Application Brief

Environmental

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# Disposable Syringes for Analysis of Per- and Polyfluoroalkyl Substances in Environmental Extracts


## Abstract

This application note describes the performance of disposable syringes for per- and polyfluoroakyl (PFAS) analysis. Use of disposable syringes provides savings in time, solvent cost, and solvent waste disposal and choice of the appropriate syringe material is critical for achieving accurate and precise quantitation of PFAS compounds. A first experiment was performed to determine the presence of any background interferences in the disposable syringes used directly out of the package without cleaning. A second experiment determined whether the syringes caused adsorption of the target compounds which would be particularly evident at low concentration.

## Introduction

Syringes are a critical element in the sample preparation workflow for per- and polyfluoroalkyl substances (PFAS). Syringes are used in combination with Luer lock adaptable filters for samples with a high potential of containing particulate matter such as soils, sediments, surface water, and wastewater. Regulated environmental methods such as USEPA Method 8237[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. These methods list reusable 10 or 25 mL glass syringes which require extensive cleaning before first use and in between uses. For example, EPA Method 8327 recommends soaking syringes in hot tap water followed by a 50 mL rinse with reagent water, with 30 mL acetonitrile, and 30 mL of methanol. For processing a large number of samples, the cleaning procedure can take a considerable amount of time and volume of high-purity solvents.

Use of disposable syringes provides significant savings in time, solvent cost, and solvent waste disposal. However, choice of the appropriate syringe material 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 syringe for sample preparation. Agilent disposable syringes are ideally suited for this analysis due to their low sorption of PFAS compounds and low background.[7]

## Experimental

The purpose of this application note is to demonstrate the performance of the Agilent 10 mL Captiva disposable syringes (p/n 9301-6474) for the analysis 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.

**Table 1.** Compound list.

| Targets | Surrogates |
|---|---|
| Perfluorobutyl sulfonic acid (PFBS) | Perfluoro-1-[1,2,3-$^{13}$C3]hexyl sulfonic acid (M3PFHxS) |
| Perfluorohexyl sulfonic acid (PFHxS) | Perfluoro-1-[$^{13}$C8]octyl sulfonic acid (M8PFOS) |
| Perfluorooctyl sulfonic acid (PFOS) | Perfluoro-*n*-[$^{13}$C4]butanoic acid (M4PFBA) |
| 1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS) | Perfluoro-*n*-[$^{13}$C5]pentanoic acid (M5PFPeA) |
| 1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS) | Perfluoro-*n*-[1,2,3,4,6-$^{13}$C5]hexanoic acid (M5PFHxA) |
| Perfluoro-1-pentanesulfonic acid (PFPeS) | Perfluoro-*n*-[1,2,3,4-$^{13}$C4]heptanoic acid (M4PFHpA) |
| Perfluoro-1-heptanesulfonic acid (PFHpS) | Perfluoro-*n*-[$^{13}$C8]octanoic acid (M8PFOA) |
| Perfluoro-1-nonanesulfonic acid (PFNS) | Perfluoro-*n*-[$^{13}$C9]nonanoic acid (M9PFNA) |
| Perfluoro-1-decanesulfonic acid (PFDS) | Perfluoro-*n*-[1,2,3,4,5,6-$^{13}$C6]decanoic acid (M6PFDA) |
| Perfluorobutanoic acid (PFBA) | Perfluoro-*n*-[1,2,3,4,5,6,7-$^{13}$C7]undecanoic acid (M7PFUnA) |
| Perfluoropentanoic acid (PFPeA) | Perfluoro-*n*-[1,2-$^{13}$C2]dodecanoic acid (MPFDoA) |
| Perfluorohexanoic acid (PFHxA) | Perfluoro-*n*-[1,2-$^{13}$C2]tetradecanoic acid (M2PFTeDA) |
| Perfluoroheptanoic acid (PFHpA) | 1H, 1H, 2H, 2H-Perfluoro-(1,2-$^{13}$C2) hexyl sulfonic acid (M2-4:2 FTS) |
| Perfluorooctanoic acid (PFOA) | 1H, 1H, 2H, 2H-Perfluoro-1(1,2-$^{13}$C2) 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-[$^{13}$C8]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) | |

## Results and discussion

The first experiment was used to determine the presence of any background interferences in the disposable syringes used directly out of the package without cleaning. Following the extraction procedures,[2-4] syringes were filled with 10 mL of 50/50 methanol/water and 20 µL of ammonium hydroxide (pH 9 to 10), capped, and tumbled for one hour on a rotator. After one hour, the extraction solvent was acidified to pH 3 to 4 with acetic acid and a 1 mL aliquot was transferred to an autosampler vial for LC/MS/MS analysis. 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 without cleaning the syringes.

The second experiment was used to determine if the syringes caused adsorption of the target compounds which would be particularly evident at low concentration. The syringes were filled with 10 mL of 50/50 methanol/water and spiked with the target compounds at the LLOQ (5ng/L), surrogates at the midlevel concentration (80 ng/L), and 20 µL of ammonium hydroxide (pH 9 to 10). The syringes were capped and tumbled for one hour on a rotator. After one hour, the extraction solvent was acidified to pH 3 to 4 with acetic acid and a 1 mL aliquot was transferred to an autosampler vial for LC/MS/MS analysis. 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 are within acceptable limits.

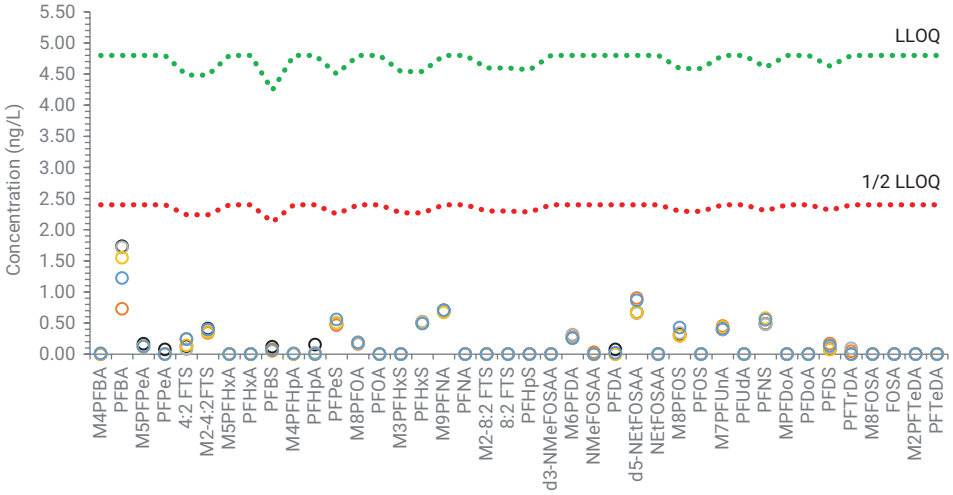


**Figure 1.** Background measurement for five replicate double blank measurements of Agilent Captiva disposable syringes. Dotted lines represent the LLOQ and blank limit threshold of ½ LLOQ.

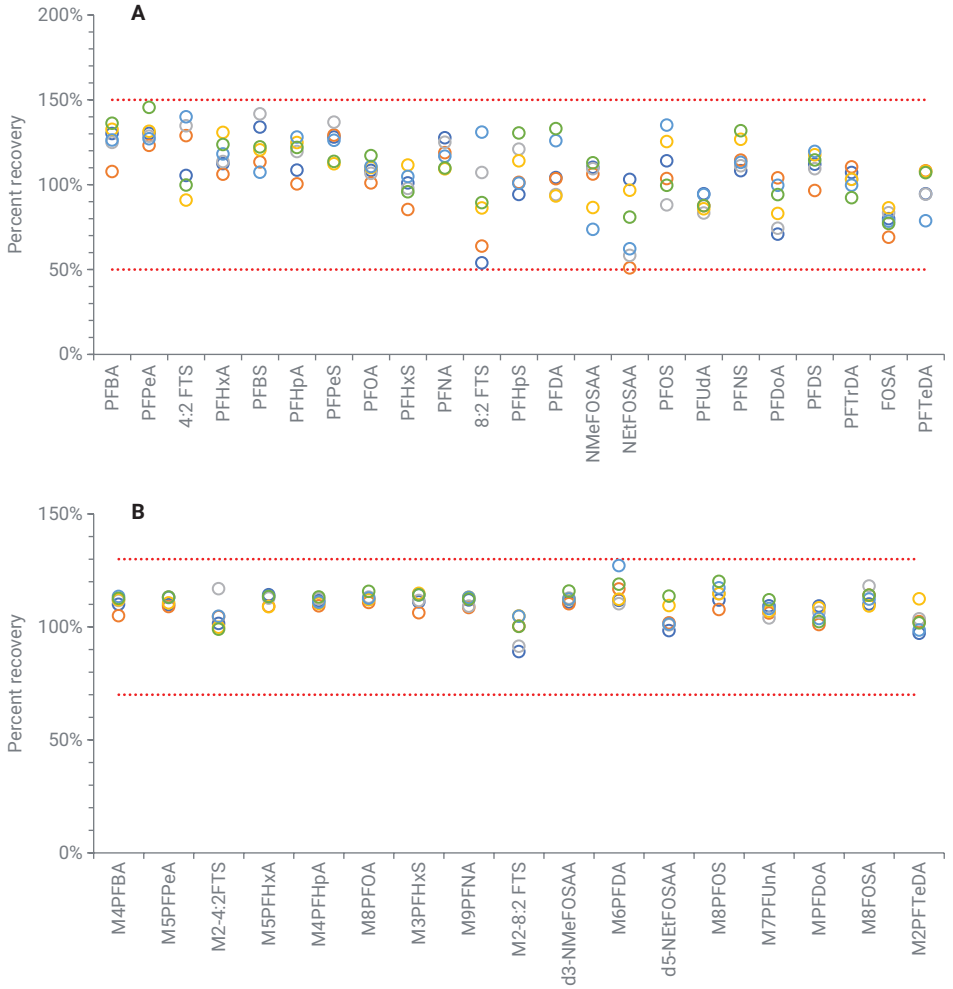


**Figure 2.** Agilent Captiva disposable syringe spike recoveries at the target LLOQ of 5 ng/L (A) and the surrogate midlevel at 80 ng/L (B).

## Conclusion

Agilent Captiva disposable syringes provide excellent performance for PFAS analysis. These syringes are free from background interferences and sorption losses. For the compounds studied, the syringes can be used without the extensive cleaning, resulting in time and cost savings in solvent use and solvent waste disposal.

## References


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Printed in the USA, August 6, 2020
5994-2247EN


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