

Perfluorinated chemicals (PFCs) are persistent, ubiquitous, and bioaccumulative in the environment, and are potentially harmful to human health. Because of their lipid and water repellent characteristics, they have been widely used in various products for more than fifty years. The processes of drinking water treatment are ineffective in removing these chemicals. Humans are primarily exposed to PFCs via drinking water, food and household dust.

This study developed a method to determine 10 PFCs in drinking water, milk, fish, beef and liver by automated solid-phase extraction (SPE) and ultra-high performance liquid chromatography/tandem mass spectrometry (UHPLC-MS/MS). The 10 PFCs were separated on a Kinetex C18 column (2.1 x 50 mm, 2.6 μ m) at 40°C and the flow rate was 0.9 mL/min; the total chromatographic time was 5.6 minutes. The mobile phase was composed of methanol and 10 mM *N*-methylmorpholine. Trace amount of perfluorohexanoic acid (PFHxA) (0.96 pg) and perfluorooctanoic acid (PFOA) (1.68 pg) was observed in backgrounds and the major contamination source was identified as the lines in the instrument of UHPLC-MS/MS.

Milk was digested with 0.5-N potassium hydroxide in Milli-Q water, after adjusted to pH 3.5 and filtration, the sample was extracted with an Atlantic HLB disk by automated SPE. Drinking water (adjusted to pH 3.5) was directly extracted with the Atlantic HLB disk. Solid food samples were homogenized and digested by 0.5-N

potassium hydroxide in methanol for two hours. After centrifugation at 3,000 rpm (1,410 xg) for 30 minutes, the supernatant of the samples were diluted with 100-fold Milli-Q water then was extracted with the Atlantic HLB disk. After loading the samples, the disks were washed with 40% methanol/60% water, and then were eluted with 0.1% ammonium hydroxide in methanol. In the concentration step, perfluorooctanesulfonamide (PFOSA) and *N*-methylperfluorooctanesulfonamide (*N*-MeFOSA) were found to be evaporated when the eluent was concentrated to barely dry; therefore the extracts were only concentrated down to one milliliter.

Ion suppression of most analytes was below fifty percentages (-29–49%), and was generally lower in fish and drinking water but was higher in liver. Recoveries of sample preparation of most analytes were higher than 50% (52–121%) in five matrixes, but only small portions of PFOSA and *N*-MeFOSA remained after sample preparation (1.8–34%). The limits of detection (LODs) for most analytes were sub-ng/L (0.29–0.85 ng/L) in drinking water, and were from 1.8 to 11 ng/L in milk. LODs of most analytes were 0.15–0.50 ng/g net weight of solid food samples. LODs of PFOSA and *N*-MeFOSA were higher than other analytes because they suffered higher ion suppression and loss at the concentration step. This study compared the accuracy and precision in five matrixes between using two and six isotope-labeled internal standards to quantify the ten analytes. Quantitative accuracy and precision on almost all analytes

were better by using all the six internal standards than those using only two of them.

The method was simple, bettered the detection sensitivity, and saved time and labor.

Use of suitable isotope-labeled internal standards for each analyte was crucial for the quantitative precision and accuracy. This method can be applied to measure these chemicals in a variety of food samples.

Keyword: perfluorinated chemicals; UHPLC-MS/MS; solid-phase extraction; isotope-dilution techniques; drinking water; food