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Sample Preparation for DTA – TGA, DSC

DTA-TGA Thermal Analyzer

a. The aluminum pan was filled with approximately 10–15 mg of the prepared sample and the sample was heated from the ambient temperature up to 600°C with heating rate of 10°C/min.
b. The analysis was done under nitrogen atmosphere with flow rate of (50 cm3/min) and, accordingly, the corresponding weight loss was recorded.
c. The differential scanning calorimetry analyses were carried out using Shimadzu DSC-60 (Shimadzu, Japan).
d. The heating and cooling program was from 30 °C to 220 °C and from 220 °C to 30 °C, respectively, at a rate of 10 °C/min and holding time of 3 min.
e. The degree of crystallinity (Xc) was calculated by using equation,

$$Xc = \frac{\Delta H_m}{(1-\Phi)\,\Delta H_m^0} \times 100\ \% \qquad (1)$$

where ΔHmis the calculated enthalpy of melting for the composite, Φ is the weight portion of the filler.

Methodology And Principle Of Dsc

This is a technique in which the difference in the amount of heat required to increase the temperature of a sample and reference are measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned.

The main application of DSC is in studying phase transitions, such as melting, glass transitions, or exothermic decompositions. These transitions involve energy changes or heat capacity changes that can be detected by DSC with great sensitivity.

Detection Of Phase Transitions

The basic principle underlying this technique is that, when 2g of the sample undergoes a physical transformation such as phase transitions, more or less heat will need to flow to

it than the reference to maintain both at the same temperature. Whether less or more heat must flow to the sample depends on whether the process is exothermic or endothermic. For example, as a solid sample melts to a liquid it will require more heat flowing to the sample to increase its temperature at the same rate as the reference. This is due to the absorption of heat by the sample as it undergoes the endothermic phase transition from solid to liquid. Likewise, as the sample undergoes exothermic processes (such as crystallization) less heat is required to raise the sample temperature. By observing the difference in heat flow between the sample and reference, differential scanning calorimeters are able to measure the amount of heat absorbed or released during such transitions. DSC may also be used to observe more subtle phase changes, such as glass transitions. It is widely used in industrial settings as a quality control instrument due to its applicability in evaluating sample purity and for studying polymer curing.

DSC curves

The result of a DSC experiment is a curve of heat flux versus temperature or versus time. This curve can be used to calculate enthalpies of transitions. This is done by integrating the peak corresponding to a given transition. It can be shown that the enthalpy of transition can be expressed using the following equation:

$\Delta H = KA$

where ΔH is the enthalpy of transition, K is the calorimetric constant, and A is the area under the curve. The calorimetric constant will vary from instrument to instrument, and can be determined by analyzing a well-characterized sample with known enthalpies of transition.

Applications

Differential scanning calorimetry can be used to measure a number of characteristic properties of a sample. Using this technique it is possible to observe fusion and crystallization events as well as glass transition temperatures T_g. DSC can also be used to study oxidation, as well as other chemical reaction

Glass transitions may occur as the temperature of an amorphous solid is increased. These transitions appear as a step in the baseline of the recorded DSC signal. This is due to the sample undergoing a change in heat capacity; no formal phase change occurs.

As the temperature increases, an amorphous solid will become less viscous. At some point the molecules may obtain enough freedom of motion to spontaneously arrange themselves into a crystalline form. This is known as the crystallization temperature (T_c). This transition from amorphous solid to crystalline solid is an exothermic process, and results in a peak in the DSC signal. As the temperature increases the sample eventually reaches its melting temperature (T_m). The melting process results in an endothermic peak in the DSC curve. The ability to determine transition temperatures and enthalpies makes DSC a valuable tool in producing phase diagrams for various chemical systems.

Differential scanning calorimetry can also be used to obtain valuable thermodynamics information about proteins. The thermodynamics analysis of proteins can reveal important information about the global structure of proteins, and protein/ligand interaction. For example, many mutations lower the stability of proteins, while ligand binding usually increases protein stability. Using DSC, this stability can be measured by obtaining Gibbs Free Energy values at any given temperature. This allows researchers to compare the free energy of unfolding between ligand-free protein and protein-ligand complex, or wild type and mutant proteins. DSC can also be used in studying protein/lipid interactions, nucleotides, drug-lipid interactions In studying protein denaturation using DSC, the thermal melt should be at least to some degree reversible, as the thermodynamics calculations rely on chemical equilibrium.

Sincerely,

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Executive Marketing Director