



Lab Report XRF 129

S2 PUMA

● Accurate and Flexible Analysis of Iron Ore as Fused Beads by EDXRF

Introduction

Iron ore is one of the most important commodity on Earth due to its high demand in the steel industry. However, some of the components in iron ore can interfere with the production process or have a negative impact on the final product. Therefore monitoring the chemical composition of iron ore is not only important for the control of the mining process but also to optimize the feeding process of melting furnaces during steel production.

A number of elements must be monitored in iron ore samples. Beside the major element Fe, other elements of interest are Si, Al, Ti, Ca, Mg, Mn, K,

S, and P. Even though some of these elements are only present in low concentrations, they have a significant impact on the steel manufacturing process and the final steel quality.

All these major and minor elements can be analyzed using the S2 PUMA energy dispersive X-ray fluorescence (EDXRF) spectrometer. This instrument offers a straight forward but accurate means of controlling both the mining process and any subsequent manufacturing process. This lab report shows the performance of this instrument for the analysis of iron ore samples, prepared as fused beads.

Instrument

The EDXRF spectrometer S2 PUMA has been used for these measurements. It is a benchtop spectrometer with an easy-to-use TouchControl™ interface. The system is equipped with an XFlash® LE silicon drift detector (SDD) and an Ag target X-ray tube.

The XFlash detector is able to handle high count rates, which minimizes the statistical errors and leads to very precise results. Its excellent energy resolution permits different elements to be clearly separated and guarantees that even small concentrations can be detected alongside the main components. Fig. 1 shows a typical spectrum of an iron ore sample and indicates that all peaks are clearly resolved.

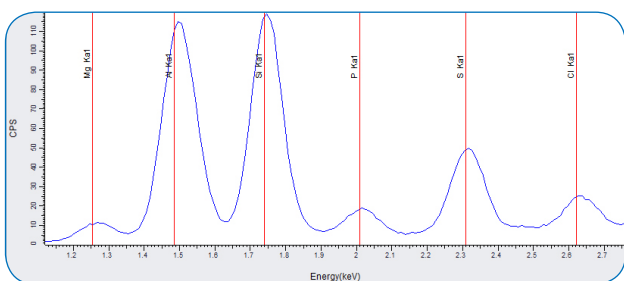


Fig. 1: Spectrum of an iron ore sample in the energy range of 1.2 to 2.7 keV

Sample preparation

In order to measure iron ore from different mining sites the samples have been prepared as fused beads. This sample preparation technique eliminates the mineralogical effects of samples from different sites.

The standards were dried in a drying oven at 110° C for one hour. For sample preparation 0.9 g standard material was mixed with 8.8 g lithium tetraborate 50 % + lithium metaborate 50 % + LiBr 0.2 % and 0.6 g NH₄NO₃ and prepared as fused beads (Fig. 2).

Measurement parameters

Two measurement regions were defined. The tube current was optimized and fixed in order to gain maximum count rate for the various elements. Tab. 1 shows the detailed measurement parameters. All measurements were performed under vacuum.



Fig. 2: Samples prepared as fused beads

Elements	Tube voltage [kV]	Tube current [μA]	Filter	Measurement time [s]
Mg, Al, Si, P, S	20	130	none	100
K, Ca, Ti, Mn, Fe	40	125	500 μm Al	100

Tab. 1 Measurement parameters for the different elements

Calibration

A set of 14 international certified reference materials (CRMs) were used to prepare the calibration for Mg, Al, Si, P, S, K, Ca, Ti, Mn, and Fe. Tab. 2 shows the concentration ranges of the different iron ore CRMs used to carry out the calibration.

	Minimum concentration [%]	Maximum concentration [%]
Fe ₂ O ₃	43.6	100
SiO ₂	2.2	16.8
CaO	0.02	19.2
Al ₂ O ₃	0.27	4.69
TiO ₂	0.03	0.21
MgO	0.02	3.32
P ₂ O ₅	0.01	3.69
Mn ₂ O ₃	0.01	3.34
SO ₃	0.01	2.5
K ₂ O	0.04	0.14

Tab. 2: Concentration ranges used for the iron ore calibration

Fig. 3 to 5 show the calibration curves for the major oxides Fe_2O_3 , SiO_2 , and P_2O_5 , respectively.

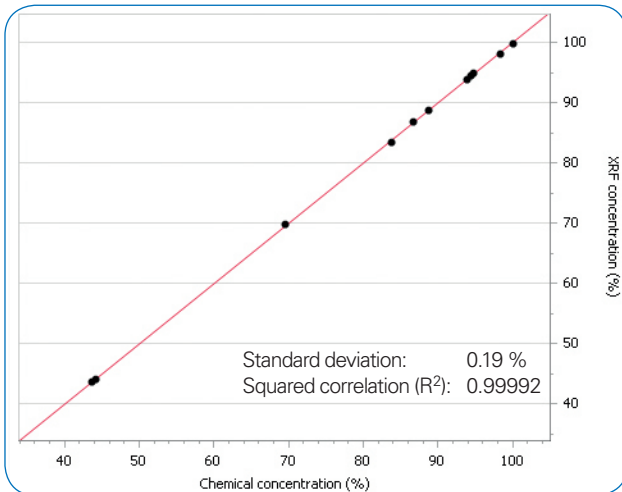


Fig. 3: Calibration curve for Fe_2O_3

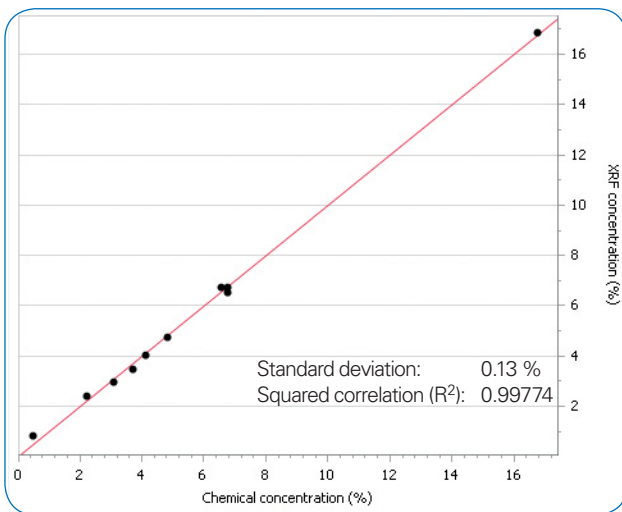


Fig. 4: Calibration curve for SiO_2

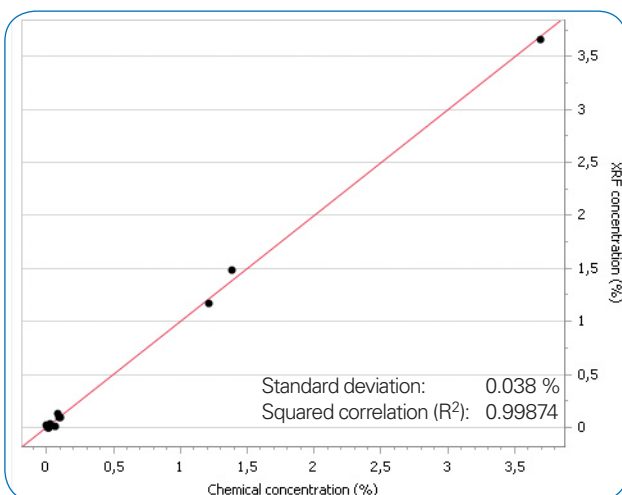


Fig. 5: Calibration curve for P_2O_5



Fig. 6: S2 PUMA XY

Results

To prove the accuracy of the calibration for iron ore samples a number of certified reference materials was measured and evaluated. Tab. 3 shows a comparison of the measured and certified concentrations for CRM 682-1 and MO7-1 from this accuracy test.

	CRM 682-1		MO7-1	
	Measured conc. [%]	Certified conc. [%]	Measured conc. [%]	Certified conc. [%]
Fe_2O_3	98.23	98.28	44.2	44.17
SiO_2	0.506	0.48	6.523	6.57
CaO	0.055	0.02	19.296	19.22
Al_2O_3	0.436	0.38	4.672	4.69
TiO_2	0.031	0.05	0.188	0.21
MgO	0.020	0.03	1.244	1.28
P_2O_5	0.070	0.08	1.15	1.21
Mn_2O_3	0.234	0.27	0.3373	0.33
SO_3	0.015	0.01	0.133	0.12
K_2O	0.024	0.01	t	t

Tab. 3: Accuracy test of iron ore calibration with two different CRMs

The precision of the system has been proven by 10 repetitive measurements of the standard reference material ECRM-601-1. For each measurement the sample has been unloaded and re-loaded into the measurement chamber. Tab. 4 shows the results obtained for these 10 measurements and compares it to the certified values of the CRM.

ECRM-601-1	Fe ₂ O ₃ [%]	SiO ₂ [%]	CaO [%]	Al ₂ O ₃ [%]	TiO ₂ [%]	MgO [%]	P ₂ O ₅ [%]	Mn ₂ O ₃ [%]	SO ₃ [%]	K ₂ O [%]
Rep_01	85.36	2.24	1.41	0.68	0.08	0.52	0.08	3.27	0.09	0.03
Rep_02	85.45	2.24	1.37	0.70?	0.08	0.49	0.07	3.26	0.08	0.03
Rep_03	85.42	2.19	1.38	0.73	0.07	0.54	0.07	3.28	0.08	0.06
Rep_04	85.33	2.29	1.35	0.69	0.08	0.55	0.09	3.27	0.09	0.07
Rep_05	85.32	2.24	1.40	0.66	0.08	0.54	0.08	3.27	0.08	0.04
Rep_06	85.29	2.23	1.42	0.69	0.08	0.49	0.07	3.31	0.08	0.04
Rep_07	85.46	2.26	1.40	0.68	0.08	0.54	0.06	3.30	0.09	0.07
Rep_08	85.42	2.34	1.35	0.68	0.07	0.55	0.07	3.31	0.09	0.08
Rep_09	85.41	2.29	1.30	0.72	0.08	0.55	0.06	3.28	0.07	0.08
Rep_10	85.38	2.31	1.46	0.67	0.07	0.53	0.09	3.29	0.07	0.03
Mean measured value	85.38	2.26	1.38	0.69	0.077	0.53	0.074	3.28	0.082	0.05
Abs. standard deviation	0.058	0.044	0.045	0.022	0.005	0.023	0.011	0.015	0.008	0.02
Certified value	85.302	2.225	1.455	0.640	0.080	0.531	0.060	3.34	0.082	0.032
Difference to ref. value	0.082	0.038	0.071	0.050	0.003	0.001	0.014	0.06	0.000	0.02

Tab. 3: Precision test of ten repetitive measurements of ECRM-601-1

Conclusion

The data shown proves the excellent performance of the S2 PUMA with XFlash® detector. With a set of 14 standard reference materials, the 10 most important elements in iron ore are determined in the required concentration range for major and minor elements. Samples have been prepared as fused beads in order to overcome any mineralogical effects due to differing mineral compositions between samples from different mining sites. In comparison to other spectroscopic techniques such as atomic absorption spectroscopy (AAS) or

inductively coupled plasma optical emission spectrometry (ICP-OES) the sample preparation required for XRF is not time consuming and does not require any chemical sample digestion steps. The high degree of accuracy and precision demonstrate the suitability of this instrument for monitoring the chemical composition of all iron ores.

Authors

Dr. Soodabeh Durali-Müller, Frank Portala, Bruker AXS, Karlsruhe, Germany

Links

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Bruker AXS GmbH

Karlsruhe · Germany
 Phone +49 721 50997-0
 Fax +49 721 50997-5654
 info.baxs@bruker.com

www.bruker.com