XRF Calibration of Fluorine in Cement Using Borate Fusion

Introduction

The analysis of fluorine with XRF is a continuous challenge, as the element is characterized by a very weak fluorescence yield, which leads to a low sensitivity [1]. In connection with this, is the very low depth of analysis for the fluorine Kα line of just a few micrometers in the samples to be analyzed. This results in very large errors in pressed pellets due to grain size effects. Traditionally, the analysis using borate fusion was difficult due to the volatility of fluorine and to the high dilution.

Fig. 1: Electrical Fusion Machine with 4 Stations for XRF and ICP.

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The method presented here takes advantage of the capabilities of the new electrical fusion machine (EFM) from FLUXANA:

- Stable temperature control
- High precision
- Fusion with covers
- Low Dilution Method

Fig. 2: Crucible for electrical fusion instrument with removable cover.

Procedure

Sample preparation of the cement samples, which were dried at 105 °C, was conducted using borate fusion according to the Low Dilution Method [2]. This means that the ratio between sample and flux was reduced to 1:2. In this way, it was possible to achieve a much higher sensitivity for fluorine as with a conventional dilution of, for example, 1:10.

Sample Preparation

Cement sample	dried	3g
Flux FX-X65*		6g

*66% Lithium tetraborate + 34% lithium metaborate

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Calibration

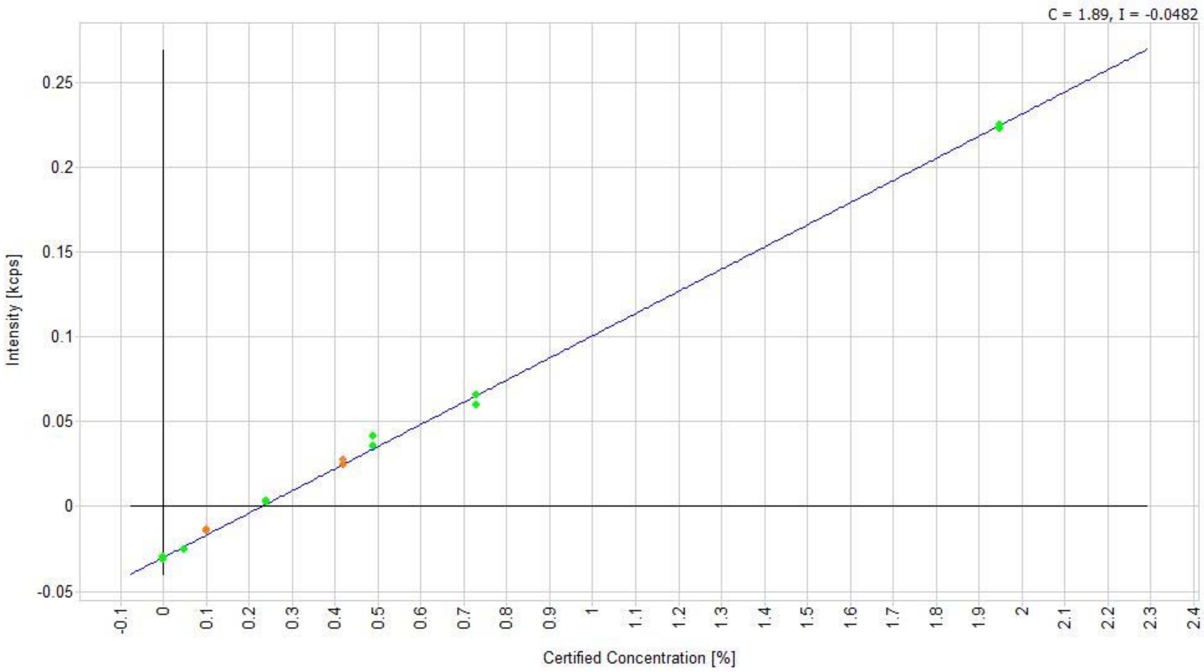

Fig. 3: Fluorine calibration in cement: Calibration samples are displayed in green; validation samples in orange. Calibration error, RMS = 0.02%.

The calibration samples were synthetically produced from the pure chemicals, calcium carbonate, calcium fluoride and silicon oxide, using the electrical fusion machines from FLUXANA. All samples were fused as duplicates. The calibration error for fluorine is 0.02%.

Validation

The cement NIST 1887b and the granite NIM-G were each prepared twice and measured with XRF to test the fluorine calibration. The results are displayed in Table 1.

Fluorine %	Cement NIST 1887b	Granite NIM-G
Certificate	0.101	0.42
Prep. 1	0.12	0.42
Prep. 2	0.12	0.44
Average	0.12	0.43

Table 1: Measured values for fluorine in the validation samples compared to the certified concentrations.

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Summary

The results presented here clearly confirm that borate beads can be produced with highest precision using the new electrical fusion machine from FLUXANA. Volatile elements, such as, for example, fluorine, for which the precision depends on the temperature stability of the fusion machine, can be satisfactorily analyzed. Through use of the Low Dilution Method, it is possible to additionally increase the sensitivity and, with it, the precision.

References

[1] Rainer Schramm, X-Ray Fluorescence Analysis: Practical and Easy - 2nd edition, FLUXANA (2017).

[2] Johnson, D.M., Hooper, P.R., Conrey, R.M., XRF Analysis of Rocks and Minerals for Major and Trace Elements on a Single low Dilution Li-tetraborate Fused Bead, Adv. X.-ray, 843 (1999).