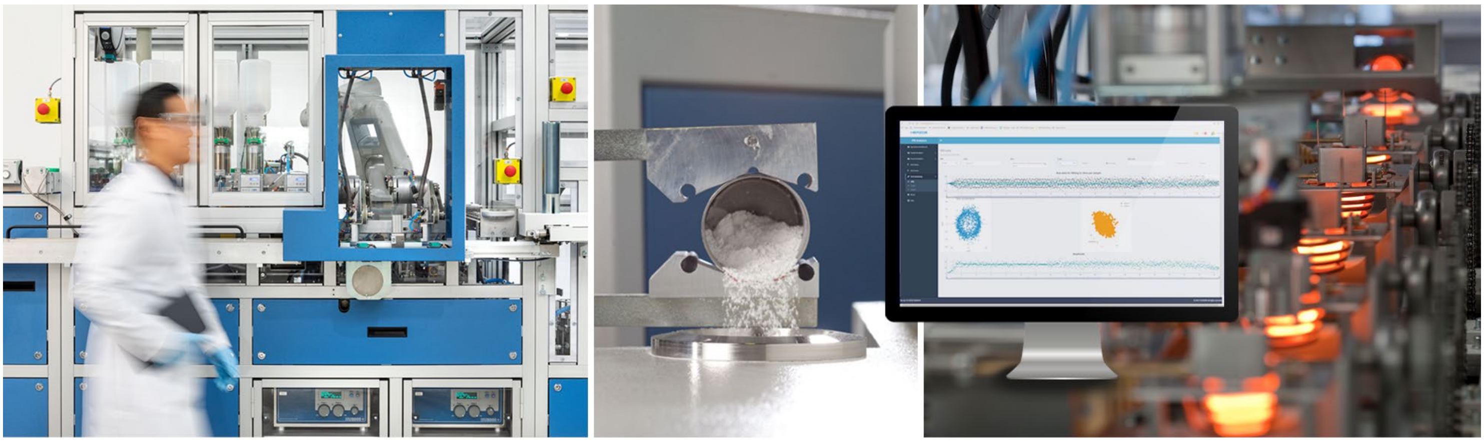

Evaluating the Precision of Automated Induction Fusion for Cement Using ISO 29581-2 Standards

Abstract

This study compares the precision of fully automated and manual sample preparation for ordinary Portland cement (OPC) according to ISO 29581-2 standards. Two series of glass beads were prepared using the HAG-HF automatic fusion system and the manual Bead One HF. X-ray fluorescence (XRF) analysis confirmed that both methods meet ISO expert precision limits. These findings demonstrate that the fully automatic inductive fusion device, HAG-HF, achieves precision comparable to that of manual preparation by experienced laboratory technicians. This paper reinforces previous findings, showcasing high accuracy and precision in accordance with ASTM C114 standards.

Key words

• Borate Fusion • Induction • Cement • Precision • ISO 29581-2

Introduction

In a previous application note [1], we demonstrated that the automatic induction fusion using the HAG-HF meets the precision and accuracy requirements outlined in ASTM C114 for chemical analysis. The results of fully automated fusion were equivalent to manual sample preparation carried out by an experienced technician.

In this study, we aim to investigate the precision of both fully automated and manual sample preparation following ISO 29581-2 standards. Two series of glass beads were produced from ordinary Portland cement: one using the HAG-HF automatic sample preparation system and the other via manual preparation with the Bead One HF. The glass beads were then analyzed using XRF, following the ISO 29581-2 method.

Introduction

The methodology follows the approach detailed in the previous application note [1]. Fusion beads were prepared in parallel using the fully automatic HAG-HF (Figure 1) and the manual Bead One HF fusion devices. Prior to fusion, temperature calibration of the Pt/Ag crucible was conducted for both machines.

For this study, 10 glass beads were produced from ordinary Portland cement (OPC). X-ray fluorescence spectroscopy was then performed on all beads, prepared both automatically with the HAG-HF and manually with the Bead One

HF. Data was generated using a Bruker-AXS S8 TIGER II 4 kW sequential wavelength-dispersive X-ray spectrometer (Bruker, Karlsruhe, Germany), equipped with a 75-position automatic sample changer and a rhodium end-window X-ray tube.

Figure 1: *Automatic induction fusion machine HAG-HF including automated dosing and cleaning*

To assess repeatability, we computed the mean and relative standard deviation (RSD_A for automatic, RSD_M for manual preparation) for all eleven elements from the XRF analysis. Subsequently, precision was assessed according to ISO 29581-2 [2], where differences between the analytical results of two successive glass bead preparations were calculated (nine values for ten beads). The maximum difference was used to determine whether the precision in each series of measurements met the normal or expert level.

Results

Determination of Repeatability

Figure 2 shows the relative standard deviation (RSD) of the measured analytes for both manual and automatic preparation of the 10 beads from ordinary Portland cement. Except for Na_2O (RSD_A = 2.37%; RSD_M = 2.53%), the RSD for all elements was below 1%. Elements with higher concentrations, such as Al_2O_3 (RSD_A = 0.22%; RSD_M = 0.15%), SiO_2 (RSD_A = 0.19%; RSD_M = 0.14%), and CaO (RSD_A = 0.06%; RSD_M = 0.04%), displayed RSD values below 0.5%. Automatic preparation resulted in lower RSD values for P_2O5, SO_3, K_2O, and TiO_2.

Deviation	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	Mn_2O_3	Fe_2O_3
1	0.011	0.006	0.005	0.022	0.001	0.025	0.002	0.069	0.001	0.001	0.007
2	0.009	0.004	0.008	0.047	0.001	0.009	0.002	0.119	0.002	0.001	0.002
3	0.005	0.003	0.010	0.061	0.000	0.011	0.004	0.055	0.002	0.002	0.002
4	0.002	0.001	0.002	0.005	0.000	0.007	0.000	0.048	0.000	0.001	0.001
5	0.004	0.007	0.016	0.053	0.001	0.020	0.001	0.027	0.001	0.001	0.000
6	0.001	0.007	0.018	0.022	0.000	0.022	0.002	0.007	0.001	0.000	0.003
7	0.001	0.000	0.031	0.049	0.000	0.005	0.001	0.017	0.000	0.000	0.003
8	0.005	0.008	0.034	0.063	0.001	0.009	0.001	0.060	0.001	0.001	0.007
9	0.002	0.004	0.022	0.001	0.000	0.001	0.001	0.021	0.000	0.002	0.009
Max. Deviation	0.011	0.008	0.034	0.063	0.001	0.025	0.004	0.119	0.002	0.002	0.009
Normal limit	0.057	0.080	0.172	0.335	0.057	0.135	0.057	0.587	0.057	0.057	0.110
Expert limit	0.023	0.032	0.069	0.134	0.023	0.054	0.023	0.235	0.023	0.023	0.044
	Expert	**Expert**	**Expert**	**Expert**	**Expert**	**Expert**	**Expert**	**Expert**	**Expert**	**Expert**	**Expert**

Table 1: *Results of the ISO 29581-2 precision test for the automatic sample preparation. According to the ISO, the results must be within the normal limits at least. All differences between the prepared glass beads are within the expert limits.*

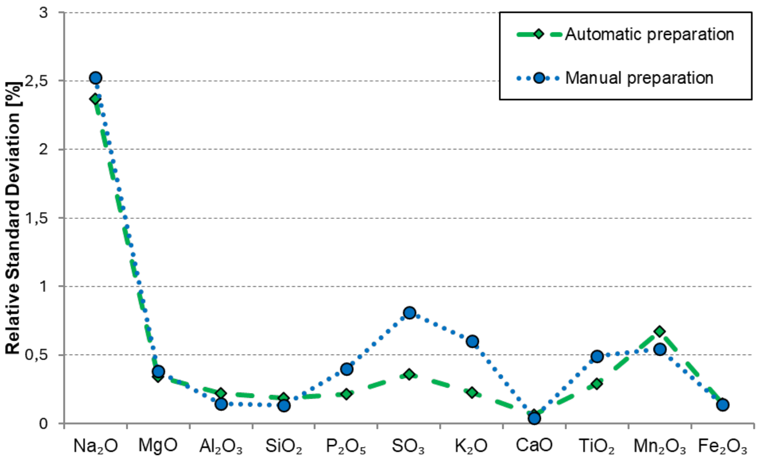

Figure 2: *Relative standard deviation of the analysis for the manual and automatic preparation.*

Determination of Precision According to ISO 29581-2

Tables 1 (automatic preparation) and 2 (manual preparation) show the results of the ISO 29581-2 precision test for the 10 beads prepared from ordinary Portland cement. The deviation was calculated by the difference between two successive preparations, and the ISO expert and normal limits were also displayed. For the automatic method, all deviations were within the ISO expert limits. In the manual method, almost all results were within expert limits, except for one SO_3 value (#6, 0.072), which remained within the ISO normal limits of 0.135.

Deviation	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	Mn_2O_3	Fe_2O_3
1	0.004	0.006	0.012	0.030	0.000	0.035	0.000	0.020	0.001	0.000	0.001
2	0.007	0.000	0.008	0.030	0.000	0.028	0.000	0.040	0.002	0.001	0.006
3	0.002	0.003	0.019	0.020	0.000	0.001	0.010	0.030	0.002	0.000	0.002
4	0.006	0.006	0.010	0.010	0.001	0.050	0.010	0.060	0.002	0.000	0.001
5	0.012	0.004	0.006	0.040	0.000	0.012	0.000	0.020	0.000	0.000	0.004
6	0.003	0.004	0.008	0.050	0.002	***0.072***	0.010	0.050	0.001	0.001	0.002
7	0.006	0.011	0.005	0.100	0.001	0.017	0.010	0.020	0.002	0.002	0.007
8	0.003	0.007	0.004	0.060	0.000	0.003	0.010	0.020	0.002	0.001	0.005
9	0.004	0.006	0.017	0.030	0.002	0.045	0.010	0.030	0.003	0.001	0.005
Max. Deviation	0.012	0.011	0.019	0.100	0.002	0.072	0.010	0.060	0.003	0.002	0.007
Normal limit	0.057	0.080	0.172	0.335	0.057	0.135	0.057	0.587	0.057	0.057	0.110
Expert limit	0.023	0.032	0.069	0.134	0.023	0.054	0.023	0.235	0.023	0.023	0.044
	Expert	**Expert**	**Expert**	**Expert**	**Expert**	**Normal**	**Expert**	**Expert**	**Expert**	**Expert**	**Expert**

Table 2: *Results of the ISO 29581-2 precision test for the manual sample preparation. The highlighted number represents the difference between two sulfur values, which is within normal limits. The other differences lie within the ISO expert limits.*

Discussion

This study demonstrates that fully automatic fusion using the HAG-HF meets ISO 29581-2 expert limits. The results from the HAG-HF closely matched those of manual sample preparation using the Bead One HF, with only one sulfur value failing to meet the expert limit in the manual process.

These test results were further supported by repeatability measurements, as demonstrated by the calculation of relative standard deviation.

In automatic sample preparation using the HAG-HF, RSD_A values were mostly below 0.5%. A significant deviation was observed only for Na_2O (2.5%), which was also present in manual preparation. Due to the low concentration of Na_2O even small differences may cause a significant increase in the relative standard deviation.

Overall, the results indicate that the entire automatic preparation process of the HAG-HF, including dosing and homogenizing, is comparable to manual procedures carried out by experienced laboratory technicians.

The precision results from this study align with the findings of Bouchard et al. [3], although the latter used JCA reference material, while our study employed commercially available OPC. While the cement used in our study may not be as homogenous as reference material, the precision was within the range observed in Bouchard et al.'s study.

In summary, the HAG-HF induction fusion system enables fast, reliable, and fully automatic creation of glass beads, providing excellent analytical precision and accuracy according to ASTM C114 and ISO 29581-2 standards.

References

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