High-precision automatic dosing of hygroscopic fluxes for analysis with XRF or ICP-OES

Abstract

Borate fusion is an effective preparation method for analysis by using XRF, ICP-OES, ICP-MS or AA. In order to achieve the highest possible analytical accuracy and precision both sample and fusion agent need to be dosed very precisely. In this application note we aimed at investigating the precision of the gravimetric dosing unit implemented in the fusion machine model HAG-HF. Hence, we assessed the dosing performance for sodium tetraborate (STB), a lithium tetraborate (LTB) mixture and sodium potassium carbonate (SPC). At a target weight of 10 g, we found a standard deviation of 0.3 mg or less for all three substances. This shows that the automatic dosing unit of the HAG-HF is suitable even for hygroscopic or deliquescent material like, e.g., the LTB mixture or SPC.

Key words

• **Automatic dosing** • **Borate Fusion** • **HAG-HF** • **Weighing** • **Induction Furnace**

Introduction

Fusion is an efficient method to prepare various types of samples for the analysis by X-ray fluorescence (XRF) [1, 4, 7, 9], inductively coupled plasma (ICP) [3, 5] or atomic absorption (AA) [8]. Due to the elimination of e.g. matrix and surface effects, it is considered to be more precise than pressed powder pellets [2]. The borate fusion technique consists of mixing an oxidized sample with a borate flux inside a platinum-gold-crucible and heating to 1000 – 1050 °C for a few minutes. The solidified melt can then be analyzed as a glass bead in an XRF instrument or can be poured into diluted acid and subsequently measured by ICP or AA.

For peroxide fusion, the powder sample is mixed with an alkali fusion flux like, e.g., sodium peroxide (Na_2O_2) inside a nickel or zirconium crucible. Then the mixture is heated up to approx. 700 °C until the reaction is complete and the sample is dissolved.

In any case, the successful performance of the fusion process relies on highly precise dosing of sample, flux and other components such as internal standards, non-wetting agents and oxidizers. Notably the properties of the powder material can significantly affect the accuracy of gravimetric dosing. Hygroscopic substances attract water from the surrounding environment and tend to become damp and cakey. Substances which absorb a sufficient amount of water to form aqueous solutions are called deliquescent. As a result, inter-particle cohesion and adhesion to surfaces increase while the powder flowability decreases. These changes in physical characteristics may impede accurate material metering, whether by a lab technician or an automated dosing system. As a rule of thumb, it may be assumed that the total weighing error should not exceed 0.5 mg in order to perform a proper fusion process.

In this application note we aim at investigating the precision of the dosing mod-ule implemented in the HAG-HF, the fully automatic system for sample preparation by induction fusion. In order to evaluate the influence of the material on the dosing performance we assessed three different fusion components with divergent material properties.

Methods

For this test series we used a HAG-HF model (Figure 1) with a dosing unit that can handle three different powders like, e.g., an internal standard, oxidizer and borate flux.

Figure 1: *Automatic high-frequency fusion system model HAG-HF for XRF sample preparation. The machine also includes the dosing unit used for this study.*

The storage bins of the powders are effectively protected against the humidity of the air by built-in pinch valves (Figure 2). The weighing is performed by a balance placed underneath the storage bins with an accuracy of 0.1 mg. A specially developed PLC algorithm allows the fast and precise dosing and can be easily adapted to the particular properties of each material.

Figure 1: *Detail photograph of the dosing unit of the HAG-HF with bins for (1) internal standard, (2) oxidizer, (3) borate flux, (4) liquid dosing of non-wetting agent.*

For this study we used three different powders, each of them was placed in one of the three powder storage bins:

(1) Lithium tetraborate + sodium nitrate + lithium bromide (LTB mixture)

(2) Sodium tetraborate (STB)

(3) Sodium potassium carbonate (SPC)

It is to be noted that the LTB mixture contains hygroscopic additives whereas the SPC is a deliquescent substance.

All dosing trials were carried out with an identical platinum-gold crucible. After each trial, the crucible was released by the handling robot, emptied by the operator, carefully cleaned and returned to the input position of the HAG-HF. For each material, the target weight was 10 g. For powder 2 we performed 20 dosing trials, for powder 1 and 3 we performed 40 dosing trials. The final weight after each dosing trial was automatically stored for later statistical evaluation. For each powder we calculated the mean average final weight and the standard deviation.

Results

All three fluxes could be automatically dosed without major problems or outliers (Figure 3).

The dosing of the LTB mixture resulted in a mean final weight of 10000.0 mg ± 0.1 mg. For STB and SPC the mean final weights were 10000.1 ± 0.2 mg and 10000.2 ± 0.3 mg, respectively.

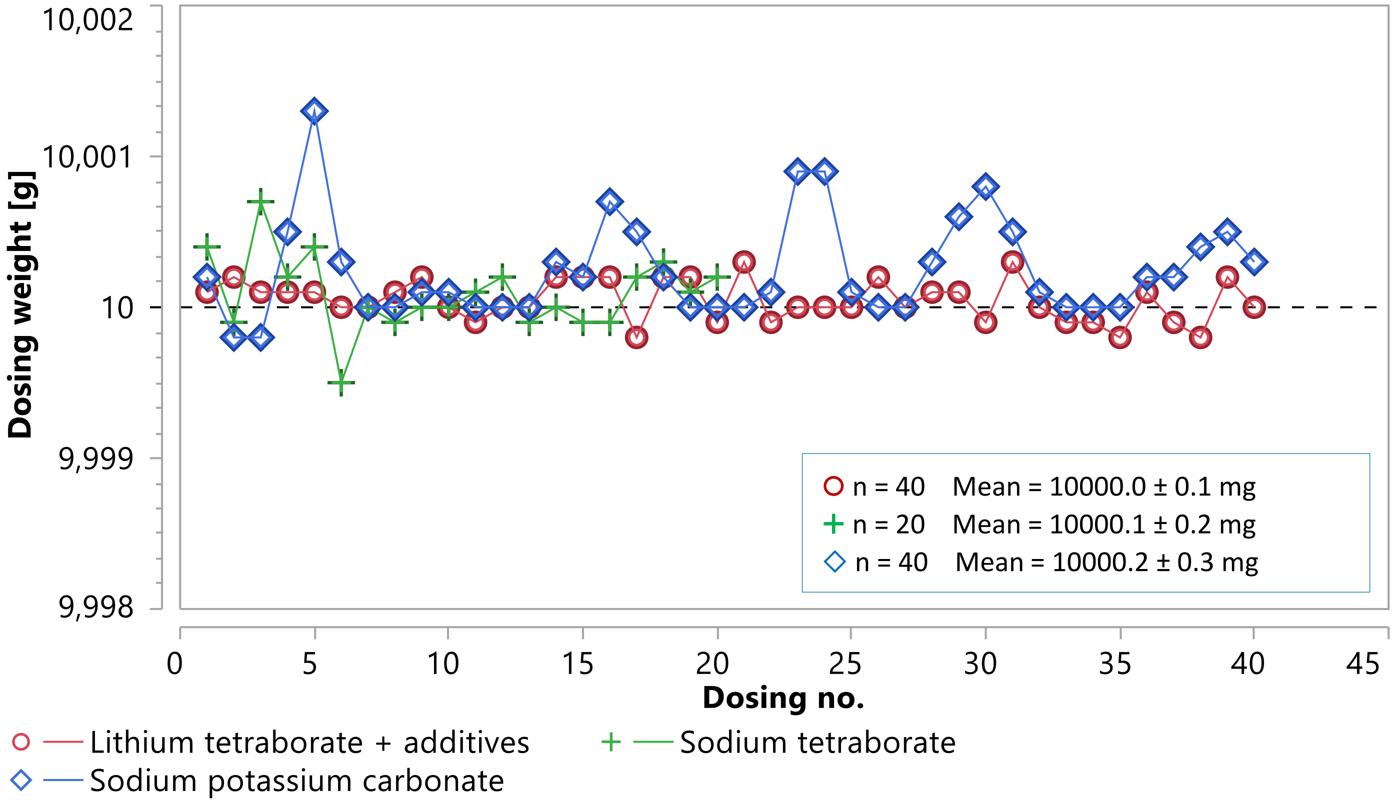

Figure 3: *Graphical representation of the individual results of the dosage of lithium tetraborate (LTB) mixture, sodium tetraborate (STB) and sodium potassium carbonate (SPC),*

Discussion

The dosing results presented here are comparable to a previous study. Perreault and Bouchard achieved optimal analytical results for Fe2O3, SiO2, Al2O3 and CaO using automatic dispensing balances in ratio mode which follows the same principle as our dosing unit [6]. In their experiments, they made use of non-hygroscopic substances as lithiumtetra- and metaborate flux mixtures. Based on the outcome of their study they concluded that the weight tolerance for fluxes should be in the range of 1.0 mg.

Our results are not only in the same order of magnitude but even lower than the flux tolerance of 1.0 mg. Interestingly, our study also included hygroscopic and deliquescent substances like the LTB mixture and SPC. This shows that the automatic dosing as implemented in the HAG-HF is also capable of handling powder with complex material features.

Automatic dosing of sample material and flux has many advantages over manual dosing by an operator. First, the present and other studies indicate that the precision in reaching the target weight is equal or superior to manual dosing. Second, Automatic dosing enables traceability of weight and sample-to-flux ratio, thus enabling continuous documentation of the sample preparation process. Also the dosing weight can be automatically transferred to the XRF analyzer resulting in significant simplification of analytical processes. Third, user safety is improved as direct contact with potentially harmful substances is prevented. Eventually, the laboratory personnel may perform more demanding tasks instead of executing tedious work like weighing sample and flux.

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