1 Introduction
Perfluorinated and polyfluorinated substances (PFAS) are synthetic compounds, widely used in industrial and consumer products. There is growing health concern to those chemicals due to their highly toxic properties. The widely used method for PFAS analysis is LC-MS/MS due to its high sensitivity and selectivity. Coupling SPE with LC-MS/MS is a popular approach and has been employed in EPA methods. Recently, a high throughput approach for preparing PFAS has been appreciated using direct injection without SPE, which not only achieves high sample throughput, reduces time and cost, but also maintains analyte loss and contamination from SPE processes, as demonstrated by this study using QqQ 480 mass spectrometer coupled with UPLC for trace PFAS analysis in drinking and surface water samples.

2 Experimental Conditions

2.1 Hardware and Software
Chromatography and mass spectrometer interfacing was conducted by a PerkinElmer QqQ 480 high-performance liquid chromatography (HPLC) system and the data was processed by MassHunter Workstation B.03.00. A triple-quadrupole mass spectrometer equipped with a dual detection source. All instrument control, data acquisition and data processing were performed using Simplex/LC²²² by Software.

2.2 Method Parameters

LC Method and MS Source Conditions

The LC method and MS source parameters are shown in Table 1. The LC gradient program is shown in Table 2. The LC method developed in this study, was used in a likely column to separate polar PFASs from the LC system. another was used in analytical column separation to separate polar PFASs. The multiple reaction monitoring mode (MRM) transition of PFAS and other optimized parameters are shown in Table 3. During method development, the source parameters for PFASs and the matrix-matched calibration standards from LC system and matrix-matched standards identified and separated from analysis peaks using a delay column. As shown in Figure 1, Analytical column was well separated within the same contaminant peaks. The data shown in Table 4, analytical column source parameters for PFASs is determined using MRM with the retention times and corresponding transition time windows for all PFASs.

2.3 Quality Control Sample Preparation

To test possible interferences or contamination from sample matrices and blanks, and to check the sample preparation procedures. An elution method Blank LFB was prepared for each method. The values of LFB should be close in size at or at least within the range of the values. For each analysis, sample blanks were prepared and processed in the same manner as the samples. Blank samples were prepared following the same water sample preparation procedure and was spiked with a known amount of analyte solution. During method validation, LFB samples were prepared using the LC analysis at three concentrations levels (LOQ, 10x LOQ and 1000x LOQ) respectively. To evaluate matrix effects and recovery from the real water sample matrices, a Laboratory Prepared Matrix Standard (LPM) sample was prepared per week (day). The LFB sample was prepared by spiking the same water sample preparation procedures spiked with a known amount of analyte. The matrix effect analysis was calculated by the percentage difference between the LFB and the matrix spike. To check the matrix effect/day, the recovery of a single sample was determined at the various matrix contamination levels from 10x LOQ, 100x LOQ, and 1000x LOQ. Quality Control Sample Preparation was prepared following the same water sample preparation procedure spiked with a known amount of analyte. The matrix effect analysis was calculated by the percentage difference between the LFB and the matrix spike. To check the matrix effect/day, the recovery of a single sample was determined at the various matrix contamination levels from 10x LOQ, 100x LOQ, and 1000x LOQ.

4 Results and Discussion

Compared with other LC-MS/MS methods, this LFC-MS/MS method has been developed and validated in the analysis of 17 PFASs in drinking water and surface water samples at sub-parts-per-trillion levels by coupling LC-QqQ 480 mass spectrometer equipped with UPLC technology.

The method has been applied for real water sample analysis with good accuracy and high sensitivity, and it showed a wide linear dynamic range and eliminated the HPLC sample preparation processes. Therefore not only the optimal and tested LC method for sample analysis, but also prevented potential contamination from PFAS sample preparation steps.

4.1 Standards, Solvents and Sample Preparation

Fourteen PFASs were obtained from Washington, D.C. and Florida, Ottawa, LC-MC grade methanol (MeOH) and water were obtained from Fisher Scientific. A mixture-addition standard solution was prepared at concentrations of 0.5, 1.0, 5.0, 10.0, 50.0, 100.0, 500.0, and 1000.0 mg/L (i.e., 5.0, 10.0, 50.0, 100.0, 500.0, and 1000.0 ng/L) in water. The water and solvent were purchased from Fisher Scientific. A mixture-addition standard solution was prepared at concentrations ranging from 0.5 to 1000.0 ng/L. A mixture of sample was put into a glass bottle with a known amount of matrix matrix contamination levels from 10x LOQ, 100x LOQ, and 1000x LOQ.

5 Summary

The developed LC-MS/MS method has been validated and validated in the analysis of 17 PFASs in drinking water and surface water samples at sub-parts-per-trillion levels by coupling LC-QqQ 480 mass spectrometer equipped with UPLC technology.

The method has been applied for real water sample analysis with good accuracy and high sensitivity, and it showed a wide linear dynamic range and eliminated the HPLC sample preparation processes. Therefore not only the optimal and tested LC method for sample analysis, but also prevented potential contamination from PFAS sample preparation steps.

References