SUMMARY

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BUDAPEST

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Title : Investigation of mobile phase additives in HPLC-MS/MS method used for the determination of PFAS in drinking water

This thesis work discusses mobile phase additives in order to investigate the signal of PFAS compounds. Per- and poly-fluoroalkyl substances (PFAS) are a large group of environmental pollutants. They are widespread throughout the world's ecosystem, including in animals and humans and also can be found in drinking water. The new limit has been established by Drinking Water Directive in 2021 in the EU for a 'Sum of PFAS' of 0.1 μg/L, which limit value must be measured by all member states from 2026. In order to make the compounds measurable with HPLC- MS/MS method with as low a concentration as possible, the aim of this work was to examine the effect of different mobile phase additives on signal generation of multi-PFAS standard solution. The methodology was based on tandem mass spectrometry (MS/MS) and ultrahigh-pressure liquid chromatography (UHPLC) methods.

In a preliminary experiment, two substances—PFAO and hexafluoropropylene oxide-dimer acid, also referred to as GenX—were tested first before the multi-standard PFAS solution was used. The selection of multi-standard PFAS (18 compounds) was based on the United Environmental Protection Agency's (EPA) Method 537.1: Determination of Selected PFAS in Drinking Water by Solid Phase Extraction (SPE) and LC/MS/MS (2018/2020). The investigation involved Flow Injection Analyses (FIA) with no column and a slow flow rate. The analyser was applied in selected ion monitoring (SIM) mode measuring the precursor ions. Besides that, chromatographic analyses with column installed was also used, and the separation and detection were done in optimized with multiple reaction monitoring (MRM) mode.

This study involved a series of eluents with buffer salts including the different concentrations of ammonium acetate and ammonium hydrogen carbonate. The suggestion about ammonium

hydrogen carbonate was based on the research of Lauren Mullin et al., 2018. Additionally, the effect of mobile phase pH on signal generation was also investigated.

The results show that the addition of ammonium hydrogen carbonate and acetic acid to the mobile phase significantly improves the detection of PFASs in negative ion mode. The best mobile phase additive for detecting PFASs using UHPLC/MS/MS in negative ion mode was a combination of (eluent A) 4mM ammonium hydrogen carbonate and 0.01% acetic acid and (eluent B) MeOH.

As a continuation of the work, by transforming the developed method into a dynamic MRM method, we can measure a multi-PFAS solution with a concentration of 1ng/ml with the appropriate intensity (Figure 1)

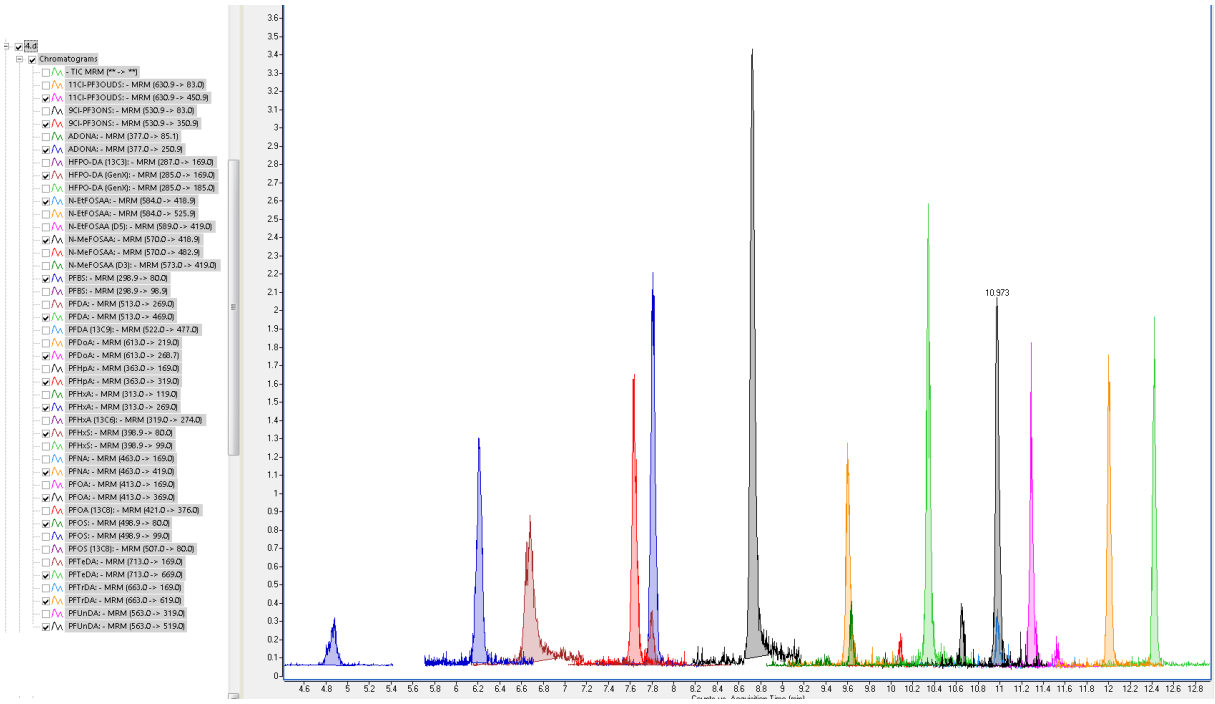

Figure 1. : Overlayed total ion chromatogram of 1 ng/mL multi-PFAS standard solution using (A) 4mM ammonium hydrogen carbonate (ELU_B) with 0.01% AA and (B) MeOH mobile phase in dMRM measuring mode (only the quantitative transitions are illustrated)