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Application News

Shimadzu Energy Dispersive X-ray Fluorescence Spectrometers EDX-7200/8100

Slag Powder Analysis by EDXRF

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User Benefits

- Slag can be easily analyzed by simply setting it into a sample cell.
- Additional calculations based on the quantitated values, such as basicity, can also be included in reports.
- By press molding samples, fluorine (F) can also be analyzed (EDX-8100).

■ Introduction

Slag is a byproduct of the metal smelting process that contains a mixture of reduced metals, separated minerals, and residual reducing agents remaining from the smelting process. There is also what is called molten slag, which is made by melting and solidifying waste such as incineration ash and sewage sludge. The principal components in slag include silica (SiO_2), and metal oxides from the raw materials, such as alumina (Al_2O_3), iron oxides (Fe_2O_3, FeO), and calcium oxide (CaO). Slag can also contain trace quantities of heavy metals and fluorine (F). Slag components are analyzed using an X-ray fluorescence spectrometer, which is important for reusing the slag or managing the conditions of slag formation. Conventionally, wavelength dispersive X-ray fluorescence (WDXRF) spectrometer was commonly used to analyze slag, but now energy dispersive X-ray fluorescence (EDXRF) spectrometer is increasingly used due to easier operability, simpler sample handling, and higher quantitative accuracy.

This article describes an example of using EDXRF for qualitative-quantitative analysis of blast furnace slag (EDX-7200) and for quantitative analysis of fluorine (F) in electric furnace slag (EDX-8100).

1. Analysis of Blast Furnace Slag (EDX-7200)

■ Elements

Elements from $_{11}$Na to $_{92}$U were targeted.

■ Samples

Standard slag sample: The Iron and Steel Institute of Japan sample 905-1

■ Pretreatment

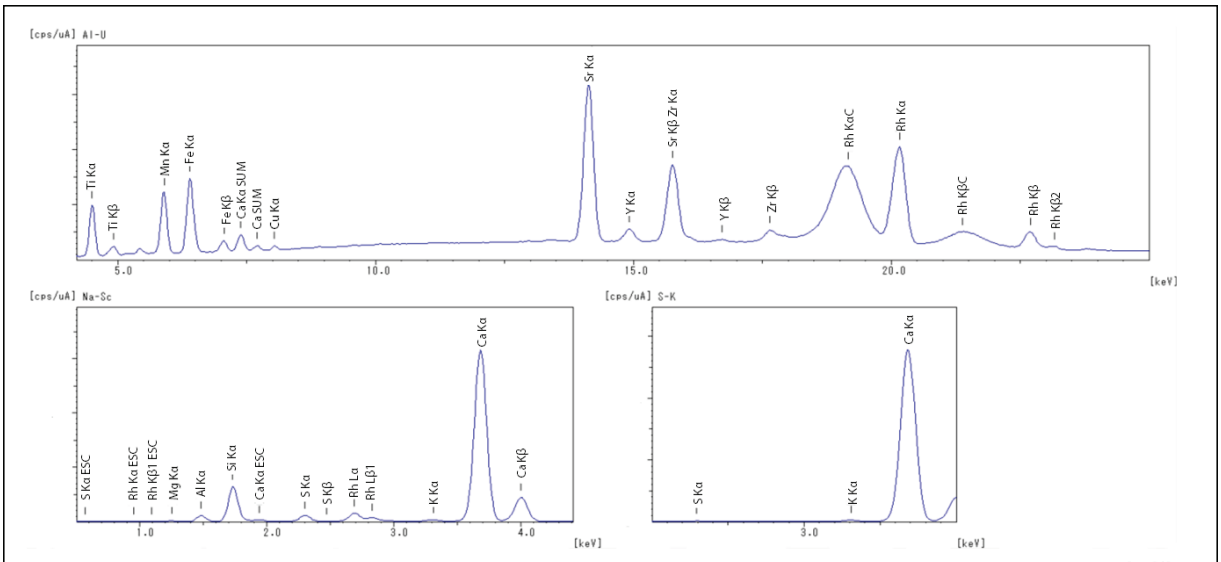
5 g of sample were placed in a sample cell, lined with 5 µm thick polypropylene film, and then compressed.

Fig. 1 shows an example of the sample.

Fig. 1 Sample

■ Qualitative-Quantitative Analysis Results

Results from the qualitative-quantitative analysis are shown in Fig. 2. Relative errors between quantitative and standard values for principal components (CaO, SiO_2, and Al_2O_3) and trace elements (MnO) were within 1%. In addition, trace elements not listed among standard values were detected, such as SrO, ZrO_2, and Y_2O_3.



[wt%]

Elements	CaO	SiO_2	Al_2O_3	MgO	SO_3	TiO_2	K_2O	MnO	Fe_2O_3	SrO	ZrO_2	Y_2O_3	CuO
Quantitative Value	41.4	35.1	13.9	6.68	1.45	0.50	0.35	0.20	0.20	0.11	0.043	0.009	0.004
Standard Value	42.1	34.2	14.2	6.80	1.39	0.56	0.37	0.22	0.24	—	—	—	—

Fig. 2 Qualitative-Quantitative Analysis Results for Standard Slag Sample (905-1)

■ Freely Modifiable Report Template

The report template included with the PCEDX software for EDXRF (same for both EDX-7200/8100 models) is created using Microsoft Excel®. Therefore, it can be edited as necessary to extract specific data required or to automatically calculate control parameters and then summarize the results to generate unique reports.

For example, one parameter relevant to slag is the basicity. If the slag is to be reused as a construction material, road subbase material, or concrete aggregate material, basicity is important for controlling slag components,[1),2)] controlling molten slag formation conditions, and so on.

Fig. 3 shows an example of calculating basicity by extracting the necessary values from the quantitated values shown in Fig. 2.

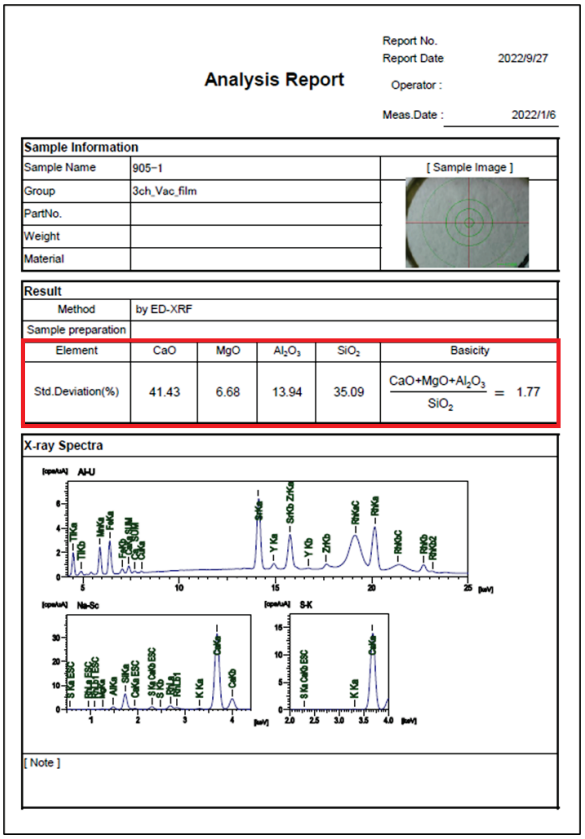


Fig. 3 Example of Basicity Report

2. Analysis of Fluorine (F) (EDX-8100)

Fluorine (F) analysis has applications in the investigation of soil and other environmental contamination. EDX-8100, which can analyze light elements such as fluorine (F), is useful for those analyses.

When a sample is set in a sample cell as shown in Fig. 1, analysis of fluorine is not possible. This is because the X-ray fluorescence of fluorine (F) measured from the sample is so low that almost all of it is absorbed by the film set on the sample cell. However, quantitation of fluorine (F) is possible by pressing the samples and analyzing them without applying a film.

■ Samples

Electric furnace slag that contained about 2 % fluorine (F).

■ Pretreatment

A PVC ring with a 22 mm internal diameter was packed with sample material and pressed for about 10 seconds at 80 kN. A photograph of the sample is shown in Fig. 4.

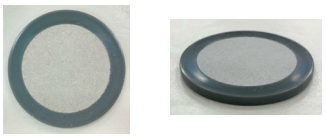


Fig. 4 Sample (Left: Top View; Right: Side View)

■ Qualitative Profiles

Fig. 5 shows a comparison of data from samples either placed in a sample cell with simple compression applied or pressed. The theoretical lower limit of detection (L.L.D.) was calculated from Equation (1) and the spectral intensity (NET, BG). For pressed samples, the L.L.D. for fluorine (F) integrated for 300 seconds was about 0.2 %. That means the EDX-8100 can quantitatively analyze fluorine (F) concentrations as low as 1 % or less.

$$L.L.D. = 3 \cdot \frac{C}{NET} \cdot \sqrt{\frac{BG}{A \cdot T}} \quad \text{—(1)}$$

NET: Intensity (cps/μA)
C: Standard F concentration (%)
A: Current value (μA)
T: Integration time (sec)

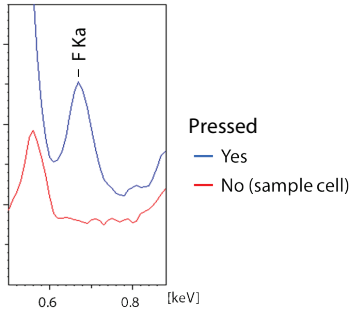


Fig. 5 Overlay of FKα

■ Conclusion

With EDX, information about the components contained in slag can be easily obtained by setting it into a sample cell. It also provides good accuracy, with a relative error of 1 % or less for principal components. Moreover, accuracy can be improved using the calibration curve method.

The EDX-8100 can also analyze fluorine (F), which is generally difficult to analyze. By pressing the sample, even content levels of 1 % or less can be quantitated.

Analytical Conditions

Table 1 Analytical Conditions

Instrument	EDX-7200	EDX-8100
Elements	$_{11}$Na to $_{92}$U	$_{9}$F
Analysis Group	Qualitative-Quantitative analysis	
Detector	SDD	
X-ray Tube	Rh target	
Tube Voltage	50 [kV] (Al to U) 15 [kV] (Na to Sc) and (S to K)	15 [kV]
Tube Current	Auto	
Collimator	10 [mmφ]	
Primary Filter	None (Al to U) and (Na to Sc) #2 (S to K)	None
Atmosphere	Vacuum	
Integration Time	100 [sec] × 3 channels	300 [sec]
Dead time	Max 30 %	

References

1) JIS A 6206 Ground granulated blast-furnace slag for concrete (2013)
2) JIS R 5211, Portland blast-furnace slag cement (2009)

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