



APPLICATION NOTE

EPA Method 1621 for Adsorbable Organic Fluorine using Metrohm's Profiler^F Analyzer for AOF

Optimized for analysis of organically bound fluorine

Per and Polyfluoroalkyl Substances (PFAS) are synthetic compounds that were created to improve the quality of industrial products. These chemicals are used for a wide range of consumables including plastics, non-stick cookware and even cosmetics. While their properties proved to be beneficial, they have become of great concern as they are slow to degrade and can remain in the environment for long periods of time. In addition, they have been shown to be a human health risk, having carcinogenic properties even at low levels.

While targeted analyses of individual PFAS compounds can provide high sensitivity and specificity, non-targeted

screening for organically bound fluorine can provide a better overall picture of the full scope of contamination from the thousands of potential PFAS and other fluorinated organic compounds.

This application highlights the Environmental Protection Agency's (EPA) non-targeted screening method for organofluoride compounds in water as adsorbable organic fluorine (AOF), using combustion ion

chromatography (CIC). Effective sample preparation and the suitability of Metrohm's Profiler^F Analyzer for AOF for use with EPA Method 1621 is demonstrated herein.

SAMPLE AND SAMPLE PREPARATION

Ultrapure water and surface water are spiked with sodium perfluoro-hexane sulfonate (PFHxS). Sample preparation is done as specified by EPA 1621. 100 mL of sample is concentrated on 100 mg of granular activated carbon (GAC) with the use of the APU sim (Fig.1). GAC is then rinsed with sodium nitrate and transferred to a ceramic combustion boat. The boat is then taken to the combustion oven by way of the auto boat driver where the sample is combusted at 1050°C. The analysis of replicate samples is used to perform an MDL study, and to determine the precision and accuracy capabilities of the instrument as it relates to EPA 1621.

EXPERIMENTAL

This analysis is performed by using the Metrohm Profiler^F Analyzer for AOF, a combustion ion chromatography system optimized for fluorine analysis (Fig.2). Using an eluent concentration of 3.2 mM sodium carbonate and 1.0 mM sodium bicarbonate, and 1 mL injection volume, samples are separated with the Metrosep A Supp 7 150/4.0 column connected to the Metrosep A Supp 5/4.0 guard. This allows for anion detection of peaks with sequentially suppressed conductivity detection and quantification at a calibration range of 0.5 to 50 ppb. All instrument control and data processing are performed using MagIC Net 4.0 software.



Figure 1. Analytik Jena APU Sim.

RESULTS

Data obtained was compared to the acceptance criteria proposed in EPA 1621 (% RSD <20%, recovery of reagent blank 70-130% and matrix spike 50 to 150%). UPW is spiked at 5 ppb and 25 ppb, while surface water was spiked at 10 ppb (Figure 3). The results for all blank reagent and sample spike fell within the required range (Table 1). The MDL study was performed by using seven replicates of both UPW and 5 ppb UPW spiked. The MDL for both was then calculated, and the highest number assigned as the MDL of system; this was found to be MDL blank at 1.36 ppb (Table 2). To ensure adequate quantification of data, an eight-point calibration curve was generated to have a quadratic fit with a %RSD of 1.14% and a correlation coefficient of 0.999 (Figure 4). Following every ten samples, calibration verifications were analyzed using two check standards at 2.5 and 10 ppb. Standards had recoveries within the required 80 to 120%, indicating acceptable performance of the instrument throughout testing.

Table 1. Results for replicate samples and spiked

Sample	Conc. (ppb)	% RSD	% Recovery
UPW	0.92	22.5	-
5 ppb UPW	4.48	4.3	90
25 ppb UPW	21.03	1.8	83
Surface Water	1.82	5.2	-
10 ppb Surface water	10.11	4.2	83

Table 2. Retention time of AOF, spike for MDL sample determination and calculated MDL blank based on seven replicates.

Analyte	Retention Time (min)	Spike Conc. (ppb)	MDL (ppb)
AOF	4.45	5.0	1.36

CONCLUSION

With increased concern over the environmental and health risks of PFAS compounds, accurate monitoring of these compounds is essential. The Profiler^F Analyzer for AOF can reliably test non-targeted adsorbable organic fluorine (AOF) as per EPA 1621. This can be done with increased sensitivity down to an MDL of 1.36 ppb, while demonstrating repeatability and accuracy to meet the proposed requirements of EPA Method 1621.



Figure 2. Profiler^F Analyzer for AOF.

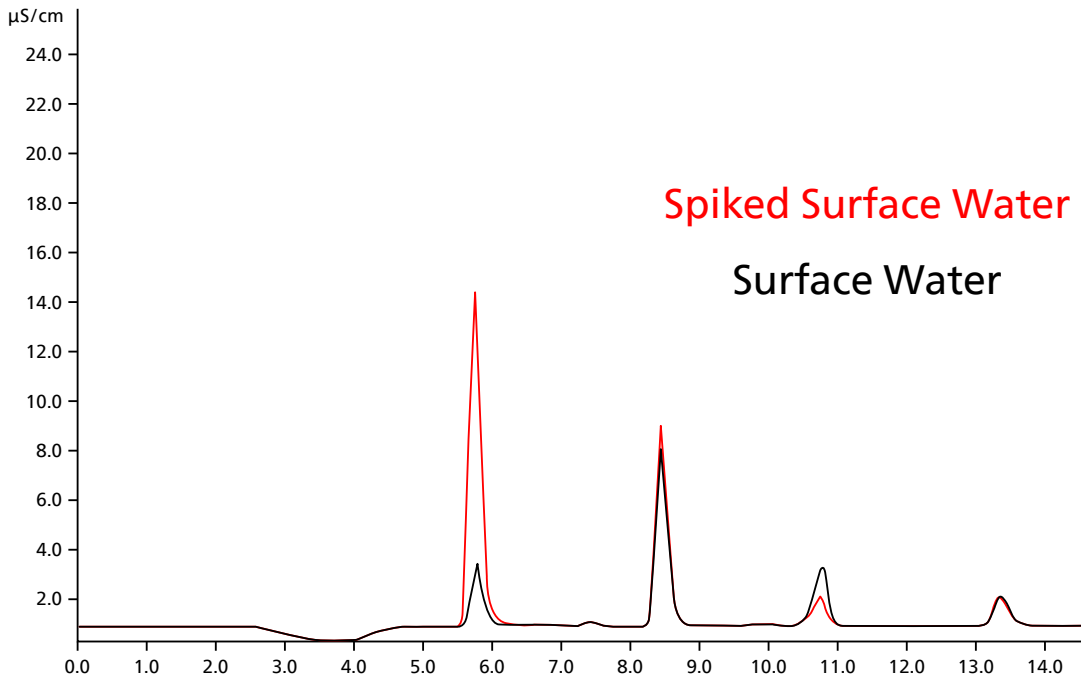


Figure 3. Chromatograms of surface water and surface water spiked- 100mL AOF sample size, 2 x 50mg carbon tube

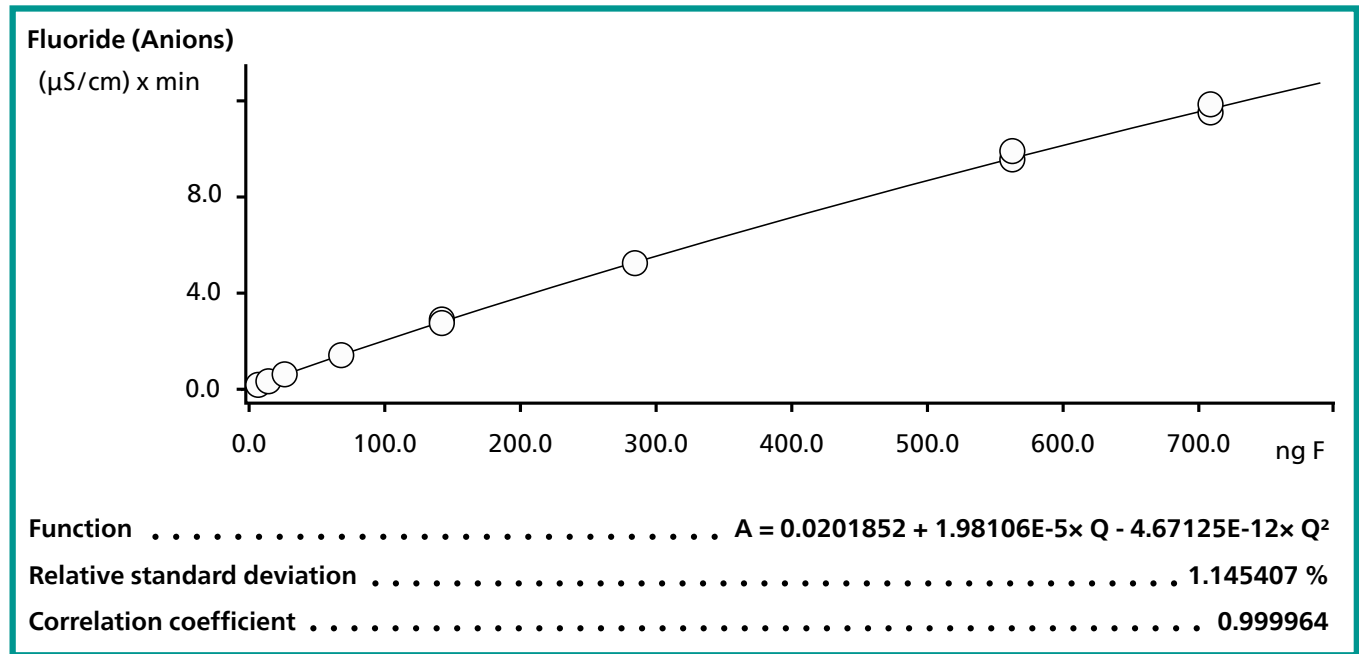


Figure 4. Calibration curve

Analytes:	Fluorine
Matrix:	Water
Method:	Ion chromatography
Industry:	Water
Standards:	USEPA 1621