TECHNICAL NOTE

Differential Scanning Calorimetry

DSC 8000 Equilibration

- Faster than ever!
- Proof of performance
- Why this is useful for your lab

Abstract/Overview

A simple experiment is suggested to demonstrate the response time of a DSC and to show how much time is needed for equilibration.

Introduction

Differential Scanning Calorimetry (DSC) has traditionally not been a rapid analysis technique. Because the predecessor technique DTA and its more recent cousin, heat-flux DSC, require time to allow the large furnaces – separated from the sample by substantial distance – enough time to equilibrate, scan rates of 10 °C per minute have been standard. In addition, to allow adequate time to reach equilibration it was suggested that scans even be started several minutes before the event of interest in order to ensure a constant scan rate and reliable temperature readout.

Long start-up times are fine if you're patient and can afford the time of a long DSC run. But what if you *need* fast rates? Or you need answers now?

More materials research and even quality control tests are being carried out at fast scan rates to better simulate processing rates, to minimize the time for unwanted changes in structure, or just to increase sample throughput. Scan rates up to several hundred degrees per minute are now in routine use. But can your DSC give *reliable* data under these conditions? This depends on the quality of your temperature control.

PerkinElmer®
For the Better

How Power Controlled DSC Works

The PerkinElmer® power controlled DSC series work under different measuring principles than all other commercial DSCs, which use the heat-flux principle, or by measuring a temperature difference between two samples heated by a single relatively large, external furnace. Power controlled DSC uses two tiny micro-furnaces intimately coupled to the sample and reference positions. It employs a different measuring circuit that uses tight feedback control to maintain the temperature of the sample and reports the power required to do so. The result is extremely tight temperature control. Here's the proof.

Temperature Control on a Power Controlled DSC

The following simple experiment proves beyond doubt the unique temperature control capability of power controlled DSC. Here we use a calibrated DSC to heat a sample of indium at 500 °C per minute from 40 °C to 152 °C, and then heat at 10 °C per minute through the indium melt which starts at 156.6 °C. See Figure 1. The details of the experiment are listed in the appendix.

Results

Figure 2 shows this *1.4 minute* test on a time scale.

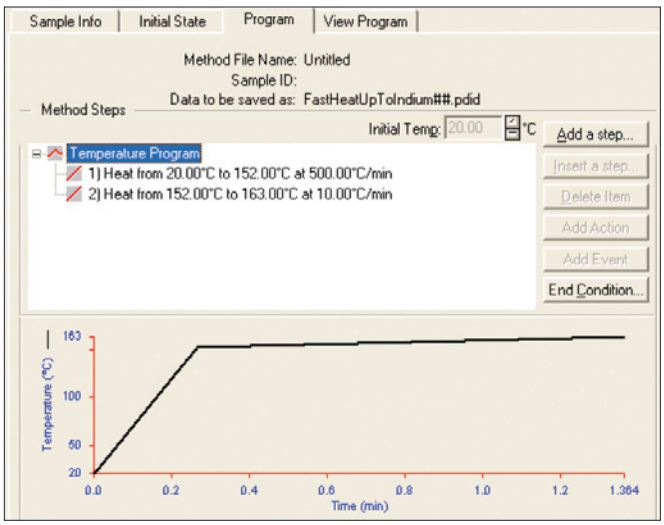

Figure 1. The DSC method: fast heat to 152 °C, then 10 °C/min through the indium melt.

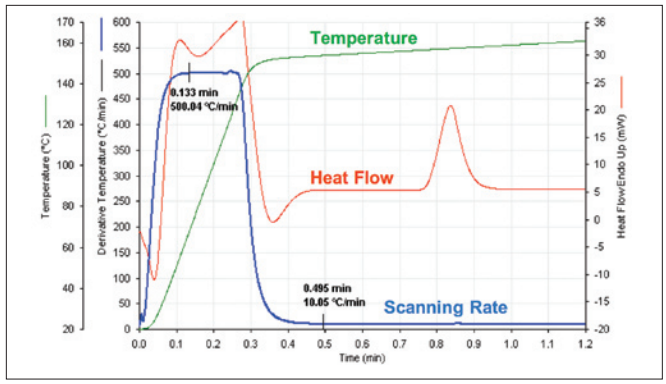

Figure 2. Indium melting after a fast heat up. The green curve is that of the sample temperature (extreme left axis), and is seen to ramp from 40 to 152 °C in a fraction of a minute. The blue curve is the derivative of the sample temperature, which is the heating rate (inner left axis). Note: temperature quickly (in 8 seconds) rises to 500 °C/min and remains constant until the control temperature reaches 152 °C. It then rapidly re-equilibrates to 10 °C/min as indium is heated through the indium melt (heat flow on right hand axis). Actually, it is obvious from the data we could have heated to an even higher temperature before switching to 10 °C/min!

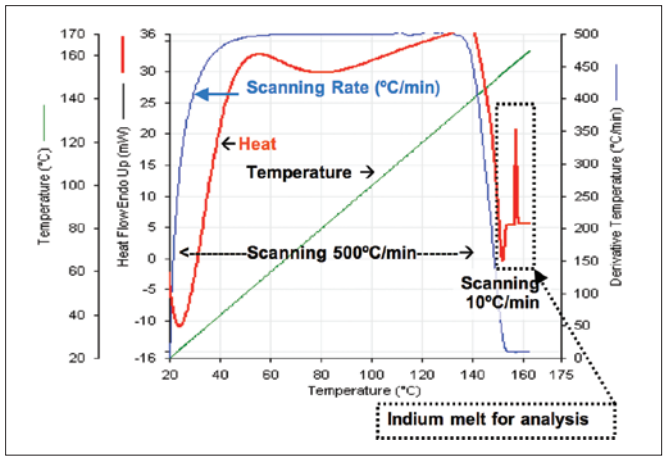

Figure 3. The same data shown versus the sample (sensor) temperature. Notice that the heating rate achieves 500 °C/min by 60 °C. This is the kind of temperature control that is needed if you want to use fast heating rates and get useful data without starting at a deep subambient temperature.

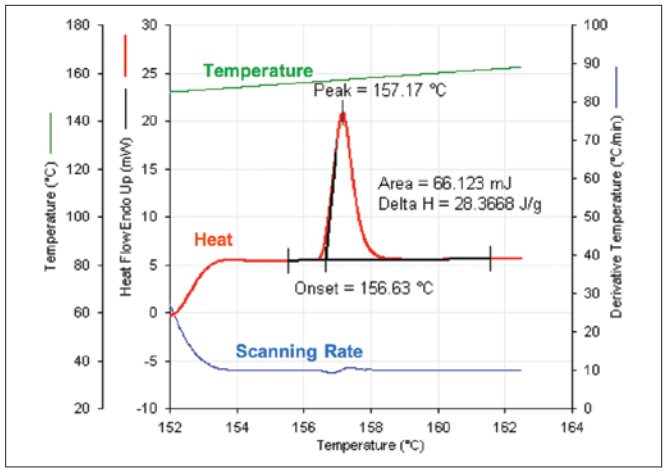

Figure 4. The same data as Figure 3 but with the indium heat flow scaled up and the melting calculation performed. Notice that despite the short time for equilibration at 10 °C/min, the temperature and melting energy are accurate.

Why is rapid, accurate temperature control like this important to you? You need:

- High sensitivity for measuring the glass transition of a low concentration amorphous component so you want to heat very rapidly
- To heat a sample very rapidly to keep it in an unstable form through a transition
- To heat a sample in a metastable state to a temperature just below a transition and immediately measure that transition at a slower rate before it changes.
- To measure the reaction kinetics of a reactive mixture by heating through the reaction at a slow rate then starting immediately at an elevated temperature
- To characterize crystallization behavior and need to stabilize the temperature rapidly after cooling from the melt
- To shock cool the sample in a controlled and reproducible way

These tests would likely not be possible with a DSC that employs a large furnace because the temperature would not stabilize in such a short time. Some instruments provide data correction for thermal lags that make the data appear that the sample is equilibrated more rapidly than it actually is. They show the data as it would be under ideal temperature control. This may mask a problem by making the data look better than it is.

With a power controlled DSC, the sample is actually controlled to a tight temperature. Therefore, there is no need to correct to a presumed model.

Appendix: Experimental Details

- The DSC was configured with a refrigerator-type cooling device and using 20 cc/min nitrogen gas purge. If helium is used as a purge gas the heat flow equilibration of the sample specimen is further improved from what has been shown. Standard (not autosampler type) platinum lids were in use.
- The DSC was calibrated in normal fashion using indium as the sole calibrant. No special conditions were used to setup the instrument. It was an old "work horse" model, but a genuine power controlled DSC.
- The indium sample (~2 mg) both for calibration and for the fast heat-up scan were crimped in a standard pan and flattened to ensure good thermal contact. One way to achieve this is to use the eraser on the end of a pencil to depress the crimped pan against a flat surface before loading.

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