High-precision borate fusion using induction heating furnaces with calibrated platinum-gold crucibles

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Insufficient sample preparation has a significant share in the total analytical error. This is particularly true for sample preparation using borate fusion where small deviations in, e.g., weighing or sample handling cause a significant increase in error. Also poor control of the fusion temperature may have a detrimental influence on the analytical accuracy and precision.

In high-frequency induction furnaces, the temperature of the platinum-gold crucible can be measured by an IR-pyrometer. For this study, we used a new method for automatic pyrometer adaptation to the specific emissivity of the crucible ('*crucible calibration*') in order to allow a more precise temperature control.

Following crucible calibration we found a maximum deviation of 5 °C from the target fusion temperature. In a second step, we tested the impact of the crucible calibration on lithium tetraborate fusion of NIST certified reference material and commercially available cement. Doublets of eight NIST specimens were prepared according to ASTM C 114 using a manual (Bead One HF, Herzog) and automatic (HAG-HF, Herzog) induction system. Prepared glass beads were examined by a WDXRF spectrometer (S8 Tiger, Bruker). For the NIST doublets, the mean coefficient of determination ($\pm \sigma$) was 0.9992 ± 0.0011 which is comparable to values in other studies. For the commercially available cement, we achieved expert limit results for the repeatability test.

The study shows that the temperature precision in induction furnaces with crucible calibration is similar to the performance of thermocouple-controlled electrical resistance furnaces with a single chamber. By contrast, it is significantly better than the precision attainable in multi-chamber electrical resistance furnaces. The WDXRF test series proves the capability of the induction fusion method with calibrated crucibles to enable results meeting highest analytical standards.