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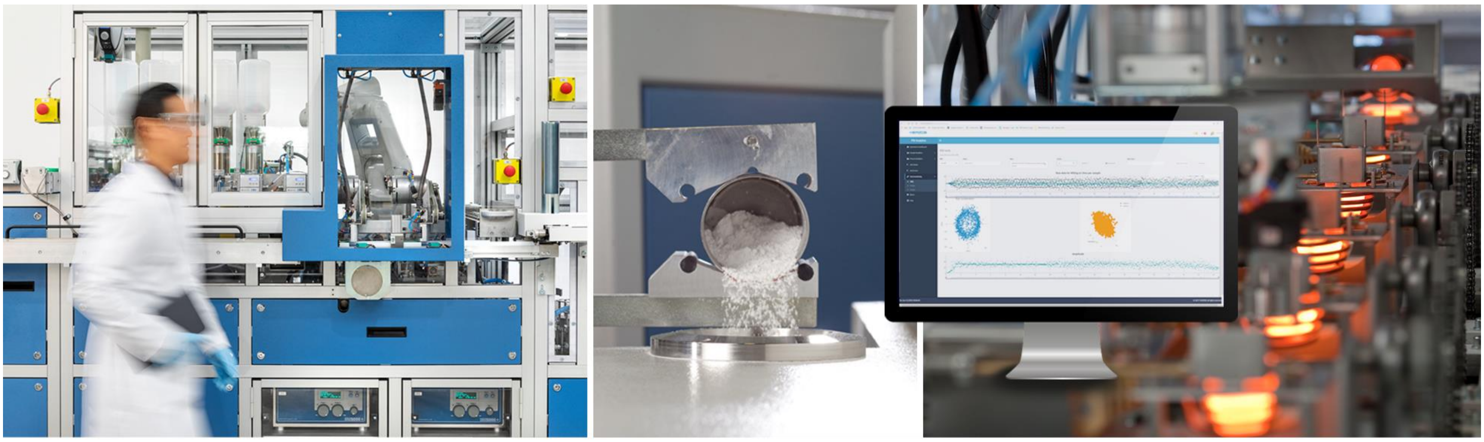

Precision and Accuracy in Automated Induction Fusion for ASTM C114 Compliance

Abstract

This application note evaluates the performance of a fully automated induction fusion system (HAG-HF) compared to a manual fusion process (Bead One HF) in meeting the precision and accuracy requirements of ASTM C114 standards for chemical analysis. Glass beads were prepared from National Institute of Standards and Technology (NIST) reference materials. The results demonstrated that both the automated and manual processes delivered comparable precision and accuracy. All measured values met the ASTM C114 limits, confirming that the fully automated fusion process ensures reliable, high-precision sample preparation for XRF analysis.

Key words

• Borate Fusion • Accuracy • Precision • ASTM C114 • NIST

Introduction

Borate fusion is a widely used method for sample preparation of powdered oxidized material. The fusion procedure results in a homogeneous and flat glass bead that is particularly suitable for analysis by X-ray fluorescence (XRF) instruments. Moreover, as the XRF is not impaired by particle size and mineralogy effects [1, 2], borate fusion results in high analytical reproducibility and accuracy.

Three main heating techniques are available to melt borate and dissolve oxides for glass bead production: electrical resistance furnaces, gas burners, and high-frequency induction heating units.

Additionally, preparatory steps, including the dosing and mixing of samples and flux, can be done manually or fully automatically.

In this study note we aimed at verifying the compliance of a fully automatic induction fusion process with the requirement of quantitative chemical analysis. At the same time, we wanted to compare the performance of automation with a manual process that has been carried out with the utmost care by an experienced operator. For this purpose, we use the reference material set of the National Institute of Standards and Technology (NIST) to determine precision and accuracy according to ASTM C 114 standard method.

Methods

For this study, 1 g of NIST reference material was fused with 6 g of borate flux to form a glass bead. We used the NIST CRM samples for this study as shown below.

NIST CRM
1880b
1881b
1884b
1885b
1886b
1887b
1888b
1889b

Simultaneous experiments were conducted using a manual (Bead One HF) and a fully automatic (HAG-HF) induction fusion device. For manual fusion, the sample and flux were weighed manually into the crucible with an accuracy of ±0.0003 g. After placing the crucible in the Bead One HF, the fusion process proceeded automatically. In contrast, the fully automatic HAG-HF system handled both dosing and fusion, achieving an accuracy of ±0.0005 g for target weights.

To enhance temperature accuracy during the fusion process, a temperature calibration of the crucible was carried out for both manual and automatic fusion (as previously described in [3]). For the automatic fusion process, we used the temperature calibration unit integrated in the HAG-HF (Figure 1). To determine the accuracy of temperature control, we performed 20 measurements using the same K-type thermocouple within the melt inside the crucible at four different target temperatures of 1050, 1075, 1100 and 1125 °C.

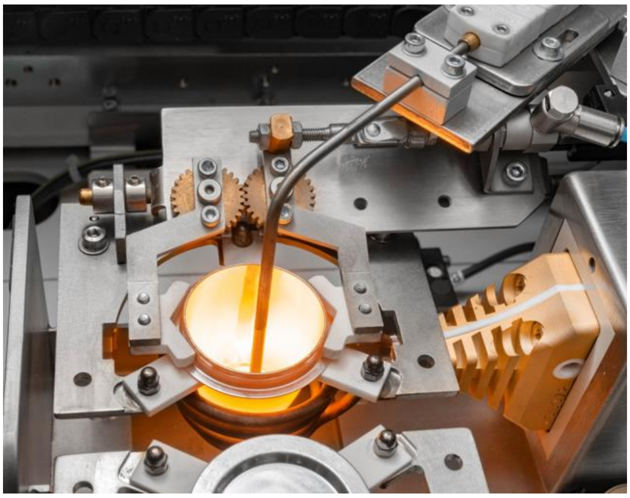

Figure 1: Automated temperature calibration unit within the HAG-HF fusion machine

Prior to fusion, the sample material was calcinated at 950 °C until constant weight. Duplicates of each CRM were prepared according to ASTM C114. For the assessment of the ASTM C114 precision test, the maximum difference of the duplicates among each other was calculated and the highest value out of 8 prepared sets of duplicates was taken for the evaluation. For determination of accuracy, the maximum difference between the 8 sets of duplicates and the reference value was calculated. For XRF analysis we used the Bruker AXS S8 Tiger II instrument (Bruker, Karlsruhe. Germany).

Results

Accuracy of temperature control

At the four targeted temperatures, the measured values were as follows: 1049.3 ± 4.5 °C (± 0.43 %); 1074.8 ± 4.6 °C (± 0.43 %); 1099.6 ± 4.8 °C (± 0.43 %) and 1125.1 ± 4.9 °C (± 0.44 %) were measured. The standard deviation remained below 5 °C at all temperatures (Figure 2).

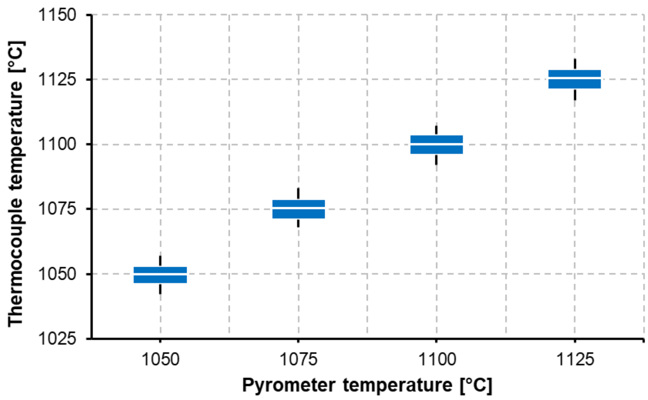

Figure 2: Boxplot diagram of the temperature measurement at four different target values

Squared correlation coefficients of XRF calibration from manual and automatic preparation

Table 1 presents the squared correlation coefficient (R^2) of the XRF calibration from manual and automatic sample preparation as well as the concentration range of this application. Overall, R^2 for the remaining elements is almost equal. SO_3 shows a higher R^2 in the automatic preparation (0.9987) than the manual preparation (0.9960) while TiO_2

shows a higher R^2 in the manual preparation (0.9991) compared to the automatic preparation (0.9971).

Element	R^2 manual preparation	R^2 automatic preparation	Concentration range [%]
SiO_2	0.9985	0.9995	18.39 - 29.05
Al_2O_3	0.9999	0.9993	3.90 - 8.81
Fe_2O_3	0.9998	0.9998	0.30 - 3.68
CaO	0.9996	0.9998	49.27 - 66.05
MgO	0.9997	0.9999	1.18 - 4.74
SO_3	0.9960	0.9987	2.63 - 4.60
Na_2O	0.9990	0.9992	0.02 - 0.79
K_2O	0.9993	0.9997	0.02 - 1.12
TiO_2	0.9991	0.9971	0.20 - 0.30
Mn_2O_3	0.9998	0.9998	0.03 - 0.20

Table 1: Squared correlation coefficient of the XRF calibration from manual and automated preparation

Maximum deviation between duplicates according to ASTM C114

Figure 3 shows the maximum difference between the duplicates according to ASTM C114. The grey bars represent the limits defined in the ASTM C114. The crosses display the maximum difference found within the manual preparation (Bead One HF) while the triangles display the maximum difference of the automatic sample preparation method (HAG-HF). For both methods, the maximum difference lies within the limits of the ASTM C114.

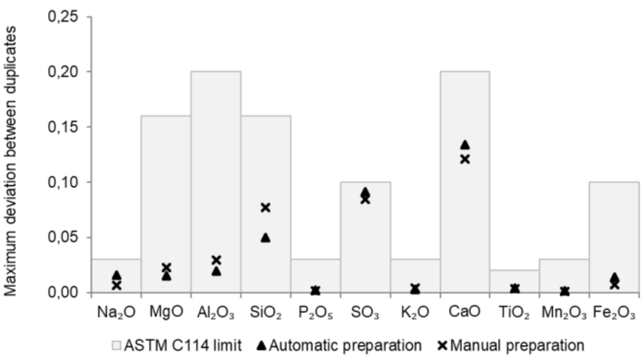

Figure 3: Display of the maximum deviation between duplicates for manual and automatic preparation

Maximum difference between the duplicates and the certificated value according to ASTM C114

In Figure 4, the grey bars represent the limits defined in the AST C114. The crosses display the maximum difference found within the manual preparation (Bead One HF) while the triangles display the maximum difference of the automatic sample preparation method (HAG-HF). The values of manual and automatic preparation lie within the limits of the ASTM C114. The maximum deviation to the certification is slightly smaller in the duplicates that were prepared automatically.

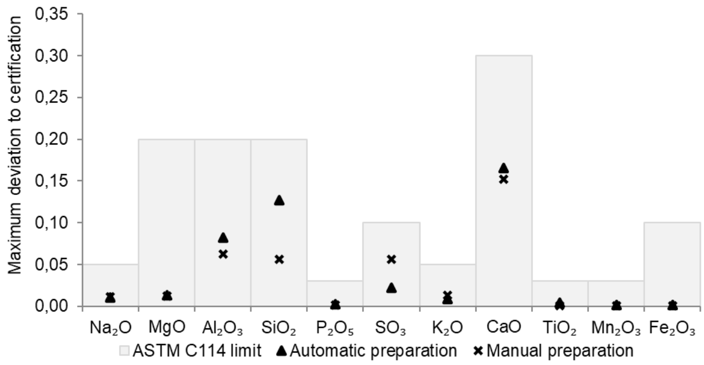

Figure 4: Display of the maximum difference between duplicate and certified value after manual and automatic preparation

Discussion

The results of this study demonstrate that fully automatic fusion using the HAG-HF meets the precision and accuracy criteria set forth in ASTM C114. These findings are consistent with those of Bouchard et al. [4], who used thermocouple-controlled electrical resistance furnaces for sample preparation together with manual dosing. Hence, our study confirms that fully automated sample preparation via induction fusion is capable of meeting the highest analytical standards.

Both automatic and manual methods produced comparable precision and accuracy, with the largest deviation observed in SO_3 values. Sulfur is known to be temperature-sensitive [1], and even minor temperature variations can affect its concentration. Nevertheless, the SO_3 values remained within ASTM C114 requirements.

One notable observation involved CaO in NIST

1889b, which showed a difference of 0.51% between the average of the duplicates and the certified value. However, the precision between the duplicates was excellent, with only a 0.001% difference. Due to the high error margin declared in the CaO certificate, we decided to exclude the value from the ASTM C114 assessment.

One of the critical factors for highly accurate analysis is exact temperature control of the fusion process. Measurements at different target temperatures between 1050 and 1125 °C showed a relative standard deviation of less than 0.5 %. Precise temperature control allows the flux to melt uniformly, without the volatile elements in the sample evaporating. In addition, induction fusion with the HAG-HF offers the advantage that the target temperatures can be reached almost immediately, so that the entire fusion process can run in a fully controlled manner.

This study demonstrates that the HAG-HF system meets the most stringent analytical demands as outlined by ASTM C114. In the accompanying application note, we will examine the performance of the HAG-HF system according to ISO 29581-2 standards.

References

[1] Spangenberg, J., Fontboté, L., & Pernicka, E. (1994): X-Ray fluorescence analysis of base metal sulphide and iron–manganese oxide ore samples in fused glass disc. X-Ray Spectrometry, 23(2), 83-90

[2] Anzelmo, J. A. (2009): “The role of XRF, inter-element corrections, and sample preparation effects in the 100-year evolution of ASTM Standard Test Method C114”, J. ASTM Int. 6, JAI101730.

[3] Herzog Application Note 23/2019: Crucible calibration for improved temperature accuracy in the induction fusion process

[4] Bouchard et al (2009): F-53 Global Cement and Raw Materials Fusion/xrf Analytical Solution. Powder Diffraction - POWDER DIFFR. 24. 10.1154/1.3176013.

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