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XRF1124 - Quantitative analysis of blast furnace slag by fusion method on the ZSX Primus III NEXT

Introduction

Blast furnace slag is formed when iron ore or iron pellet, coke and flux are melted in a blast furnace of the iron foundry. The rapid chemical analysis of a blast furnace slag is an important task to control the blast furnace. X-ray fluorescence spectrometers are the most common analysis tools to analyze powder samples in iron and steel making process.

This application note describes blast furnace slag analysis using ZSX Primus III NEXT.

Instrument

The ZSX Primus III NEXT has tube above optics, where the X-ray tube is placed above the sample. Tube above optics reduces the risk of instrument contamination or damage that can occur when pressed pellet samples break inside the spectrometer while being measured or transported to the measurement position. The ZSX Primus III NEXT is ideal for the steel industry where both bulk metal and powder samples are analyzed as part of the process control in steel manufacturing. The system software is based on Rigaku's Flowbar interface that leads the user through a series of step-by-step procedures to optimize and execute measurements. "EZ analysis", another Rigaku software innovation, greatly simplifies the analysis setup and sample analysis. In addition, the software has various kinds of statistical process control functions for the steel industry. The blast furnace slag is often analyzed by the pressed pellet method in XRF. However, fusion method preparing the powder to fused bead gives accurate results by removing the errors caused by grain size and heterogeneity effects and the method is a popular analysis technique for blast furnace slags.

Standards and sample preparation

The certified reference materials of blast furnace slag which were provided by NCS (China national analysis center), JSS (Japan), BRAMMAR (UK) and CSMU (Czech) were used to establish the calibration. The well-dried (2 hours at 105°C) samples were fused with flux of Spectroflux® 100B (4LiBO₂:1Li₂B₄O₇) supplied from Thermo Fisher Scientific Chemical, oxidation reagent (V₂O₅) and samples in the ratio 10 : 1 : 1 = 4 g : 0.4 g : 0.4 g by using a fusion machine.

Measurement and calibration

Measurements were performed using the ZSX Primus III NEXT with a 3 kW Rh target X-ray tube. The K α lines were measured for the elements with the counting time of 20 seconds and only Na is measured 40 seconds.

An LiF(200) analyzing crystal was used for all the heavy elements from K to Mn. A PET was used for Si and Al, and an optional Ge was used to provide high resolution for the measurement of P and S. A scintillation counter was used for all the heavy elements and a gas flow proportional counter was used for all the light elements. An RX26 multilayer was used for Mg and Na analysis.

The calibration curves obtained are shown in Figure 1 through Figure 11.

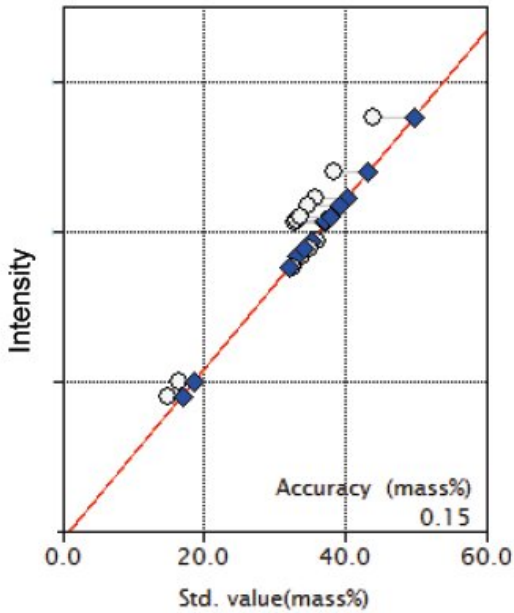


Figure 1: Calibration curve of SiO₂

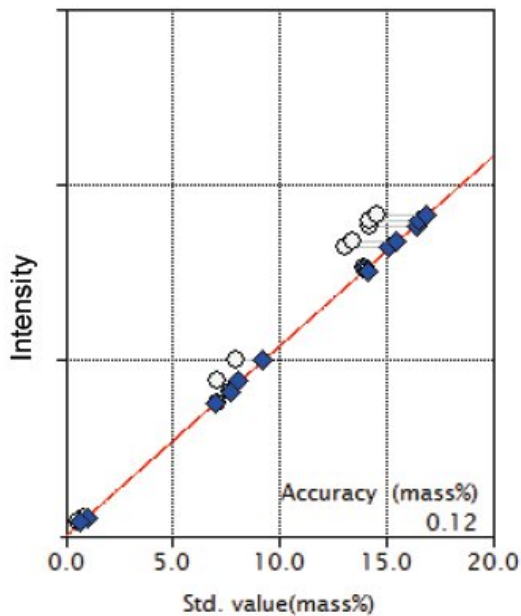


Figure 2: Calibration curve of Al₂O₃

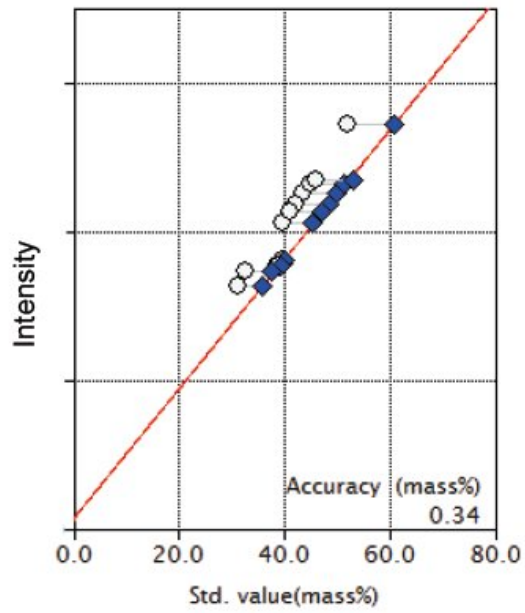


Figure 3: Calibration curve of CaO

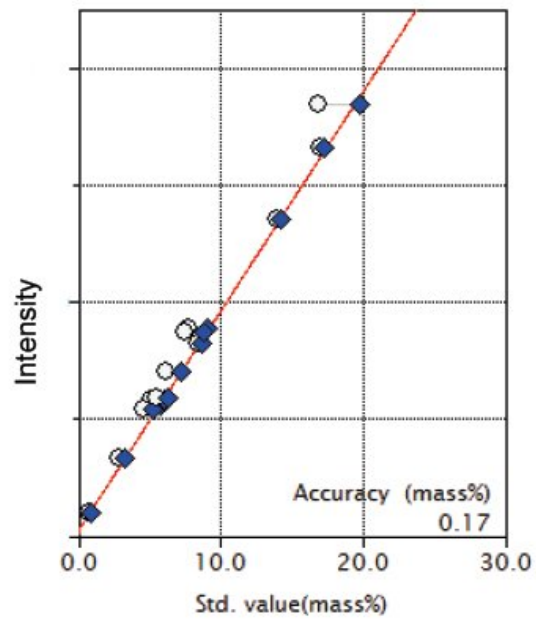


Figure 4: Calibration curve of MgO

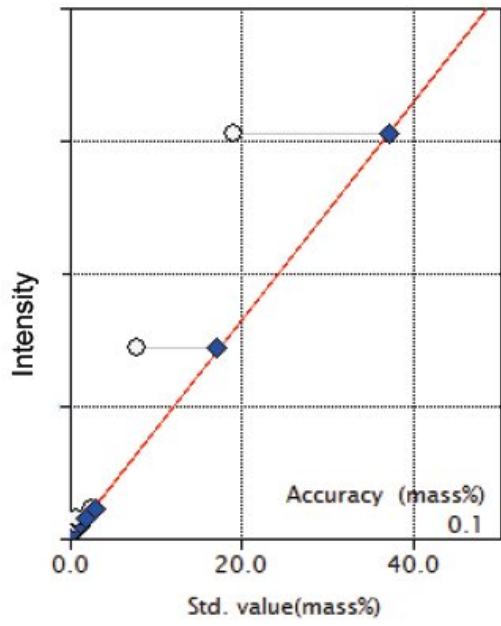


Figure 5: Calibration curve of Fe₂O₃

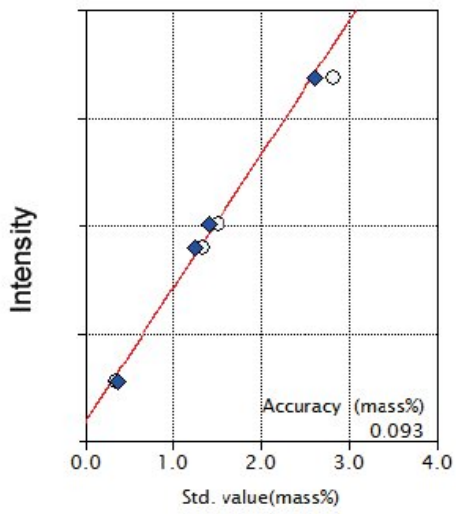


Figure 6: Calibration curve of SO₃

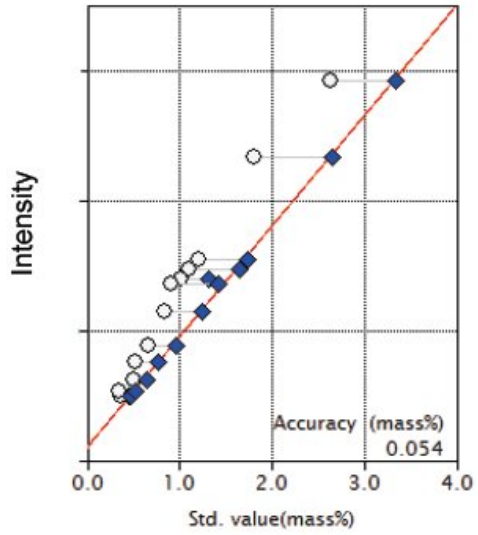


Figure 7: Calibration curve of TiO₂

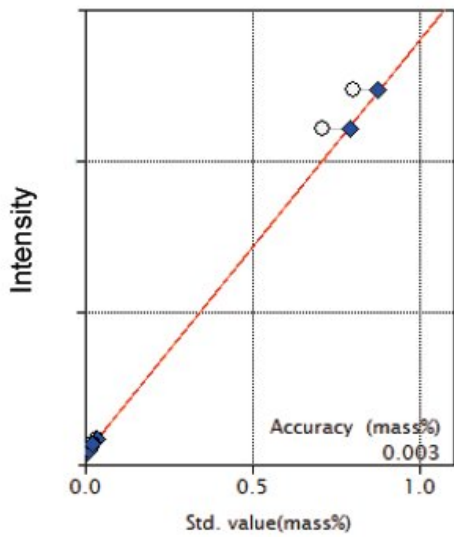


Figure 8: Calibration curve of P₂O₅

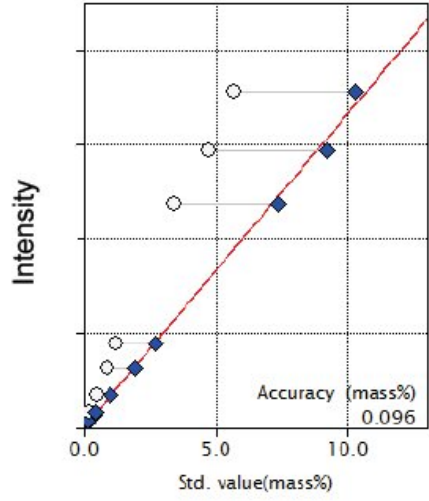


Figure 9: Calibration curve of MnO

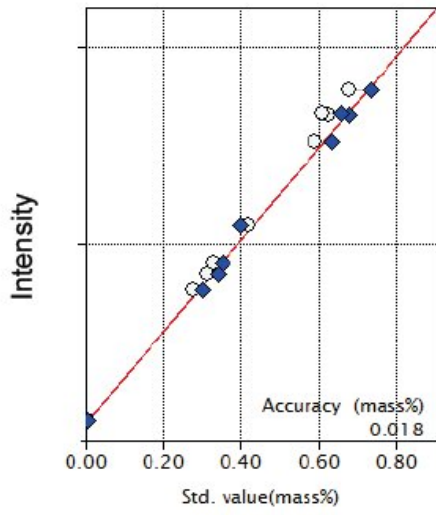


Figure 10: Calibration curve of K₂O

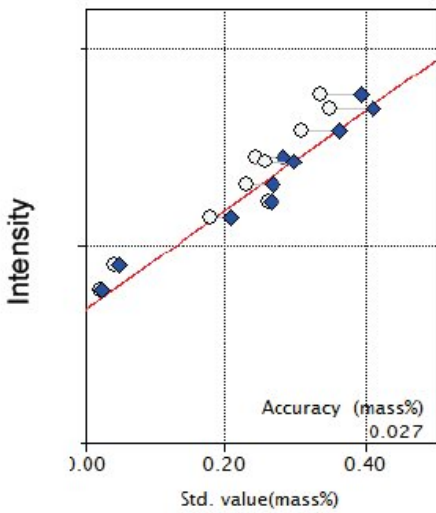
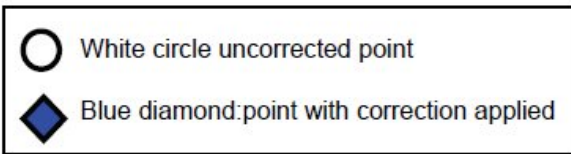


Figure 11: Calibration curve of Na₂O



Theoretical alphas obtained by the fundamental parameter method included in the software package were applied to all the calibrations to correct for absorption and enhancement effects.

Results

The accuracies of the calibration curves are listed in Table 1 and the repeatability test results (10 times) are listed in Table 2.

Table 1: Accuracy of calibration curves (unit : mass%)

Component	Concentration range	Accuracy
SiO ₂	14.9 – 44.00	0.15
Al ₂ O ₃	0.57–14.6	0.12
CaO	31.2 – 51.9	0.34
MgO	0.73 – 16.97	0.17
Fe ₂ O ₃	0 – 19.12	0.10
SO ₃	0 – 2.84	0.093
TiO ₂	0.34 – 2.63	0.054
P ₂ O ₅	0.00 – 0.80	0.003
MnO	0.07 – 5.70	0.096

K ₂ O	0.00 – 0.67	0.018
Na ₂ O	0.023 – 0.35	0.027

Table 2: Result of repeatability (unit : mass%)

Component	Certified value	Average	Std dev.	RSD%	Sample
SiO ₂	36.10	35.97	0.0384	0.10	13824
Al ₂ O ₃	7.73	7.63	0.0206	0.27	13824
CaO	39.33	38.96	0.0143	0.040	13824
MgO	13.94	13.96	0.0277	0.20	13824
Fe ₂ O ₃	1.015	1.032	0.0015	0.14	13824
SO ₃	1.512	1.581	0.0050	0.32	13824
TiO ₂	0.37	0.36	0.0044	1.2	13824
P ₂ O ₅	0.026	0.0259	0.0015	5.9	13824
MnO	0.10	0.113	0.0010	0.88	13824
K ₂ O	0.59	0.57	0.0022	0.34	7-1-010
Na ₂ O	0.18	0.16	0.0122	7.6	7-1-010

The accuracy is calculated by the following formula:

$$Accuracy = \sqrt{\frac{\sum_i (C_i - \hat{C}_i)^2}{n-m}}$$

C_i : calculated value of standard sample

\hat{C}_i : reference value of standard sample

n : number of standard samples.

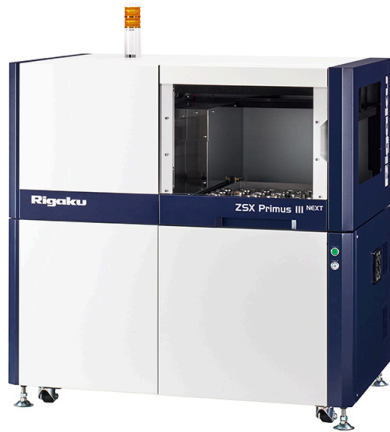
m: degree of freedom (linear 2, quad. 3)

Conclusions

The results show that, high precision and accurate analysis of the components in blast furnace slag can rapidly be performed in fusion method using ZSX Primus III NEXT. It is also possible to analyze the blast furnace slag by the pressed pellet method.

In addition, the ZSX Primus III NEXT can be applied to various kinds of materials such as iron and sintered ores, coke, limestone and pig iron for the process control in iron foundry.

Related products



ZSX Primus III NEXT

Affordable, high-end, tube-above Industrial WDXRF for the analysis of solid samples