g level, followed by the addition of 10mL acetonitrile and 10mL water. Four replicates of each matrix (milk, eggs, butter, cheese and fish) were prepared. The samples were processed by a modified QuEChERS procedure using a commercial kit (Phenomenex roQ™ Extraction Kit). An aliquot (500 µL) of the cleaned acetonitrile phase was transferred to an LC vial for analysis. **Figure 2** displays an extraction blank and the five sample types following sample preparation.

1. New Concerns about PFAS in Food (continued)

Optional Solid Phase Extraction

A dispersive SPE cleanup was used to achieve a 10-fold lower level of quantitation. Four replicate samples of the egg matrix were spiked with the PFAS analyte mix at the 0.1 ng/g level and processed by the QuEChERS procedure. Following extraction, 500 µL of the acetonitrile phase was diluted with 15 mL of water and loaded onto a preconditioned, weak-ion-exchange SPE tube (Phenomenex Strata®-X-AW 200 mg). The analytes of interest were then eluted with 4 mL of 0.3 % NH₄OH-acetonitrile. The eluate was evaporated to dryness, reconstituted with 500 µL of acetonitrile and transferred to an LC autosampler vial for analysis.

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LC-MS/MS Analysis

The chromatography was performed on an Agilent® 1290 UH-PLC system. The LC column employed was a Phenomenex Luna™ Omega 1.6 µL PS C18 operating at 40 degrees Celsius with a flow rate of 0.55 mL/min and an injection volume of 20 µL. The mass spectrometer used was an Agilent 6460 QQQ. Various LC-MS/MS conditions were explored and an ammonium acetate/acetonitrile gradient (**Table 2**) proved to be optimum, resulting in a run time of approximately 4 minutes.

Results and Discussion

System calibration showed a linear dynamic response from 0.05 ppb – 1000 ppb with a lower limit of quantization of 0.05 ppb as shown in **Figure 3** and a calibration chromatogram at the 0.05 ppb level is shown in **Figure 4**. Recovery data for the five matrix types is summarized in **Figures 5–9**. Four replicates of each matrix were spiked at the 1 ng/g level and prepared for analysis as described above (but were not subjected to the solid phase extraction process). **Figure 10** presents the recovery data for four replicates of the egg matrix spiked at 0.1 ng/g and prepared as described above, but with the addition of the solid phase extraction step to increase method sensitivity.

The recovery data show good recovery for all five matrices spiked at the 1 ng/g level, with most analytes falling into the 80 % - 120 % recovery range. Precision is generally somewhat poorer for the higher fat dairy products than for the lower fat matrices. The recoveries on tuna fish are particularly good, considering the complexity of the matrix. In comparing the analyte recoveries from eggs at the 1 ng/g and 0.1 ng/g levels (**Figure 9** and **Figure 10**), both show comparable recoveries although, as expected, the higher spike level shows greater precision. Overall, the data suggest that the method has sufficient accuracy and precision to potentially be used to assess environmental PFAS contamination of food products. Clearly, this is preliminary data and further development and multi-laboratory validation would be required to demonstrate such a purpose. However, the data clearly show that current sample preparation techniques, coupled with the power of advanced chromatography and triple-quad mass spectrometry represent a suitable workflow.

The Sequel

The earlier discussion showed the use of current analytical technology to address the challenge of environmental PFAS contamination of the food supply. However, care should be taken since experience with analytical chemistry teaches us that we will inevitably be facing further analytical challenges from the realm of the “unknown-unknowns”.

In PFAS analysis, we are currently discussing a target analyte list of 20, 30 or 40 compounds? However, the number of compounds in the PFAS universe has been estimated at 5000 - and even as high as 8,000 - which doesn't include potential degradation products. Toxicity is largely a function of the unique chemical and configurational state of a molecule that controls the biochemical interaction with the organism. So, there is much more analytical work to identify the most important PFAS compounds from a toxicity perspective.

Excellent work is being done with accurate mass and advanced data analysis to give us a broader understanding of the chemical complexity of the PFAS universe. However, given the complexity and extent of the problem of environmental PFAS contamination, it is clear that a lot of hard work has yet to be done.

Acknowledgements

The contribution of Dr. Agustin Pierri and his team at Weck Laboratories, City of Industry, California, USA is gratefully acknowledged.



1. New Concerns about PFAS in Food (continued)

Figure 1.
Pathway Model for Environmental Transmission of PFAS to Food and Consumer

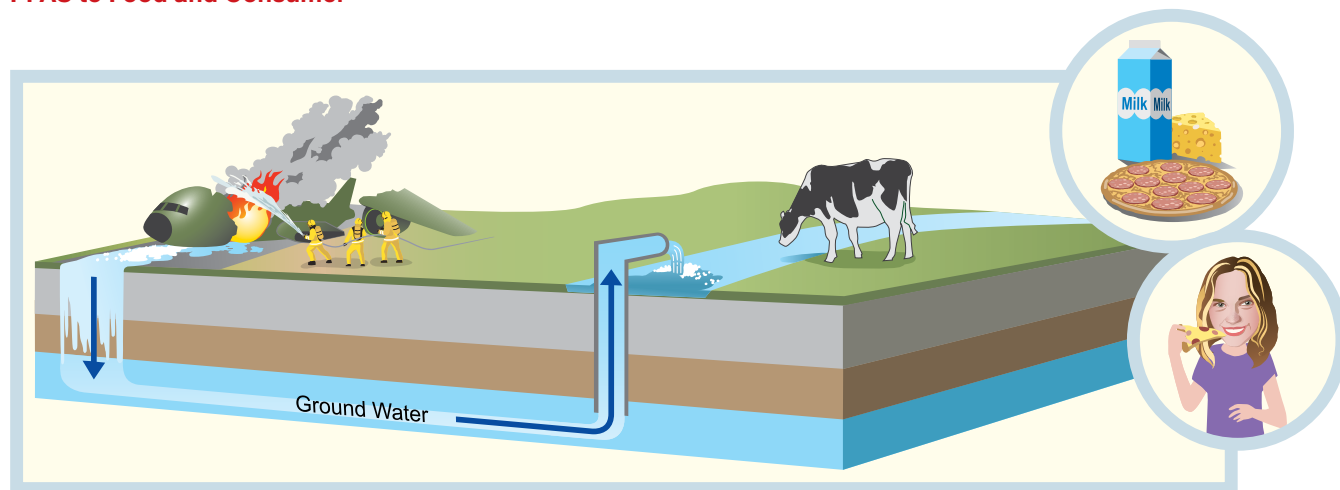
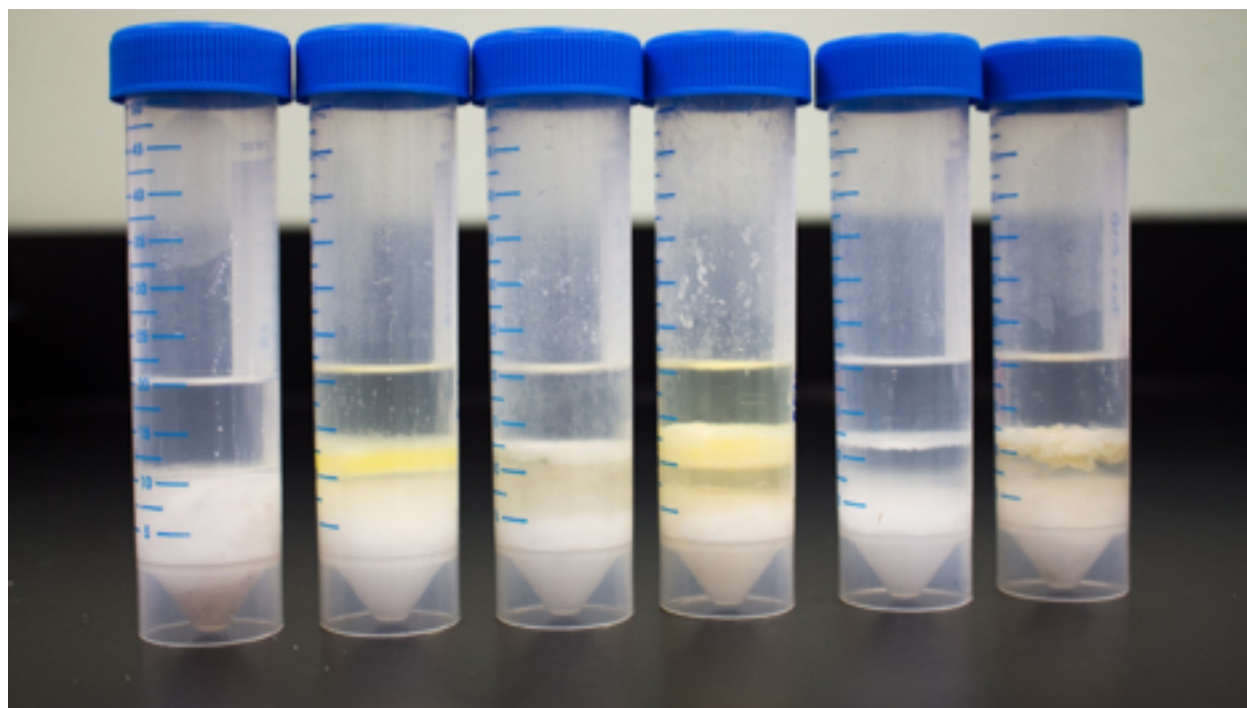


Figure 2.
Samples after QuEChERS Cleanup:
From Left to Right: Blank, Butter, Cheese, Egg, Milk and Fish



1. New Concerns about PFAS in Food (continued)

Applications

Figure 3.
System Calibration Dynamic Range (0.05 – 1000 ppb)

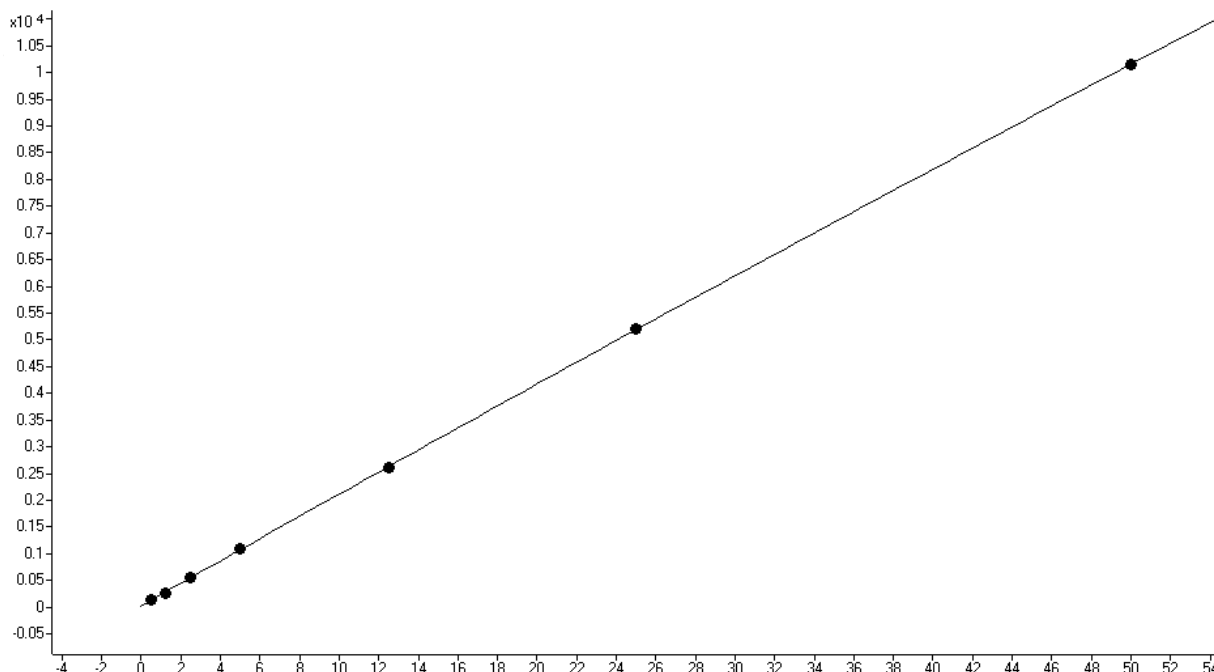
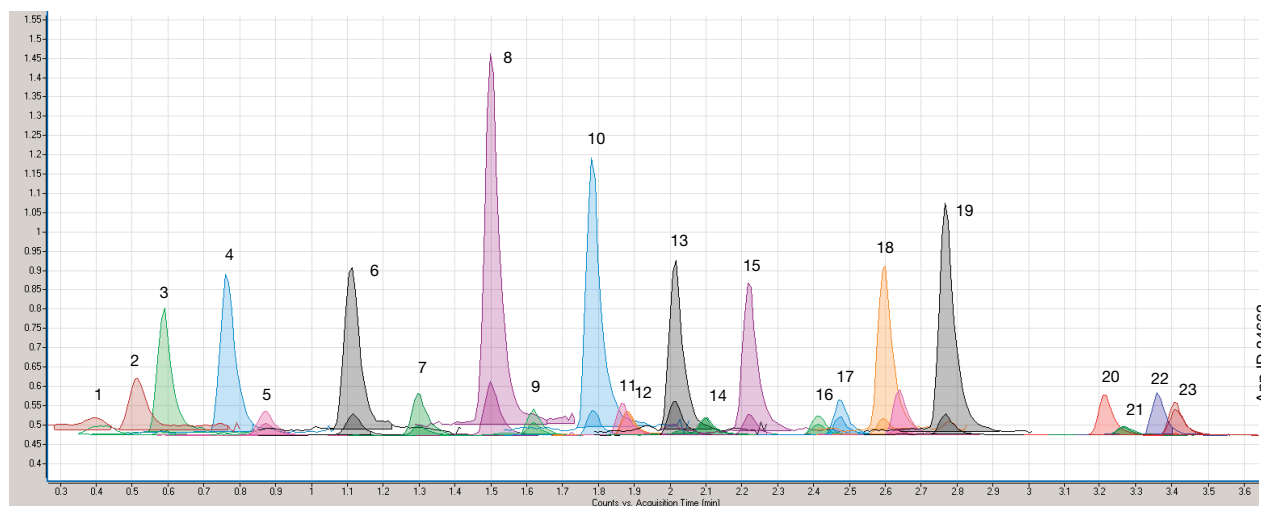


Figure 4.
Chromatogram of 0.05 ppb Lower Limit of Quantization Standard



1. New Concerns about PFAS in Food (continued)

Figure 5.
Milk Recoveries (QuEChERS: 1 ng/g, n=4)

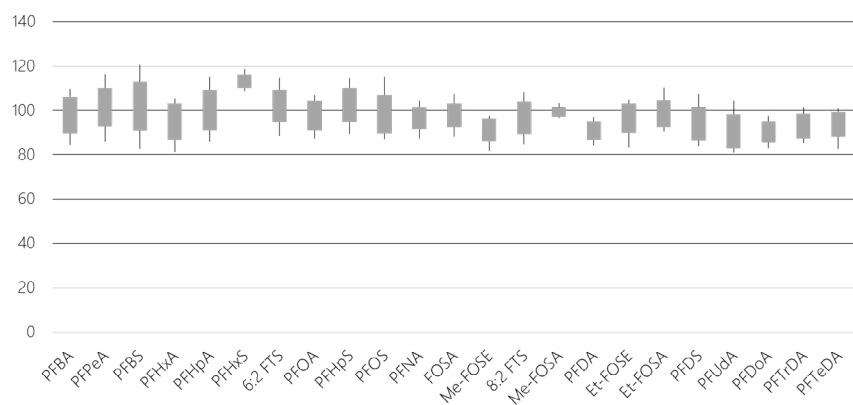


Figure 6.
Butter Recoveries (QuEChERS: 1 ng/g, n=4)

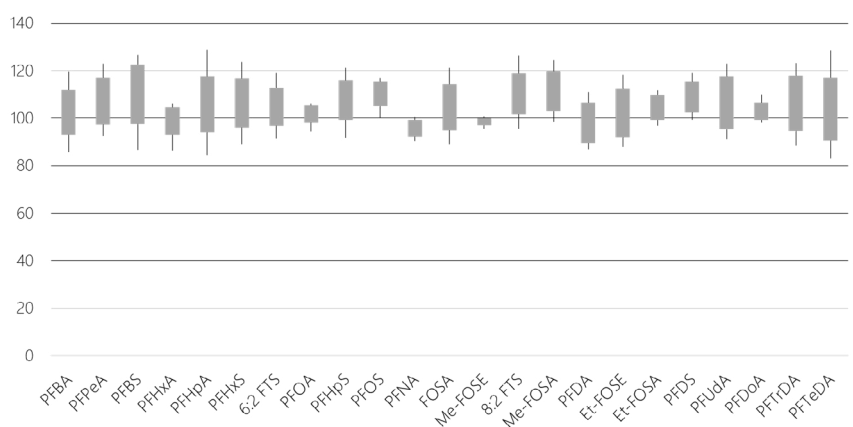
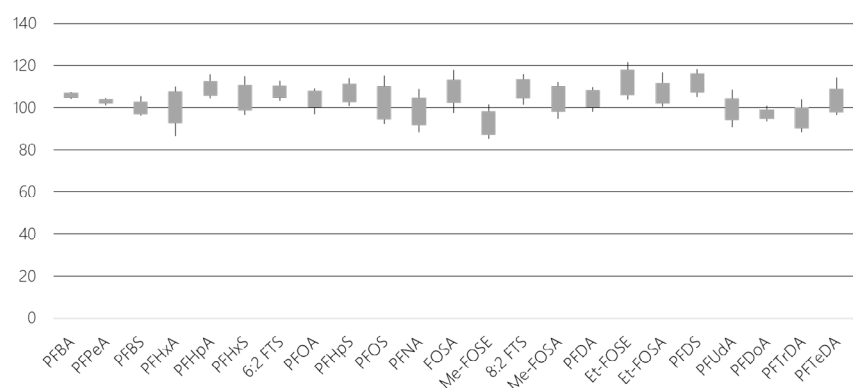


Figure 7.
Tuna Recoveries (QuEChERS: 1 ng/g, n=4)



1. New Concerns about PFAS in Food (continued)

Figure 8.
Cheese Recoveries (QuEChERS:1 ng/g, n=4)

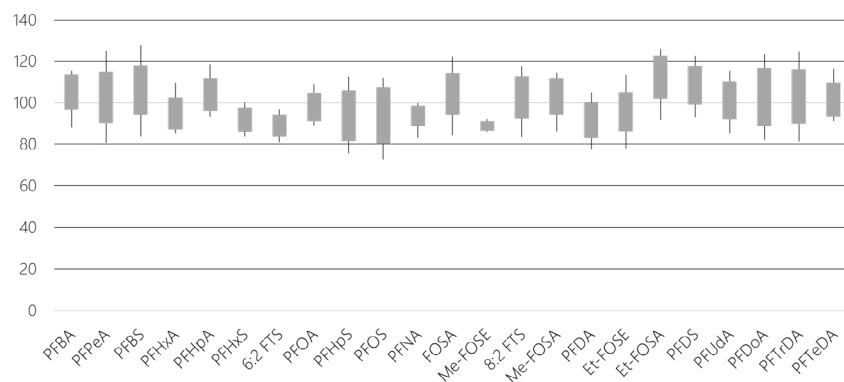


Figure 9.
Egg Recoveries (QuEChERS: 1 ng/g, n=4)

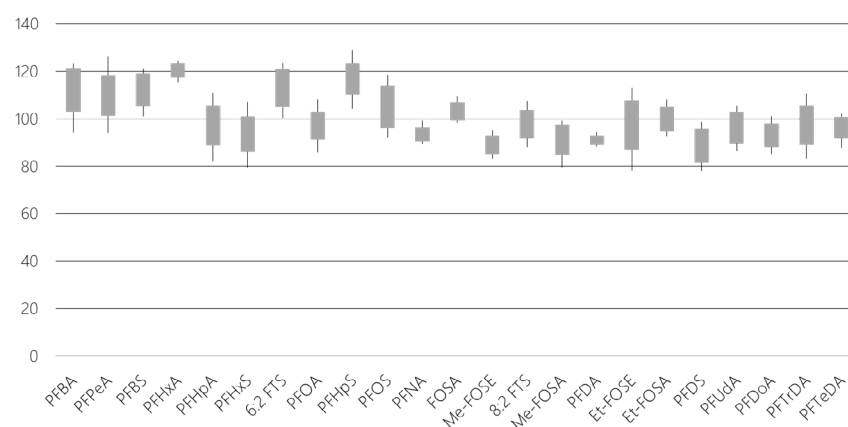
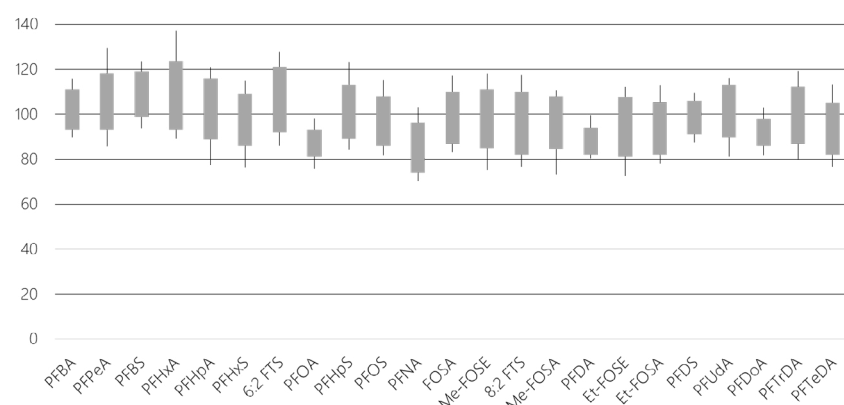


Figure 10.
Egg Recoveries (QuEChERS + SPE: 0.1 ng/g, n=4)



1. New Concerns about PFAS in Food (continued)

Table 1.
PFAS Analyte List

Analytes:		
1. PFBA	9. PFHpS	17. Et-FOSE
2. PFPeA	10. PFOS	18. Et-FOSA
3. PFBS	11. PFNA	19. PFDS
4. PFHxA	12. FOSA	20. PFDS
5. PFHpA	13. Me-FOSE	21. PFDoA
6. PFHxS	14. 8:2 FTS	22. PFTTrDA
7. 6:2 FTS	15. Me-FOSA	23. PFTeDA
8. PFOA	16. PFDA	

Table 2.
LC-MS/MS Conditions

Column:	Luna™ Omega 1.6µm PS C18		
Dimensions:	100 x 2.1 mm		
Part No.:	00D-4752-AN		
Mobile Phase:	A: 5 mM Ammonium Acetate in Water B: Acetonitrile		
Gradient:	Time (min)	% B	
	0	40	
	0.5	40	
	3	90	
	3.1	100	
	4	100	
Flow Rate:	0.55 mL/min		
Injection:	20 µL		
Temperature:	40 °C		
UHPLC System:	Agilent® 1290		
Detection:	Agilent 6460 QQQ		
Analytes:	1. PFBA	9. PFHpS	17. Et-FOSE
	2. PFPeA	10. PFOS	18. Et-FOSA
	3. PFBS	11. PFNA	19. PFDS
	4. PFHxA	12. FOSA	20. PFUdA
	5. PFHpA	13. Me-FOSE	21. PFDoA
	6. PFHxS	14. 8:2 FTS	22. PFTTrDA
	7. 6:2 FTS	15. Me-FOSA	23. PFTeDA
	8. PFOA	16. PFDA	

References

1. Analytical Results of Testing Food for PFAS from Environmental Contamination. (<https://www.fda.gov/food/chemicals/analytical-results-testing-pfas-environmental-contamination>), US Food and Drug Administration, October 20, 2020.
2. Basic Information about PFAS (<https://www.epa.gov/pfas/basic-information-pfas>), US Environmental Protection Agency, December 06, 2018.
3. Toxicological Profile for Perfluoroalkyls (<https://www.atsdr.cdc.gov/toxicoprofiles/tp.asp?id=1117&tid=237>), Agency for Toxic Substances and Disease Registry, Communicable Disease Center, September 30, 2020.
4. FDA Removes Approval for the Use of PFCs in Food Packaging Based on the Abandonment. (<https://www.fda.gov/food/cfsan-constituent-updates/fda-removes-approval-use-pfcs-food-packaging-based-abandonment>), US Food and Drug Administration, November 21, 2016.
5. Development of the Proposed Unregulated Contaminant Monitoring Rule for the Fifth Monitoring Cycle (UCMR 5): Public Meeting and Webinar (<https://www.epa.gov/sites/production/files/2019-10/documents/ucmr5-stakeholdermeeting-190830.pdf>), US Environmental Protection Agency, July 16, 2019.
6. Installations Conducting Assessments for PFAS use or Potential Release. (https://media.defense.gov/2020/Mar/17/2002265608/-1/-1/1/MAP_OF_INSTALLATIONS_CONDUCTING_ASSESSMENTS_FOR_PFAUSE_OR_POTENTIAL_RELEASE.PDF), US Department of Defense, September 30, 2019.
7. Per and Polyfluoroalkyl Substances (PFAS) (<https://www.fda.gov/food/chemicals/and-polyfluoroalkyl-substances-pfas>), US Food and Drug Administration, December 20, 2019.
8. Per- and Polyfluorinated Alkyl Substances (PFAS) from Milk, Eggs, Butter, Cheese and Fish using QuEChERS, SPE and LC-MS/MS. Agustin Pierri Weck Laboratories) and Scott Krepich (Phenomenex, Inc.). Phenomenex Technical Note TN-0124 (<https://phenomenex.blob.core.windows.net/documents/a9406d77-c88e-49cb-9347-81efd828fc25.pdf>), Phenomenex, Inc., 2018.

2. PFAS in Food Contact Materials

Identification and Quantification of PFAS in Food Contact Materials using MRM^{HR} Workflow on X500R QTOF System

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Introduction

In comparison to other surfactants, perfluorinated alkyl substances (PFAS) have stable physiochemical structures with hydrophobic and oleophobic properties. They are widely used in industrial and consumer products like plastic packaging materials for food and as coating in non-stick pans. Due to their chemical stability and low reactivity, PFAS are highly resistant to degradation even in living organisms and can therefore be accumulated in the food chain. Human exposure to PFAS residues has been implicated in incidences of cancer, obesity, endocrine system disruption and other adverse health effects. [1]

With the rapid growth in the food delivery industry in China (and globally) in the past two years, one-time-use plastic packaging materials are widely used by merchants due to their low cost and high durability [2]. One-time-use plastic has become a source of public concern and environmental pollution. Given the tremendous persistence of PFAS in the environment and the adverse effect on human health, monitoring of PFAS residue has gained traction in China and elsewhere.

In China, the level of PFOS and PFOA in food contact materials and products is regulated according to the latest National Food Safety Standard (GB 31604.35-2016). The detection limit is set at 1.0 ng/g while the quantification limit is set at 2.0 ng/g. In 2006, the European Union (EU) has set a regulation that the level of PFOS in finished products should not exceed 0.005 % of the product mass.



The X500R QTOF system has the industry's fastest scanning speed, allowing for the implementation of the unique MRM^{HR} acquisition mode to provide excellent quantitative performance using high-resolution MS/MS data. This approach to quantitation with LC-QTOF-MS/MS minimizes matrix interferences and the patented Turbo VTM ion source with curtain gas interface, twin sprayer technology and built-in automatic calibration system help to improve and maintain instrument robustness and maintain high mass accuracy results. The high resolution MS/MS spectra can also be used for qualitative analysis by calculating the ion ratio for confirmation, thus reducing false positives by taking advantage of the data acquired on the LC-QTOF platform.

Key Workflow Advantages

- PFAS quantitation using an easily established method and minimal method development
- 10-minute run time using a Phenomenex Kinetex[®] C18 column demonstrates separation of PFAS targets
- MRM^{HR} workflow using MS/MS for selectivity vs high resolution TOF MS mode provides improved signal-to-noise
- QTOF technology can be utilized for quantitative analysis of PFAS suite without compromising method performance (excellent sensitivity, linearity demonstrated)

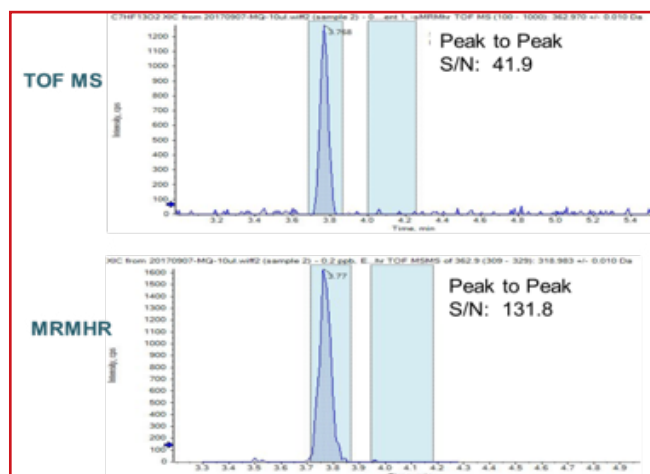


Figure 1.
Signal-to-Noise Comparison of PFHpA using TOF-MS and MRMHR Data Using a Post Spiked 0.2 ppb Matrix Blank

Monitoring the transition and the high resolution fragment ion results in greater specificity and reduced baseline, so signal-to-noise demonstrates marked improvement and method sensitivity is maximized.

2. PFAS in Food Contact Materials (continued)

Methods

Sample Preparation

The food packaging material to be tested is cut into small pieces. For coating sample, scrape it with a small knife. The sample preparation procedure was adapted from National Standard of China (document number GB 31604.35-2016) which is implemented on 19 April 2017 (Figure 2).

A total of eight samples were collected as test samples which include disposable meal box, plastic bag, beverage bottle, coating of non-stick pan, etc. Packaging materials in the collected samples were mainly polyethylene, polystyrene and polytetrafluoroethylene.

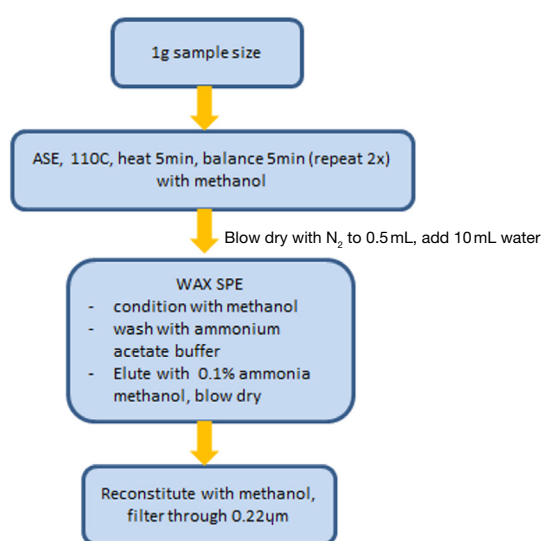


Figure 2.
Extraction and Clean-up Process Flow Diagram

Chromatography

Using the SCIEX® ExionLC™ AD System with a Phenomenex Kinetex®, 2.6µm C18, 100 X 2.0mm, compounds were separated using a gradient elution with mobile phase A of 5mM NH₄AC in water and mobile phase B of 5mM NH₄AC in methanol (flow rate of 0.3 mL/min, column temperature 40 °C).

Mass Spectrometry

The SCIEX X500R QTOF System was used to analyse the compounds operating in negative ion polarity using the Scheduled MRM^{HR} acquisition mode (Table 1). Source conditions were as follows: CUR of 30psi; CAD of 7; IS of -4500V; Temp 500 °C; GS1 of 50psi; GS2 of 55psi.

Data Processing

All data was processed with SCIEX OS Software.

Table 1.

Scheduled MRM^{HR} Method Setup in SCIEX OS

Unique RTs can be defined for each transition for each analyte.

Compound ID	Group name	Precursor ion (Da)	Fragment ion (Da)	Accumulation time (sec)	Declustering potential (V)	Collision energy (V)	Retention time (min)
1	PFBA	212.90	168.9000	0.0600	-80	-35	2.55
2	PFPeA 1	262.90	218.9000	0.0600	-80	-35	3.22
3	PFPeA 2	262.90	69.0000	0.0600	-80	-35	3.22
4	PFBS 1	298.90	80.0000	0.0600	-80	-35	3.29
5	PFBS 2	298.90	99.0000	0.0600	-80	-35	3.29
6	PFHxA 1	312.90	268.9000	0.0600	-80	-35	3.55
7	PFHxA 2	312.90	119.0000	0.0600	-80	-35	3.55
8	PFHxS 1	362.90	318.9000	0.0600	-80	-35	3.79
9	PFHxS 2	362.90	168.9000	0.0600	-80	-35	3.79

Establishing the Scheduled MRM^{HR} Quantitative Method

The SCIEX OS software is fully automated with a user-friendly interface, greatly reducing the time to establish the acquisition method. The MRM parameters can be set up easily in two different ways. For compounds which are in MS/MS spectral library, fragment ions can be imported easily from the library to build the MRM^{HR} method list. Up to 5 fragment ions can be imported at the same time using a single click. For compounds not found in the spectral library, spectra can be added easily to the library using TOF MS-IDA-MS/MS data acquired for standards of the desired targets.

MRM parameters like retention time, declustering potential (DP) and collision energy (CE) from an existing triple quadrupole method are fully transferrable.

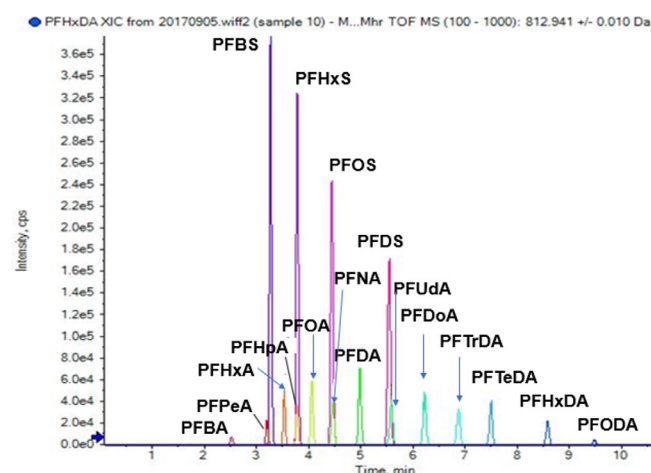


Figure 3.
TOF MS Extracted Ion Chromatogram of 17 PFAS
Good separation was achieved for most of the 17 PFAS compounds analysed.

2. PFAS in Food Contact Materials (continued)

MRM^{HR} Quantitation of PFAS

Chromatogram of 17 PFAS utilizing extracted precursor ion data from TOF-MS scan are shown (Figure 3).

High Selectivity Data

Comparing 0.2 ppb post spiked in matrix blank, PFHpA show higher selectivity in MRM^{HR} mode as compared to TOF-MS mode for quantification (Figure 1). Monitoring the high resolution fragment ion from the full scan MS/MS data collected provides greater specificity and reduced baseline, so signal-to-noise demonstrates marked improvement and method sensitivity is maximized.

Linearity and Accuracy

The 17 monitored PFAS demonstrate good linearity and accuracy (Figure 4) with the correlation coefficients above 0.99. Accuracy values are within the permissible deviation range for LOD and LOQ according to the national standards.

Ion Ratio Calculations

Ion ratios can be easily calculated using the SCIEX® OS software. Ion ratio confirmation can be visually displayed in the chromatogram and result table. Depending on the requirement, the confirmation tolerance can be defined using either constant tolerance or variable tolerance as shown in Figure 5.

Figure 5: Setting up Tolerance for Ion Ratios Confirmation

The top screenshot shows the 'Ion Ratios' settings with 'Constant Tolerance' selected. The bottom screenshot shows the 'Ion Ratios' settings with 'Variable Tolerance' selected, displaying a table of tolerance levels based on expected ion ratio calculation.

Qualitative Rule		Acceptable % Difference	Marginal % Difference	Unacceptable % Difference
Ion Ratio Lower Limit	Ion Ratio Upper Limit	(Tolerance levels based on expected ion ratio calculation)		
0	0.1	< 50	< 50	>= 50
0.101	0.2	< 30	< 30	>= 30
0.201	0.5	< 25	< 25	>= 25
0.501	1	< 20	< 20	>= 20

Figure 5.

Setting up Tolerance for Ion Ratios Confirmation

Constant tolerance (same percent difference from measured standard ion ratio) or variable tolerance (varying percent difference dependant on concentration level) can be utilized when determining whether an unknown same meets the criteria for qualitative analyte identification by ion ratio confirmation. Different levels of percent difference can be defined by the user to be flagged as within "Acceptable," "Marginal," or "Unacceptable."

Detection of PFAS in Food Contact Materials

SCIEX OS software combines both qualitative and quantitative results in one single interface (Figure 6). The result table show the retention time, concentration, peak area, ion ratio confirmation and the mass error of 0.9ppm for a sample tested positive with PFOA.

Among the eight samples, eight types of PFAS were detected as shown in Table 2. Two out of eight samples have levels which exceeded regulated level of 1 ng/g by national standard. Most of the detected PFAS are the acid derivatives of PFOA and primarily found in non-stick pan coating and disposable meal boxes. The number of actual samples collected in this test is rather small; hence statistically it does not imply that all related products are unsafe for consumers.

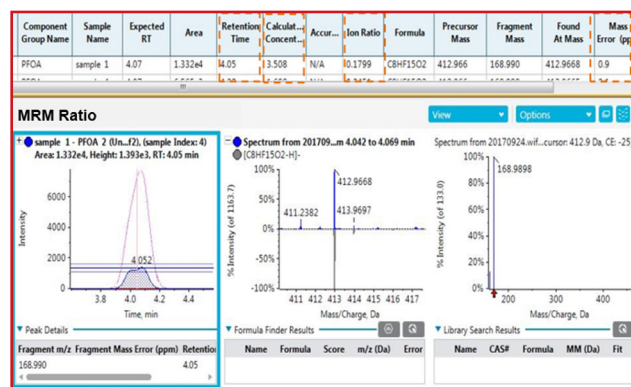


Figure 6.

PFOA Results in Actual Sample

2. PFAS in Food Contact Materials (continued)

Summary

The SCIEX® X500R QTOF system and SCIEX OS software brings powerful performance capabilities for routine testing of PFAS. The unique MRM^{HR} quantification method enables high selectivity even in real sample with matrix interference. This improves the detection and quantification of PFAS which can meet the EU regulation and national standards in China.

Although the concentration of PFAS in most of the test samples falls below the regulated level, the detection rate of perfluorinated alkyl substances is relatively high indicating that the quality of food contact/packaging materials may pose potential risks to consumer's health.

References

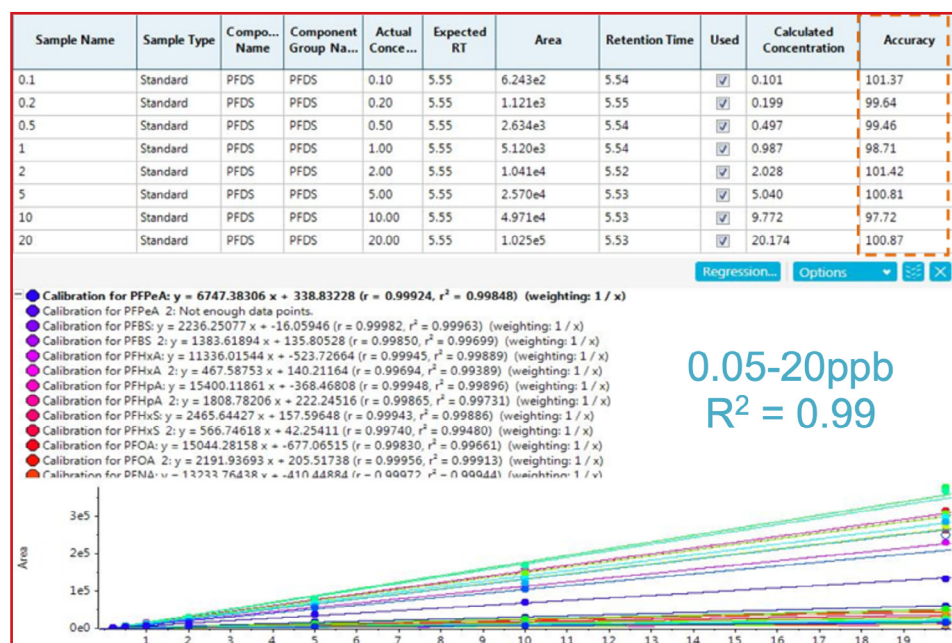
1. Xu R, Tan H, Yang H B, et al. (2014) Food and Fermentation Industries, 40(10): 205.
2. Kannan K, Tao L, Sinclair E, et al. (2005) Arch Environ Con Tox, 48: 559.
3. GB 31604.35-2016, National Food Safety Standard - Food contact materials and products - Determination of perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA).

Table 2.
PFAS Content in Different Food Contact Samples

	Detected Amount (ng/g)							
	PFHxA	PFHpA	PFOA	PFDA	PFuDA	PFDoA	PFTTrDA	PFTeDA
Meal box 1	0.14	0.16	3.15	-	-	-	-	-
Meal box 2	-	-	3.12	-	-	-	-	-
Plastic bag 1	-	-	-	-	-	-	-	-
Plastic bag 2	-	-	-	-	-	-	-	-
Drink bottle 1	-	-	-	-	-	-	-	-
Drink bottle 2	-	-	-	-	-	-	-	-
Non-stick pan 1	-	-	-	0.11	0.15	0.13	0.15	-
Non-stick pan 2	-	-	-	-	-	-	-	0.17

- Falls below the detection level of this method.

Figure 4.
Calibration Curve of 17 PFAS with Acceptable Accuracy and Linear Response



Product Guide for PFAS Analysis

Phenomenex PFAS Products Referenced or Applicable in Official Methods

Regulatory Method	Product	Part Number
USEPA 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/ Tandem Mass Spectrometry (LC/MS/MS) (5)	PFAS CRM EPA 537.1 mix 1mL 2µg/mL in methanol	AL0-101839
	PFAS CRM EPA 533 + 537.1 mix 1mL 2µg/mL in methanol	AL0-101840
	Strata™ SDB-L 500 mg/6 mL	8B-S014-HCH
	Gemini™ 3 µm C18, 50 x 3 mm or	00B-4439-B0
	Luna™ Omega 1.6 µm PS C18 100 x 2.1 mm	00D-4752-AN
USEPA Method 533: Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution, Anion Exchange Solid Phase Extraction and LC-MS/MS. (1)	PFAS CRM EPA 533 mix 1mL 2µg/mL in methanol	AL0-101838
	PFAS CRM EPA 533 + 537.1 mix 1mL 2µg/mL in methanol	AL0-101840
	Strata-X-AW 500 mg/6 mL	8B-S038-HCH
	Gemini 3 µm C18 50 x 2 mm or	00B-4758-Y0
	Luna Omega 1.6 µm PS C18 100 x 2.1 mm	00D-4752-AN
US Food and Drug Administration: Determination of 16 Perfluoroalkyl and Polyfluoroalkyl Substances(PFAS) in Food using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS). (2)	Strata-XL-AW 200 mg/3 mL	8B-S051-FBJ
US Department of Agriculture: Screening, Determination and Confirmation of PFAS by UPLC-MS-MS (3)	Luna C8(2) 3 µm 50 x 2 mm	00B-4248-B0
US Department of Defense: Quality Systems Manual (QSM) for Environmental Laboratories (4)	Strata PFAS (WAX/GCB) 200 mg/50 mg/6 mL, 30/box 500 mg/50 mg/6 mL, 30/box	CS0-9207 CS0-9208
	Gemini 3 µm C18 50 x 2 mm	00B-4439-B0

References

1. [Method 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry \(LC/MS/MS\) | Science Inventory | US EPA](#)
2. [Method 533: Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry | Methods Approved to Analyze Drinking Water Samples to Ensure Compliance with Regulations | US EPA](#)
3. [Determination of 16 Perfluoroalkyl and Polyfluoroalkyl Substances in Food using Liquid Chromatography-Tandem Mass Spectrometry \(fda.gov\)](#)
4. [Screening, Determination and Confirmation of PFAS by UPLC-MS-MS \(usda.gov\)](#)
5. <https://denix.osd.mil/edqw/documents/manuals/qsm-version-5-3-final/>

Recommended HPLC Products for Routine PFAS Analysis

Description and Function	Product	Part Number
Analytical Column (UHPLC)	Kinetex™ 5 µm EVO C18 100 x 2.1 mm	00D-4633-AN
	Luna Omega C18 1.6 µm 50 x 2.1	00B-4752-AN
	Gemini 3 µm C18 50 x 3 mm	00B-4439-Y0
Analytical Column	Gemini 3 µm C18 50 x 3 mm	00B-4439-Y0
Analytical Column (> 100 µL injection)	Gemini 3 µm C18 100 x 3 mm	00D-4439-Y0
Analytical Column (improved, low wt acids)	Luna Omega 3 µm PS C18 50 x 3 mm	00B-4758-Y0
Delay Column	Kinetex 5 µm EVO C18, 50 x 2.1 mm	00B-4633-AN 00A-4252-Y0
SecurityGuard	Luna Omega PS C18 4 x 3.0/10 pack for ID: 3.2-8.0 mm 4 x 2.0/10 pack for ID: 2.0-3.0 mm	AJ0-7606 AJ0-7605

Product Guide for PFAS Analysis (continued)

Recommended SPE Products

Description and Function	Product	Part Number
SPE Cartridge for EPA 537.1	Strata™ SDB-L 500 mg/6 mL, 30/box	8B-S014-HCH
SPE Cartridge for EPA 533	Strata-X-AW 33um Polymeric Weak Anion, 500 mg/6 mL, 30/box	8B-S038-HCH
SPE Cartridge (Rev. Phase, High Perf.)	Strata-XL 500 mg/6 mL, 30/box	8B-S043-HCH
SPE Stacked Cartridge (DOD QSM 5.3)	Strata PFAS (WAX/GCB) 200 mg/50 mg/6 mL, 30/box	CS0-9207
SPE Stacked Cartridge (DOD QSM 5.3)	Strata PFAS (WAX/GCB) 500 mg/50 mg/6 mL, 30/box	CS0-9208
SPE Cartridge (WAX for DOD QSM 5.3)	Strata-XL-AW 500 mg/6 mL, 30/box	8B-S051-HCH
GCB** Cartridge (GCB for DOD QSM 5.3)	Strata GCB 250 mg/6 mL, 30/box	8B-S528-FCH
SPE Cartridge (WAX* for FDA Method)	Strata-XL-AW 100 µm 200 mg/3 mL, 50/box	8B-S051-FBJ

(*WAX = Weak Anion Exchange)

(**GCB = Graphitized Carbon Black)

Recommended QuEChERS Products

Description and Function	Product	Part Number
QuEChERS Extraction (Soil/Sediment)	roQ QuEChERS Extraction Kit	KS0-8911
QuEChERS dSPE (Soil/Sediment)	roQ QuEChERS dSPE Kit, 15 mL	KS0-9516
QuEChERS Extraction (Dairy/Eggs/Fish)	roQ QuEChERS Extraction Kit	KS0-8910
QuEChERS dSPE (Dairy/Eggs/Fish)	roQ QuEChERS dSPE Kit	KS0-9511




Recommended Accessories

Description and Function	Product	Part Number
SPE Sample Reservoir	75 mL Sample Reservoir	H0-7005
Large Volume SPE	Adaptor Cap for 12,20, 60 mL SPE Tubes	AH0-7379
Autosampler Vials	Polypropylene, 300 µm + PE Starburst Cap	AR0-9995-12-C
Polypropylene Vials	Vial 9mm Screw Thd PP 2 mL, 1000 Pk	AR0-89C7-13
Vial Caps	Cap 9mm Solid Top Black Unlined	8B-S528-FCH
PEEK Capillary Tubing	Capillary Tubing Kit, Various Sizes	AT0-1964
PEEK Tubing Cutter	Cutter for PEEK Capillary Tubing	AT0-1110

Strata™ Solid Phase Extraction (SPE)

Strata-X

Ordering Information

Format	Sorbent Mass	Part Number	Unit
Tube			
	30 mg	8B-S100-TAK**	1 mL (100/box)
	30 mg	8B-S100-TBJ	3 mL (50/box)
	60 mg	8B-S100-UBJ**	3 mL (50/box)
	100 mg	8B-S100-FBJ	3 mL (50/box)
	100 mg	8B-S100-ECH	6 mL (30/box)
	200 mg	8B-S100-FBJ	3 mL (50/box)
	200 mg	8B-S100-FCH	6 mL (30/box)
	500 mg	8B-S100-HBJ	3 mL (50/box)
	500 mg	8B-S100-HCH	6 mL (30/box)
Giga™ Tube			
	500 mg	8B-S100-HDG	12 mL (20/box)
	1 g	8B-S100-JDG	12 mL (20/box)
	1 g	8B-S100-JEG	20 mL (20/box)
	2 g	8B-S100-KEG	20 mL (20/box)
	5 g	8B-S100-LFF	60 mL (16/box)
Teflon® Tube			
	200 mg	8B-S100-FBJ-T	3 mL (50/box)
	200 mg	8B-S100-FDG-T	12 mL (20/box)



On-line Extraction Cartridge

Description	Part Number	Unit/Box
Strata-X on-line extraction cartridge, 20 x 2.0 mm	00M-S033-B0-CB	ea
Cartridge holder, 20 mm	CH0-5845	ea

**Tab-less tubes available. Contact Phenomenex for details.

Strata-XL

Ordering Information

Format	Sorbent Mass	Part Number	Unit
Tube			
	30 mg	8B-S043-TAK	1 mL (100/box)
	60 mg	8B-S043-UBJ	3 mL (50/box)
	100 mg	8B-S043-FBJ	3 mL (50/box)
	200 mg	8B-S043-FBJ	3 mL (50/box)
	200 mg	8B-S043-FCH	6 mL (30/box)
	500 mg	8B-S043-HCH	6 mL (30/box)
Giga Tube			
	2 g	8B-S043-KDG	12 mL (20/box)
	2 g	8B-S043-KEG	20 mL (20/box)
	5 g	8B-S043-LEG	20 mL (20/box)
	5 g	8B-S043-LFF	60 mL (16/box)
	10 g	8B-S043-MFF	60 mL (16/box)
	30 mg	8E-S043-TGB	2 Plates/Box

* To control flow rate with Strata-XL, use a stopcock ([AHO-6048](#)) when processing samples with a vacuum manifold.

Gemini™ pH Flexible LC Columns

Ordering Information

3 µm Microbore, Minibore and MidBore™ Columns (mm)									SecurityGuard™ Cartridges (mm)	
Phases	50 x 1.0	20 x 2.0	30 x 2.0	50 x 2.0	100 x 2.0	150 x 2.0	50 x 3.0	100 x 3.0	150 x 3.0	4 x 2.0* /10pk
C18	00B-4439-AO	00M-4439-BO	00A-4439-BO	00B-4439-BO	00D-4439-BO	00F-4439-BO	00B-4439-YO	00D-4439-YO	00F-4439-YO	AJ0-7596

for ID: 2.0-3.0 mm

3 µm Analytical Columns (mm)						SecurityGuard™ Cartridges (mm)	
Phases	30 x 4.6	50 x 4.6	100 x 4.6	150 x 4.6	250 x 4.6	4 x 3.0* /10pk	
C18	00A-4439-EO	00B-4439-EO	00D-4439-EO	00F-4439-EO	00G-4439-EO	AJ0-7597	

for ID: 3.2-8.0 mm



Kinetex™ Core-Shell LC Columns

Ordering Information

2.6 µm Micro LC Columns (mm)						
Phases	30 x 0.3	50 x 0.3	100 x 0.3	150 x 0.3	50 x 0.5	150 x 0.5
EVO C18	—	00B-4725-AC	—	00F-4725-AC	00B-4725-AF	—

2.6 µm MercuryMS™ LC-MS Cartridges (mm)			MercuryMS Cartridge Holders		
Phases	20 x 2.0	20 x 4.0	Part No.	Description	Unit
Biphenyl	00M-4622-BO-CE	00M-4622-DO-CE	CH0-7188	Direct-Connect Cartridge Holder, 20 mm	ea
			CH0-5845	Standard Cartridge Holder, 20 mm	ea

2.6 µm Minibore Columns (mm)						SecurityGuard ULTRA Cartridges†
Phases	30 x 2.1	50 x 2.1	75 x 2.1	100 x 2.1	150 x 2.1	3/pk
EVO C18	00A-4725-AN	00B-4725-AN	—	00D-4725-AN	00F-4725-AN	AJ0-9298

for 2.1 mm ID

2.6 µm MidBore™ Columns (mm)						SecurityGuard ULTRA Cartridges†
Phases	30 x 3.0	50 x 3.0	75 x 3.0	100 x 3.0	150 x 3.0	3/pk
EVO C18	00A-4725-YO	00B-4725-YO	—	00D-4725-YO	00F-4725-YO	AJ0-9297

for 3.0 mm ID

Ordering Information

5 µm Minibore Columns (mm)					SecurityGuard™ ULTRA Cartridges‡
Phases	30 x 2.1	50 x 2.1	100 x 2.1	150 x 2.1	3/pk
EVO C18	00A-4633-AN	00B-4633-AN	00D-4633-AN	00F-4633-AN	AJ0-9298

for 2.1 mm ID

5 µm MidBore™ Columns (mm)					SecurityGuard™ ULTRA Cartridges‡
Phases	30 x 3.0	50 x 3.0	100 x 3.0	150 x 3.0	3/pk
EVO C18	00A-4633-YO	00B-4633-YO	00D-4633-YO	00F-4633-YO	AJ0-9297

for 3.0 mm ID

5 µm Analytical Columns (mm)					SecurityGuard™ ULTRA Cartridges‡
Phases	50 x 4.6	100 x 4.6	150 x 4.6	250 x 4.6	3/pk
EVO C18	00B-4633-EO	00D-4633-EO	00F-4633-EO	00G-4633-EO	AJ0-9296

for 4.6 mm ID

†SecurityGuard ULTRA Cartridges require holder, Part No.: [AJ0-9000](#)

***SemiPrep SecurityGuard Cartridges require holder, Part No.: [AJ0-9281](#)

*PREP SecurityGuard Cartridges require holder, Part No.: [AJ0-8223](#)



Luna™ One of The World's Leading LC Columns



Luna C18

Ordering Information

5 µm MidBore and Analytical Columns (mm)								SecurityGuard™ Cartridges (mm)	
Phases	30 x 3.0	50 x 3.0	150 x 3.0	250 x 3.0	30 x 4.6	50 x 4.6	75 x 4.6	4 x 2.0*	4 x 3.0*
C18(2)	00A-4252-Y0	00B-4252-Y0	00F-4252-Y0	00G-4252-Y0	00A-4252-E0	00B-4252-E0	00C-4252-E0	AJ0-4286 /10pk	AJ0-4287 /10pk
								for ID: 2.0-3.0 mm	3.2-8.0 mm

5 µm Analytical and Semi-Prep Columns (mm)					SecurityGuard™ Cartridges (mm)	
Phases	100 x 4.6	150 x 4.6	250 x 4.6	250 x 10	4 x 3.0*	10 x 10 [‡]
C18(2)	00D-4252-E0	00F-4252-E0	00G-4252-E0	00G-4252-N0	AJ0-4287 /10pk	AJ0-7221 /3pk
					for ID: 3.2-8.0 mm	9-16 mm

*SecurityGuard™ Analytical Cartridges require holder, Part No.: [KJ0-4282](#)

‡SemiPrep SecurityGuard™ Cartridges require holder, Part No.: [AJ0-9281](#)

Luna Omega PS C18 and Luna C18

Ordering Information

1.6 µm Microbore Columns (mm)			
Phases	50 x 1.0	100 x 1.0	150 x 1.0
PS C18	—	00D-4752-A0	—
C18	00B-4742-A0	00D-4742-A0	00F-4742-A0

1.6 µm Minibore Columns (mm)				SecurityGuard™ ULTRA Cartridges [‡]	
Phases	30 x 2.1	50 x 2.1	100 x 2.1	150 x 2.1	3/pk
PS C18	00A-4752-AN	00B-4752-AN	00D-4752-AN	00F-4752-AN	AJ0-9508
C18	00A-4742-AN	00B-4742-AN	00D-4742-AN	00F-4742-AN	AJ0-9502
for 2.1 mm ID					

To find complete list of part numbers visit:

www.phenomenex.com

For chat with a live technical expert at:

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Perfluoroalkyl Substances (PFAS) Testing Guide

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