SII NanoTechnology Inc.

UNDERSTANDING UNEXPECTED RESULTS BY DSC

During a DSC experiment, results can sometimes be obtained which are not expected or anticipated. The following are some tips to help better understand the unexpected.

Is the anomaly due to the instrument or the sample?

The easiest way of addressing this point is to perform a baseline experiment. This is done by heating the empty DSC cell over the given temperature range with the purge gas turned on and adjusted to a flow rate of 50 mL/min. If there is a problem with the instrument, the anomaly will be observed in the baseline scan. If the anomaly is due to the sample, then a peak-free baseline will be obtained.

Peak in the baseline scan

If a peak is observed in the DSC baseline scan, the cell may be contaminated with a sample spill-over or with moisture. Moisture effects will be observed as a small peak near 0°C. Peaks observed well away from 0°C are most likely due to sample contamination and the DSC cell should be baked out. This is done by heating the empty DSC cell to 550°C and holding for 5 minutes under an air purge.

Moisture peak

If a peak is observed near 0°C, it is best to heat the DSC cell to 200°C with the nitrogen purge gas on. If the peak is still observed after heating to 200°C, check to ensure that the purge gas is dry and that the purge lines are made of Teflon tubing and are connected securely. In-house supplies of nitrogen purge gas can oftentimes contain significant levels of moisture which may give rise to a small peak near 0°C. It is recommended to use dry, bottled nitrogen gas.

Large endothermic start-up hook

At the beginning of a DSC heating experiment, there may be a significant and rapid increase in the endothermic direction. This is known as the DSC start-up hook and is due to a heat capacity imbalance between the sample and reference. The effect is heightened at faster heating rates. The start-up hook can be reduced by counterbalancing the heat capacity of the sample and reference. An extra inert mass (aluminum metal or alumina powder) can be added to the reference pan to increase its heat capacity to match that of the sample.

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Sample's transition temperature is not correct

Check to ensure that the DSC has been calibrated for temperature and enthalpic response using high purity indium metal. Indium should have an onset melt temperature of 156.6°C. The DSC should be calibrated using the same experimental conditions (purge gas, pan type, heating rate) that will be used to analyze the sample. Switching from a nitrogen or air purge to helium will cause a large shift in the DSC calibration. Switching from standard aluminum crimped pans to the larger aluminum or silver sealed containers will also give rise to a significant shift in the temperature and enthalpic calibration.

Peak shows up in sample where it shouldn't

If the sample moves during the course of a DSC experiment, an artificial transition (exothermic or endothermic peak) may be obtained. Some samples tend to 'ball up' when heated above Tg and this movement can give rise to an artificial peak. It is best to keep the sample as thin as and flat as possible and to crimp the sample to prevent it from moving during the course of an experiment.

Numerous sharp peaks are obtained

The observation of sharp, narrow peaks is usually due to the evolution of volatiles during the course of the DSC experiment. This could be due to the degradation of the sample or due to the evolution of gases trapped in the material. Weight loss can be easily determined by comparing the mass of the sample prior to and after the experiment. A significant decrease in the mass of the sample indicates that volatiles were evolved during the DSC experiment. The final program temperature should be decreased or a sealed container (Al or Ag) should be used to analyze the sample.

Large shift occurs in DSC baseline after endothermic peak

A large shift in the DSC baseline is indicative of the loss of weight of the sample during heating. With DSC experiments, it is assumed that the system is *closed*, which means that the mass should be constant throughout the course of the experiment. A loss of sample mass due to volatilization will cause the baseline to shift in the exothermic direction since the heat capacity difference between the sample and reference is effectively lessened. The loss of water or low temperature volatiles will cause a shift in the baseline.

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