Application Brief

Environmental

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Filtration for the Analysis of Per- and Polyfluoroalkyl Substances in Environmental Extracts

Introduction

Filtration is a critical element in the sample preparation workflow for per- and polyfluoroalkyl substanceS (PFAS) for samples with a high potential of containing particulate matter such as soils, sediments, surface water, and wastewater. Regulated environmental methods such as US EPA Method 8327[1] and 8329[2] (draft), ASTM D7968-17a,[3] and ASTM D7979-19[4] specify the use of syringe filtration as a final step preceding instrumental analysis.

Choice of the appropriate filtration membrane and cartridge housing materials is critical for achieving accurate and precise quantitation of PFAS compounds. Some PFAS compounds are widespread and may be present in common laboratory supplies containing polytetrafluoroethylene, leading to contamination of extracts.[5] In addition, some PFAS compounds have been demonstrated to irreversibly adsorb on common surfaces such as glass, leading to quantitative losses.[6] Therefore, care must be taken in selecting the appropriate filtration device for sample preparation. Agilent Captiva premium regenerated cellulose (RC) syringe filters are ideally suited for this analysis due to their low sorption of PFAS compounds and outstanding filtration properties.[7]

Experimental

The purpose of this application brief is to demonstrate the performance of the 25 mm diameter, 0.2 µm Agilent Captiva premium RC syringe filters (p/n 5190-5110) for the analysis of 23 PFAS compounds and 17 surrogates, listed in Table 1. Procedures for extraction and quantitation followed those listed in ASTM methods D7968-17a and D7979-19 and the draft EPA method 8329.

Results and discussion

The first experiment was used to determine the presence of any background interferences in the syringe filters by passing 10 mL of extraction solvent acidified methanol/water (50/50, pH 3 to 4) through the cartridges (double blank). Figure 1 shows the results of five replicate blanks. According to the methods,[1-4] the blanks are considered acceptable if the concentration is less than half the lower limit of quantitation (LLOQ). In this case, the LLOQ was nominally 5 ng/L with an acceptable limit background limit of less than 2.5 ng/L. For all compounds measured, the blank requirement was easily achieved.

The second experiment was used to determine if the syringe filters cause irreversible adsorption of the target compounds, which would be particularly evident at low concentrations. This was measured by filtering 10 mL of extraction solvent spiked with target compounds at the LLOQ concentration (5 ng/L) and surrogates at the midlevel concentration (80 ng/L). The results for five replicate measurements are shown in Figure 2. According to the methods, recovery at the LLOQ should be within 50 to 150% and between 70 and 130% at the midpoint level.[1,2] For all the compounds, recoveries were with acceptable limits.

Table 1. Compound list.

Target	Surrogate
Perfluorobutyl sulfonic acid (PFBS)	Perfluoro-1-[1,2,3-$^{13}C_3$]hexyl sulfonic acid (M3PFHxS)
Perfluorohexyl sulfonic acid (PFHxS)	Perfluoro-1-[$^{13}C_8$]octyl sulfonic acid (M8PFOS)
Perfluorooctyl sulfonic acid (PFOS)	Perfluoro-*n*-[$^{13}C_4$]butanoic acid (M4PFBA)
1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS)	Perfluoro-*n*-[$^{13}C_5$]pentanoic acid (M5PFPeA)
1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS)	Perfluoro-*n*-[1,2,3,4,6-$^{13}C_5$]hexanoic acid (M5PFHxA)
Perfluoro-1-pentanesulfonic acid (PFPeS)	Perfluoro-*n*-[1,2,3,4-$^{13}C_4$]heptanoic acid (M4PFHpA)
Perfluoro-1-heptanesulfonic acid (PFHpS)	Perfluoro-*n*-[$^{13}C_8$]octanoic acid (M8PFOA)
Perfluoro-1-nonanesulfonic acid (PFNS)	Perfluoro-*n*-[$^{13}C_9$]nonanoic acid (M9PFNA)
Perfluoro-1-decanesulfonic acid (PFDS)	Perfluoro-*n*-[1,2,3,4,5,6-$^{13}C_6$]decanoic acid (M6PFDA)
Perfluorobutanoic acid (PFBA)	Perfluoro-*n*-[1,2,3,4,5,6,7-$^{13}C_7$]undecanoic acid (M7PFUnA)
Perfluoropentanoic acid (PFPeA)	Perfluoro-*n*-[1,2-$^{13}C_2$]dodecanoic acid (MPFDoA)
Perfluorohexanoic acid (PFHxA)	Perfluoro-*n*-[1,2-$^{13}C_2$]tetradecanoic acid (M2PFTeDA)
Perfluoroheptanoic acid (PFHpA)	1H, 1H, 2H, 2H-Perfluoro-(1,2-$^{13}C_2$) hexyl sulfonic acid (M2-4:2 FTS)
Perfluorooctanoic acid (PFOA)	1H, 1H, 2H, 2H-Perfluoro-1(1,2-$^{13}C_2$) decyl sulfonic acid (M2-8:2 FTS)
Perfluorononanoic acid (PFNA)	N-Methyl-d3-perfluoro-1-octanesulfonamidoacetic acid (d3-N-MeFOSAA)
Perfluorodecanoic acid (PFDA)	N-Ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid (d5-N-EtFOSAA)
Perfluoroundecanoic acid (PFUdA)	Perfluoro-1-[$^{13}C_8$]octanesulfonamide (M8FOSA)
Perfluorododecanoic acid (PFDoA)	
Perfluorotridecanoic acid (PFTrDA)	
Perfluorotetradecanoic acid (PFTeDA)	
N-Ethylperfluoro-1-octanesulfonamidoacetic acid (NEtFOSAA)	
N-Methylperfluoro-1-octanesulfonamidoacetic acid (NMeFOSAA)	
Perfluoro-1-octanesulfonamide (FOSA)	

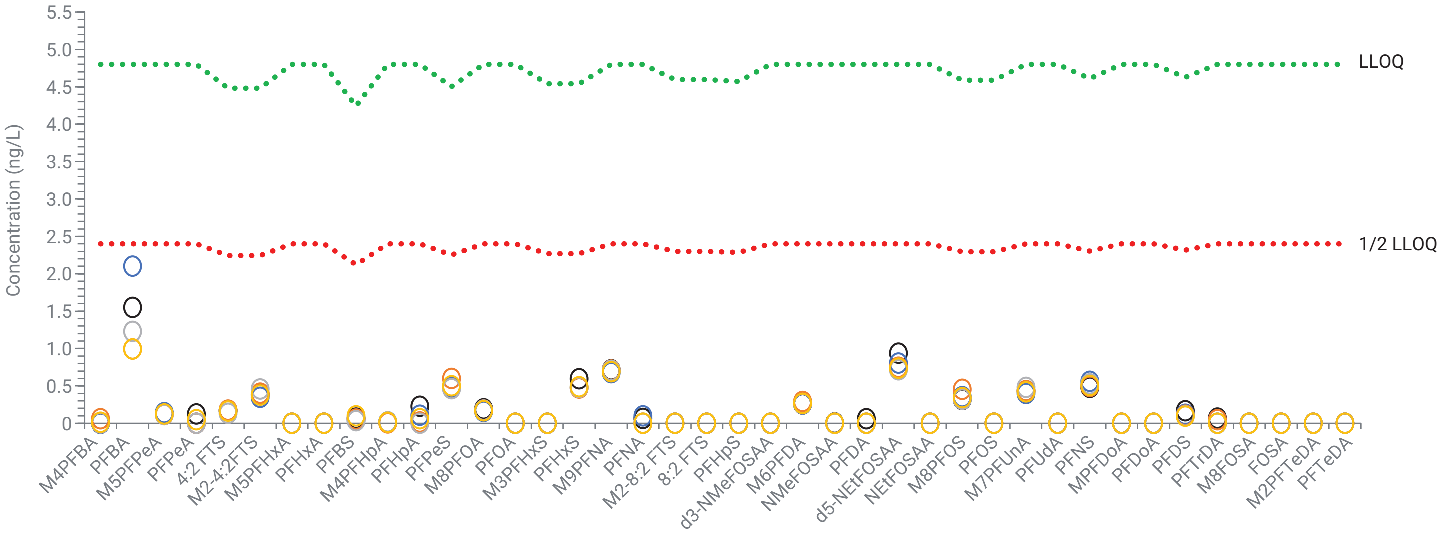

Figure 1. Background measurement for five replicate double blank measurements. Dotted lines represent the LLOQ and blank limit threshold of ½ LLOQ.

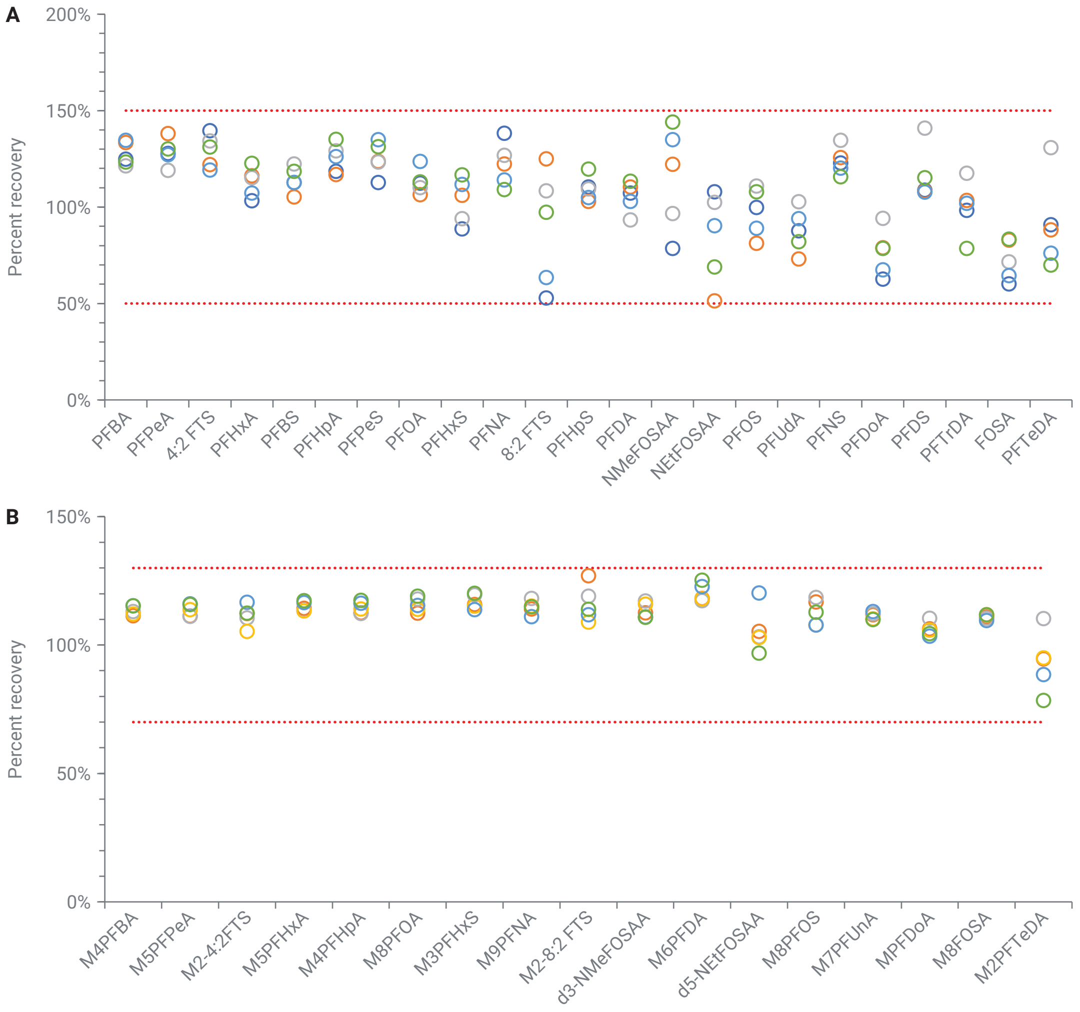

Figure 2. Agilent Captiva premium regenerated cellulose filters spike recoveries at the target LLOQ of 5 ng/L (A) and the surrogate midlevel at 80 ng/L (B).

Conclusion

Captiva premium RC syringe filters provide excellent performance for PFAS analysis. These syringe filters are free from background interferences and sorption losses.

References

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