

The Evolving Landscape of PFAS Detection, an Outline of Methods

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Introduction

Perfluoroalkyl substances are a group of man-made chemicals widely used in industrial applications and consumer products. Their widespread usage and resistance to degradation has resulted in PFAS being a ubiquitous environmental contaminant and its potential health effects are of growing concern. While many of the long-chain PFAS have been recognized as harmful, alternative compounds have emerged in their place. Short-chain PFAS compounds are less bio accumulative and therefore thought to be less toxic than long-chain PFAS compounds, but their widespread use has resulted in their increased environmental accumulation. In this work, several methods will be outlined to meet the evolving landscape of PFAS analysis. These methods include EPA methods 1633, 533, 8327, and 537.1 as well as a method for the analysis of ultra-short through short-chain PFAS(C1-C4).

EPA 1633 Aqueous, Solid, Biosolids, and Tissue Samples

In EPA 1633 the potential interference from bile acids to PFOS is outlined along with guidelines to follow for the analysis of 40 PFAS compounds. This potential interference from the bile acids to PFOS is affected by the organic modifier used in the analytical method. The document states that when using acetonitrile as the organic modifier, the bile acid taurodeoxycholic acid (TDCA) needs to be monitored and resolved with at least one minute or greater retention time difference between it and PFOS. If using any other organic modifier, such as methanol, it is necessary to also include taurochenodeoxycholic acid (TCDCA) and tauroursodeoxycholic acid (TUDCA) and achieve one minute or greater retention time difference between PFOS and all three bile acids. In addition to the retention time requirement, the bile acids may also not coelute with any of the PFAS analytes. A method was developed to meet these guidelines using acetonitrile.

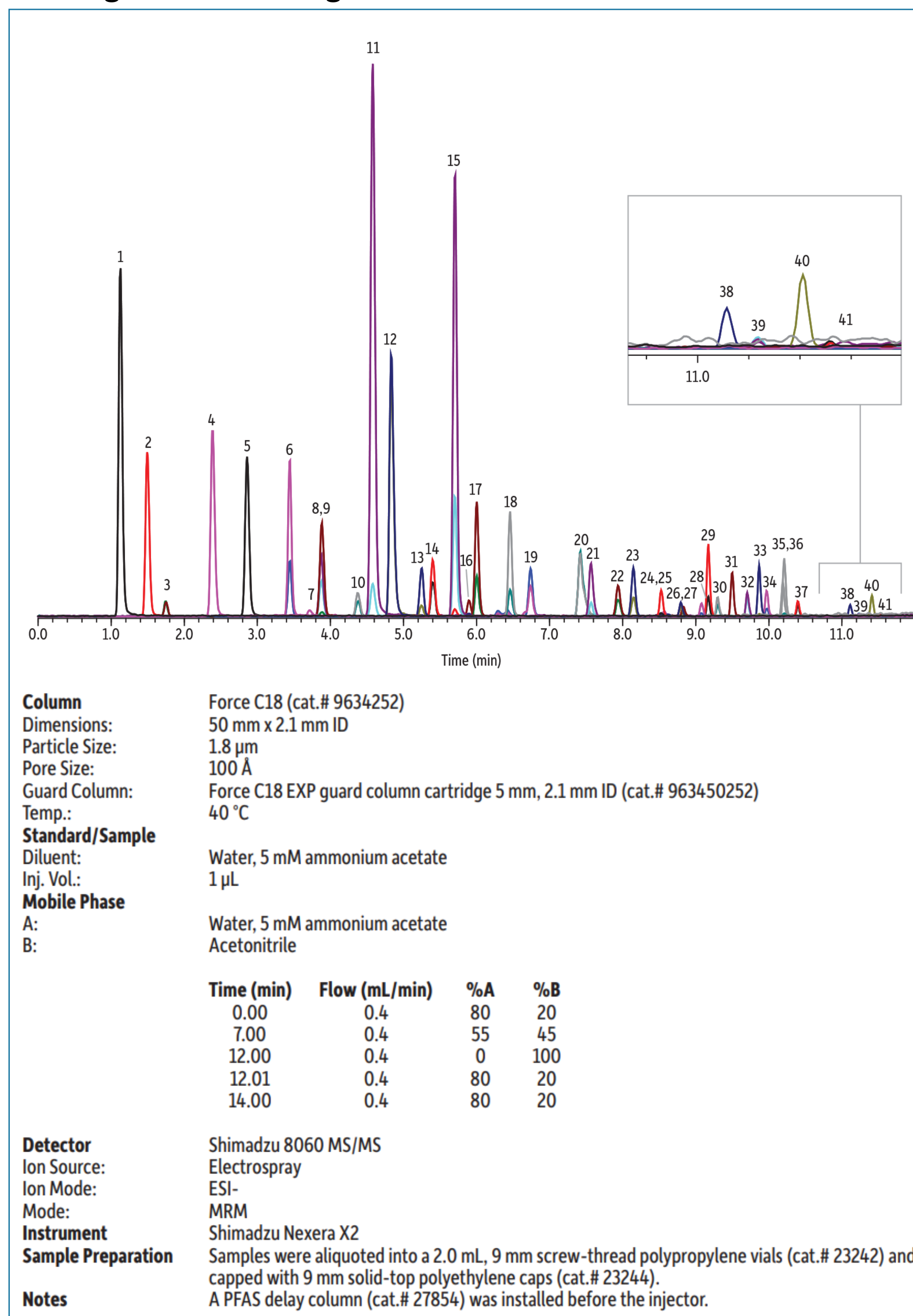


Figure 1: Chromatogram and method conditions for EPA 1633 using acetonitrile to resolve the matrix interference TDCA (peak 16) and PFOS (peak 26) with >1 minute retention using a Force C18 column.

EPA 537.1 Drinking Water

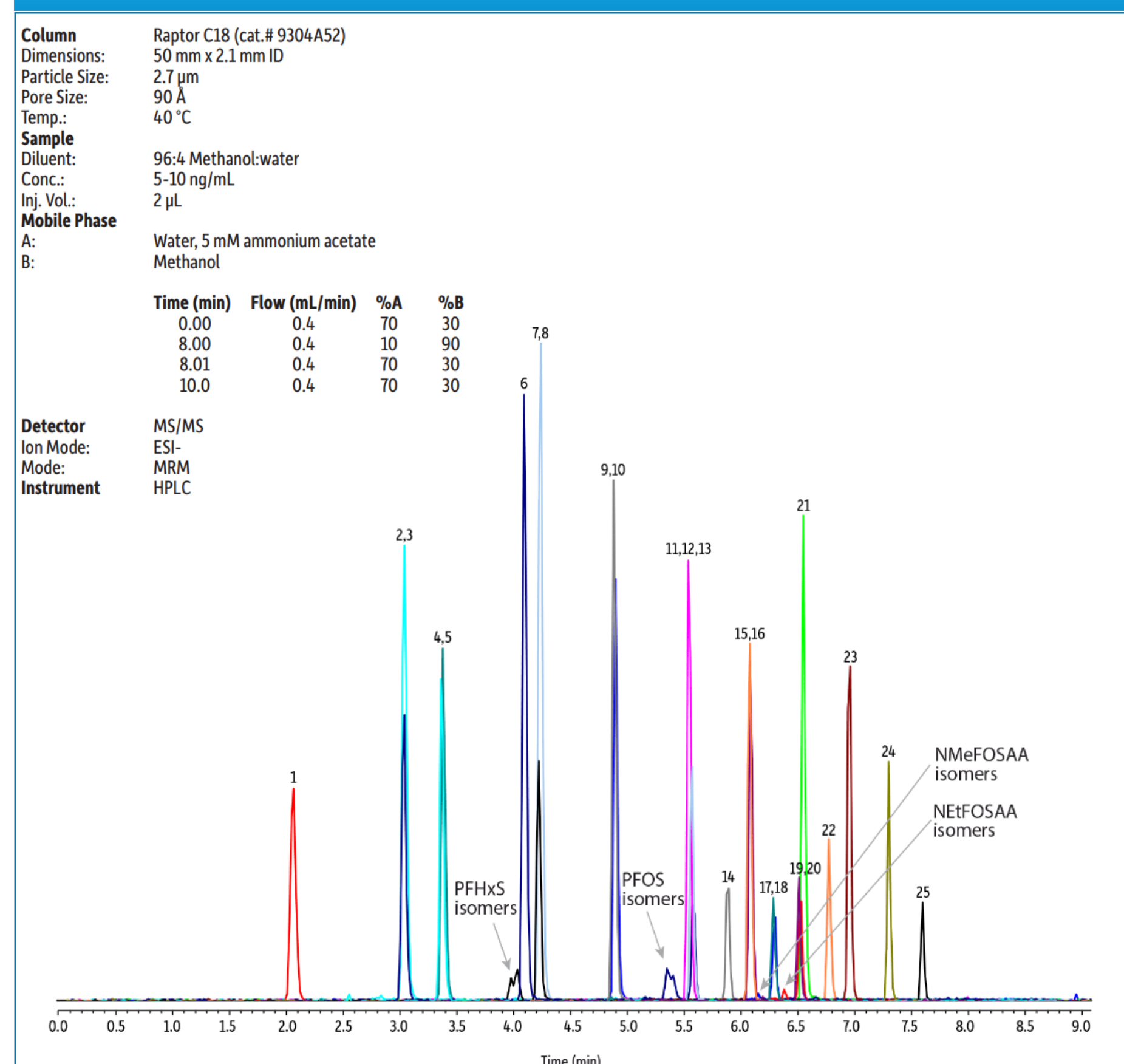


Figure 2: Chromatogram and method conditions for EPA 537.1 using Restek standard Cat# 30735 on a Raptor C18 column.

EPA 533 Drinking Water

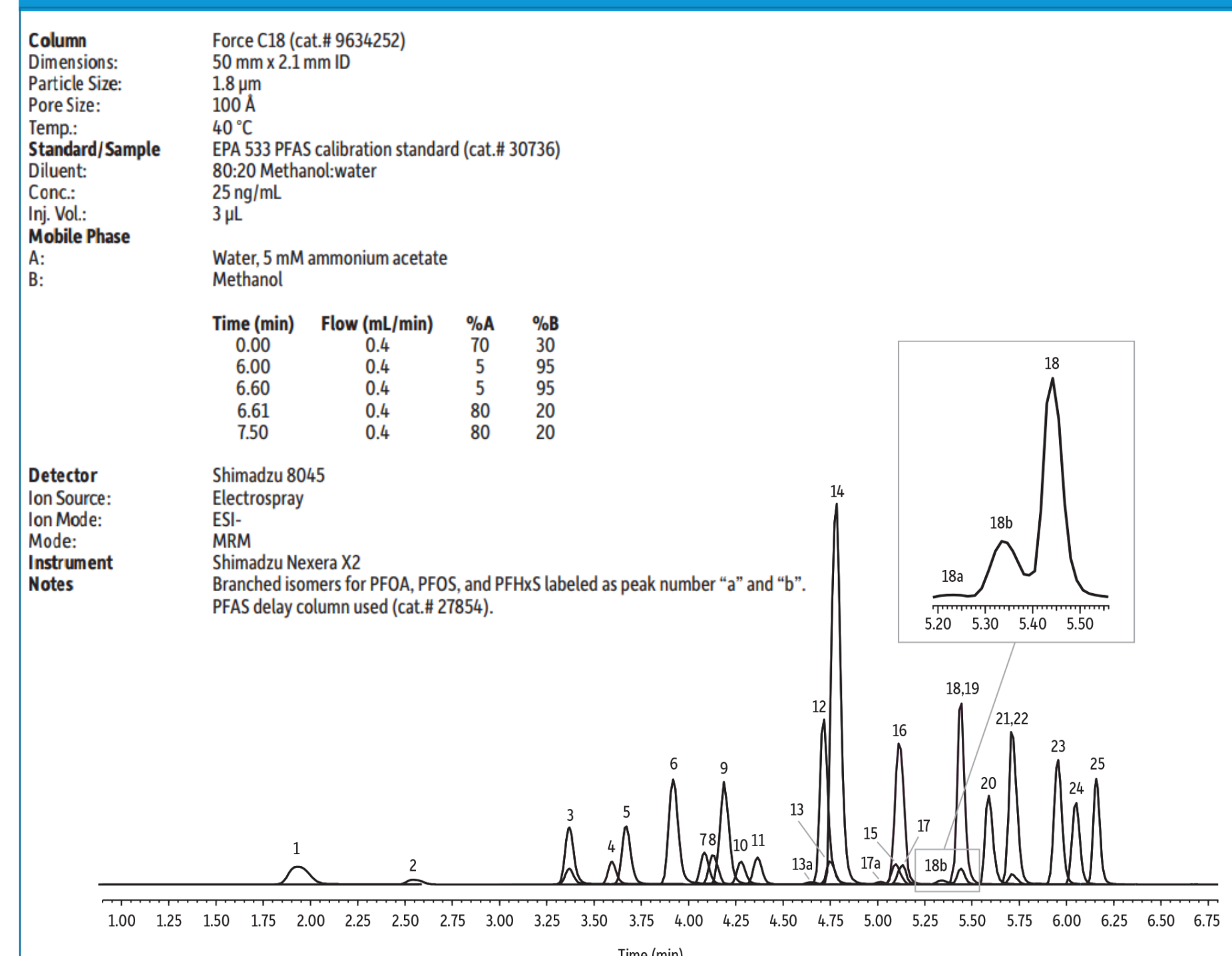


Figure 3: Chromatogram and method conditions for EPA 533 using Restek standard Cat#30736 on a Force C18 column.

EPA 8327 Surface Water, Groundwater, & Wastewater

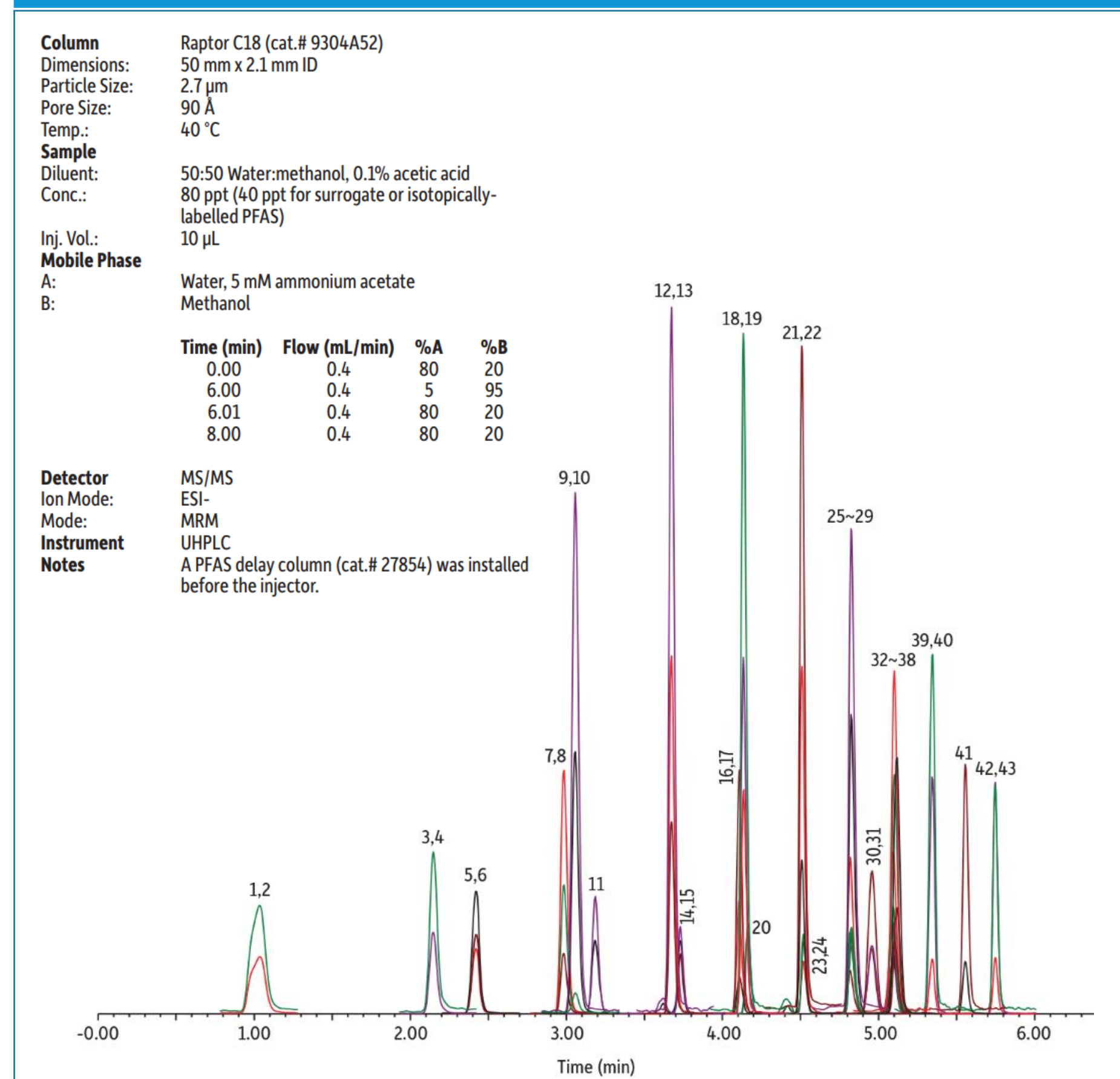


Figure 4: Chromatogram and method conditions for EPA 8327 by direct injection. Restek standard Cat# 30733 for 24 analytes and Cat#30734 for 28 analytes can be used and analyzed on a Raptor C18 column.

Ultrashort Through Short-chain (C1-C4)

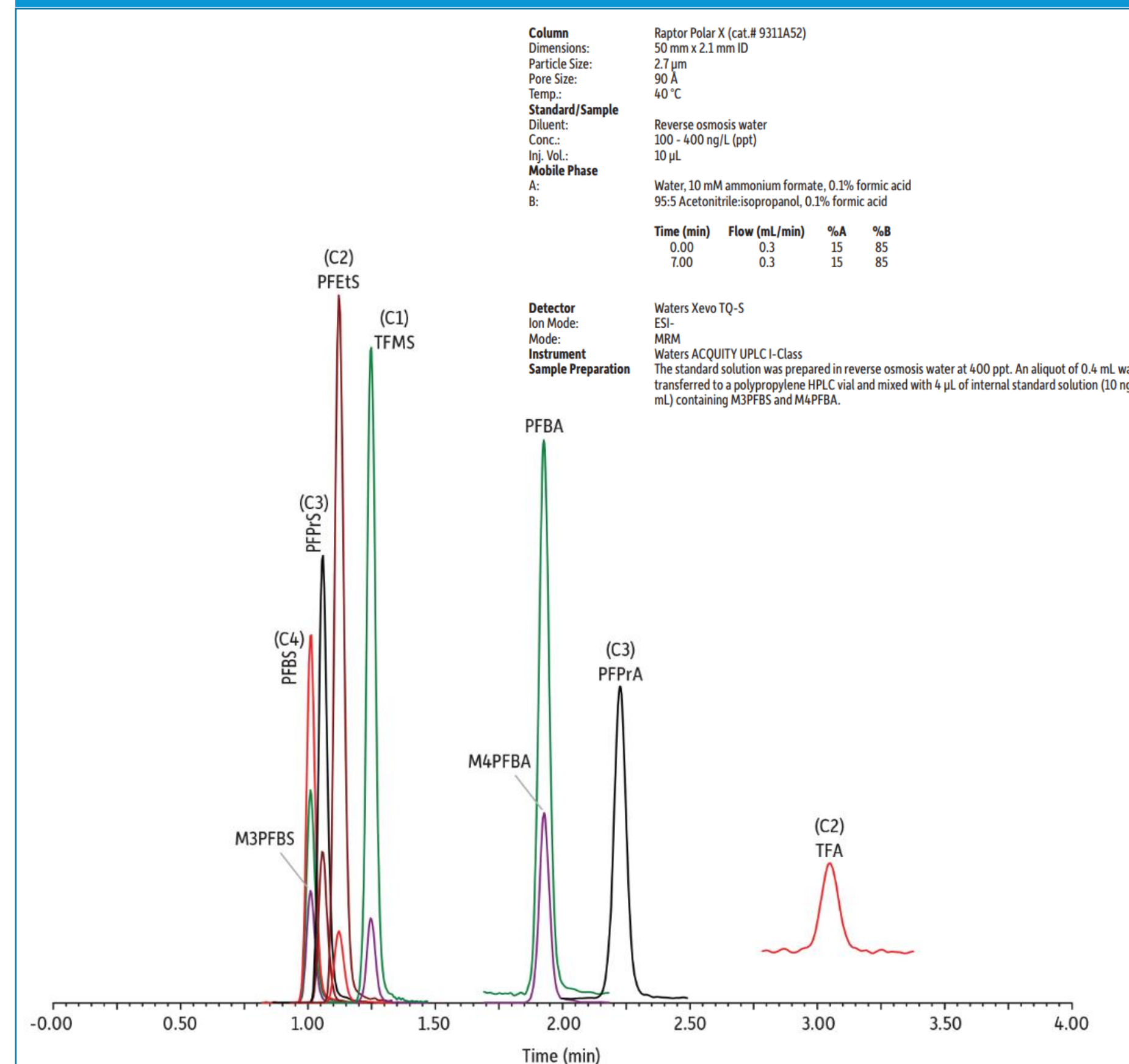


Figure 5: Chromatogram and method conditions for C1-C4 PFAS using a unique hybrid HILIC/ion-exchange stationary phase, Polar X.

Ultrashort Through Long-chain (C2-C8)

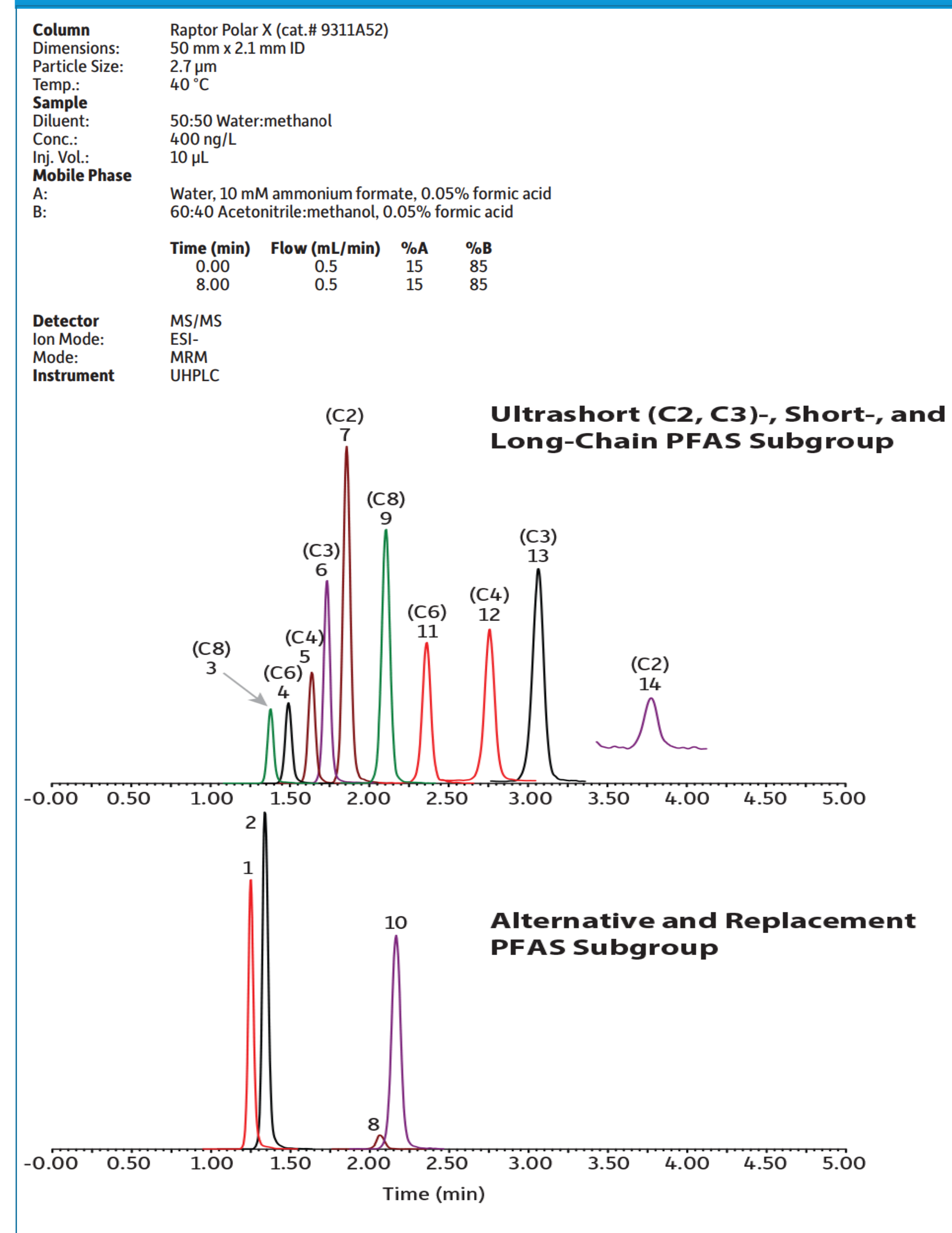


Figure 6: Chromatogram and method conditions for ultrashort, short, long, alternative, and replacement PFAS using a unique hybrid HILIC/ion-exchange stationary phase, Polar X.

Conclusions

While the majority of PFAS methodologies utilize a C18 column, a unique hybrid HILIC/ion-exchange stationary phase, Polar X, can be useful for the analysis of C2-C8 compounds. When using a C18 phase, a PFAS delay column needs to be installed before the injector to retain contaminants from the mobile phase and the system. For the analysis of ultrashort and short chain PFAS (C1-C4), the Polar X column can be used with isocratic conditions using water containing 10 mM ammonium formate and 0.1% formic acid as mobile phase A and 95:5 acetonitrile:isopropanol containing 0.1% formic acid as mobile phase B. If C2-C8 are the target analytes mobile phase B can be diluted with methanol (60:40 acetonitrile:methanol) to increase the retention of C6-C8.