AN APPLICATION REPORT FROM
SPECTRO ANALYTICAL INSTRUMENTS

XRF-132, REV. 0



SPECTRO XEPOS (XEP05)

Analysis of Raw Material Prepared as Fused Bead in the Cement Industry

Introduction

X-ray Fluorescence is an accepted technique for analyzing cement and other materials in the cement making process. The performance requirements for analyzing cement are described in various documents. The report describes the calibration and validation of the SPECTRO XEPOS for the simultaneous analysis of the SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, P_2O_5, SO_3, K_2O, Na_2O, TiO_2, Mn_2O_3, ZnO and SrO in raw materials for the cement industry prepared as fused bead.
The SPECTRO XEPOS can be offered as a package of:

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

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The SPECTRO XEPOS simultaneous XRF spectrometer provides a fast, reliable and economic solution to analyze raw material prepared as fused bead for the cement industry.

Fused bead

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 lithium tetraborate 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.

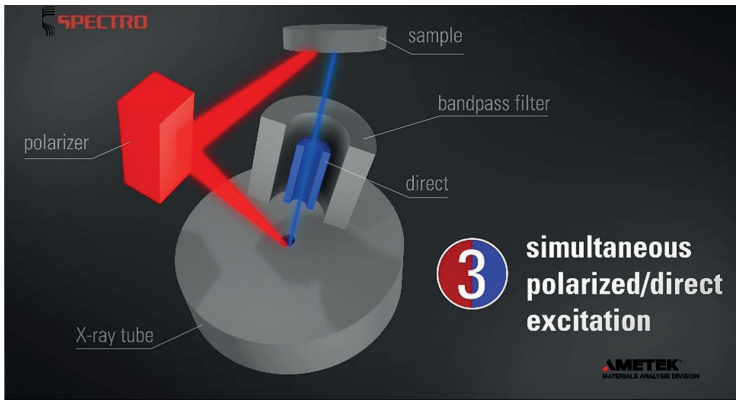

Illustration 1: Schematics of the combined direct/polarized excitation

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 bench top housing with a small footprint. All measurement parameters are controlled by the system PC.

Calibration and Validation Samples

For 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 based on a fundamental parameters method for matrix correction purposes. 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 2.

Table 2: Calibration ranges, root mean square error of calibration and correlation coefficients for components in raw materials

Oxide	Range	RMS %	r
SiO_2	0.01 – 97.9	0.28	0.9999
Al_2O_3	0.01 – 85.3	0.22	0.9999
Fe_2O_3	0.01 – 100	0.15	0.99998
CaO	0.01 – 97.9	0.31	0.9999
MgO	0.01 – 24.3	0.08	0.9999
P_2O_5	0.01 – 7.3	0.06	0.9995
SO_3	0.01 – 57.8	0.12	0.9999
K_2O	0.01 – 5.0	0.09	0.9991
Na_2O	0.03 – 3.4	0.08	0.9938
TiO_2	0.01 – 3.8	0.012	0.9995
Mn_2O_3	0.01 – 0.8	0.009	0.9991
ZnO	0.001 – 0.056	0.001	0.9986
SrO	0.001 – 0,47	0.005	0.9984

Line overlap and matrix corrections were applied where necessary. A basic requirement for low RMS errors can is the excellent sensitivities of the SPECTRO XEPOS for the given elements (oxides). The following graphs show as examples the correlation for CaO, SiO_2, Al_2O3, and Fe_2O_3. Those graphs also show the results of the analyses of the validation samples.

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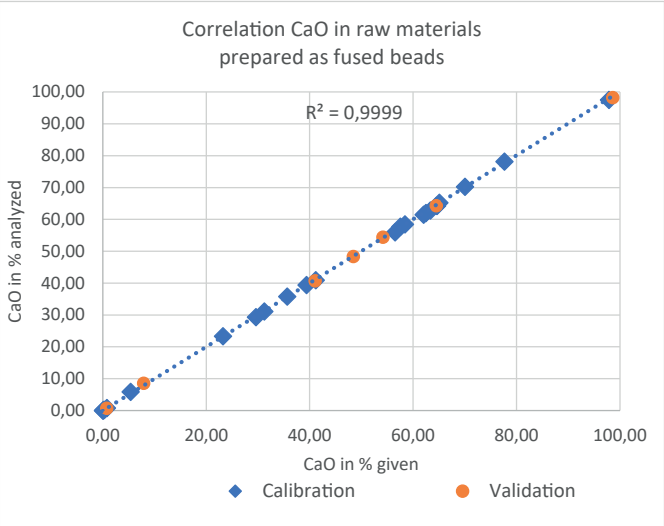

Figure 1: Correlation plot for CaO

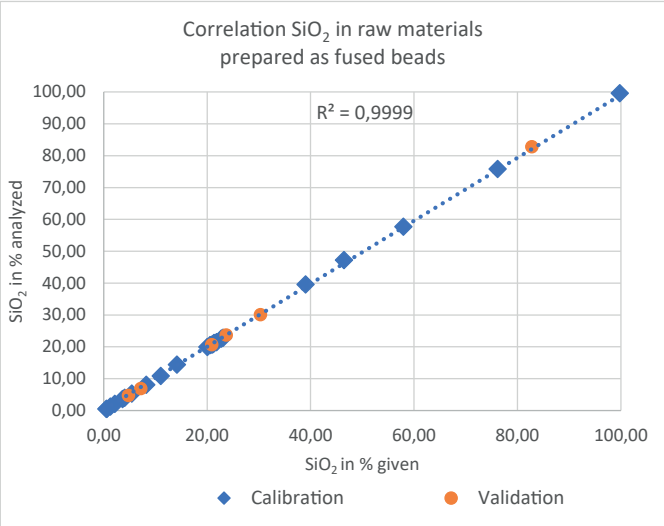

Figure 2: Correlation plot for SiO_2

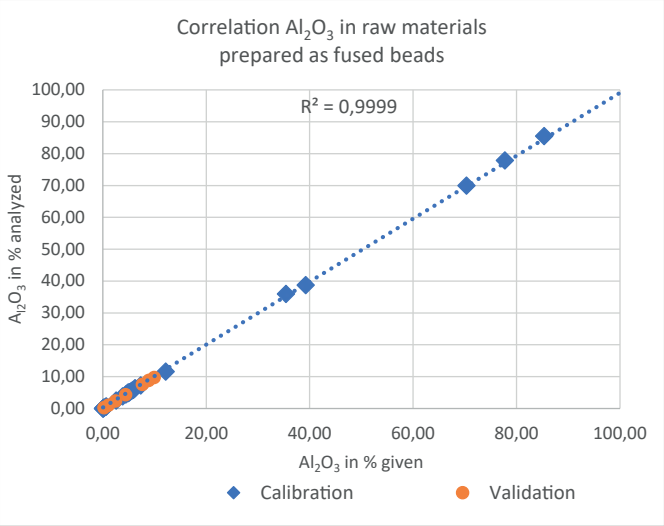

Figure 3: Correlation plot for Al_2O_3

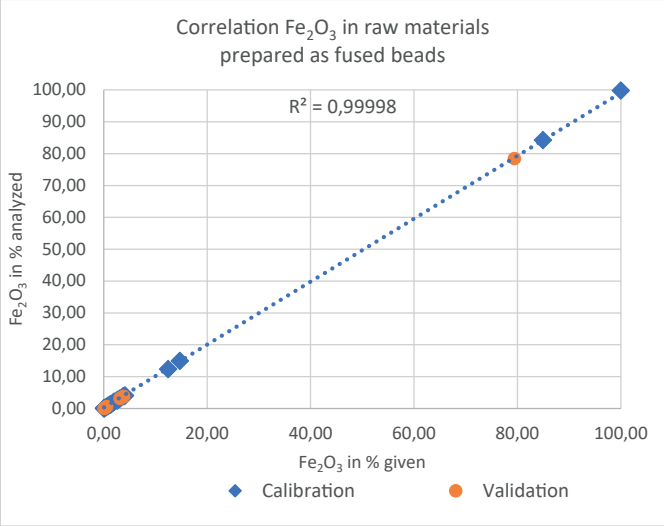

Figure 4: Correlation plot for Fe_2O_3

Summary

All results show that SPECTRO XEPOS is capable to analyze raw material for the cement industry prepared as fused beads with good accuracy. 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 require-ment 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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