X-Ray Fluorescence Analysis of Chlorine in Cement and Hot Meal Using Borate Fusion and the VITRIOX® ELECTRIC

Introduction

The analysis of materials containing chlorine with XRF is a continuous challenge, because chlorine displays high volatility during borate fusion. This leads to poor repeatability with conventional fusion systems.

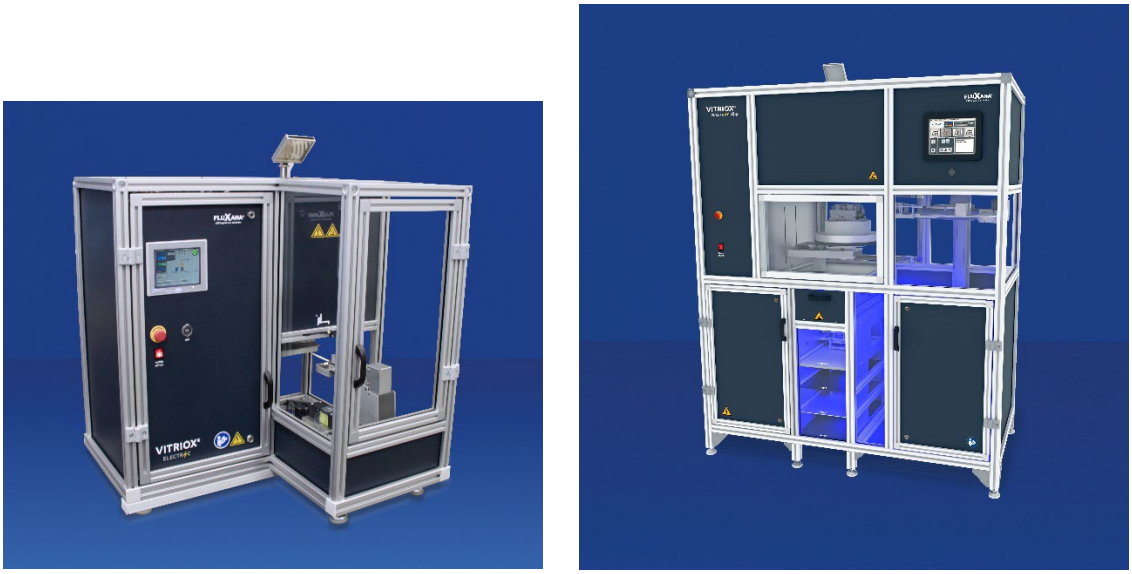

Fig. 1: VITRIOX® ELECTRIC Mono and 4+ for XRF and ICP.

The method presented here takes advantage of the capabilities of the VITRIOX® ELECTRIC from FLUXANA [1]:

- Precise temperature control
- High precision
- Fusion with covers

Fig.2: Cup for VITRIOX® ELECTRIC with removable cover.

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Procedure

Sample preparation of the sample, which had been dried at 105 °C, was conducted using borate fusion. The ratio of sample to flux was 1:8. In this way, it was possible to achieve high sensitivity for chlorine.

Sample Preparation

Cement, hot meal dried	1 g
Flux FX-X65*	8 g

*66% lithium tetraborate + 34% lithium metaborate

1. Example: Calibration of Chlorine in hot meal

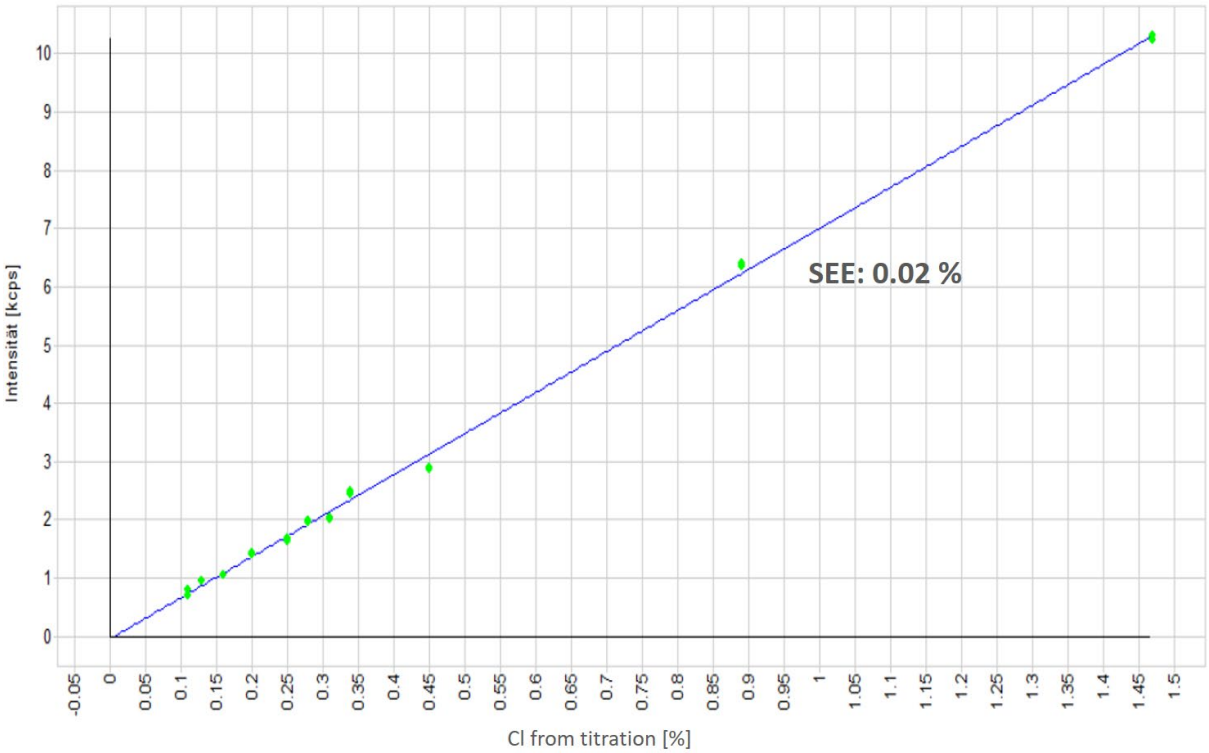

Fig. 3: Calibration for chlorine in hot meal, calibration error RMS = 0.02%. Reference values were performed with titration.

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The calibration samples were real samples provided by a customer analyzed by wet chemistry. They were fused using the VITRIOX® ELECTRIC from FLUXANA. Duplicates of all samples were produced. The calibration error achieved for chlorine was 0.02%.

2. Example: Determination of chlorine in cement

Table 1 shows the results for repeat preparations with the VITRIOX® ELECTRIC of cement samples containing low chlorine concentrations.

Table 1: Determination of chlorine in cement using fused bead method.

Preparation#	Chlorine mass%
1	0,037
2	0,036
3	0,038
4	0,035
5	0,036
6	0,035
7	0,037
8	0,036
9	0,034
10	0,035
11	0,034
12	0,041
Mean	0,036
Std.dev.	**0,002**

Summary

The results presented here clearly confirm that borate beads can be produced with highest precision using FLUXANA's electrical fusion machine VITRIOX® ELECTRIC. Volatile elements, such as chlorine, for which the precision depends on the temperature stability of the fusion machine, can be satisfactorily analyzed.

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References

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