# Crucible calibration enhances accuracy and precision of temperature control in induction fusion


## Abstract

In this study we assessed the impact of adjustment of the pyrometer to the emissivity of the Pt/Au crucible (“crucible calibration”, patent pending). Following crucible calibration we evaluated the deviation from the target temperature by measurement of the melt temperature using a thermocouple. For the temperature range between 1050°C and 1100°C, the mean deviation from the target temperature was between 1.0°C and 3.5°C corresponding to a percentage deviation between 0.15 and 0.30%. The standard deviation was between 1.9°C and 2.2°C. These values demonstrate the excellent accuracy and precision of temperature control following crucible calibration.



## Key words

• **Fusion** • **Induction heating** • **Crucible** • **Calibration** • **Infrared pyrometer**


## Introduction

Borate fusion by induction heating is a fast and flexible method for production of glass beads used for X-ray fluorescence spectroscopy. It is a major advantage of Herzog induction heating systems that they allow the direct and continuous temperature measurement of the platinum/gold crucible by an infrared pyrometer. This makes it possible to reach the preset temperature within seconds and keep it stable and unaffected by interfering influences from outside.

In order to further improve the accuracy of the temperature control Herzog introduced a new method of crucible calibration (patent pending).

By using a special calibration device (“calibrator”), it is possible to simultaneously record the melt temperature and the pyrometer reading at various temperature stages. Based on these values the emissivity for each crucible can be determined at each temperature level. In all subsequent fusion procedures, the PLC of the fusion machine automatically adjusts the pyrometer to the emissivity of the specific crucible.

In the previous application note 23/2019 we explained the basic principle of crucible calibration. Here we evaluate, by repeated measurement at four different temperatures, how crucible calibration influences accuracy and precision in reaching the targeted temperature.

## Method

All trials were performed on a Bead One HF benchtop induction fusion device. The crucible calibration was performed by melting a defined amount of Lithium tetraborate. Temperature recording and calculation of emissivity were carried out automatically by the PLC of the fusion machine. During the calibration, the PLC simultaneously recorded the pyrometer reading and the temperature within the melt at four different temperature steps. Based on these reference values for pyrometer and melt temperature, the system determined the exact emissivity values of the crucible at each temperature level. In all subsequent fusion operations using this crucible, the PLC automatically selects the correct emissivity value of the pyrometer for this crucible.

The reference temperature values within the melt were obtained using a special calibration device ("calibrator"). The calibrator inserts the thermocouple into the melt and ensures that it is always placed in the same position for absolutely reproducible measurements.

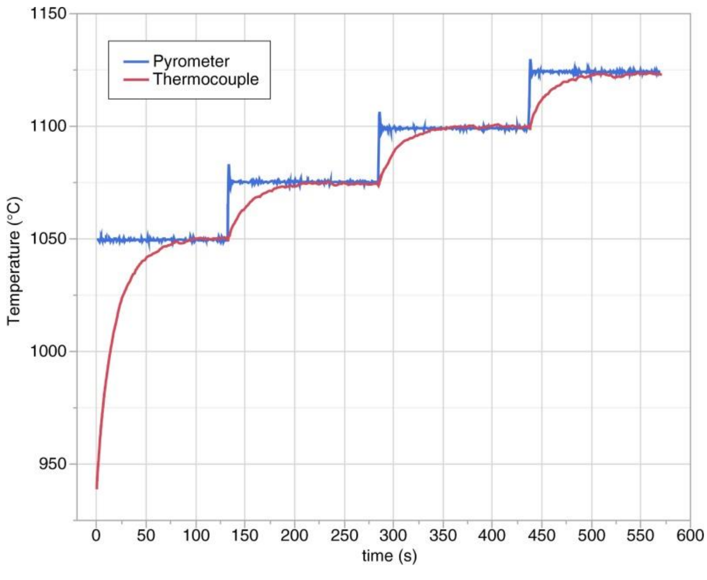


**Figure 1:** *Example for evaluation of the deviation of the melt temperature (measured by thermocouple, red) from the target temperature (measured by pyrometer, blue). Four temperature stages (1050°C, 1075°C, 1100°C, 1125°C) were assessed subsequently.*

In semi-automatic machines like the Bead One HF, the HMI panel guides through the process and prompts the operator to insert the calibrator. This can be done from outside the machine without any contact to the heating unit. In most cases, the calibration is performed only once and takes less than 10 minutes.

After crucible calibration we performed twenty trials to evaluate temperature accuracy and precision. In each trial, the crucible was filled with 10g (±5 mg) of Lithium tetraborate. Subsequently, the crucible was heated up to four different target temperatures (1050°C, 1075°C, 1100°C and 1125°C) as measured by the pyrometer (example in Figure 1). In each trial, the order of the target temperatures was randomized.

When the first of four target temperature was reached and stable for 30 s, the thermocouple (type K) was introduced into the crucible in order to measure the melt temperature (Figure 1, time point= 0 s). The melt temperature was recorded when the thermocouple temperature was stable for 30s. This was usually the case 90 s after the pyrometer reached the target temperature. Figure 1 reveals that the melt temperature curve showed a non-linear increase and reached the target temperature approximately 60 s after the pyrometer temperature. This is due to the fact that the induction system primarily heats the crucible wall which transfers the temperature to the molten glass. The heat distribution takes place via convective motions within the melt.

After passing through the first temperature level the crucible was heated up or cooled down to the other target temperatures without removing the thermocouple element. Again, the melt temperature was recorded as soon as it remained stable for 30 s.

## Results

For the target temperature of 1050°C, the mean temperature in the melt was 1049.0 ± 1.9°C as measured by the thermocouple. The maximum upward deviation from the target temperature was 2°K, the maximum downward deviation was 4°K. For the target temperature of 1075°C, the measured mean temperature was 1073.5 ± 2.2°C (range 1070 to 1076°C). For the target temperature of 1100°C, the measured mean temperature was 1098.6 ± 2.5°C (1095 to 1103°C) and for a target of 1125°C, the measured temperature was 1121.5 ± 2.2°C (1118- 1124°C) (Figure 2).

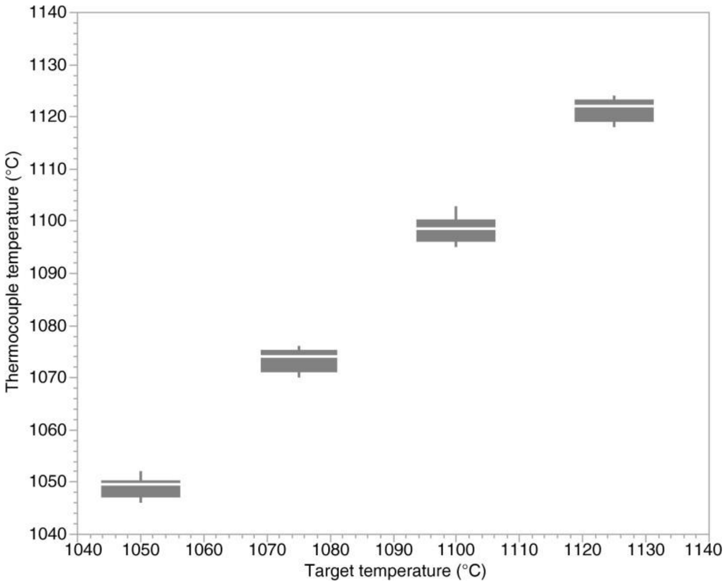


**Figure 2:** *Boxplot diagram showing the temperature of the melt as measured by thermocouple for four different target temperatures (1050°C, 1075°C, 1100°C and 1125°C).*

## Discussion

In this application note, we show that calibration of the crucible leads to an excellent accuracy and precision in temperature control of the fusion process. For the temperature stages between 1050 to 1100°C, the deviation from the targeted temperature was less than 2°C. This corresponds to a percentage deviation of less than 0.15 % from the target value. For the temperature level of 1125°C, the mean deviation was 3.5°C corresponding to a percentage deviation of 0.30 %. The low standard deviation between 1.9°C and 2.5°C as well as the small value range (maximum 8°C for a target temperature of 1100°C) demonstrates the high precision of our approach.

The study confirms that adjustment of the pyrometer to the emissivity of the crucible ensures a pinpoint temperature control of the fusion process. Reproducibility and accuracy in temperature control are important factors to further reduce the bias in XRF-analysis of glass beads. Deviations from the target temperature have impact on various processes during fusion like, e.g., volatilization of the flux, viscosity of the melt, loss of volatile elements etc. Therefore, there is a clear beneficial effect on analytical quality if variations of these processes due to temperature fluctuations can be avoided.

A high proportion of the total analytical error is caused by poorly controlled sample preparation. The Herzog fusion systems make a significant contribution to sample preparation and analysis under identical conditions. At the same time, the software provides all relevant KPI's like to enable the operator to further optimize the laboratory processes.


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