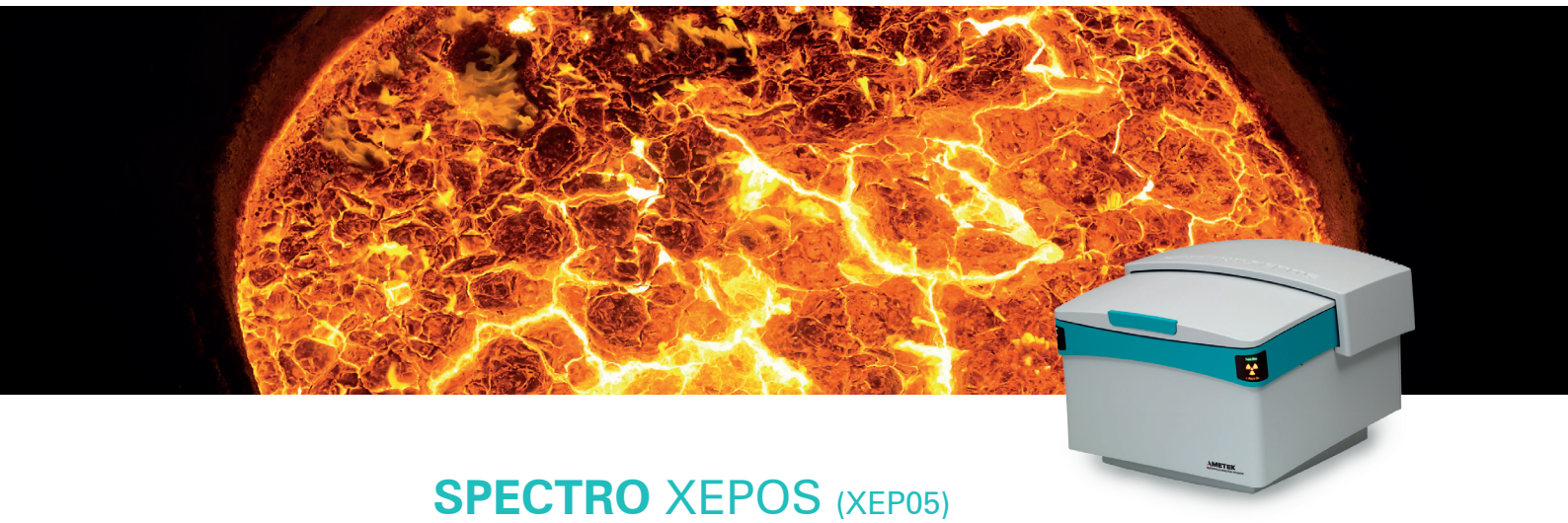
AN APPLICATION REPORT FROM
SPECTRO ANALYTICAL INSTRUMENTS

XRF-138, REV. 0



SPECTRO XEPOS (XEP05)

Analysis of Slag Prepared as Fused Beads

Introduction

X-ray Fluorescence is an accepted technique for analyzing slag. For the analysis of slag the samples should be prepared as pressed pellets or fused beads.
For the analysis of pressed pellets, dedicated calibrations should be carried out for different types of slag.
This report describes the calibration and validation of the SPECTRO XEPOS for the simultaneous analysis of the Al_2O_3, SiO_2, Fe, CaO, MgO, S, K_2O, Na_2O, TiO_2, Cr_2O_3, Mn_2O_3, CuO, and ZnO in different kinds of slag prepared as fused beads.

The SPECTRO XEPOS can be offered as a package consisting of:

- Instrument
- Fusion technique
- Calibration samples
- Validation samples
- Consumables
- Installation
- Calibration

2 Analysis of Slag Prepared as Fused Beads



The SPECTRO XEPOS simultaneous XRF spectrometer provides a fast, reliable and economic solution to analyze slags prepared as fused beads.

Fused beads

The samples were prepared as fused beads. For the preparation the material was ignited and the loss on ignition was determined. 1 g of the ignited material was mixed with 8 g of Lithiumtetaborate flux and fused to a bead with 40 mm diameter in an automatic fluxer.

Instrumentation

The samples were excited for 600 s by an air cooled low power thick binary Pd/Co alloy anode end window X-ray tube combined with an innovative monochromator, which also polarizes the primary tube spectrum.

The illustration 1 shows the schematic setup of this excitation principle. The main benefit for this monochromatic excitation is the boost in sensitivity for the analysis of the light elements Na-Cl.

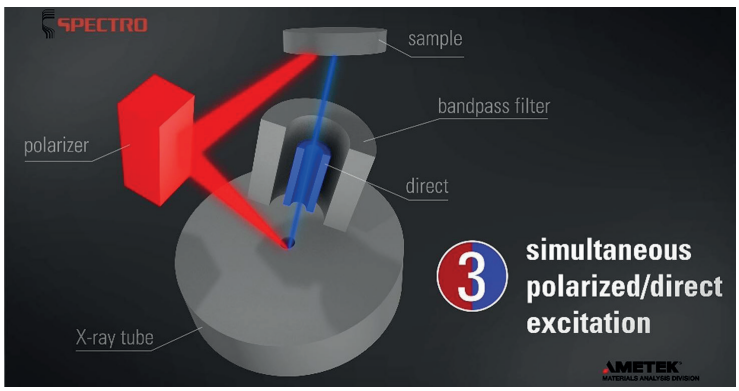


Illustration 1: Schematics of the combined direct/polarized excitation

A high resolution SDD was used to record the fluorescence radiation from the sample. The resolution for the SDD used was ≤130 eV for the Mn-Kα-line. The measurements were done in vacuum.

The components are packaged in a compact benchtop housing with a small footprint. All measurement parameters are controlled by the system PC.

Calibration and Validation Samples

For the calibration a set of commercially available fused beads from Fluxana was used. This set also includes several validation samples.

Calibration and Validation

The calibrations were performed by measuring the set of commercially available reference samples from Fluxana based on a matrix correction method using influence coefficients. The concentration ranges, the RMS-error of calibration:

$$RMS = \sqrt{\frac{\sum_{i=1}^{n}\left(C_{certified} - C_{calculated}\right)^2}{n - f}}$$

and the correlation coefficients, r, are given in Table 1.

Table 1: Calibration ranges, root mean square error of calibration and correlation coefficients for components in raw materials

	Range %	RMS %	r
CaO	64.52	0.27	0.9999
SiO_2	58.06	0.32	0.9997
Al_2O_3	35.92	0.19	0.9997
Fe	69.90	0.13	0.9999
Na_2O	3.01	0.11	0.9997
MgO	28.12	0.28	0.9993
S	1.18	0.07	0.9873
K_2O	2.53	0.02	0.9997
TiO_2	12.25	0.04	0.9999
V_2O_5	0.95	0.02	0.9987
Cr_2O_3	17.60	0.02	0.9999
MnO	13.16	0.06	0.9998
NiO	0.239	0.004	0.9994
ZnO	4.01	0.02	0.9991

4 Analysis of Slag Prepared as Fused Beads

A basic requirement for low RMS errors is the excellent sensitivity of the SPECTRO XEPOS for the given elements (oxides).

The following graphs show as examples the correlation for Al_2O_3, CaO, SiO_2, and Fe. Those graphs also show the results of the analyses of the validation samples.

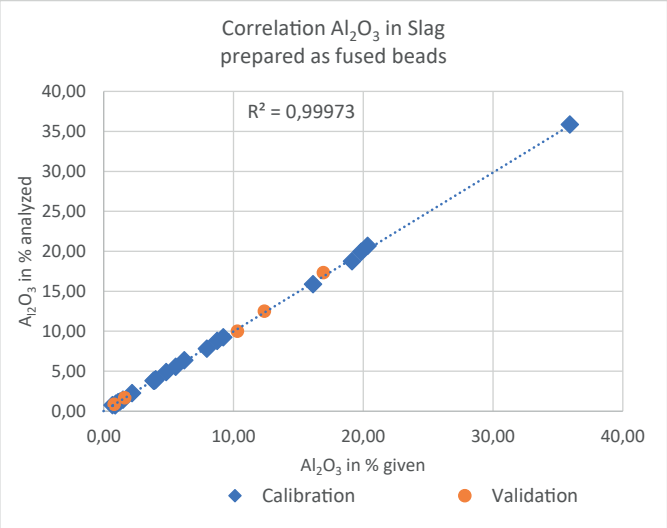


Figure 1: Correlation plot for Al_2O_3

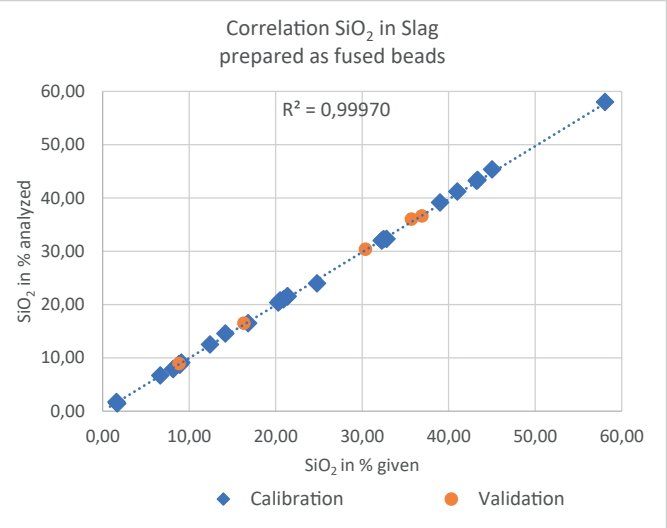


Figure 2: Correlation plot for SiO_2

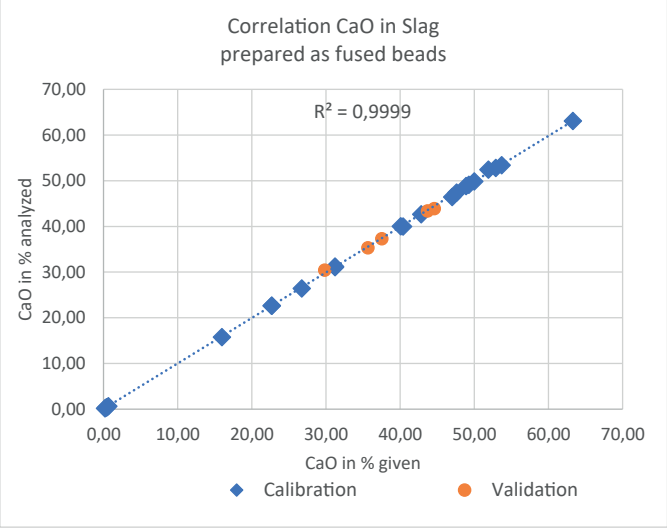


Figure 3: Correlation plot for CaO

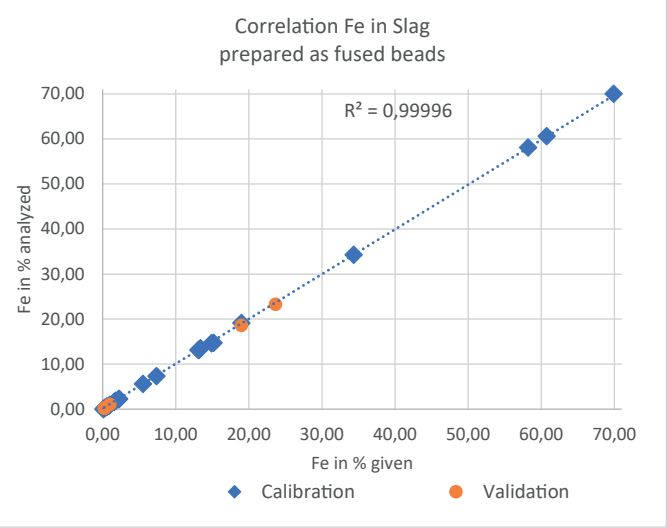


Figure 4: Correlation plot for Fe

The precision of the analyzer was tested by analyzing one sample ten times. The results of this repeatability test are displayed in Table 2.

Table 2: Results of a ten times repeated analysis

	MgO %	Al_2O_3 %	SiO_2 %	S %	CaO %	TiO_2 %	MnO %	Fe %
CMSI 1756 repeat	21.27 ± 0.07	3.93 ± 0.02	21.43 ± 0.03	0.0327 ± 0.000	22.52 ± 0.01	0.187 ± 0.001	13.05 ± 0.01	13.06 ± 0.01
CMSI 1756 repeat R02	21.27 ± 0.07	3.94 ± 0.02	21.47 ± 0.03	0.0328 ± 0.000	22.49 ± 0.01	0.186 ± 0.001	13.02 ± 0.01	13.04 ± 0.01
CMSI 1756 repeat R03	21.38 ± 0.07	3.94 ± 0.02	21.49 ± 0.03	0.0331 ± 0.000	22.50 ± 0.01	0.188 ± 0.001	13.02 ± 0.01	13.07 ± 0.01
CMSI 1756 repeat R04	21.33 ± 0.07	3.92 ± 0.02	21.43 ± 0.03	0.0329 ± 0.000	22.49 ± 0.01	0.190 ± 0.001	13.02 ± 0.01	13.07 ± 0.01
CMSI 1756 repeat R05	21.34 ± 0.07	3.92 ± 0.02	21.47 ± 0.03	0.0328 ± 0.000	22.49 ± 0.01	0.190 ± 0.001	13.03 ± 0.01	13.04 ± 0.01
CMSI 1756 repeat R06	21.35 ± 0.07	3.94 ± 0.02	21.43 ± 0.03	0.0326 ± 0.000	22.49 ± 0.01	0.188 ± 0.001	13.03 ± 0.01	13.09 ± 0.01
CMSI 1756 repeat R07	21.30 ± 0.07	3.93 ± 0.02	21.44 ± 0.03	0.0326 ± 0.000	22.51 ± 0.01	0.187 ± 0.001	13.03 ± 0.01	13.05 ± 0.01
CMSI 1756 repeat R08	21.32 ± 0.07	3.96 ± 0.02	21.52 ± 0.03	0.0326 ± 0.000	22.47 ± 0.01	0.189 ± 0.001	13.01 ± 0.01	13.09 ± 0.01
CMSI 1756 repeat R09	21.31 ± 0.07	3.94 ± 0.02	21.53 ± 0.03	0.0325 ± 0.000	22.48 ± 0.01	0.187 ± 0.001	13.02 ± 0.01	13.08 ± 0.01
CMSI 1756 repeat R10	21.27 ± 0.07	3.96 ± 0.02	21.44 ± 0.03	0.0329 ± 0.000	22.46 ± 0.01	0.187 ± 0.001	13.02 ± 0.01	13.06 ± 0.01
Average	21.31	3.94	21.47	0.0327	22.49	0.188	13.03	13.07
Std. dev.	0.04	0.01	0.04	0.0002	0.02	0.001	0.01	0.02
Certified	21.18	4.00	21.35	0.036	22.65	0.18	13.16	13.12

Summary

The results show that SPECTRO XEPOS is capable to analyze a wide range of different slag samples prepared as fused beads with good accuracy and precision. The calibrations show low values for rms and high correlation coefficients. The results of the validation samples show a good agreement between expected and analyzed concentrations.

In view of size, power and cost the SPECTRO XEPOS meets also the requirement of modern laboratories.

Choosing an ED-XRF analyzer

ED-XRF technology keeps getting better and better. Today's most advanced instruments can provide a quantum leap in performance, even over earlier top-ranked models. Look for the following benefits:

High sensitivity and precision. In multi-element analysis of major, minor, and trace element concentrations, it's critical to maximize spectrometric sensitivity and precision. Example: the newest SPECTRO XEPOS analyzers combine exclusive new excitation technology with innovative detector and tube designs. These help deliver up to 10X greater sensitivity and 3X better precision than previous models. So users get fast, accurate analysis of a wide range of elements, from sodium to uranium.

Long-term stability. Most ED-XRF analyzers shut down their X-ray tube between measurements. Unfortunately, resulting temperature variations can negatively affect repeatability and accurate readings. To ensure stability, look for an instrument that maintains constant tube power.

Low detection limits. Lower limits of detection (LODs) improve performance with minor and trace element concentrations. The best new models combine high sensitivity with minimized backgrounds, achieving exceptionally low LODs for a wide range of elements.

Consumables flexibility. Analysis in an inert-gas environment sometimes produces better results. But constant gas purging becomes expensive. SPECTRO XEPOS instruments offer low-volume helium purging only when required for analysis of the contents of light elements like Mg, Al, Si, P, and S. A qualitative analysis of elemets starting from C and a quantitative analysis of light elements starting from F can be done for pressed pellets, fused beads or other solid samples and is typically performed in a vacuum atmosphere in the measurement chamber.

Lower costs. Today, an advanced ED-XRF analyzer such as SPECTRO XEPOS exhibits significantly lower costs — of initial investment and long-term ownership — than wavelength dispersive X-ray fluorescence (WD-XRF) spectrometers. Yet it generally provides comparable performance for many applications.

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