APPLICATION NOTE

Determination of Total Fluorine in Carpet Samples Using Combustion Ion Chromatography

Monitoring PFAS and PFAA in carpet samples

Carpets are widely used all over the world to cover and protect the flooring of residential homes, commercial buildings and vehicles. With continuous use over the years, carpets provide comfort and beauty by way of décor. As carpets are frequently used in high traffic areas, it is essential that manufacturers provide a product that is rugged, often adding features like stain resistance or waterproofing. These features are achieved with the use of chemicals, specifically Per and polyfluoroalkyl substances (PFAS). While they help to maintain the lifetime of carpets, their use in the manufacture, and eventual disposal of these products have proven hazardous to human health and the environment.

Being very stable, PFAS compounds remain in the environment for long periods of time and can enter the food supply and water ways. Therefore, it is important that these chemicals be monitored. This application note highlights the non-targeted analysis of the sum of inorganic fluoride and organic fluorine in carpet samples with the use of Metrohm combustion ion chromatography.

Metrohm

SAMPLE AND SAMPLE PREPARATION

Samples are prepared by using various pieces of each carpet sample. Weighed samples of the carpet fiber, carpet backing and carpet fiber with backing are analyzed at a target mass of 25 to 35 mg. The fibers are placed into quartz sacrificial vials before combustion while the backings and fiber/backing combinations are weighed directly onto the boats and combusted (Figure 1).

Figure 1. Sample preparation of carpet samples.

EXPERIMENTAL

This analysis is performed by using a Metrohm combustion ion chromatography system. With fluoride being the target analyte and the high concentration of sulfate in samples, this application runs without using matrix elimination (Fig. 2). Using an eluent concentration of 3.2 mM sodium carbonate and 1.0 mM sodium bicarbonate, and 200 µL injection volume, samples are separated with the Metrosep A Supp 5 150/4.0 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 an instrument calibration range of 0.1-5 ppm. The on-column result is then used to calculate the final concentration of fluoride in samples using Equation 1. All instrument control and data processing are performed using MagIC Net 4.0 software.

$$\text{Conc in Sample (mg/kg)} = \frac{\textit{Conc from IC}\left(\frac{mg}{L}\right) \times \left(\frac{1L}{1000mL}\right) \times \textit{Total Vol abs soln (mL)}}{\textit{Sample aliquot mass (mg)}} \times (1{,}000{,}000\ mg/1kg)$$

$\textit{Conc from IC}\left(\frac{mg}{L}\right)$: Off column concentration

$\textit{Total Vol abs soln (mL)}$: Total Absorber solution (Absorber holds 2-12 mL)

$\textit{Sample aliquot mass (mg)}$: Mass of sample combusted in mg

Equation 1. Calculation of total concentration in sample

RESULTS

Data obtained confirmed the presence of fluorine in the samples (Fig 3). Replicate measurements demonstrated an RSD of ≤ 7% for fibers only and backing only. Carpet fibers and backing combined showed ≤ 5% RSD with a single outlier (Table 1). Sample homogenization may improve instances of higher sample to sample variation. Samples were quantified based on a 6-point, quadratic curve that had a relative standard deviation of 1.01 % and correlation coefficient of 0.9999 indicating very little deviation of the points from the curve and good response to the concentration introduced to the system (Figure 4). Potassium nonfluoro-1-butanesulfonate was used as a check standard with 100% recovery.

Table 1. Data representing analysis of triplicate sample runs

Sample	Fibers		Backing		Fibers+ Backing	
	F		F		F	
	Avg (ppm)	RSD%	Avg (ppm)	RSD%	Avg (ppm)	RSD%
1	385.3	4.9	117.9	3.5	200.2	4.9
2	1196.8	4.0	118.6	3.0	415.5	4.1
3	504.9	6.8	135.5	6.8	183.3	23

CONCLUSION

With the prevalent use of fluorinated compounds in the carpet industry, monitoring these compounds is essential for protecting human health and the environment. The Metrohm combustion ion chromatography system has been demonstrated as an effective tool for non-targeted analysis of fluorine in carpet samples. This application note demonstrated acceptable performance of the instrument through 100% recovery of check standard and repeatability of triplicate runs, while highlighting the importance of homogenous sample preparation.

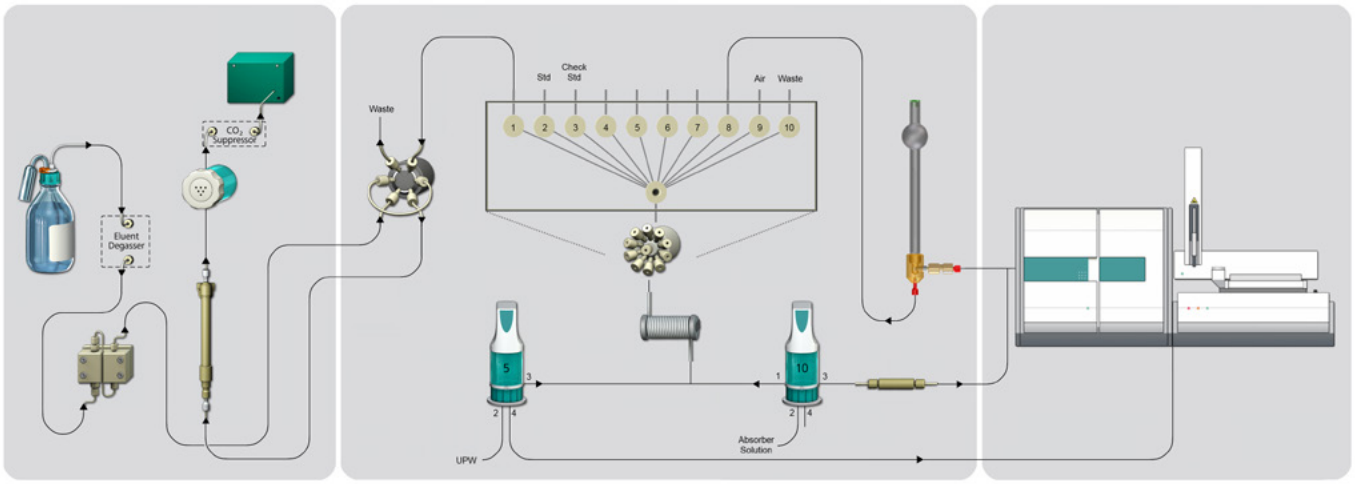

Figure 2. Schematic of standard combustion ion chromatography with matrix elimination out of line.

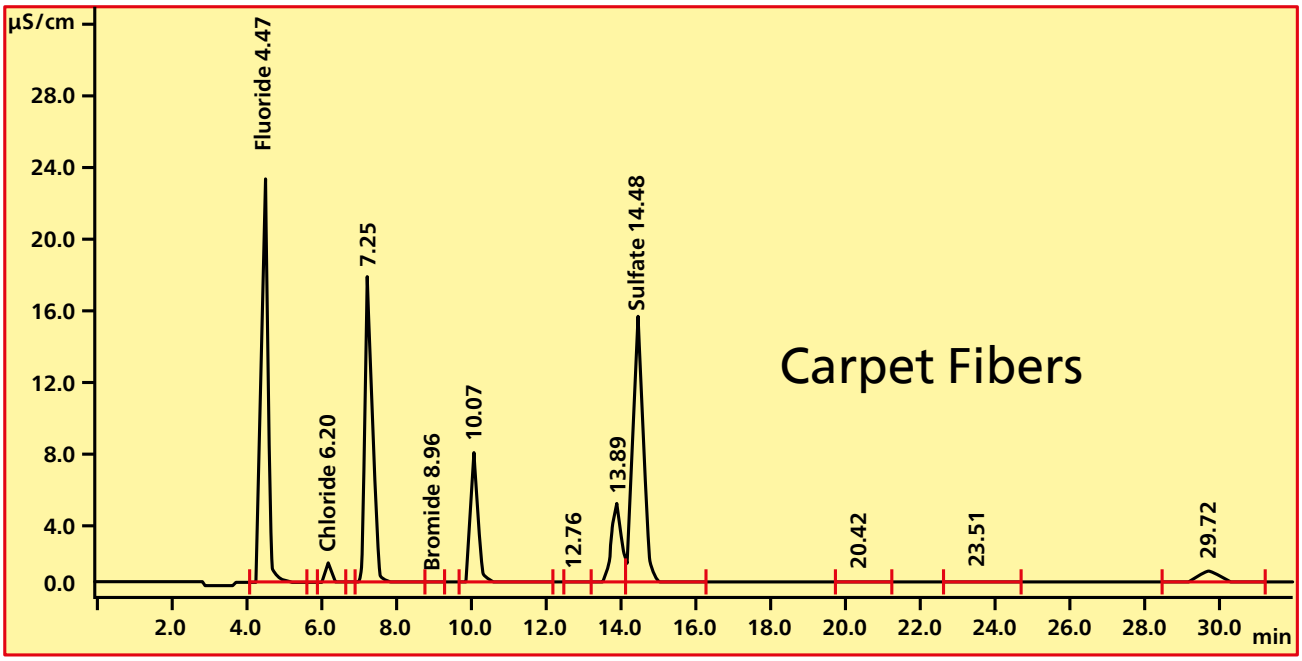

Figure 3. Chromatogram of carpet fiber sample

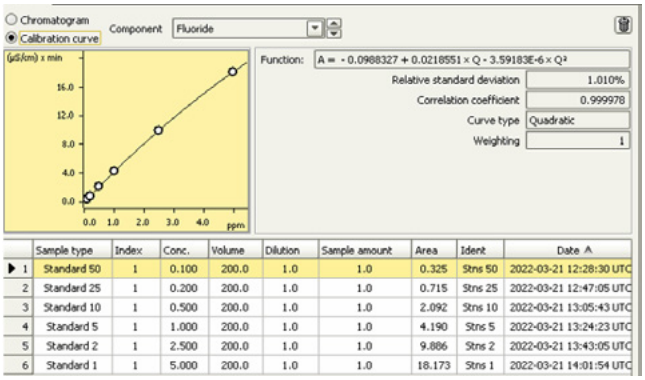

Figure 4. Calibration Curve