TECHNICAL NOTE

Thermal Analysis

TGA 8000
Temperature Performance

Obtaining reliable thermogravimetric data depends on having a Thermogravimetric Analyzer (TGA) with accurate and responsive temperature control and read-out. The TGA 8000™ utilizes an improved, more robust thermocouple and improved temperature calibration and control algorithms. Once it has been calibrated using materials with known transition points, the temperature accuracy can be demonstrated. Then, the thermocouple signal can be used to demonstrate temperature control and precision.

Introduction

The gold standard method of TGA temperature calibration has been the use of magnetic standards, developed by PerkinElmer. This makes use of the observation that ferromagnetic materials lose their magnetism when heated above an intrinsic "Curie temperature" that has been accurately determined for certain pure materials and alloys. By scanning a selection of these materials in a TGA with an imposed magnetic field, one can use the apparent weight-loss temperatures to calibrate the TGA in the exact position occupied by the sample.

The TGA 8000 is provided with a new temperature calibration algorithm that allows the TGA to be accurately calibrated over a range of heating and cooling rates after determining the Curie transitions at two different heating rates. This can be accomplished in one multistep program method. While it is true that the TGA 8000 comes standard with an accurate temperature calibration, most users will want to carry out a careful calibration using the experimental setup conditions they will be using for their most critical TGA tests. The calibration method used here takes slightly longer to perform, but it has the advantage that the calibration is most accurate for heating rates in the range of 20 °C/min and 0 °C/min (isothermal operation), which is called for in many dwell-time tests. Most tests that use fast rates use them to get quickly from one isothermal separation temperature to the next, and it is the accuracy of the isothermal temperatures that affect the precision of the test results. The method of calibration is also recommended for use of the AutoStepwise and Variable Rate programming steps since the heating rate in these methods is variable and most of the weight loss occurs at a slow scan rate.

TGA 8000 Temperature Specifications

Scanning Accuracy	1.5 °C
Scanning Precision	1 °C
Isothermal Precision	0.1 °C

Covering the T-range between 154 °C - 770 °C and heating rates up to 10 °C/min

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The Calibration Procedure

The sample preparation and instrument setup procedure is detailed in the on-screen help file with the calibration samples used being the primary PerkinElmer-supplied Curie point materials: Alumel, Nickel, Perkalloy, and Iron. The method to obtain accurate isothermal temperatures requires obtaining the apparent Curie transitions by heating the uncalibrated TGA at two heating rates: 2 °C/min and then 20 °C/min. To save time, the 2 °C/min scan can be performed by alternating between transitions at a fast scan rate, then scanning at 2 °C/min up to, and through, each Curie transition. After getting the 2 °C/min data, the method calls for cooling to 30 °C, equilibrating, then heating at 20 °C/min to 840 °C. After taking the data, the Curie transitions were calculated and entered into the Temperature Calibration tab, as shown in Figure 1. To verify the temperature calibration, a similar method was run with additional heating ramps, and the Curie data analyzed and plotted, shown in Figure 2. What can be seen from this data is that:

- When heating at 2 °C/min, 10 °C/min or 20 °C/min all of the Curie transitions fall within two degrees of their literature values.
- All the 2 °C/min heating data is within 1 degree of the literature values.
- If the data is interpolated to 0 °C/min heating rate (isothermal) the data is within one degree of literature value. An example of these interpolations can be seen in Figure 3.

These data demonstrate the absolute temperature accuracy for isothermal measurements for the temperature range of 154 °C to 770 °C bracketed by the Curie transition points.

Thermocouple Data

Having established the accuracy of the temperature in the position occupied by the sample at multiple scan rates and isothermal, we can now examine the equilibration and precision characteristics of the TGA from the sensor signal. Figure 4 shows isothermal equilibration characteristics when scanning to 550 °C from room temperature at 50 °C/min and 500 °C/min and cooling from 750 °C at 200 °C/min. All signals are within 0.1 °C of 550 °C within 1.5 minutes. This level of precision would be achieved at any temperature. However, the equilibration time will be somewhat longer at lower temperatures.

Figure 5 shows the sensor temperature after 1.5 minutes equilibration as the temperature is programmed at 200 °C/min between two isotherms. This shows the reproducibility, the precision, of sensor temperature after equilibration in an isotherm. The equilibration is easily within 0.1 °C within 1.5 minutes of set-point arrival. Based on this data, the precision of sensor temperature is a few hundredths of a degree. Again, at lower temperatures the precision would be similar after a somewhat longer equilibration time. This shows the reproducibility of the TGA 8000 equilibrium isothermal temperature control.

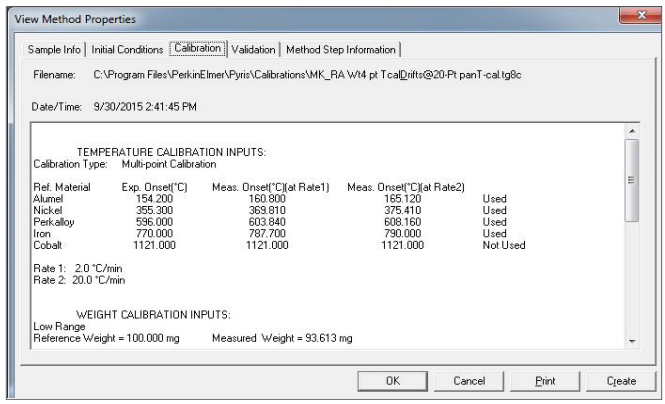

Figure 1. Temperature calibration parameters.

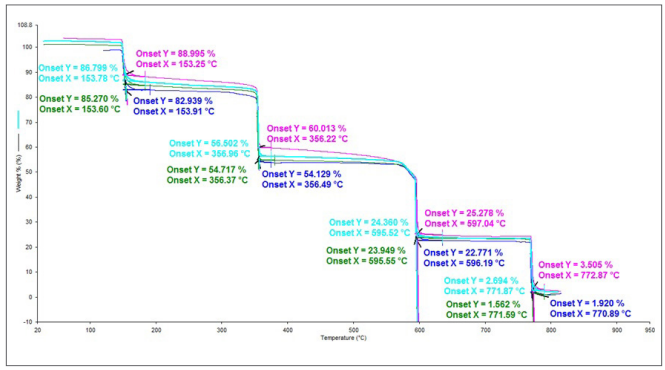

Figure 2. Alumel, Nickel, Perkalloy and Iron Curie Transitions after calibration. Pink is cooling at 10 °C/min. Blue is heating 2 °C/min. Green and aqua are 10 °C/min and 20 °C/min.

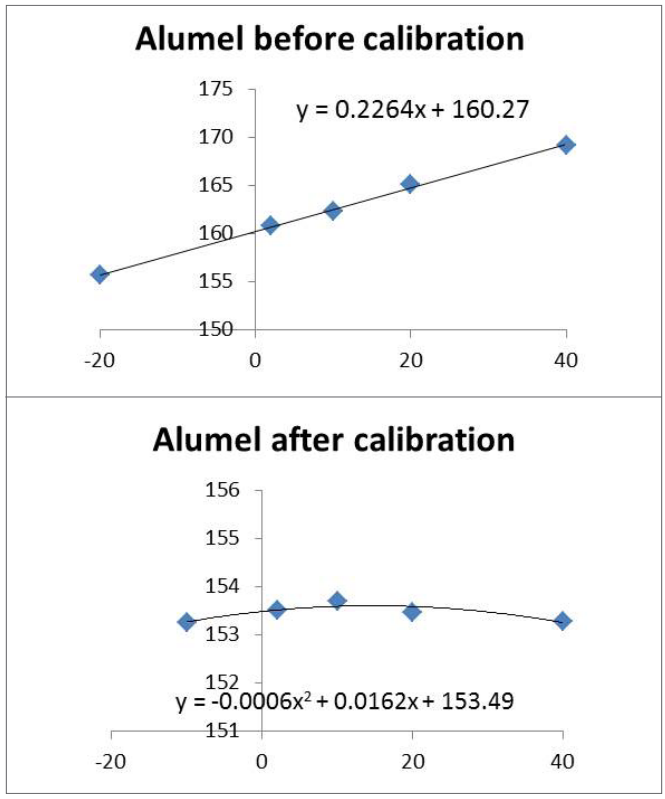

Figure 3. Temperature vs. scan rate, before and after calibration, at the alumel transition.

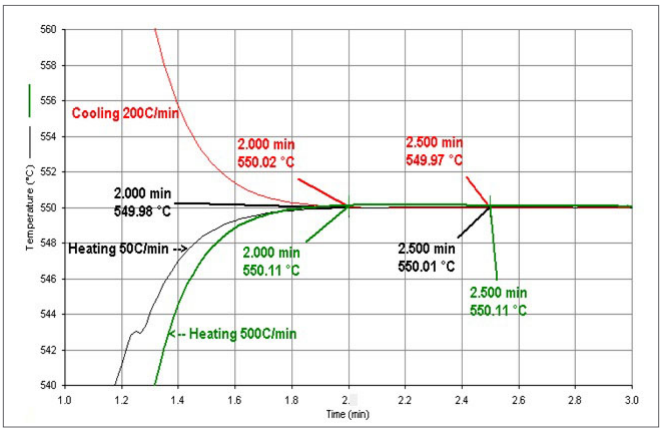

Figure 4. TGA 8000 Temperature equilibration characteristics.

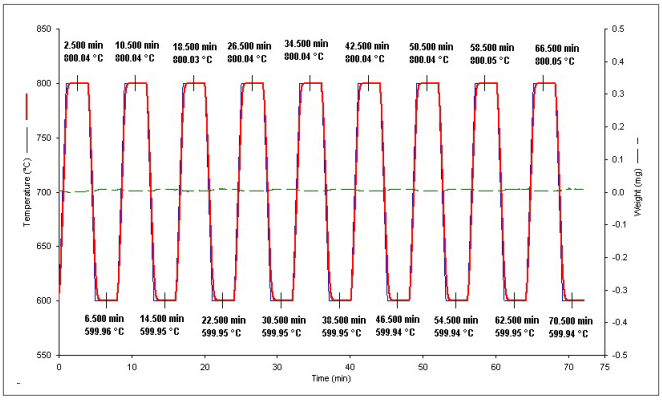

Figure 5. Precision of Temperature measurement between 600°C and 800°C scanning 200°C/min.

Extrapolations and Caveats

The thermal measurements made to demonstrate this performance were made under commonly used experimental conditions, but chosen to optimize isothermal accuracy. The pans used were standard platinum pans with the calibration materials covered with alumina powder. The purge gas was nitrogen at 60 cc/min total purge. It is possible to calibrate the TGA using other conditions and other materials in order to optimize accuracy over a higher or lower temperature range, higher heating rates, or using other experimental conditions, including vacuum operation. The temperature precision and accuracy can be expected to be different under such conditions.

Conclusion

For many thermogravimetric analyses, the accuracy and precision of the temperature control and read-out are important aspects of test measurement accuracy and reliability. The TGA 8000 temperature control algorithm has been upgraded over that used in the Pyris™ 1 TGA, and the thermocouple has been replaced by one of even better quality. These steps, together with baseline improvements, make the TGA 8000 the obvious choice for high-performance gravimetric analysis. While it is easy to make claims of temperature accuracy and precision, it is another matter to demonstrate as the TGA 8000 does that these specifications can be achieved over a wide range of conditions, including at isothermal equilibration.

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