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Application News

EDX-8100 Energy Dispersive X-ray Fluorescence Spectrometer

Analysis of Fluorine by EDXRF: Simple Check for Use of PFAS for Textile and Kitchen Supplies

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User Benefits

- EDXRF can detect the element fluorine (F) in fluorine resin coatings.
- Measurement does not require complicated sample preparation, and can be started simply by placing the sample in the instrument.
- Checking for the presence of fluorine in products by EDXRF is a simple technique for investigating the possibility that PFAS were used.

■ Introduction

The organofluorine compounds known collectively as PFAS (per- and polyfluoroalkyl substances) have been used widely as water repellents and surface treatment agents, taking advantage of their water repellency, stability, and other useful properties. However, since they are highly resistant to degradation and easily accumulate in the environment and living organisms, environmental persistence and bioaccumulation are concerns. Against this background, PFAS analysis is carried out using liquid chromatograph-mass spectrometer (LC-MS) or ion chromatograph, particularly in fields such as drinking water, foods, and soil.

In response to limits on the use of PFAS, analysis of PFAS in products is also required. For example, perfluorohexanoic acid (PFHxA) and fluorotelomer alcohol (FTOH) are used respectively in various types of products as a water repellent and an oil repellent.

Detection of the element fluorine (F) by EDXRF allows quick and simple investigation of the possibility that PFAS were used in products.

This article introduces examples of analysis of water-repellent and oil-repellent cloth products and cooking products used in familiar products such as clothing, furniture, and kitchen products.

1. Water-Repellent Cloth

■ Sample Materials

The sample materials were four water-repellent-treated synthetic fabrics, A and B (polyester) and C and D (nylon). Fig. 1 shows an image of a sample.

Fig. 1 Water-Repellent Cloth (Sample B)

■ Sample Preparation

The sample materials were cut to a size that covers the analysis diameter of 10 mm, and were used directly in the analysis.

■ Qualitative/Quantitative Analysis

Fig. 2 and Table 1 show the results of the qualitative analysis and quantitative analysis, respectively. Peaks of fluorine were clearly detected in samples A and B (Fig. 2). On the other hand, fluorine was not detected in samples C and D,, which is considered to be below the lower limit of detection (LOD, 486 ppm*1). Thus, fluorine can be detected at approximately the 1 % level in 1 minute.

*1 : The 3 σ of sample D was used as the LOD.

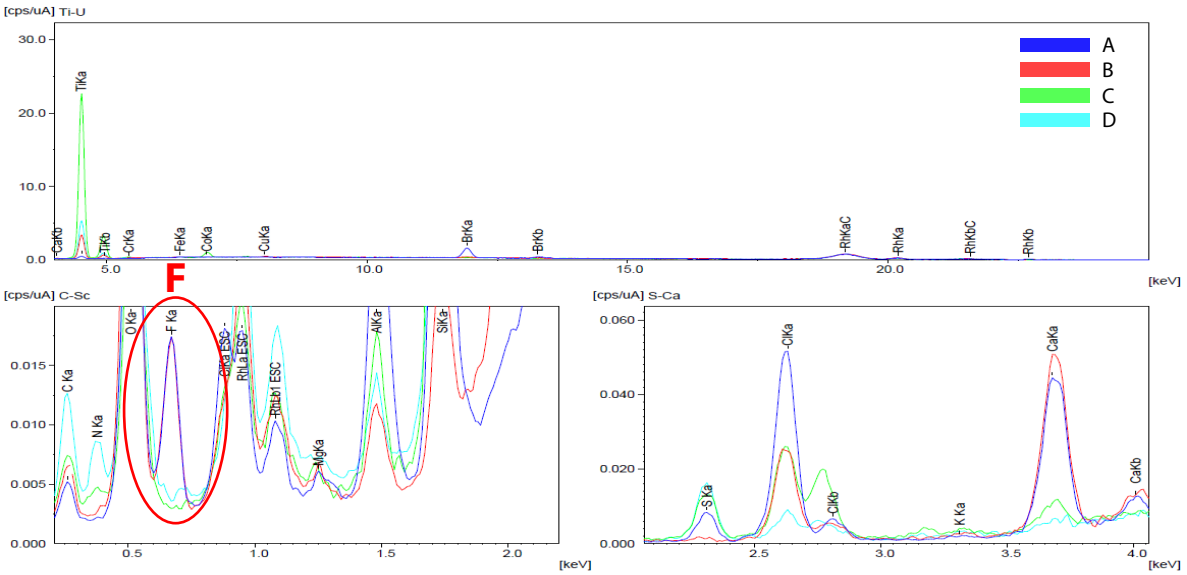

Fig. 2 Results of Qualitative Analysis of Water-Repellent Cloths

Table 1 Results of Quantitative Analysis of Water-Repellent Cloths [%]

Sample	F	Ti	S	Cl	Si	P	Al	Cu	Mg	Fe	Ca	K	Co	Cr	Br	$C_{10}H_8O_4$*2	$C_6H_{11}NO$*3
A	2.05	0.013	0.070	0.252	0.048	-	0.042	0.002	0.006	0.003	0.033	0.001	0.002	0.001	0.050	97.43	-
B	1.53	0.11	0.008	0.099	0.009	0.009	0.008	0.003	0.006	0.003	0.031	0.001	-	-	0.005	98.18	-
C	-	0.75	0.063	0.087	0.043	0.005	0.010	0.002	0.005	0.002	0.004	0.002	0.017	0.004	-	-	99.01
D	-	0.17	0.054	0.018	0.012	0.006	0.004	0.003	0.003	0.002	0.001	0.001	-	-	-	-	99.73

– : Not detected.
*2: Quantitative calculation of the balance (residual amount), assuming samples A and B are polyester ($C_{10}H_8O_4$).
*3: Quantitative calculation of the balance (residual amount), assuming samples C and D are nylon ($C_6H_{11}NO$).

2. Water- and Oil-Repellent Cooking product

■ Sample Materials

The sample products were a toaster bag and a BBQ sheet. Fig. 3 shows an image of the toaster bag.

Fig. 3 Toaster Bag

■ Sample Preparation

The samples were cut to a size that covers the analysis diameter of 10 mm and analyzed.

■ Qualitative Analysis

Fig. 4 shows the results of the qualitative analysis. Fluorine was detected in both the toaster bag and the BBQ sheet. These products have been treated with a water- and oil-repellent coating to prevent food from sticking and staining. In this experiment, the element fluorine in these coatings could be detected by a simple test using EDXRF, and confirmed the possibility that PFAS were used.

■ Conclusion

EDXRF can detect the element fluorine (F) in water-repellent-treated cloth and water- and oil-repellent-treated cooking product without complicated sample preparation. Development of fluorine-free alternatives to these water- and oil-repellent materials is now underway. Analysis by EDXRF is useful as a simple and quick test method for confirming the possibility that PFAS were used in products.

* In this Application News Article, PFAS are used as a collective term for organofluorine compounds. Note that EDXRF can detect the presence of elemental fluorine (F), but cannot identify the type of organofluorine compound.

■ Analysis Conditions

Table 2 shows the analysis conditions used in "1. Water-Repellent Cloth" and "2. Water- and Oil-Repellent Cooking product". The measurement time of the "C-Sc" measurement channel, which includes fluorine, is 60 seconds.

Table 2 Analysis Conditions

Measurement channel	Ti-U	C-Sc	S-Ca*4
Analysis line	Kα, Kβ, Lα, Lβ . . .		
X-ray tube	Rh target		
Tube voltage	50 kV	15 kV	
Tube current	Automatic control		
Collimator	10 mm		
Integral time	20 s	60 s	20 s
Dead time	Max. 30 %		
Filter	None		#2
Detector	SDD semiconductor detector		
Atmosphere	Vacuum		
Quantitative analysis method	FP method		

*4 : Only for water-repellent cloths.

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2. 01-00786-EN, Analysis of AOF (Adsorbable Organic Fluorine) According to EPA Method 1621
3. 01-00808-EN, Analysis of Per-and Polyfluoroalkyl Substances (PFAS) in Drinking Water in Accordance with EPA Method 537.1
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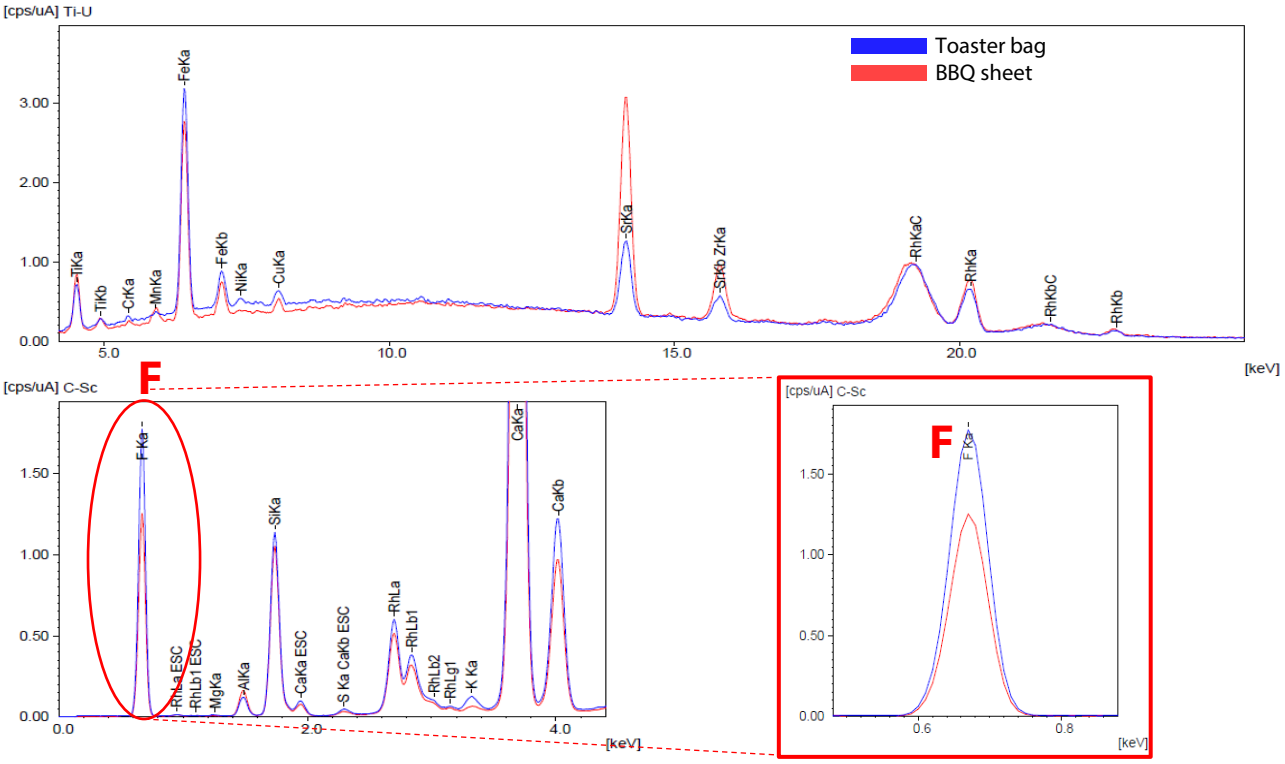

Fig. 4 Results of Qualitative Analysis of Cooking Products

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