Bead One R for accurate and precise analysis (ED-XRF) in cement applications

Abstract

For quantitative analysis of cement samples, HERZOG offers the Bead One R, a new benchtop fusion model with electrical resistance heating. In this application note, we assess the fusion performance of the Bead One R for cement reference material (JCA-601B). Assessment of precision and accuracy according to ISO 29581-2 showed results on expert level.

Key words

• **Borate Fusion** • **XRF** • **Quantitative Cement Analysis** • **Accuracy** • **Precision**

Introduction

The borate fusion is a sample preparation method widely used in many quality control laboratories of various industries.

Borate fusion allows the precise analysis of the elemental composition of a raw material, semi-finished or finished products. Dissolution of the material in a flux e.g., lithium borate eliminates particle size and mineralogical effects. With its features, the new BeadOne R is a precise and reliable fusion system for process control meeting the latest standards of modern cement plant operation.

Method

Weighing precision of sample and flux was at least ± 0.0003 g to minimize preparation error.

The sample: flux ratio was 1: 10 resulting in perfectly shaped and homogeneous beads. For a 40 mm diameter bead, 6 g Lithiumtetraborate

Figure 1:

Perfectly homogeneous cement fusion bead

For cement applications, commonly used fluxes are lithium tetraborate (LiT) or a mixture of lithium tetraborate and lithium metaborate (LiM). For the vast majority of cement samples, LiT is sufficient and therefore was used for this test series. Table 1 shows commonly used fluxes for cement applications.

The HERZOG Bead One R was used for fusion. For this specific application, a fusion temperature of 1075 °C with a total fusion time of 15 minutes was applied. Loss On Ignition (LOI) was determined according to **ISO 26845.** Figure 3 shows the platinum crucible and the dish with the melt inside at the passive cooling stage.

Fluxes	Ratio	Melting Point	Application
Lithium Tetraborate (LiT)	1	930 °C	Basic Oxides, Carbonates, Aluminiumsilicates, Mixtures of Cement, Magnesites
Lithium Metaborate (LiM)	1	845 °C	Acidic Oxides, Silica, Sand, Clay, Phosphates, Sulphates
LiT + LiM	50:50	870 °C	Silicates, Calcreous Products (Dolomite/Magnesites)
LiT + LiM	12:22	850 °C	Aluminiumsilicates, Acidic Oxides

Table 1: Types of fluxes for cement fusion

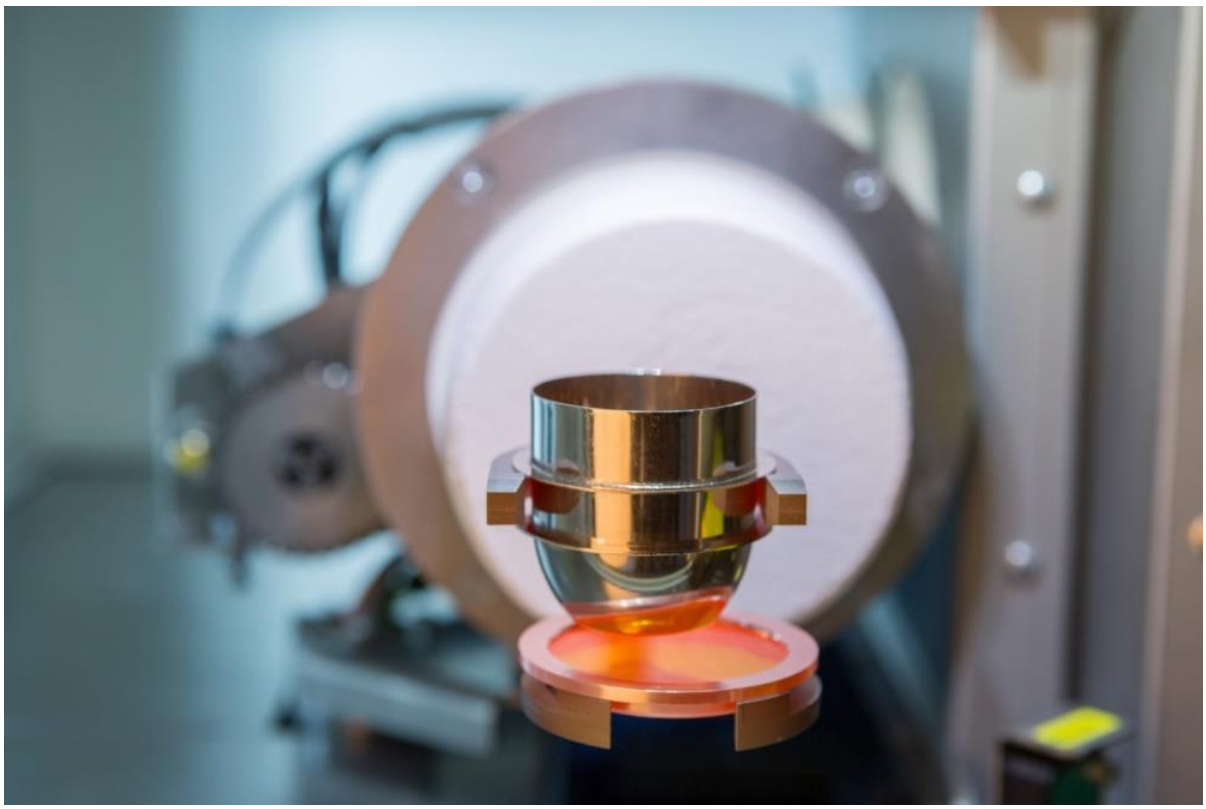

Figure 2: Hot platinum crucible and dish after casting

Results- Calibration

For XRF analysis we used a PANalytical Epsilon 3XL EDXRF. Calibration of the device was set up by using JCA-601B RM with 15 samples. Total measurement time for the XRF was set to 5 minutes for each sample.

Figure 3 shows 6 out of 12 calibration lines. Table 2 gives an overview about all squared correlation coefficients of all measured elements.

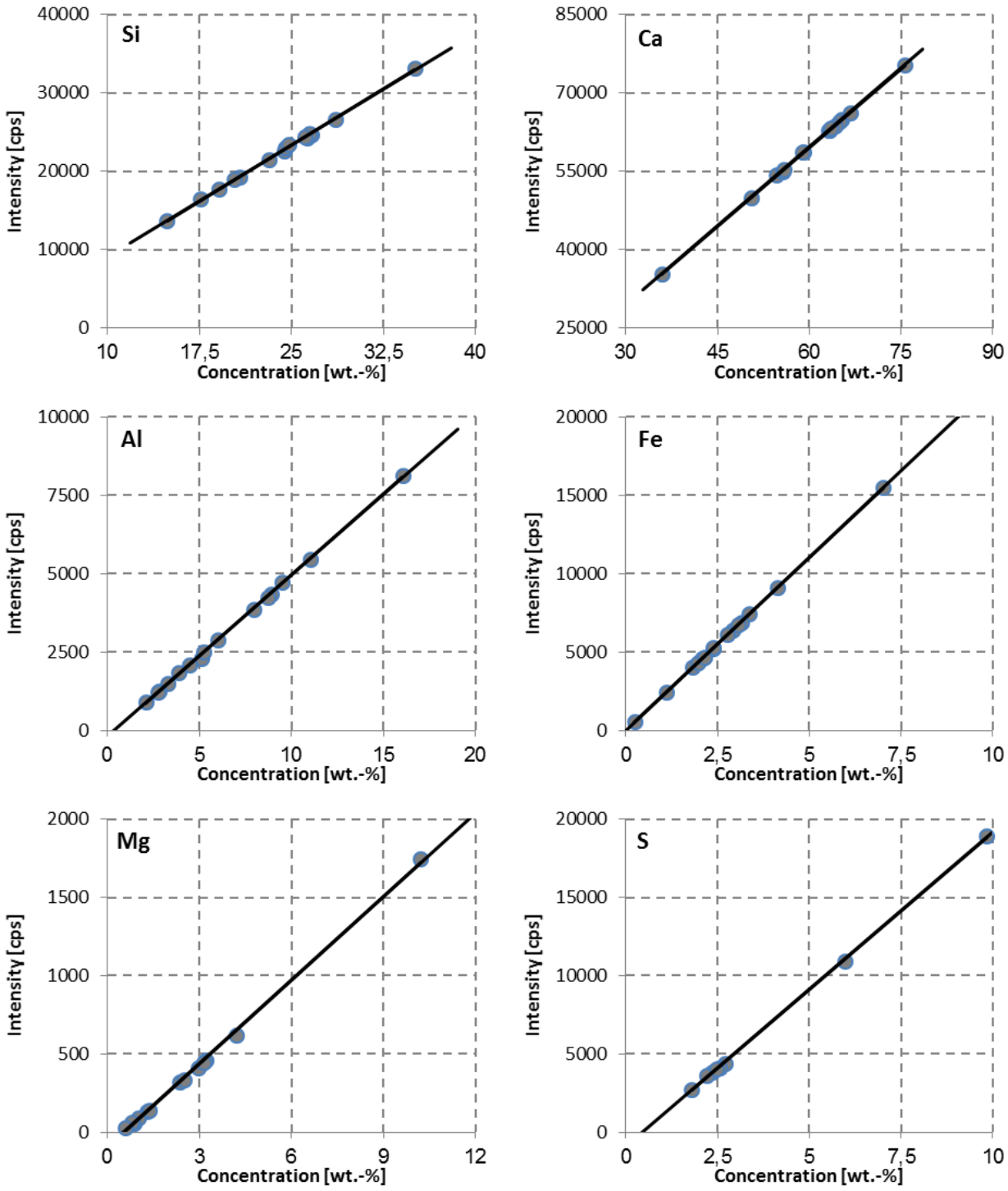

Figure 3: Calibration curves for Si, Ca, Al, Fe, Mg und S

Element	Emission line	R^2	Correction
Al	Kα	0,9995	-
Ca	Kα	0,9999	-
Fe	Kα	0,9999	Fixed α
K	Kα	0,9972	-
Mg	Kα	0,9999	-
Mn	Kα	0,9997	Fixed α
Na	ROI	0,9804	-
P	Kα	0,9994	-
S	Kα	0,9998	-
Si	Kα	0,9995	-
Sr	Kα	0,9988	Fixed α
Ti	Kα	0,9994	Fixed α

Table 2: Measured elements for cement calibration and squared correlation coefficient

Validation of method

The fusion method was validated according to **ISO 29581-2**. For assessment of accuracy and precision, the reference material JCA-601B RM was used. For industrial applications, standard limits are sufficient.

For scientific use, expert limits are recommended. Table 3 shows the results for accuracy and precision using JCA 601B no. 14. Both tests show that nearly all elements are not only within the ISO standard but also expert limits. The only exception is sodium with an accuracy level only within the standard limit.

Precision

Analyte	Na2O	MgO	Al2O3	SiO2	P2O5	K2O	CaO	Ti2O	MnO	Fe2O3	SrO
Maximum Difference	0,023	0,047	0,045	0,112	0,017	0,004	0,095	0,006	0,004	0,023	0,002
Expert Limit	0,023	0,096	0,116	0,175	0,023	0,023	0,175	0,023	0,023	0,023	0,023
Standard Limit	0,057	0,240	0,290	0,437	0,057	0,057	0,437	0,057	0,057	0,057	0,057

Accuracy

Analyte	Na2O	MgO	Al2O3	SiO2	P2O5	K2O	CaO	Ti2O	MnO	Fe2O3	SrO
Maximum Difference	0,050	0,086	0,147	0,168	0,015	0,013	0,125	0,010	0,003	0,015	0,004
Expert Limit	0,020	0,120	0,150	0,200	0,020	0,020	0,200	0,020	0,020	0,020	0,020
Standard Limit	0,050	0,300	0,350	0,500	0,050	0,050	0,500	0,050	0,050	0,050	0,050

Table 3: Results of accuracy and precision test using JCA 601B 14

Discussion

This study on cement samples demonstrates that borate fusion using the Bead One R leads to highly consistent and accurate results. All elements but sodium are within the ISO expert limit. The deviation of sodium can be explained by its low atomic number and the low EDXRF sensitivity towards small sodium concentrations. Using a WDXRF will certainly meet the requirements for analysis of small sodium concentrations.

The Bead One R is the optimal fusion device for a wide range of applications requiring the highest degree of precision and accuracy. The high concentration range of e.g. silicon and calcium of the calibration allows its application on many different materials.

References

[1] ISO 29581-2 Cement Test methods Part 2: Chemical analysis by X-ray fluorescence

[2] ISO 26845 Chemical analysis of refractories - General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods

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