Thermal Analysis & Rheology

THERMAL APPLICATIONS NOTE

TGA TEMPERATURE CALIBRATION USING CURIE TEMPERATURE STANDARDS

TGA temperature calibration is most commonly accomplished using melting point standards (1,2). However, a second calibration method exists which utilizes the magnetic transition of ferromagnetic materials. In this procedure, a magnetic material is placed inside an empty, tared TGA pan located near a strong magnet. The material is then heated. At the Curie Temperature, the magnetic properties disappear (i.e., the material goes from diamagnetic to paramagnetic) and the reduced attraction for the magnet results in a sharp apparent weight loss or gain (depending on the TGA design). This point (temperature) is sharp and reversible, and hence ideal for calibration. Specific details for this Curie Temperature calibration procedure are described below for the TGA 51, TGA 2050, TGA 2950, and SDT 2960.

<u>TGA 51</u>

- 1. Prepare the TGA. Place a piece of Curie Temperature standard material into a clean, tared TGA pan.
- 2. Start heating the TGA using a method which heats at the same rate and under the same purge environment that will be used in subsequent experiments.
- Slowly lower a bar magnet down onto the furnace until a weight loss of approximately 2% is detected (as seen on the Real-Time Plot or the Signal Control screen). If necessary, secure the magnet in position with wooden shimes (spatulas).

Maintain the magnet in this position for the duration of the experiment.

Note: The magnet should be positioned such that its long axis is parallel with the long axis of the furnace.

4. Continue heating until an "S" shaped curve is observed. Use the TGA Standard or General data analysis software program to determine the extrapolated endpoint of this "S" shaped curve. A typical curve is displayed in Figure 1.



5. Enter the temperature calibration point in the table on the Signal Control screen. TCA = 2050/2050

TGA 2050/2950

- 1. Prepare the TGA. Place a piece of Curie Temperature standard material into a clean, tared TGA pan.
- 2. Start heating the TGA using a method which heats at the same rate and under the same purge environment that will be used in subsequent experiments.
- 3. Select "Furnace Closed" as a method end condition on the Module Parameters screen. The furnace will not be able to open fully with the magnet in place and can cause damage to the instrument.
- Position the bar magnet under the furnace and slowly raise one end of the magnet until a weight gain of approximately 2% is detected (as seen on the Real-Time Plot or the Signal Control screen). Maintain The magnet in this position for the duration of the experiment.
- 5. Continue heating until an "S" shaped curve is observed. Use the TGA Standard or General data analysis software program to determine the extrapolated endpoint of this "S" shaped curve. A typical curve is displayed in Figure 2.
- 6. Enter the temperature calibration point in the table on the Signal Control screen.

SDT 2960

- 1. Prepare the SDT. Place a piece of Curie Temperature standard material into a clean, tared SDT pan on the sample platform.
- 2. Start heating the SDT using a method which heats at the same rate and under the same purge environment that will be used in subsequent experiments.
- Slowly lower a bar magnet down onto the furnace until a weight loss of approximately 2% is detected (as seen on the Real-Time Plot or the Signal Control screen). If necessary, secure the magnet in position with wooden shimes (spatulas). Maintain the magnet in this position for the duration of the experiment. Note: For best results, rest the left end of the magnet directly on the black furnace cover and the right end of the

magnet on the remaining portion of the metal cover (right side of the furnace).

- 4. Continue heating until an "S" shaped curve is observed. Use the SDT Standard or General data analysis software program to determine the extrapolated endpoint of this "S" shaped curve. The typical curve obtained is similar to that obtained for the TGA 51 (Figure 1).
- 5. Enter the temperature calibration point in the table on the Signal Control screen.



GENERAL COMMENTS:

- Remember to reset the temperature calibration table displayed on the Signal Control screen before beginning temperature calibration.
- When entering additional temperature calibration points on the Signal Control screen, enter all calibration points in the table after all the experiments have been completed.
- · Note: Some Curie Temperature materials will oxidize and gain weight when an oxidizing purge environment is used.
- · Magnet Specifications:

magnet #B61-6 from PERMAG (516) 822-3311 or equivalent.

The most serious deficiency of this Curie Temperature calibration technique and the reason it is less frequently used is the lack of readily available standard materials with known Curie temperatures. However, simultaneous TGA-DTA devices (like the SDT 2960) offer a means to accurately determine the transition temperatures of these materials by calibrating them against known melting point standards (3). This is achieved by first temperature calibrating the SDT using two melting points standards which bracket the Curie Temperature transition. An additional experiment is then conducted to determine the Curie Temperature as well as to verify the melting points by placing all three materials in a single pan (without contacting one another). Typical results are shown in Figure 3.

TA Instruments Service Representatives in the U.S. are currently offering TGA calibration with certified Curie Temperature standards to their customers. Contact our Service department at (302) 427-4050 for additional details.

References:

- 1. TA Instruments TGA 2950 Brochure.
- 2. R.L. Blaine and P.G. Fair, *Thermochica Acta*, <u>67</u>, 233-240 (1983)





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