XRF Application Solutions



# XRF Analysis of Feldspar and Silicates in the Glass Industry Using Borate Fusion

## Introduction

The analysis of raw materials with XRF is a common standard method [1,2]. The samples are prepared as borate beads to ensure high precision and, thus, accuracy. Here it is important that the sample is fused with the borate to 100% and that a homogenous fusion bead is produced. FLUXANA's electrical fusion machine guaranties both of these requirements. The element silicon is an important indicator for the performance. It is only homogenously distributed in the bead when both the temperature remains stable and the stirring mechanism works properly.

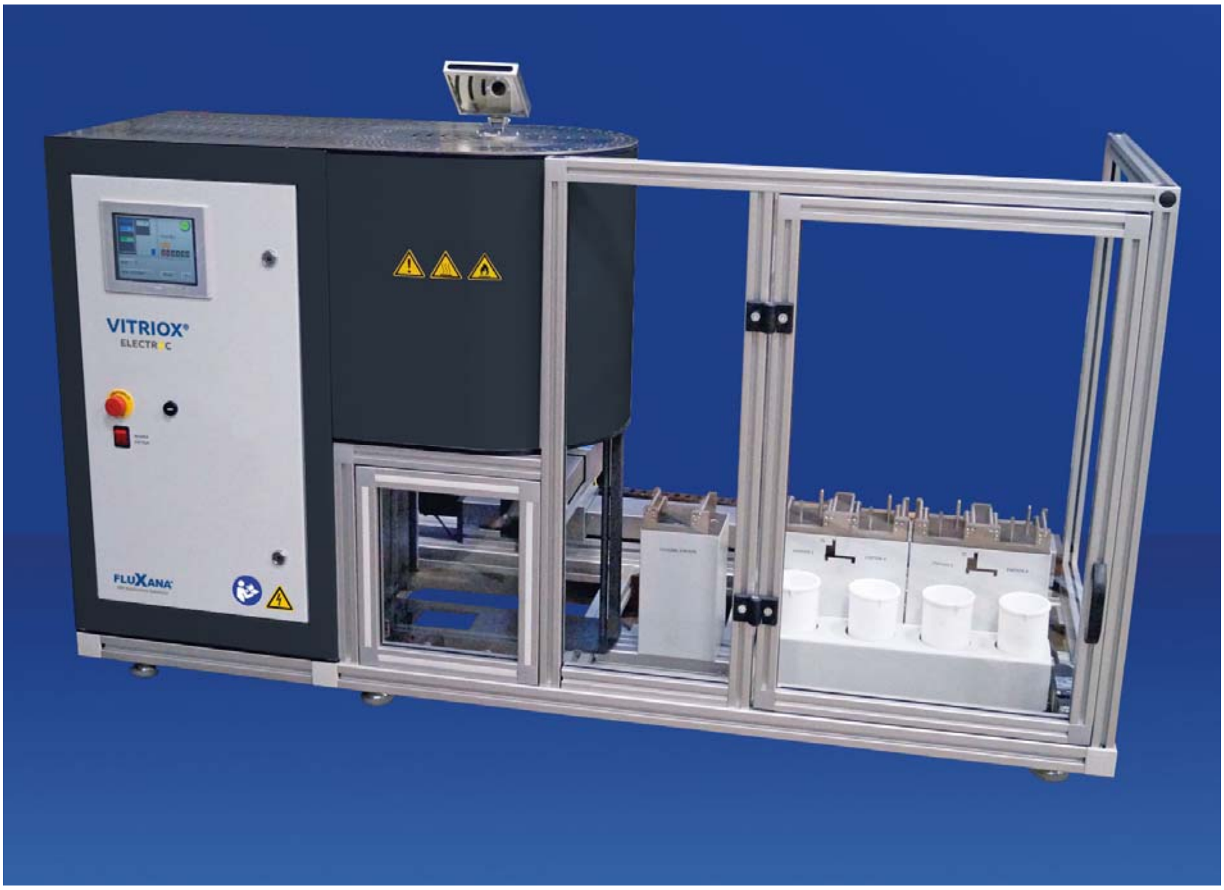


**Fig. 1: Electrical Fusion Machine with 4 Stations for XRF and ICP.**

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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

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

## Procedure

Sample preparation of the samples, which had been annealed for 1 hour at 950 °C, was conducted using borate fusion. The ratio of sample to flux was defined as 1:8. In this way, it was possible to achieve high sensitivity and precision for all elements.

## Sample Preparation

| | |
|---|---|
| Sand, Feldspar annealed* | 1g |
| Flux FX-X65** | 8g |

*1h at 950 °C

**66% lithium tetraborate + 34% lithium metaborate

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## Results

**Fig. 3 Multiple Preparation of the CRM Sample NIST 1413. Comparison with Certified Concentrations.**

| Sand sample (NIST1413) conc. mass % | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **Preparation No.** | **Al2O3** | **BaO** | **CaO** | **Cr2O3** | **Fe2O3** | **K2O** | **MgO** | **Mn2O3** | **Na2O** |
| 1 | 9.93 | 0.12 | 0.74 | 0.006 | 0.270 | 3.92 | 0.045 | 0.006 | 1.68 |
| 2 | 9.87 | 0.11 | 0.73 | 0.005 | 0.268 | 3.92 | 0.059 | 0.006 | 1.69 |
| 3 | 9.92 | 0.11 | 0.73 | 0.006 | 0.269 | 3.92 | 0.049 | 0.007 | 1.67 |
| 4 | 9.88 | 0.13 | 0.74 | 0.006 | 0.273 | 3.90 | 0.056 | 0.005 | 1.67 |
| 5 | 9.93 | 0.12 | 0.73 | 0.005 | 0.269 | 3.92 | 0.042 | 0.006 | 1.68 |
| 6 | 9.91 | 0.12 | 0.74 | 0.004 | 0.269 | 3.91 | 0.056 | 0.005 | 1.68 |
| 7 | 9.89 | 0.11 | 0.73 | 0.006 | 0.271 | 3.91 | 0.056 | 0.005 | 1.67 |
| 8 | 9.92 | 0.11 | 0.74 | 0.006 | 0.269 | 3.92 | 0.062 | 0.006 | 1.67 |
| 9 | 9.92 | 0.11 | 0.73 | 0.004 | 0.273 | 3.91 | 0.064 | 0.006 | 1.70 |
| 10 | 9.91 | 0.11 | 0.74 | 0.006 | 0.272 | 3.90 | 0.058 | 0.008 | 1.67 |
| **Mean** | **9.91** | **0.12** | **0.74** | **0.005** | **0.270** | **3.91** | **0.054** | **0.006** | **1.68** |
| Std dev | 0.02 | 0.01 | 0.01 | 0.001 | 0.002 | 0.01 | 0.007 | 0.001 | 0.01 |
| **Certificate** | **9.9** | **0.12** | **0.74** | | **0.24** | **3.94** | **0.06** | | **1.75** |

| Sand sample (NIST1413) conc. mass % | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **Preparation No.** | **P2O5** | **SiO2** | **SrO** | **TiO2** | **V2O5** | **ZnO** | **ZrO2** | **LOI** | **Sum** |
| 1 | 0.039 | 82.83 | 0.010 | 0.111 | 0.009 | 0.005 | 0.007 | 1.52 | 99.7 |
| 2 | 0.041 | 82.77 | 0.015 | 0.113 | 0.007 | 0.004 | 0.011 | 1.49 | 99.6 |
| 3 | 0.040 | 82.79 | 0.010 | 0.110 | 0.007 | 0.004 | 0.008 | 1.50 | 99.6 |
| 4 | 0.040 | 82.76 | 0.012 | 0.112 | 0.006 | 0.004 | 0.004 | 1.49 | 99.6 |
| 5 | 0.039 | 82.83 | 0.009 | 0.114 | 0.007 | 0.004 | 0.009 | 1.55 | 99.7 |
| 6 | 0.041 | 82.68 | 0.005 | 0.113 | 0.007 | 0.004 | 0.005 | 1.55 | 99.5 |
| 7 | 0.038 | 82.72 | 0.014 | 0.110 | 0.008 | 0.004 | 0.006 | 1.59 | 99.7 |
| 8 | 0.043 | 82.86 | 0.012 | 0.112 | 0.008 | 0.004 | 0.015 | 1.58 | 99.7 |
| 9 | 0.039 | 82.83 | 0.010 | 0.116 | 0.008 | 0.004 | 0.009 | 1.62 | 99.8 |
| 10 | 0.039 | 82.67 | 0.015 | 0.121 | 0.009 | 0.004 | 0.012 | 1.52 | 99.5 |
| **Mean** | **0.040** | **82.77** | **0.011** | **0.113** | **0.008** | **0.004** | **0.009** | **1.54** | **99.6** |
| Std dev | 0.001 | 0.07 | 0.003 | 0.003 | 0.001 | 0.000 | 0.003 | 0.04 | 0.1 |
| | | **82.77** | | **0.11** | | | | | |

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**Fig. 4 Multiple Preparation of Two Feldspar Samples.**

| Feldspar (128) conc. mass % | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **Preparation No.** | **Al2O3** | **BaO** | **CaO** | **Cr2O3** | **Fe2O3** | **K2O** | **MgO** | **Mn2O3** | **Na2O** |
| 1 | 20.17 | 0.000 | 0.87 | 0.001 | 0.041 | 0.19 | 0.005 | 0.003 | 10.60 |
| 2 | 20.21 | 0.008 | 0.88 | 0.003 | 0.039 | 0.18 | 0.009 | 0.002 | 10.63 |
| 3 | 20.19 | 0.004 | 0.88 | 0.002 | 0.040 | 0.19 | 0.030 | 0.002 | 10.63 |
| 4 | 20.07 | 0.009 | 0.89 | 0.002 | 0.054 | 0.19 | 0.018 | 0.002 | 10.62 |
| 5 | 20.13 | 0.007 | 0.88 | 0.003 | 0.047 | 0.19 | 0.010 | 0.002 | 10.62 |
| 6 | 20.14 | 0.004 | 0.88 | 0.001 | 0.044 | 0.19 | 0.022 | 0.003 | 10.64 |
| 7 | 20.18 | 0.005 | 0.89 | 0.003 | 0.038 | 0.19 | 0.020 | 0.003 | 10.66 |
| 8 | 20.19 | 0.009 | 0.87 | 0.002 | 0.039 | 0.19 | 0.012 | 0.002 | 10.63 |
| 9 | 20.11 | 0.006 | 0.87 | 0.001 | 0.040 | 0.19 | 0.022 | 0.003 | 10.63 |
| 10 | 20.09 | 0.004 | 0.88 | 0.002 | 0.039 | 0.19 | 0.021 | 0.003 | 10.59 |
| **Mean** | **20.15** | **0.006** | **0.88** | **0.002** | **0.042** | **0.19** | **0.017** | **0.003** | **10.62** |
| Std dev | 0.05 | 0.003 | 0.01 | 0.001 | 0.005 | 0.00 | 0.008 | 0.001 | 0.02 |

| Feldspar (128) conc. mass % | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Preparation No.** | **P2O5** | **SiO2** | **SrO** | **TiO2** | **V2O5** | **ZnO** | **LOI** | **Sum** |
| 1 | 0.008 | 68.31 | 0.052 | 0.038 | 0.005 | 0.001 | 0.14 | 100.2 |
| 2 | 0.008 | 68.38 | 0.039 | 0.025 | 0.006 | 0.001 | 0.14 | 100.3 |
| 3 | 0.007 | 68.34 | 0.047 | 0.025 | 0.005 | 0.001 | 0.13 | 100.1 |
| 4 | 0.009 | 68.26 | 0.035 | 0.040 | 0.005 | 0.001 | 0.12 | 100.1 |
| 5 | 0.008 | 68.20 | 0.041 | 0.020 | 0.005 | 0.001 | 0.12 | 100.1 |
| 6 | 0.008 | 68.36 | 0.036 | 0.022 | 0.005 | 0.002 | 0.12 | 100.1 |
| 7 | 0.009 | 68.37 | 0.053 | 0.022 | 0.004 | 0.001 | 0.12 | 100.3 |
| 8 | 0.008 | 68.28 | 0.044 | 0.024 | 0.004 | 0.001 | 0.12 | 100.3 |
| 9 | 0.012 | 68.40 | 0.032 | 0.023 | 0.005 | 0.001 | 0.11 | 100.2 |
| 10 | 0.010 | 68.14 | 0.040 | 0.021 | 0.005 | 0.001 | 0.11 | 99.9 |
| **Mean** | **0.008** | **68.30** | **0.042** | **0.026** | **0.005** | **0.001** | **0.12** | **100.2** |
| Std dev | 0.001 | 0.08 | 0.007 | 0.007 | 0.001 | 0.000 | 0.01 | 0.1 |

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**Feldspar (129) conc. mass %**

| Preparation No. | Al2O3 | BaO | CaO | Cr2O3 | Fe2O3 | K2O | MgO | Mn2O3 | Na2O |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 16.46 | 0.13 | 0.14 | 0.002 | 0.120 | 10.81 | 0.031 | 0.004 | 2.08 |
| 2 | 16.57 | 0.13 | 0.13 | 0.004 | 0.123 | 10.74 | 0.031 | 0.004 | 2.08 |
| 3 | 16.68 | 0.14 | 0.14 | 0.003 | 0.126 | 10.80 | 0.023 | 0.002 | 2.10 |
| 4 | 16.55 | 0.13 | 0.13 | 0.002 | 0.121 | 10.81 | 0.024 | 0.004 | 2.11 |
| 5 | 16.62 | 0.13 | 0.14 | 0.002 | 0.129 | 10.80 | 0.028 | 0.003 | 2.08 |
| 6 | 16.54 | 0.13 | 0.15 | 0.002 | 0.121 | 10.78 | 0.026 | 0.003 | 2.10 |
| 7 | 16.57 | 0.13 | 0.14 | 0.003 | 0.125 | 10.80 | 0.033 | 0.004 | 2.09 |
| 8 | 16.62 | 0.14 | 0.13 | 0.002 | 0.137 | 10.82 | 0.028 | 0.004 | 2.09 |
| 9 | 16.58 | 0.13 | 0.14 | 0.003 | 0.124 | 10.82 | 0.026 | 0.003 | 2.09 |
| 10 | 16.52 | 0.14 | 0.15 | 0.002 | 0.118 | 10.81 | 0.032 | 0.003 | 2.08 |
| **Mean** | **16.57** | **0.13** | **0.14** | **0.002** | **0.124** | **10.80** | **0.028** | **0.003** | **2.09** |
| Std dev | 0.06 | 0.00 | 0.01 | 0.001 | 0.005 | 0.02 | 0.004 | 0.001 | 0.01 |

**Feldspar (129) conc. mass %**

| Preparation No. | P2O5 | SiO2 | TiO2 | V2O5 | ZnO | ZrO2 | LOI | Sum |
|---|---|---|---|---|---|---|---|---|
| 1 | 0.073 | 70.01 | 0.030 | 0.006 | 0.002 | 0.008 | 0.44 | 99.7 |
| 2 | 0.072 | 70.12 | 0.052 | 0.005 | 0.002 | 0.004 | 0.44 | 99.8 |
| 3 | 0.074 | 70.15 | 0.030 | 0.007 | 0.001 | 0.004 | 0.47 | 100.3 |
| 4 | 0.073 | 70.04 | 0.040 | 0.005 | 0.001 | 0.004 | 0.47 | 99.9 |
| 5 | 0.071 | 70.16 | 0.038 | 0.007 | 0.001 | 0.006 | 0.45 | 100.1 |
| 6 | 0.071 | 70.01 | 0.036 | 0.007 | 0.001 | 0.005 | 0.45 | 99.9 |
| 7 | 0.070 | 70.03 | 0.039 | 0.006 | 0.001 | 0.003 | 0.45 | 100.0 |
| 8 | 0.071 | 70.06 | 0.038 | 0.006 | 0.002 | 0.003 | 0.45 | 100.1 |
| 9 | 0.073 | 70.12 | 0.035 | 0.006 | 0.001 | 0.003 | 0.42 | 99.9 |
| 10 | 0.071 | 70.09 | 0.035 | 0.005 | 0.001 | 0.005 | 0.42 | 100.0 |
| **Mean** | **0.072** | **70.08** | **0.037** | **0.006** | **0.001** | **0.004** | **0.45** | **99.9** |
| Std dev | 0.001 | 0.06 | 0.006 | 0.001 | 0.000 | 0.002 | 0.02 | 0.2 |

The results display the very high precision and accuracy. The software-regulated temperature control in the electrical fusion machine is responsible for this. The high speed circular movement during mixing ensures that even difficult to dissolve elements, such as silicon, are homogeneously distributed in the fused bead. Volatile elements, like sodium or potassium, are retained by the closed oven design and, therefore prevented from being lost.

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**Summary**

The results presented here clearly confirm that borate beads can be produced with high precision using FLUXANA's new electrical fusion machine. Volatile elements, such as the alkalines, for which the precision depends on the temperature stability of the fusion machine, can be satisfactorily analyzed. Even elements that are difficult to dissolve, such as silicon, can be determined with high precision.

**References**

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

[2] www.fluxana.com