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TA Instruments
Thermal Analysis & Rheology

DSC 2910 Differential Scanning Calorimeter



DSC: The Technique

Differential Scanning Calorimetry (DSC) measures the temperatures and heat flows associated with transitions in materials as a function of time and temperature. Such measurements provide quantitative and qualitative information about physical and chemical changes that involve endothermic or exothermic processes, or changes in heat capacity.

DSC is the most widely used of all thermoanalytical techniques. It is used primarily to characterize polymers and other organic materials, but is also applicable to metals, ceramics and other inorganics.

What DSC Can Tell You

DSC provides important information that can be used to characterize materials. Specific measurements made by DSC include:

- Glass transitions
- Thermal stability
- Oxidative stability
- Percent crystallinity
- Reaction kinetics
- Heats of fusion and reactions
- Crystallization time and temperature
- Purity
- Rate of cure
- Degree of cure
- Boiling points
- Melting points
- Specific heat

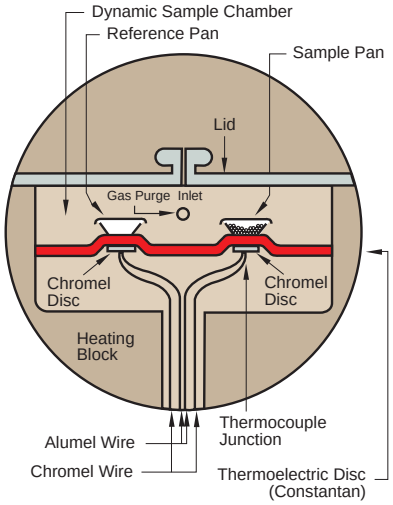
DSC 2910

The DSC 2910 is a product of nearly three decades of TA Instruments' leadership in calorimetry, enhanced by state-of-the-art electronics and instrument technology. The result is a module that is smarter, more sensitive, more versatile, and easier to use than any of its predecessors. In business and economic terms, the DSC 2910 offers its users the potential for strengthened competitive positioning through reduced costs, new and improved products, and reduced time lag between product conception and the market.

The complete DSC system consists of the DSC analysis module (base cabinet with interchangeable analysis cells), a computer-based controller/data analyzer, and a printer/plotter for preparation of hard-copy reports. The 2910 is designed to operate a standard DSC cell, pressure DSC cell, and a high-temperature (1600°C) DTA cell.

In addition to plug-in mountings for the interchangeable cells, the module cabinet contains the electronics and operating software needed to control the system, perform experiments and store results. A random access memory (RAM) with battery back-up assures integrity of operation and data. A GPIB interface provides communication with the controller/analyzer, which performs data analysis functions and provides for permanent data storage. A keypad/display on the front of the cabinet permits local

Figure 1. DSC Cell Schematic



start/stop control and displays real-time information on the experiment. The cabinet also contains connections for ancillary services, including purge gases, liquid nitrogen cooling, and automated end-of-run cell cool down.

Compatibility with accessories for differential photocalorimetry and Modulated DSC™*, as well as a 62-sample autosampler, further broaden the versatility of the DSC 2910.

Principle of Operation

A cross section diagram of the TA Instruments Standard DSC Cell is shown in Figure 1. The cell is based on a "heat flux" design which uses a constantan disk as the primary means of transferring heat to the sample and reference positions. The sample contained in a metal pan, and the reference (an empty pan) sit on raised platforms formed in the constantan disk. As heat is transferred through the disk, the differential heat flow to the sample and reference is measured by area thermocouples formed by the junction of the constantan disk and chromel wafers which cover the underside of the platforms. Chromel and alumel wires attached to the chromel wafers form thermocouples which directly measure the sample temperature. This continuous direct measurement of sample temperature accounts for high transition temperature repeatability and accuracy not available with alternative heat flux or power compensation DSC designs which determine sample temperature by calculation.

Constant calorimetric sensitivity is maintained throughout the usable temperature range of the cell by electronic linearization of the cell calibration constant. The temperature environment of the sample is controlled by a sophisticated feedback-control temperature programmer with its own thermocouple system located in the silver heating block. This allows the temperature of the sample to be held isothermal, or raised or lowered at a variety of pre-programmed rates. Purge gas is admitted to the sample chamber through an orifice in the heating block wall midway between the two raised platforms. The gas is preheated by circulation through the block before entering the sample chamber. The result is a uniform, stable thermal environment which assures excellent baseline flatness and exceptional sensitivity (signal-to-noise).

* U.S. Patent Nos. B1 5,224,775; 5,248,199; 5,335,993; 5,346,306; 5,439,291; 5,474,385
European Patent No. 0559362
Canadian Patent No 2,089,225

Features & Benefits

The DSC 2910 is designed with the operator in mind. In addition to retaining all of the proven attributes of its predecessors, the DSC 2910 offers new levels of sensitivity, versatility, automation, and ease-of-use. In combination with a Thermal Analyst Controller, the DSC 2910 is an excellent cost-effective DSC system. Key features and benefits include:

High calorimetric sensitivity (1μW), permitting measurement of low enthalpy transitions and the use of small samples resulting in high resolution and optimum temperature accuracy.

Direct measurement of sample temperature, assuring accurate and precise transition temperatures. In addition, temperature calibration can be based on one to five standards assuring maximum temperature accuracy.

Superior baseline stability, facilitating measurement of weaker transitions and assuring reproducibility and reliability of the data.

Modular design, with the economy and convenience of a single cell-base cabinet accommodating three different analysis cells-standard DSC, pressure DSC and high temperature DTA.

Methods versatility, permitting a wide choice of temperature programming (heating/cooling) and atmosphere conditions for obtaining maximum information from a single sample. An unlimited number of methods each containing up to 60 segments selected from 18 available functions can be stored at one time in system memory. Unique segments are available for controlling the LNCA and RCS cooling accessories and stopping (aborting) a segment when a specific measurement signal is achieved. Temperature programming options include heating and cooling at rates from 0.01 to 200°C/minute, step heating and cooling, and isothermal operation. Data collection rate can be adjusted to maximize data storage effectiveness. Furthermore, the DSC 2910 can be readily upgraded to perform Modulated DSC™ experiments.

Availability of specialized accessories, permitting specific experiments to be performed. These accessories include:

- *Quench Cooling Accessory:* for simple nonprogrammed rapid cooling
- *Liquid Nitrogen Cooling Accessory (LNCA):* for automated quench or programmed cooling to -150°C.
- *Refrigerated Cooling System (RCS):* a mechanical refrigeration device for controlled cooling to -70°C.
- *Gas Switching Accessory (GSA):* for programmed or manual switching of purge gases.
- *Autosampler Accessory:* for unattended evaluation of up to 62 samples.
- *Differential Photocalorimetry (DPC) Accessory:* for characterization of the photosensitive properties of materials.

Compatibility with other thermal techniques, broadening the range of materials and the types of measurements which can be performed. These other techniques include thermogravimetric analysis (TGA), simultaneous TGA-DTA, thermomechanical analysis (TMA), dynamic mechanical analysis (DMA) and dielectric analysis (DEA). These techniques can be run individually, or in multimodule configurations, by the Thermal Analyst Controllers to provide complete materials characterization.

APPLICATIONS

The broad capability of the TA Instruments DSC 2910 for characterizing materials is illustrated by these representative applications. These examples also illustrate many of the benefits inherent in the DSC 2910 System.

Evaluation of Subtle Transitions

Sensitivity and resolution are two important parameters associated with obtaining precise and accurate DSC results. Generally, optimizing both of these parameters simultaneously is difficult because sensitivity is increased by larger sample sizes and faster heating rates, while resolution is improved by smaller sample sizes and slower heating rates. The exceptional sensitivity of the DSC 2910, because of low noise and a flat baseline, however, facilitates obtaining both good resolution and sensitivity simultaneously. Figure 2, which shows the multiple phase transition peaks for a liquid crystal, illustrates this capability. These curves were run using approximately 1 milligram of material at 1°C/minute heating rate. The transition at 153°C is still detected and readily quantifiable even through the peak height associated with the transition is less than 20μW.

Thermal History of Thermoplastic Materials

The internal structure of thermoplastics is strongly affected by the thermal history imparted during processing. In particular, the rate of cooling from the melt can result in either a crystalline (more ordered) or amorphous (more random) internal structure. The presence of a glass transition in DSC indicates that some amorphous structure exists, while the presence of an endothermic melting peak indicates that some crystalline structure exists. Figure 3 shows the DSC heating profiles for two samples of a typical thermoplastic material, polyphenylene sulfide (PPS), that were previously subjected to different thermal histories. The solid curve represents the material after quench cooling from the molten state. The broken curve represents the material after slow, controlled cooling. The quenched material exhibits a totally amorphous internal structure (as indicated by a strong glass transition) which rearranges on heating to the more stable crystalline structure with an associated exothermic crystallization peak and subsequent melting peak. The slowly cooled material, on the other hand, yields a highly crystalline structure as evidenced by the presence of only a melting peak and a very weak glass transition on reheating. As these results indicate, DSC provides a convenient method for evaluating the effects of different processing conditions (thermal history) and is a valuable aid for choosing optimum processing conditions for obtaining a specific product.

In addition, the amount of crystalline structure (% crystallinity) can be quantified directly from the DSC melting endotherm by comparing the measured heat of fusion with that for a standard of known crystallinity. Alternatively, in polymer blends it is often possible to quantify blend composition based on the relative size of the crystalline melting peaks, provided thermal history effects are constant. Blends of polyethylene and polypropylene are a typical example.

Thermoset Cure Evaluation

Thermosets are another broad class of polymers which initially are powders or liquids, but which undergo a chemical reaction with time and temperature to form rigid, final materials. This chemical reaction process is called curing and involves crosslinking, that is, formation of new bonds in the material. Once curing occurs, thermosets, unlike thermoplastics, cannot be melted and reformed.

Since thermoset curing is accompanied by the evolution of heat (it's an exothermic reaction), DSC can be used to evaluate partially or fully cured thermosets. This is important because

Figure 2. Liquid Crystal Transitions

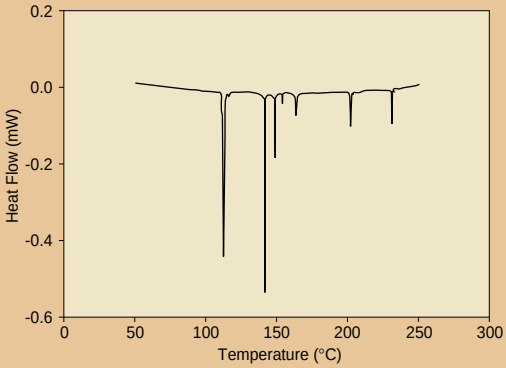


Figure 3. Thermal History Effects

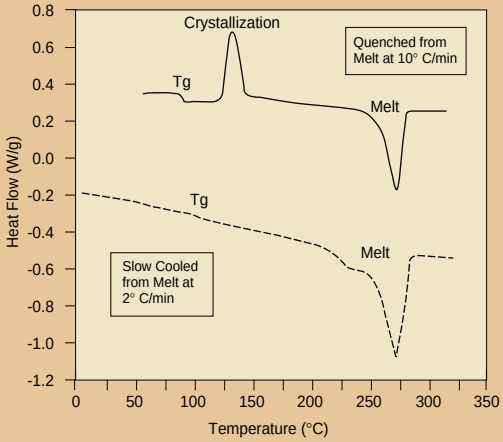


Figure 4. Thermoset Cure Determination

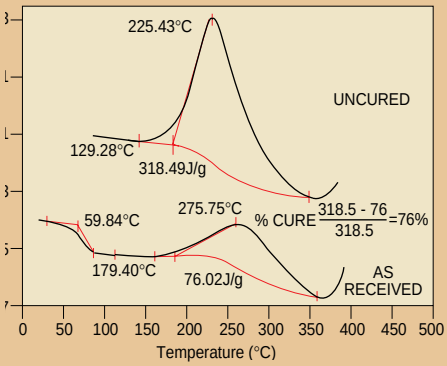
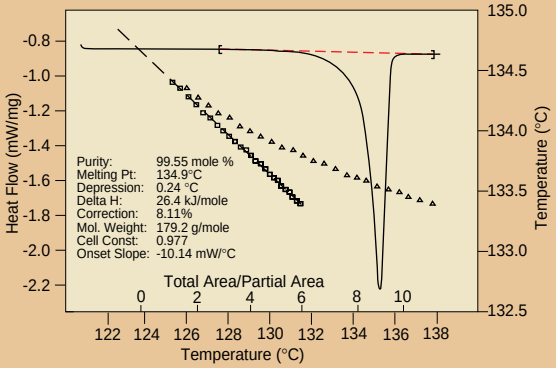


Figure 5. Purity of Pharmaceuticals



thermosets are often processed initially to a low level of cure (B stage) to facilitate storage and handling, and then later completely cured into the desired final form. Figure 4 shows the DSC curve for an epoxy resin "as received". Also shown is the DSC cure for a completely uncured sample of the same epoxy. By measuring the heat evolved by the uncured material (in this case 318 J/g), it is possible to determine the degree of cure for the "as received" resin by comparing its remaining heat of cure to the uncured material's. As shown in the Figure, the "as received" material is 76% cured. Notice that the partially cured resin exhibits a glass transition at 59.8°C. For thermosets, this glass transition temperature can also be used to determine the degree of cure provided suitable calibration curves are run.

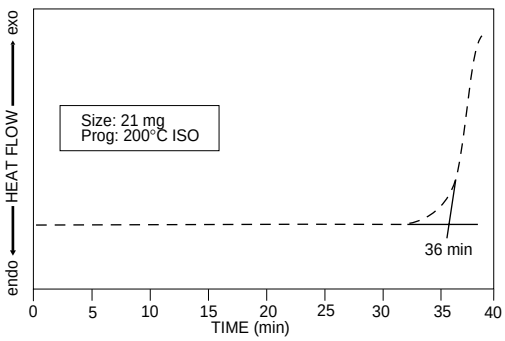
Determining the Calorimetric Purity of Pharmaceuticals

Using calorimetric purity software, the DSC 2910 can accurately determine the purity of highly pure (>95 mole % pure) chemicals. The technique is based on the principle that the concentration of impurity in a material is inversely proportional to its melting point; thus, an increase in the sample's impurity content decreases the melting point and broadens the melting range. Only a few milligrams of material are needed for an accurate determination without the need for pure reference materials. The analyst simply selects the baseline points, then the software calculates the mole percent purity. Results of an evaluation of a phenacetin sample are shown in Figure 5.

Oxidative Stability of Materials

Oxidative stability is an important in-use property for a wide range of materials including plastics, oils and lubricants, and foods. Although other factors such as temperature and exposure to ultraviolet light can adversely affect a material over a long period of time, attack by oxygen in the atmosphere is usually the key factor in determining the material's lifetime. Special compounds, called antioxidants, are often added to a base material to improve its resistance to attack by oxygen (its oxidative stability). Suppliers of the base materials are interested in comparing the effects of different antioxidants, as well as different levels of the same antioxidant, so that they can achieve the best compromise between increased lifetime of the base material and cost. Since oxidative stability is an exothermic process, DSC provides a convenient way to determine when significant oxidative degradation and material failure begins as shown in Figure 6 for a polyethylene wire coating.

Figure 6. Oxidative Stability



Specifications

Standard DSC Cell System

Atmosphere:	non-corrosive inert, reducing or oxidizing
Dynamic Gas Purge (preheated):	up to 100 mL/min at pressures from 300 Pa (2 torr) to atmospheric
Temperature Range:	
Inert Atmosphere:	ambient to 725°C
Air/Oxygen:	ambient to 600°C
With Quench Cooling Accessory:	-180 to 725°C
With Refrigerated Cooling System:	- 70 to 400°C
With Liquid Nitrogen Cooling Accessory:	- 150 to 725°C
Temperature Accuracy:	±1°C or 1%, whichever is greater
Temperature Reproducibility (using metal standards):	±0.1°C
Programmable Heating Rate:	0.01°C to 200°C/min
Maximum Sensitivity:	1 µW (2:1 signal-to-noise)
Calorimetric Precision:	±1% (based on metal samples)

Specifications are subject to change.

TA Instruments Commitment

The DSC 2910 Differential Scanning Calorimeter is designed and engineered to assure easy, reliable, trouble-free operation. It is supported by a full range of services, including an applications laboratory, publications, training courses, seminars, training and applications CD's, an Internet website and a telephone Hotline for customer consultation. Highly qualified service personnel, specialized in thermal analyzer/rheometer maintenance and service, are available throughout the world. All of these items reflect TA Instruments commitment to providing thermal analysis & rheology products and related services that deliver maximum value for your investment.

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