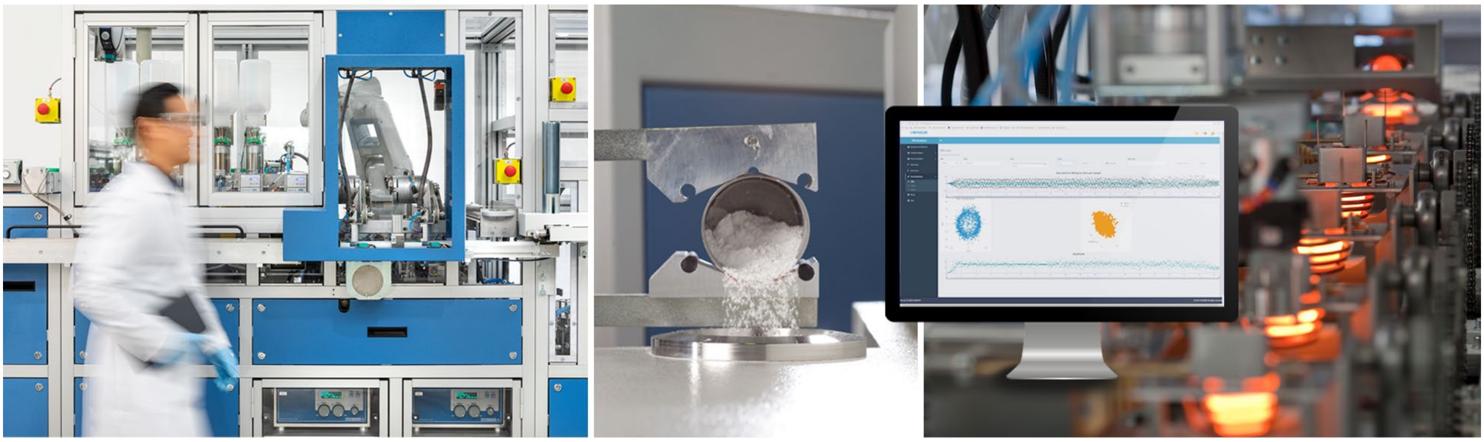

Precision and Accuracy in Sample and Flux Dosing: Evaluation of HP-GCD and HP-GID Dosing Units for Powder

Abstract

This study assesses the performance of the HP-GCD and HP-GID high-precision dosing units in achieving dilution ratios of sample and flux for borate fusion. Experiments were conducted on slag and cement samples using the automated fusion device HAG-HF with integrated dosing units. The HP-GCD dispenses sample materials, while the HP-GID handles flux dosing. As a measure for precision, the relative standard deviation of the obtained dilution was 0.007 % for slag and 0.011 % for cement. The percentage deviation from the target dilution, indicating accuracy, was 0.006 % for slag and 0.008 % for cement. These results demonstrate that the HP-GCD and HP-GID ensure highly accurate and precise powder dosing, even with variable sample properties.

Key words

• Gravimetric Dosing • Accuracy • Precision • Sample • Flux

Introduction

A critical aspect of sample preparation in many analytical methods is the precise and accurate dosing of powders, including both sample material and additives. This is especially important in procedures like borate fusion, where highly accurate dosing of the sample and flux is required for precise XRF analysis. While accurate dosing of individual components is essential, the exact dilution ratio between the sample and flux plays an even more significant role. Typically, a dilution ratio in the range of 5 to 10 is used to ensure an efficient fusion process.

In this application note, we evaluate the performance of two high-precision dosing units: the HP-GCD and the HP-GID. The HP-GCD is designed to dispense sample materials from a specialized cup into crucibles or other containers such as vials, while the HP-GID is tailored for dosing homogeneous additives from storage containers.

In a previous application note [1], we reported on the accuracy and precision of the HP-GID for various additives. Here, we extend that work by assessing the accuracy and precision of both the HP-GCD and HP-GID in achieving the target dilution ratios of flux and sample. This study focuses on two sample materials: slag and cement.

Methods

All experiments were conducted using the HP-GCD and HP-GID dosing units, which were integrated into the HAG-HF automatic fusion device. We performed 22 dosing experiments with finely ground slag (Material A) and 37 experiments with commercially available ordinary Portland cement (Material B).

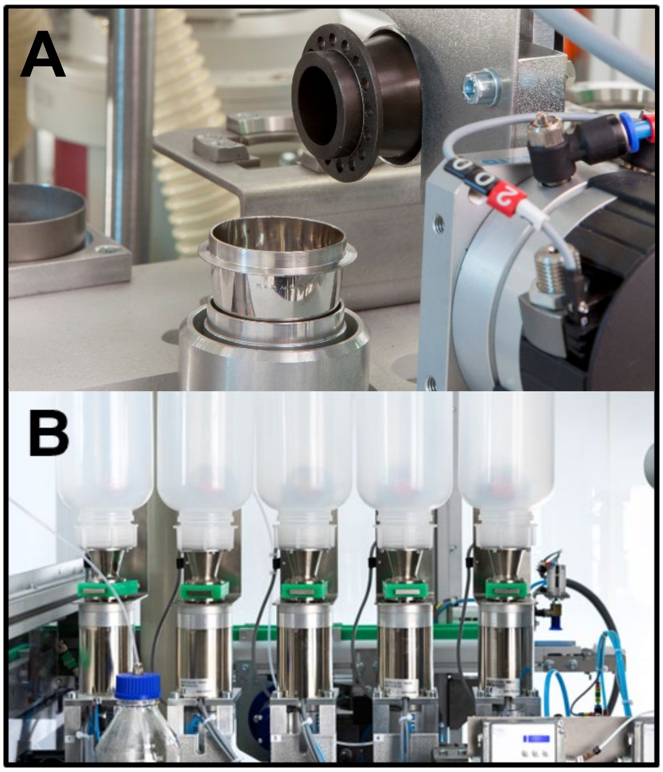

Figure 1: Photographs of the dosing units HP-GCD for sample material (A) and HP-GID for additives (B).

In the first step, the sample material was dosed gravimetrically from a special sample cup into a platinum/gold crucible using the HP-GCD (Figure 1 A). At this stage, a dosing tolerance of up to 0.05 g was acceptable since the final dilution ratio was obtained by the precise dosing of the flux, which has a more homogeneous grain size. Next, the borate flux was dosed using the HP-GID (Figure 1 B). For both experiments, we used a 50:50 mixture of lithium tetraborate and lithium metaborate. For Material A, the target dilution ratio of flux to sample material was 6.5, while for Material B, it was set at 6.

All processes were executed automatically in the HAG-HF using the PrepMaster control software, which also automatically tracked all relevant data, including weight information.

Results

Dosing of Sample Material and Flux

For Material A (slag), the mean sample weight (± standard deviation) was 0.8681 ± 0.0059 g, resulting in a relative standard deviation (RSD) of 0.685 %. The mean flux weight was 5.6397 ± 0.0383 g, with an RSD of 0.680 %. The individual data of the test series for slag samples are shown in Figure 2 A (dosing of sample) and C (dosing of flux).

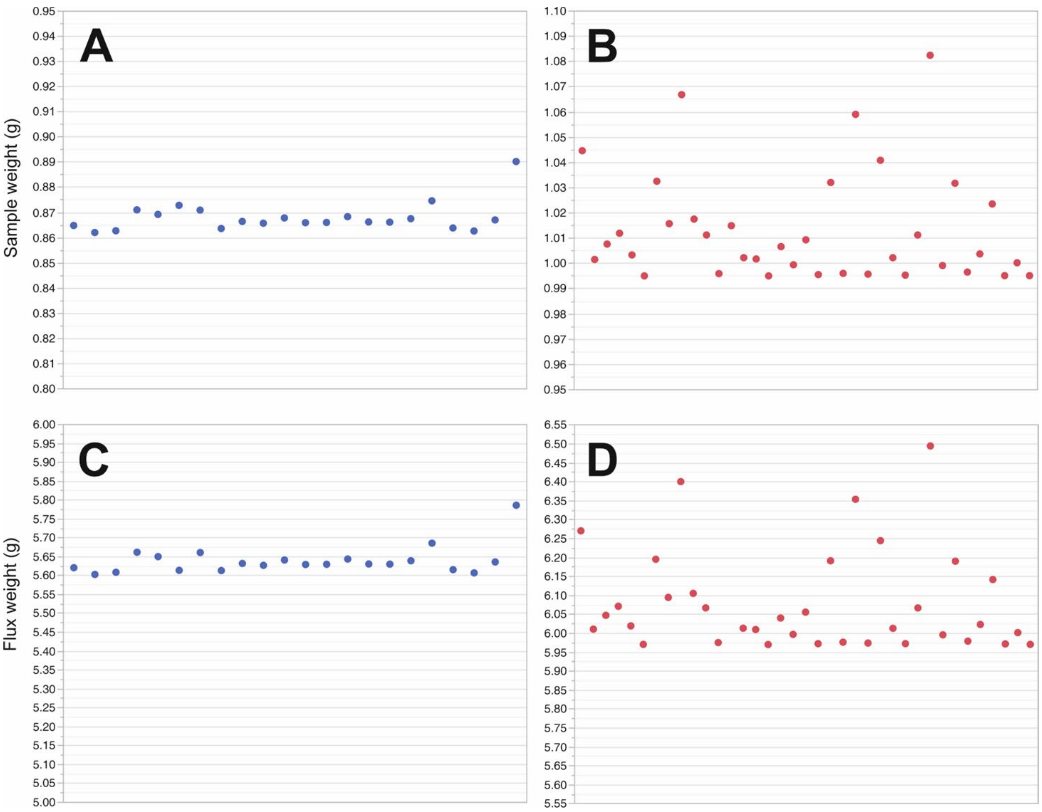

Figure 2: Diagrams of the individual measuring points of the weights (g) obtained for the sample material slag and cement, as well as the flux.

(A) Weights after dosing of slag sample material,

(B) Weights after dosing of the flux for a target dilution of flux and slag sample of 6.5

(C) Weights after dosing of cement sample material

(D) Weights after dosing of the flux for a target dilution of flux and cement sample of 6.0

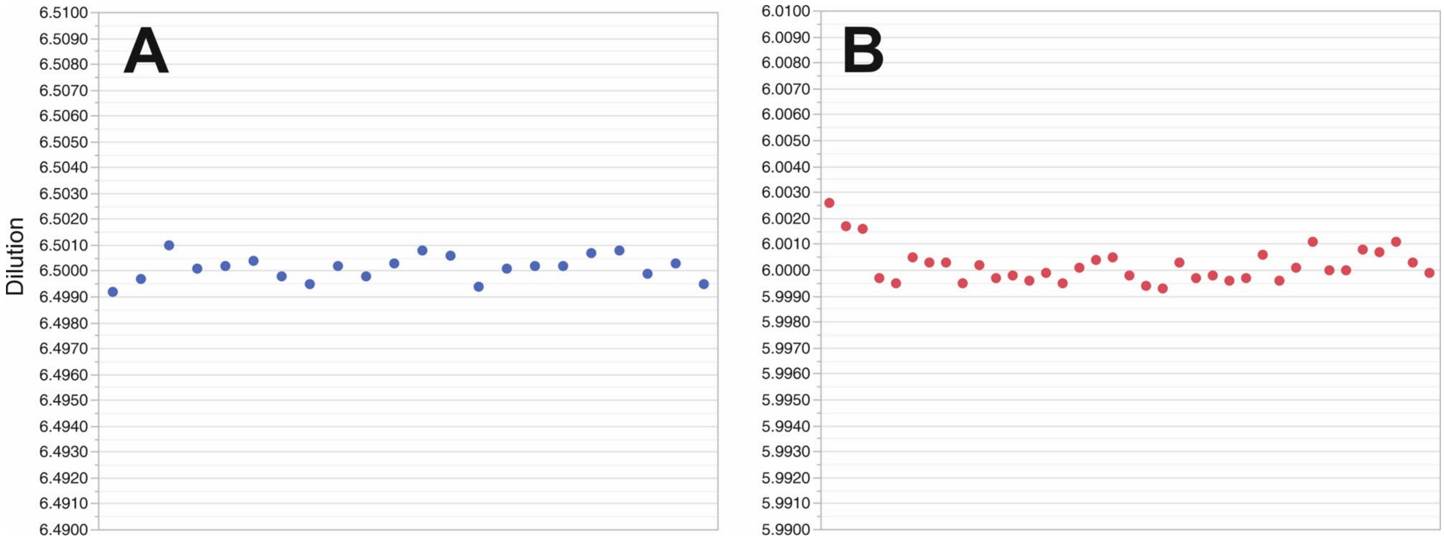

Figure 3: *Diagrams of the individual data points for the dilution of flux and sample material. (A) Dilution ratio of flux and slag samples (target value 6.5), (B) Dilution ratio of flux and cement samples (target value 6.0)*

For Material B (cement), the mean sample weight was 1.0132 ± 0.0218 g, yielding an RSD of 2.157 %. The mean flux weight was 5.9638 ± 0.7159 g, with an RSD of 12.005 %. The single data are shown in Figure 2 B and D.

Dilution of Sample Material and Flux

For Material A, the mean dilution ratio (± standard deviation) was 6.5001 ± 0.0005, with a corresponding RSD of 0.007 % (original data in Figure 3, A). The mean deviation from the target dilution was 0.0004 ± 0.0002 g, equating to a mean percentage deviation of 0.006 % (Figure 4).

For Material B, the mean dilution ratio was 6.0001 ± 0.0007, with an RSD of 0.011 % (original data in Figure 3, B).

Figure 4: Boxplot diagram of the percentage deviation from the target dilution for slag and cement.

The mean deviation from the target was 0.0005 ± 0.0005 g, corresponding to a mean percentage deviation of 0.008 % (Figure 4). The boxplot diagram in Figure 4 shows that the somewhat higher percentage deviation from the target dilution for cement is mainly caused by three outliers at the beginning of the test series.

Discussion

The results of this study demonstrate that the HP-GCD and HP-GID dosing units provide excellent accuracy and precision in preparing dilutions of sample and flux. Precision, as measured by the relative standard deviation of the obtained dilution ratios, was 0.007% for Material A and 0.011% for Material B. Accuracy, reflected in the percentage deviation from the target dilution, was 0.006% for Material A and 0.008% for Material B.

The slightly lower precision and accuracy observed for the cement sample compared to slag is likely due to the higher variability in cement dosing. The RSD for cement sample weight was 2.157%, while for slag, it was only 0.685%. However, this variability in cement dosing was largely mitigated by the precise flux dosing from the HP-GID, resulting in only a marginally higher RSD for the cement dilution compared to slag.

The increased variability in cement dosing may be attributed to the formation of agglomerates within the sample material, leading to less consistent filling of the target vessel compared to the more homogeneously grained slag.

Furthermore, it was shown that the higher variability was mainly caused by three values at the beginning of the test series. Subsequently, a very uniform course with lower variability was observed. Therefore, procedural or material-related causes at the beginning of the test series cannot be ruled out as the cause of the slightly lower precision and accuracy for material B.

In a previous application note, we demonstrated the high precision and accuracy of the HP-GID for dosing additives such as fluxes. This study further shows that even powder blends can be prepared with exceptional accuracy and precision. The combined use of the HP-GCD and HP-GID effectively compensates for unfavorable sample properties that might otherwise compromise dosing accuracy.

References

[1] HERZOG Application Note 42/2021: High-precision automatic dosing of hygroscopic fluxes for analysis with XRF or ICP-OES.

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