SII NanoTechnology Inc.

TIPS FOR OBTAINING THE BEST POSSIBLE DSC DATA

The following guidelines should help to optimize the overall quality of DSC data in terms of general appearance and noise reduction.

- **Sample mass.** It is recommended that a mass between 10 and 20 mg be used for the analysis of most materials. For materials which yield highly endo- or exothermic responses during heating, it is best to use lower masses. If a sample has a very weak transition, or is highly filled (with glass or some other inert material), it may be best to increase the sample mass. Best results are obtained on a thinner specimen as opposed to a thicker sample, since this reduces thermal gradients across the sample. It should be noted that the peak temperature may increase somewhat with increasing sample mass.

- **Starting temperature.** It is important to pick an initial temperature which is well below the expected transition temperature. A good rule of thumb is to allow three minutes of run time before the transition is encountered during heating. For example, if a Tg is expected at -10°C at a heating rate of 20°C/min, then the starting temperature for this experiment should be -70°C to yield a good baseline going into the transition.

- **Counterbalancing.** It is always recommended to balance, as closely as possible, the heat capacity of the sample and pan with an inert material, such as aluminum, on the reference side. This helps to reduce the overall noise level and also minimizes the 'start-up hook'. For a quick estimate of the necessary countermass, simply add aluminum lids to the reference pan to balance the total mass of the sample and sample pan. The counterbalancing procedure should not be used if actual heat capacity measurements are to be performed.

- **Baseline subtraction.** If a very weak transition is expected, it may help to subtract the baseline response (empty DSC cell with no pans) from the sample results. The baseline experiment should be performed under the same exact experimental conditions as used for the sample.

- **Heating rate.** For most samples, a heating rate of 10 or 20°C/min is recommended. The maximum heating rate for ODSC experiments is 5°C/min. It should be noted that for polymeric melting peak temperatures or for thermoset curing exotherms, there will be a significant increase in the peak temperature with respect to the heating rate.

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net

- **Crimping.** It is generally a good idea to crimp the sample when using the standard sample pans. Avoid, however, over- crimping which can result in a rounding of the bottom of the sample pan. If rounding occurs, the pan can rock in the DSC cell giving rise to noise. If a small amount of rounding occurs after crimping, this can be eliminated by placing the pan on a smooth, hard surface and pushing down with the blunt side of the tweezers.

  Rubbery samples, such as elastomers, are best handled by lightly crimping. If excessive noise is observed on an elastomer through Tg, the sample is probably moving in the pan (due to expansion) and should be reanalyzed in an open pan.

  If analyzing a material which may give off volatiles, it is best to punch a pin hole in the lid of the sample pan to provide a means for the volatiles to easily escape.

  A lid should never be permitted to 'float' on a sample. It is important that the lid be crimped to prevent it from moving or rocking during the experiment.

- **Sealed pans.** If analyzing samples where the volatiles are wished to be contained, the sealed pans should be used. For samples containing water and which are to be heated above 40°C, the Ag sealed pans should be used. Use only the 15 μL pans with the DSC220.

PID Settings. Ensure that the PID values are set to the proper values especially when changing from standard DSC to Oscillating DSC. For standard DSC, D = 10 and for ODSC, D = 0.

- **Purge gas.** It is recommended that a purge gas (nitrogen) be used with the DSC. The flow rate of the purge should be 50 to 80 mL/min. The purpose of the purge is to vent corrosive volatiles (such as HCl, from PVC) if a sample would happen to emit these during heating. Of course, it is best to avoid decomposition of the sample in the DSC. Also, the purge prevents water from entering the DSC cell when operating under subambient conditions. For special experiments, such as oxidative stabilities, air or oxygen purges can be used. If a purge gas is not available, the purge port on the back of the DSC module should be blocked off using the supplied plug.

Cell lid. Ensure that the silver lid placed on the DSC cell is flat. If the lid is accidentally dropped and its shape becomes distorted, it should be placed on a smooth hard surface, and flattened by pushing down on the lid. If necessary, the bottom surface of the lid can be further smoothed by polishing with emory paper.

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ODSC Conditions. When operating in the Oscillating DSC mode, the following conditions are recommended to yield the best results: frequency 0.015 Hz; amplitude 5°C; heating rate 5°C/min (2.5°C/min for heat capacity measurements); data collection rate of 0.7 seconds at 0.015 Hz; D = 0 under PID. Remember to avoid using data in the melting regions since there is a phase shift which occurs during melting which makes the ODSC data difficult to interpret.

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