

Application News

No. X267

X-Ray Analysis

X-Ray Fluorescence Analysis of Light Elements in Liquid Samples – EDX-8100 and Helium Purge Unit –

Shimadzu's EDX-8100 is an energy dispersive X-ray fluorescence spectrometer with high sensitivity for light elements and can be configured with an optional helium purge unit. The helium purge unit enables high-sensitivity analysis of light elements in samples that cannot be depressurized to a vacuum state such as solutions and samples that generate gas. As one effect, detection of fluorine (F) in liquid samples is possible with the EDX-8100, whereas it was not possible with the conventional EDX-8000.

The detection limits of the elements F to K in liquid samples in a He atmosphere and differences depending on the film used to hold the samples were evaluated.

S. Yada, N. Ichimaru

Elements and Samples

The samples are shown in Table 1. Pure water was used to calculate the background intensity.

Table 1 Elements, Contents, and Solutions

Element	Content [ppm]	Solution
F	94,962	Preparation
Na	20,000	Preparation
Mg	20,000	Preparation
Al	20,000	Preparation
Si, P, S, Cl, K	1,000	Atomic Absorption Standard Solution
-	-	Pure Water

Sample Pretreatment

Approximately 5 mL of each sample was introduced directly into a film-covered sample container. The three films shown in Table 2 were used.

Table 2 Sample Holder Films

Name	Thickness (μm)	Compositional Formula
Prolene® (Chemplex)	4	C ₃ H ₆
Polypropylene	5	C ₃ H ₆
PET	6	C ₁₀ H ₈ O ₄

Detection Limits

(1) Atmosphere: Air/He

Fig. 1 (next page) shows the measured spectra in air and a He atmosphere when using the polypropylene film. Table 3 shows the detection limits*1 calculated from the intensity and measurement conditions (current and integration time). The He atmosphere enabled detection of F and Na, and improved detection of Mg by 40 times, Al and Si by 8 times, and P and S by approximately 2 times.

Table 3 Detection Limits (1) Differences Depending on Atmosphere [ppm]

Atmosphere	⁹ F	¹¹ Na	¹² Mg	¹³ Al	¹⁴ Si	¹⁵ P	¹⁶ S	¹⁷ Cl	¹⁹ K
Air	-	-	3,300	210	91	25	16	23	6.3
He	41,000	680	84	26	11	11	7.8	15	5.1

Table 4 Detection Limits (2) Differences Depending on Film [ppm]

Film	⁹ F	¹¹ Na	¹² Mg	¹³ Al	¹⁴ Si	¹⁵ P	¹⁶ S	¹⁷ Cl	¹⁹ K
Prolene® (Chemplex)	18,000	480	72	22	10	9.7	7.5	14	4.7
Polypropylene	41,000	680	84	26	11	11	7.9	15	5.1
PET	-	5,400	340	55	54	25	24	18	5.8

(2) Differences Depending on Film

Three types of films were used in measurements in a He atmosphere. Fig. 2 (next page) shows the spectra, and Table 4 shows the detection limits. Prolene (Chemplex Inc.) with a thickness of 4 μm displayed the highest sensitivity, and the influence of the film thickness and composition on absorption increased with lighter elements.

Conclusion

Measurements of liquid samples by EDX are possible regardless of conditions such as concentration, organic or inorganic material, suspension, and viscosity.

This technique is not limited to solutions, but is also effective with samples such as the following, which are difficult or impossible to depressurize to a vacuum.

- Teeth and other biomaterials (fracture, alteration) (Care is necessary in handling, for example, considering infection.)
- Porous materials such as zeolite (time required to reach measurement vacuum)
- Fibers and clothing (porous, wet)
- Ultrafine particles such as graphite powder (scattering)
- Sealed and enclosed materials (bursting)

This technique is effective for quickly determining the presence and content of light elements in the above-mentioned materials.

Measurement Conditions

Instrument	: EDX-8100
X-ray tube	: Rh target
Detector	: SDD
Tube voltage	: 50 [kV]
Tube current	: 260 to 483 [μA] (Auto)
Primary filter	: None (F to S) / #2 (Cl, K)
Collimator	: 10 [mmφ]
Measurement atmosphere	: Air/He
Integration Time	: 100 [sec]
Dead time	: 30 max. [%]

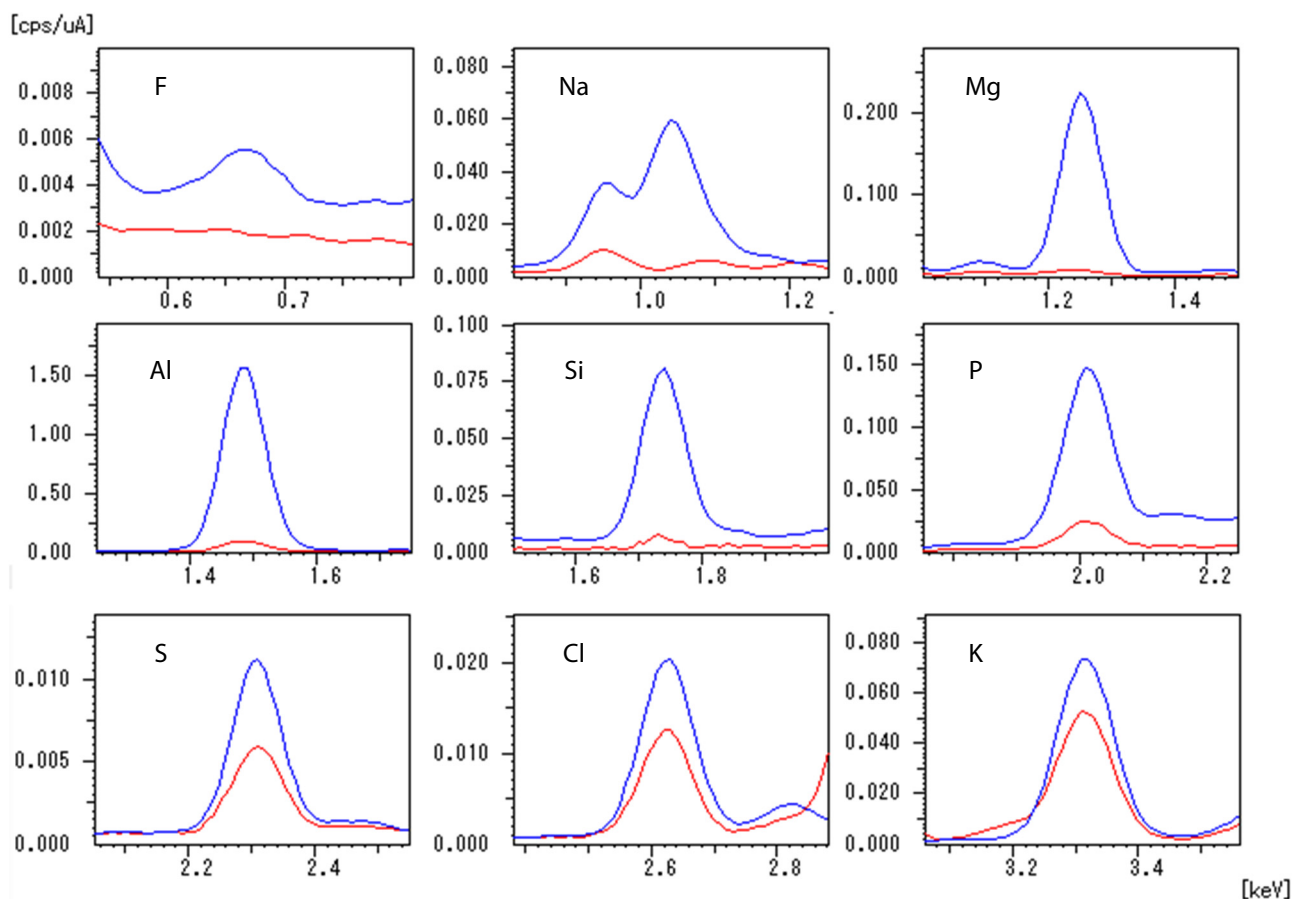


Fig. 1 X-Ray Fluorescence Spectra for F to K [Blue: He Atmosphere, Red: Air]

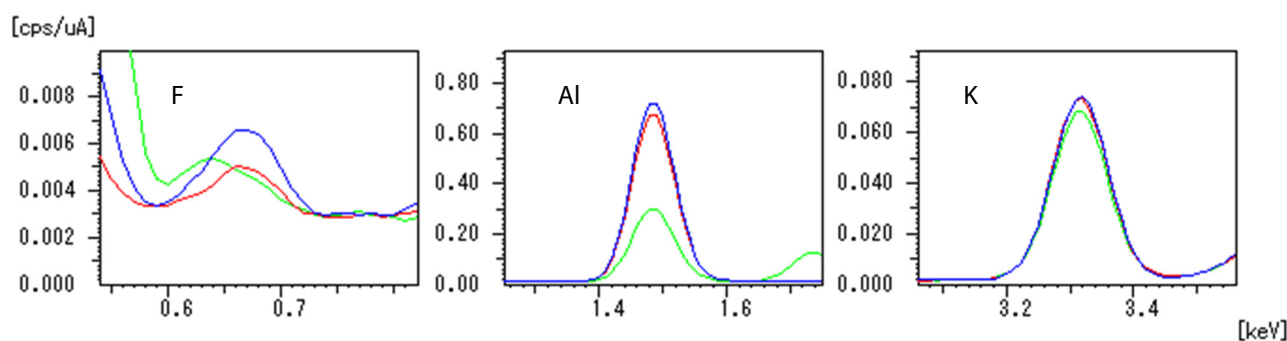


Fig. 2 X-Ray Fluorescence Spectra for F, Al, and K [Blue: Prolene (Chemplex), Red: Polypropylene, Green: PET]

*1 Calculation of detection limit

$$\text{Detection limit} = 3 \times \frac{\text{Content}}{\text{X-ray fluorescence intensity}} \times \sqrt{\frac{\text{Background intensity} \times \text{Integration time} \times \text{Current}}{\text{Integration time} \times \text{Current}}}$$

$$\text{(Example: Case of Table 3, P in He atmosphere)} = 3 \times \frac{1,000_{[ppm]}}{1,217437_{\left[\frac{cps}{\mu A}\right]}} \times \sqrt{\frac{0.536691_{\left[\frac{cps}{\mu A}\right]} \times 100_{[sec]} \times 283_{[\mu A]}}{100_{[sec]} \times 283_{[\mu A]}}} = 10.73_{[ppm]} \approx 11_{[ppm]}$$

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