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## DETECTION AND QUANTIFICATION OF PFAS CONTAMINATION IN WATER: ADVANCEMENTS IN LC-MS/MS METHODOLOGY

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### **Introduction**

PFAS are a group of synthetic compounds composed of carbon and fluorine atoms linked in a chain, widely used in the production of various industrial and household goods. They are classified into polymer and non-polymer PFAS, with the non-polymer ones further categorized as perfluoroalkyl carboxylates (PFCA) and perfluoroalkyl sulfonates (PFSA). PFAS have raised concerns due to their presence in the environment and the potential health risks they pose. Recent regulations aim to restrict and monitor the levels of PFAS in drinking water, with US EPA proposing MCLs for six PFAS including PFOA and PFOS. The EU and various countries have also implemented restrictions on PFAS, highlighting the global concern over these substances. To meet the new requirements imposed by the current legislation, a newly developed LC-MS/MS method for the detection of PFAS in water samples has shown promising results, with detection limits ranging from ultra-trace levels for drinking water to low ng/L levels for surface water and wastewater. The method has proven effective in isolating and concentrating the target analytes from water matrices. This study contributes to the ongoing efforts to monitor and regulate PFAS contamination in water sources, showcasing the importance of addressing these emerging contaminants for environmental and public health protection.

### **Materials and methods**

High-purity methanol (LC-MS grade) was sourced from Merck (Darmstadt, Germany). Ammonium acetate was acquired from Sigma-Aldrich (Germany). The perfluoroalkyl acid (PFAS) standards were purchased from Sigma-Aldrich (Germany): perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorooctanoic acid (PFOA), perfluorooctansulfonate acid (PFOS), perfluorononanoic acid (PFNA), perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFNuDA). Labeled internal standard (IS) perfluorooctanoic acid 13C8 (PFOA-13C8) was also acquired from Sigma-Aldrich (Germany). Polymeric weak-anion solid phase extraction cartridges (500 mg, 6 mL) were obtained from Phenomenex. Ultra-pure Milli-Q water was utilized as the solvent.

Drinking water samples were collected from the drinking water distribution system in various locations throughout Bucharest, thereby facilitating the evaluation of the presence and concentration of PFAS compounds in the water supply.

A liquid chromatography-mass spectrometry (LC-MS/MS) system was used to quantify PFAS. The system consisted of an Agilent 1260 series LC system and an Agilent 6410B triple-quadrupole mass spectrometer. The chromatographic column was a Zorbax Eclipse C18 (2.1 × 100 mm, 3.5 μm) maintained at 30°C. The mobile phase gradient was composed of five mM ammonium acetate and methanol (60/40, v/v) at a flow rate of 0.200 mL/min. Multiple reaction monitoring (MRM) was used to detect PFAS. The gas flow was set at 8 L/min and the capillary voltage at 2500 V.

### Results and conclusions

The analysis of 11 per- and poly-fluoroalkyl substances (PFAS) in water samples collected from the distribution network revealed the presence of 9 compounds above the quantification limit of the method (Fig. 1). The detection frequency of PFOA was 100%, while PFHpA and PFHxA were detected at frequencies of 91% and 73%, respectively. PFBA, PFPeA, and PFNA were detected at a frequency of 45%, and PFOS was detected at a frequency of 36%. PFOA was found to be the most abundant compound, with concentrations ranging from 3.65 to 23.9 ng/L. The other 8 compounds were identified with values approximately one order of magnitude lower than those of PFOA and PFHpA, with concentrations ranging from 0.5 to 1.72 ng/L. The total sum of the 9 PFAS compounds was found to range from 5.4 to 26.8 ng/L, with a median value of 21.8 ng/L.

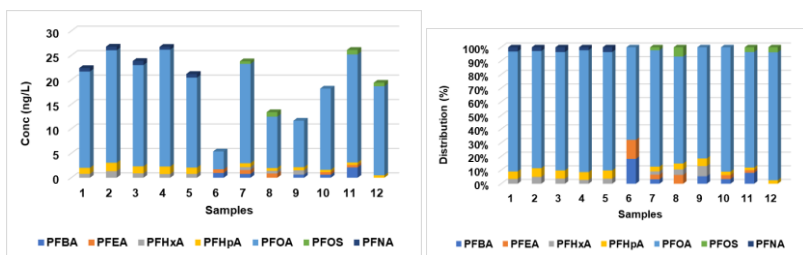


Figure 1. PFAS concentration (a) and percentual distribution (b) in drinking water samples

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