

Application News

X-ray Analysis

No.X242A

EDXRF Analysis of Sulfur and Other Elements in Oil

The concentrations of sulfur substances in fuel oil, which greatly contribute to air pollution and acid rain, are regulated by law in many countries, and these regulations are becoming stricter with the growing concern about their impact on the environment and the consequences for society.

Here, using the easy-to-operate EDX-720 energy dispersive X-ray fluorescence spectrometer, we determined the limit of detection and limit of

■ Standard

Oil standards of MBH Analytical Ltd.
Sulfur content: 0, 10, 25, 50, 100, 200, 300 ppm

■ Sample Preparation

Approximately 6 mL of sample was placed in a container covered with 5 μm thick polypropylene film, and analyzed.

A photograph of the sample is shown in Fig. 1.



Fig. 1 Photograph of Oil Standard Sample

■ Calibration Curve

Fig. 2 shows calibration curves of sulfur measured in atmospheres of He and Air, respectively. $\text{SK}\alpha$ was used as the analysis line.

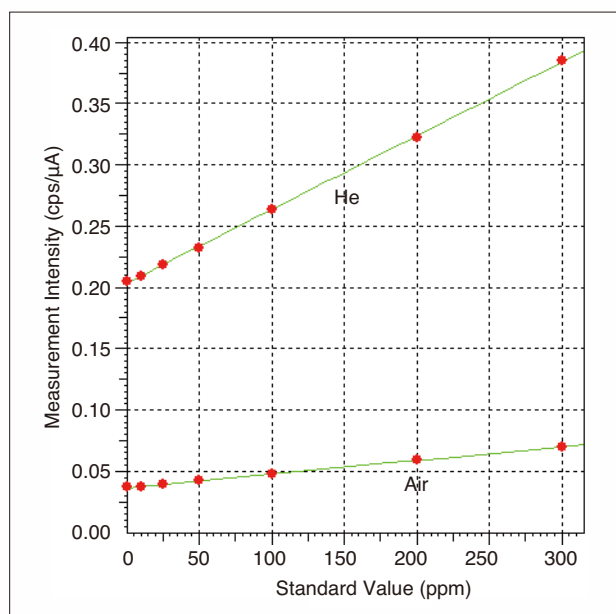


Fig. 2 Calibration Curve of S [He, Air]

quantitation of sulfur.

The energy dispersive X-ray fluorescence spectrometer can be used for simultaneous multi-element analysis of the elements from Na (atomic no. 11) to U (atomic no. 92).

Next, we conducted simultaneous analysis of the elements in Conostan[®] S-21, a commercially-available oil standard, and determined the limit of detection for each element detected.

■ Calibration Curve, Limits of Detection and Limits of Quantitation

The limits of detection and limits of quantitation for sulfur in air and He atmospheres calculated from the calibration curves of Fig. 2 are shown in Table 1.

Table 1 Limits of Detection and Limits of Quantification for S

Measurement Atmosphere	He	Air
Spectrum	$\text{SK}\alpha$	$\text{SK}\alpha$
Limit of Detection (3σ)	5.9 ppm	13.0 ppm
Limit of Quantitation (10σ)	19.9 ppm	43.6 ppm

■ Results of Qualitative Analysis

Fig. 3 shows the qualitative profile for the 200 ppm sample. The analytical conditions are shown in Table 2.

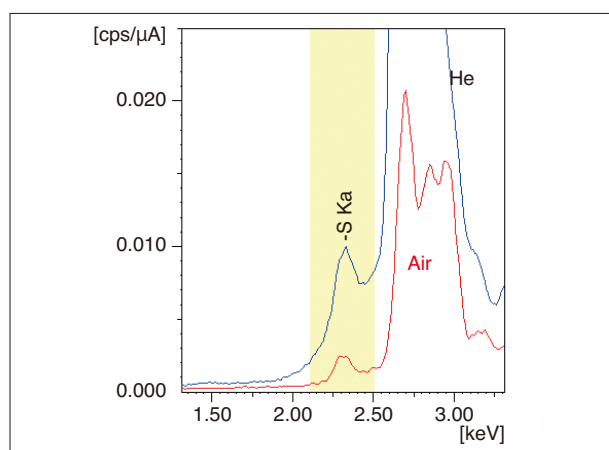


Fig. 3 Profile of S (200 ppm)

Table 2 Analytical Conditions

Instrument	: EDX-720
X-ray Tube	: Rh target
Filter	: Without
Voltage-Current	: 15 kV- (Auto) μA
Atmosphere	: He, Air
Measurement Diameter	: 10 mm ϕ
Measurement Time	: 300 sec
Dead time	: Max 40 %

Quantitative Analysis of Conostan® S-21

We conducted qualitative analysis of the Conostan® S-21 standard sample, and then calculated the limit of detection for each element.

Sample

Conostan® S-21, 100 ppm

Elements

Al, Si, P, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Mo, Ag, Cd, Sn, Pb

Sample Preparation

Approximately 6 mL of sample was placed in a container covered with 5- μ m thick polypropylene film, and analyzed.

A photograph of the sample is shown in Fig. 4.

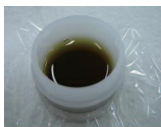


Fig. 4 Conostan® S-21, 100ppm

Qualitative Profile

The results of qualitative analysis of Conostan® S-21 are shown in Fig. 5.

Table 3 Limits of Detection

Element	Analysis Line	Detection Limit (ppm)	Primary Filter	Atmosphere
¹³ Al	K α	31.0	—	He
¹⁴ Si	K α	19.0	—	He
¹⁵ P	K α	12.8	—	He
²⁰ Ca	K α	2.9	#1	He
²² Ti	K α	1.9	#1	Air
²³ V	K α	2.1	#1	Air
²⁴ Cr	K α	2.0	#1	Air
²⁵ Mn	K α	3.3	#2	Air
²⁶ Fe	K α	2.4	#2	Air
²⁸ Ni	K α	2.2	#3	Air
²⁹ Cu	K α	1.6	#3	Air
³⁰ Zn	K α	1.3	#3	Air
⁴² Mo	K α	1.9	#3	Air
⁴⁷ Ag	K α	1.8	#4	Air
⁴⁸ Cd	K α	2.3	#4	Air
⁵⁰ Sn	K α	3.6	#4	Air
⁸² Pb	L α	1.2	#3	Air

* Calculation Expression for Limit of Detection

C: Standard value (ppm)

Inet: Net intensity (Counts)

Iback: Background intensity (Counts)

Limits of Detection

The limits of detection for the respective elements calculated from the qualitative analysis results are shown in Table 3. It was understood from these results that detection of the heavy elements is possible at the ppm level in Air atmosphere.

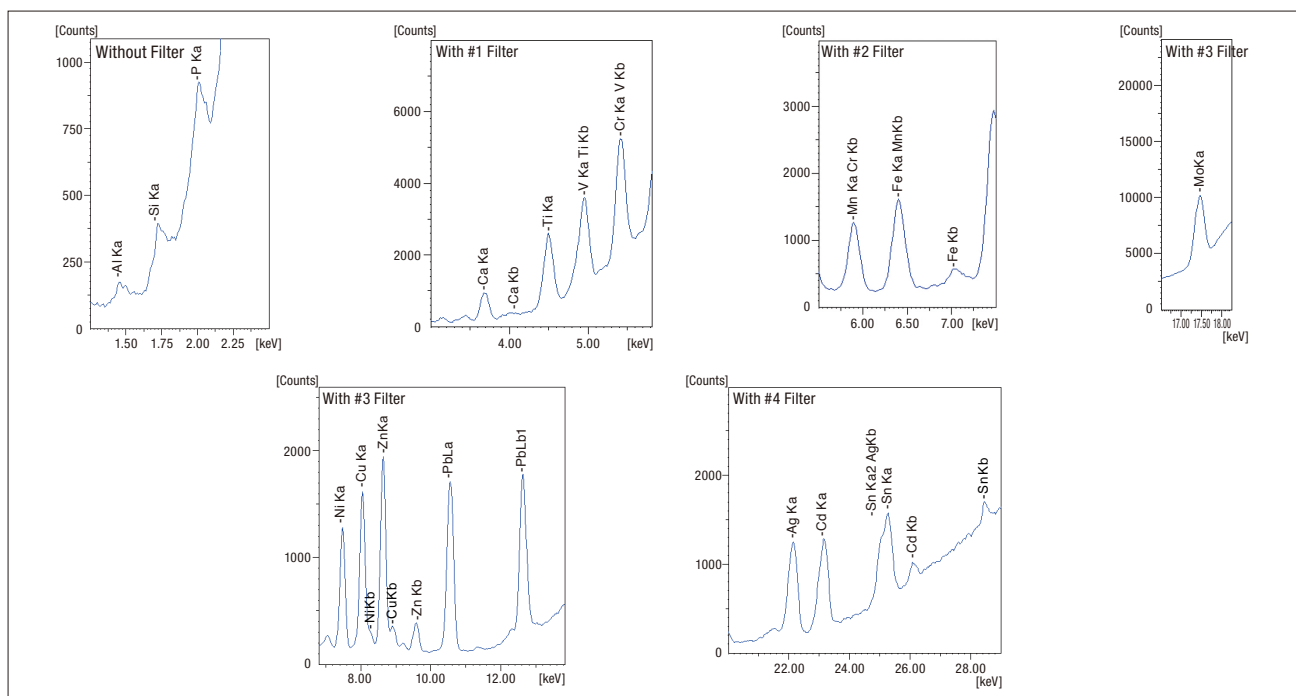


Fig. 5 Qualitative Analysis of Standard Oil Sample, Conostan® S-21

Table 4 Analytical Conditions (Qualitative Analysis)

Instrument : EDX-720	Voltage-Current : Al, Si, P, Ca, Ti, V, Cr : 15kV-(Auto) μ A
X-ray Tube : Rh target	Mn, Fe, Mo, Ni, Cu, Zn, Pb, Ag, Cd, Sn : 50kV-(Auto) μ A
Filter : Without [Al, Si, P], Filter #1 [Ca, Ti, V, Cr], Filter #2 [Mn, Fe]	Atmosphere : He, Air
Filter #3 [Mo, Ni, Cu, Zn, Pb], Filter #4 [Ag, Cd, Sn]	Measurement Diameter : 10 mm ϕ
Dead time : Max 40 %	Measurement Time : Each 300 sec



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