



APPLICATION NOTE

Determination of Total Fluorine & Chlorine in Food Packaging using Combustion Ion Chromatography

Cardstock, cardboard, and epoxy coating

Per and Polyfluoroalkyl Substances (PFAS) are synthetic compounds that were created to improve the quality of industrial products but have since become an environmental and health concern. One use of PFAS is in the grease proofing of food packaging material. This can range from fast-food wrappers and take-out paperboard containers to pet food bags. With the Food and Drug Administration banning PFAS used in grease proofing of food packaging in the United States and California and limiting the amount of PFAS intentionally used in food

packaging, it is essential that we monitor for these compounds to ensure safety of consumers. In addition to monitoring for total fluorine, total chlorine is also often monitored as this is a corrosive and can lead to long term issues with production processes.

This application explores the testing of various pieces of food packaging and the epoxy used to coat food packaging for total fluorine and chlorine using the Metrosep A Supp 19 – 150/4.0 column. Samples were weighed into ceramic boats, combusted and analyzed by IC using sequentially suppressed conductivity detection.

EXPERIMENTAL

This analysis is performed using a Metrohm combustion ion chromatography system, with fluoride and chloride being the target analytes. This application was run without using matrix elimination. Samples were weighed into ceramic boats and introduced into the combustion oven where they were combusted at 1050°C. The post combustion gases are then condensed and collected in 7 mL of absorber solution in a 920 absorber module. 1000 µL of absorber solution is injected into a 940 Professional IC, where separation takes place using A Supp 19 150/4.0 analytical and A Supp 19/4.0 guard columns under standard eluent conditions (8 mM sodium carbonate/0.25 mM sodium bicarbonate). Peaks are detected and quantified using sequentially suppressed conductivity detection. The on-column result is then used to calculate the final concentration of fluoride and chloride in samples using Equation 1. All instrument control and data processing are performed using MagIC Net 4.2 software.

SAMPLE AND STANDARD PREPARATION

Approximately 30-60 mg of samples were weighed into ceramic boats with quartz wool to aid in combustion. Standards were prepared from a single stock of fluoride and chloride and calibration was done through the IC (Fig 1).

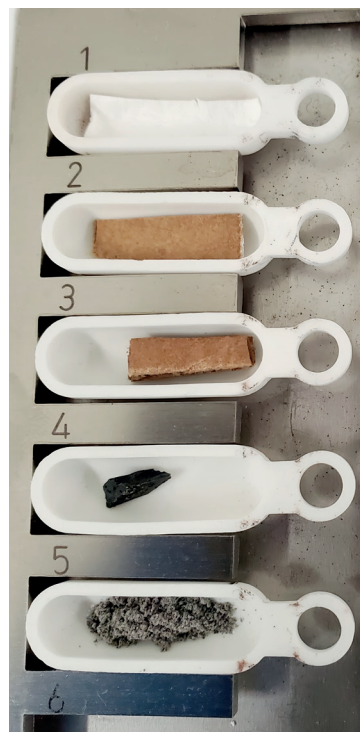


Figure 1: Samples loaded into ceramic boats

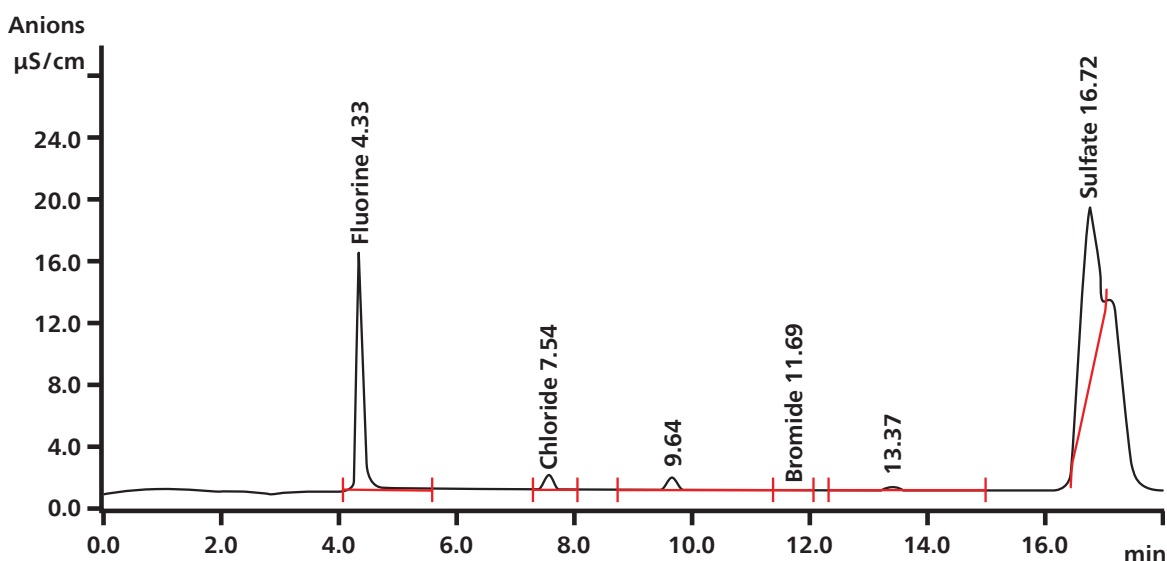


Figure 2: Cardstock treated with PFAS

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

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

Total Vol abs soln (mL) : Total Absorber solution (Absorber holds 2-12 mL)

Sample aliquot mass (mg) : Mass of sample combusted in mg

Equation 1: Calculation of total concentration in sample

RESULTS

Standards for all analytes tested generated a quadratic curve with relative standard deviation less than or equal to 5% and a correlation coefficient of 0.999. To ensure that sample data was accurate, two calibration curves were used, with low and high ranges. Samples were run in triplicate demonstrating good repeatability of fluoride and chloride measurements. The results also show comparable data for epoxy that was cryoground vs combusted as pieces, indicating very little need for sample preparation prior to combustion. Check standards were analyzed using PFBS and ERM 680M, which gave fluoride and chloride recoveries within the 80 – 120 % expected range, indicating accuracy of the method and instrument.

CONCLUSION

With the prevalent use of fluorinated compounds in the food packaging industry, monitoring these compounds is essential to protect human health and the environment. The Metrohm combustion ion chromatography system has been demonstrated as an effective tool for non-targeted analysis of total fluorine and chlorine in food packaging and the epoxy used to coat paper in these products. This application demonstrated acceptable performance of the instrument through excellent recovery of check standard and repeatability of sample triplicate runs, while also highlighting the importance and efficiency of combustion as a means of sample preparation.

Table 1: Calibration data

Standard	Range mg/L	RSD %	Correlation Coefficient
Fluoride (Low)	0.05-0.25	3.81	0.999
Chloride (Low)		2.09	0.999
Fluoride (High)	0.2-10	1.72	0.999
Chloride (High)		1.74	0.999

Table 2: Repeatability data

Sample (n=3)	F		CL	
	Avg (mg/kg)	RSD%	Avg (mg/L)	RSD%
Cardstock	3.6	3.14	-	-
Cardstock (Treated)	132.8	5.96	-	-
Cardboard	8.37	4.20	-	-
Epoxy	0.6	0	334.4	0.5
Epoxy (Ground)	0.7	7.5	333.4	1.13

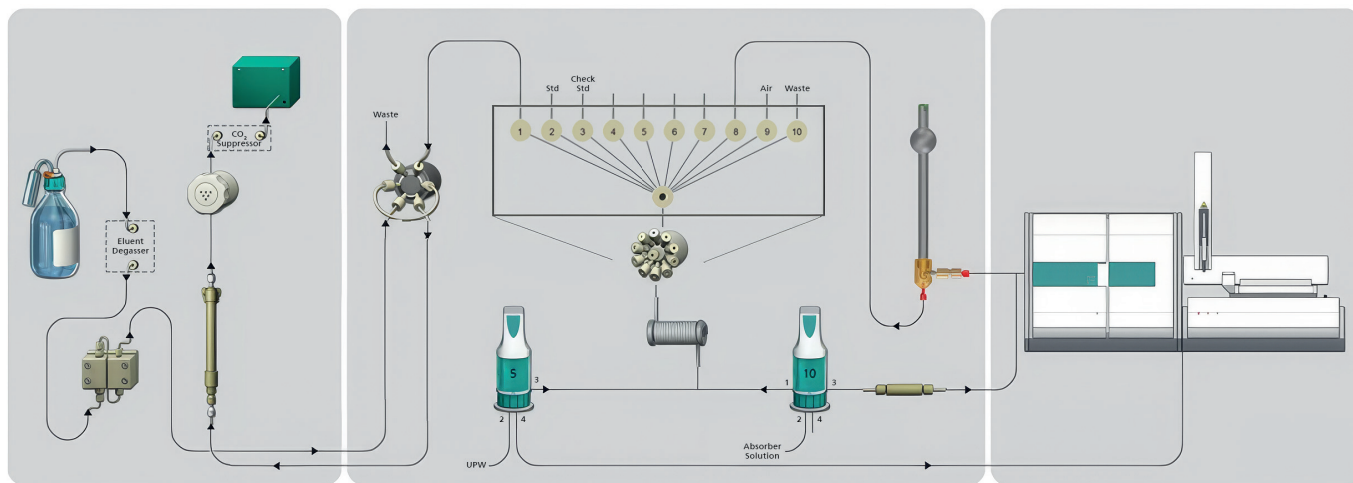


Figure 1: Sample flow path

Analytes:	Fluorine & Chlorine
Matrix:	Cardboard, cardstock & epoxy
Method:	Ion chromatography
Industry:	Food and Beverage