X-Ray Fluorescence Analysis of NaCl and KCl Using Borate Fusion and the VITRIOX®

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.

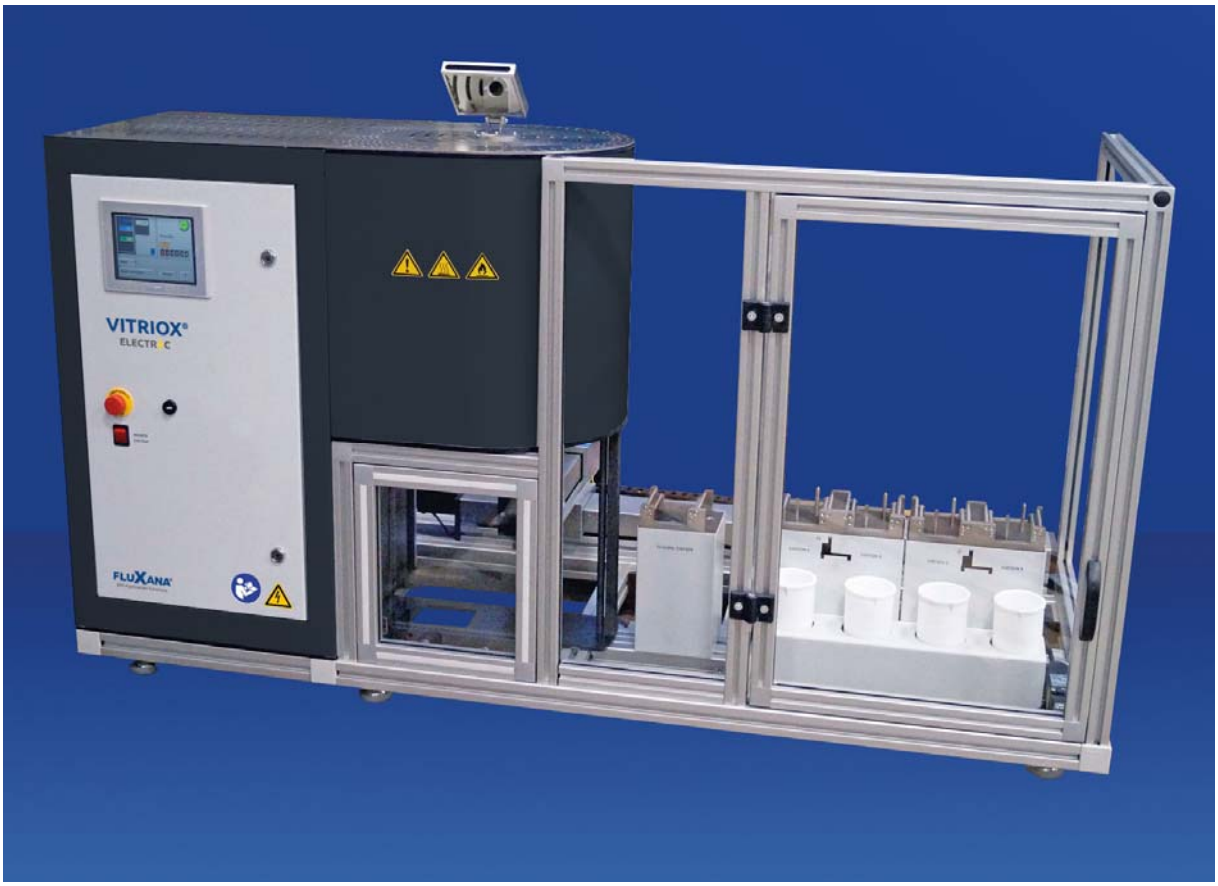

Fig. 1: Electrical Fusion Machine with 4 Stations for XRF and ICP.

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

- Precise temperature control
- High precision
- Fusion with covers

Fig.2: Cup for electrical fusion machine with removable cover.

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 as well as for sodium and potassium.

Sample Preparation

NaCl, KCl	dried	1 g
Flux FX-X65*		8 g

*66% lithium tetraborate + 34% lithium metaborate

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Calibration

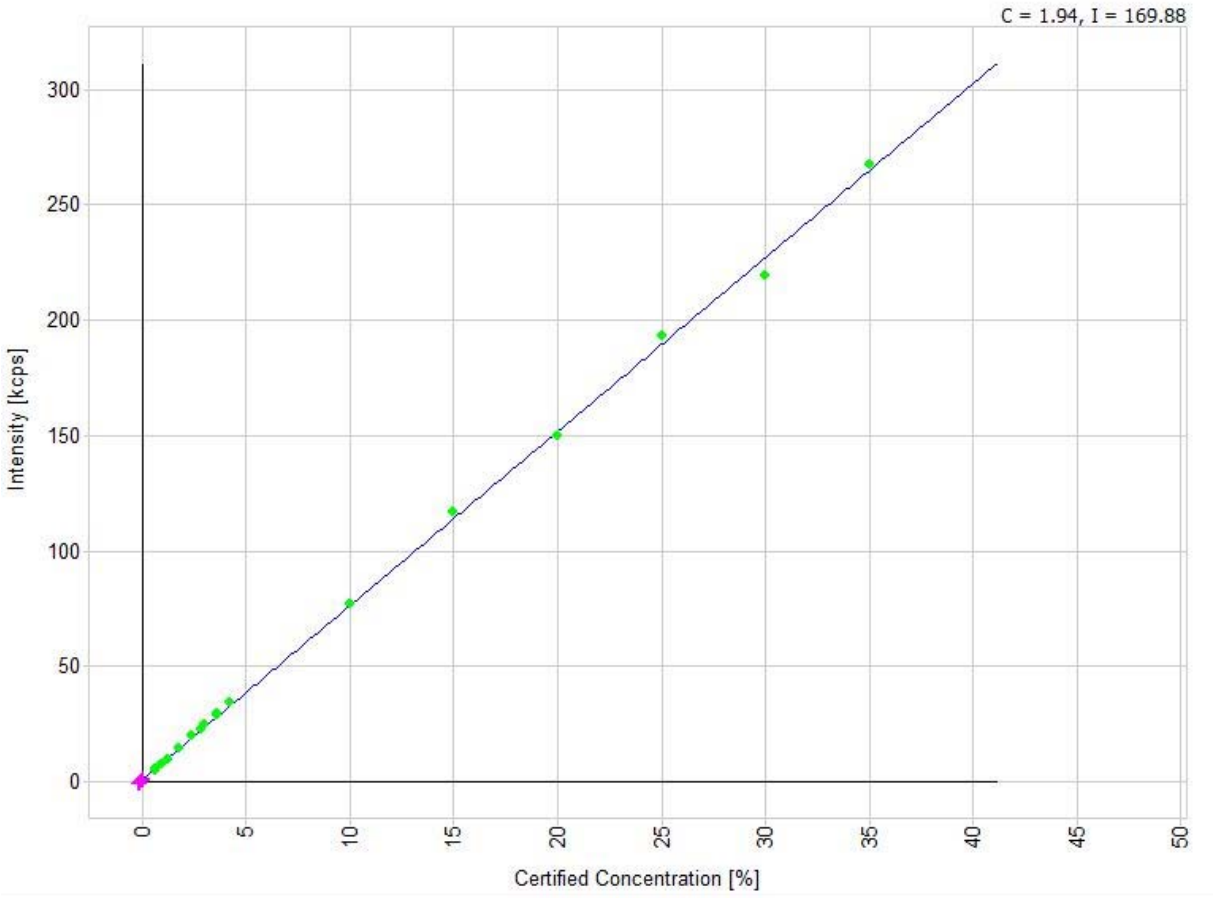

Fig. 3: Calibration for chlorine in raw materials, FLUXANA Application Package RAW-CL, calibration error RMS = 0.25%. The upper points are NaCl standards; the lower KCl.

The calibration samples were synthetically manufactured from the pure chemicals NaCl and KCl using the electrical fusion machine from FLUXANA. Duplicates of all samples were produced. The calibration error achieved for chlorine was 0.25%. There is a systematic deviation between KCl and NaCl, which is due to the differing volatility of the compounds.

Validation of the Sample Preparation

Table 1 shows the results for repeat preparations with the VITRIOX of samples containing high NaCl and KCl concentrations.

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Table 1: Repeatability of chloride in samples with high NaCl and KCl concentrations.

%	Cl	Na_2O	SiO_2	%	Cl	K_2O	SiO_2
SiO_2-NaCl Prep. 1	30.30	26.97	44.44	SiO_2-KCl Prep. 1	23.53	32.49	44.22
SiO_2-NaCl Prep. 2	30.39	27.04	44.45	SiO_2-KCl Prep. 2	23.36	32.44	44.20
SiO_2-NaCl Prep. 3	30.28	26.96	44.44	SiO_2-KCl Prep. 3	23.41	32.62	44.13
SiO_2-NaCl Prep. 4	30.30	26.98	44.40	SiO_2-KCl Prep. 4	23.36	32.46	44.22
SiO_2-NaCl Prep. 5	30.17	26.98	44.43	SiO_2-KCl Prep. 5	23.40	32.43	44.21
Average	**30.29**	**26.99**	**44.43**	**Average**	**23.41**	**32.49**	**44.20**
Standard dev.	**0.08**	**0.03**	**0.02**	**Standard dev.**	**0.07**	**0.08**	**0.04**

Summary

The results presented here clearly confirm that borate beads can be produced with highest precision using FLUXANA's new electrical fusion machine. Volatile elements, such as chlorine, for which the precision depends on the temperature stability of the fusion machine, can be satisfactorily analyzed. A systematic error depending on the bonding structure of chlorine remains. This must be considered during planning of the analysis.

References

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