TA Instruments

THERMAL ANALYSIS

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Lindon, UT USA
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Bangalore, India
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Differential Scanning Calorimeters offering superior performance and unmatched flexibility for the widest range of applications

DSC Q20 **SPECIFICATIONS**

The Q20 (Q20, AQ20, Q20P) is a cost-effective, easy-to-use, general-purpose DSC module, with calorimetric performance superior to many competitive research-grade models. These are entry-level instruments not based on performance, but on available options. The Q20 is ideal for research, teaching, and quality control applications that require a rugged, reliable, basic DSC. The AQ20 is designed for unattended analysis of up to 50 samples in a sequential manner. The Q20 and AQ20 include dual digital mass flow controllers and are available with MDSC®. The Q20P is designed for studies of pressure-sensitive materials or samples that may volatilize on heating.

Hardware Features	Q20	AQ20	Q20P
Tzero® Cell (fixed position)	Included	Included	—
User Replaceable Cell	—	—	Yes
50-Position Autosampler	—	Included	—
Autolid	—	Included	—
Dual Digital Mass Flow Controllers	Included	Included	—
Full Range of Cooling Accessories (LNCS, RCS90, RCS40, FACS, QCA)	Available	Available	QCA Only
Pressure DSC	—	—	Yes
Platinum Software	—	Included	—
MDSC	Available	Available	—

Performance	Q20	AQ20	Q20P
Temperature Range	Amb to 725 °C	Amb to 725 °C	Amb to 550 °C
With Cooling Accessories	-180 to 725 °C	-180 to 725 °C	-130 to 550 °C
Temperature Accuracy	+/- 0.1 °C	+/- 0.1 °C	+/- 0.1 °C
Temperature Precision	+/- 0.05 °C	+/- 0.05 °C	+/- 0.05 °C
Calorimetric Reproducibility (indium metal)	+/- 1 %	+/- 1 %	+/- 1 %
Calorimetric Precision (indium metal)	+/- 0.1 %	+/- 0.1 %	+/- 0.1 %
Dynamic Measurement Range	+/- 350 mW	+/- 350 mW	+/- 350 mW
Digital Resolution	>0.04μW	>0.04μW	>0.04μW
Baseline Curvature (-50 to 300 °C)	<0.15 mW	<0.15 mW	—
Baseline Reproducibility	< 0.04 mW	<0.04 mW	—
Sensitivity	1.0 μW	1.0 μW	1.0 μW
Indium Height / Width (mW/°C)*	8.0	8.0	—

*Indium height/width ratio: 1.0 mg In heated at 10 °C/min in N_2 atmosphere. (A larger number denotes better performance).

Q SERIES™ DSC **TECHNOLOGY**

Tzero® Cell Design

The Tzero cell is designed for excellence in both heating and cooling. The heat flow sensor is machined for symmetry from a single piece of durable, thin wall, high response constantan and directly brazed to the silver heating block. Design benefits include faster signal response, flat and reproducible baselines, superior sensitivity and resolution, improved data precision, and unmatched ruggedness.

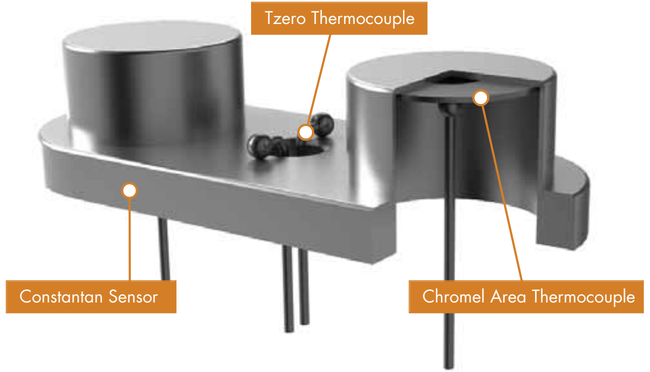
A chromel/constantan Tzero thermocouple is located symmetrically between the sample and reference sensor platforms, and acts as an independent measurement and furnace control sensor. Matched chromel area thermocouples are welded to the underside of each sensor platform, providing independently measured sample and reference heat flows that result in superior DSC and MDSC® results.

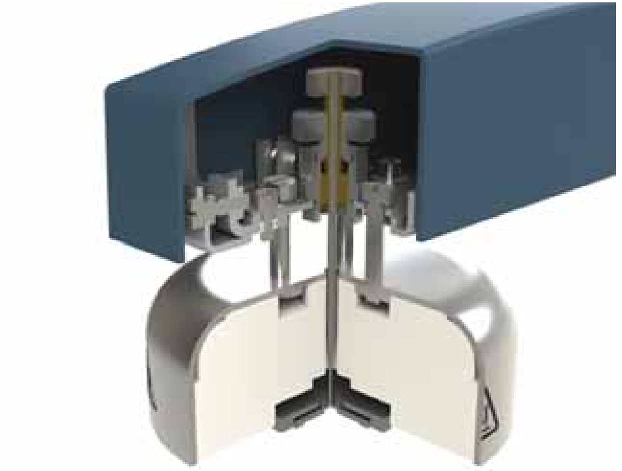
Auto Lid

The Q2000 and AQ20 have a new and improved auto lid assembly that consists of dual silver lids, an innovative lifting/venting mechanism, and a dome-shaped heat shield. More accurate and reproducible measurements result from improved thermal isolation of the cell.

Mass Flow Controllers

High quality DSC experiments require precise purge gas flow rates. Mass flow controllers, along with integrated gas switching, provide flexible control as part of individual methods. Purge gas flow rates are settable from 0-240 mL/min in increments of 1 mL/min. The system is precalibrated for helium, nitrogen, air and oxygen and suitable calibration factors may be entered for other gases.





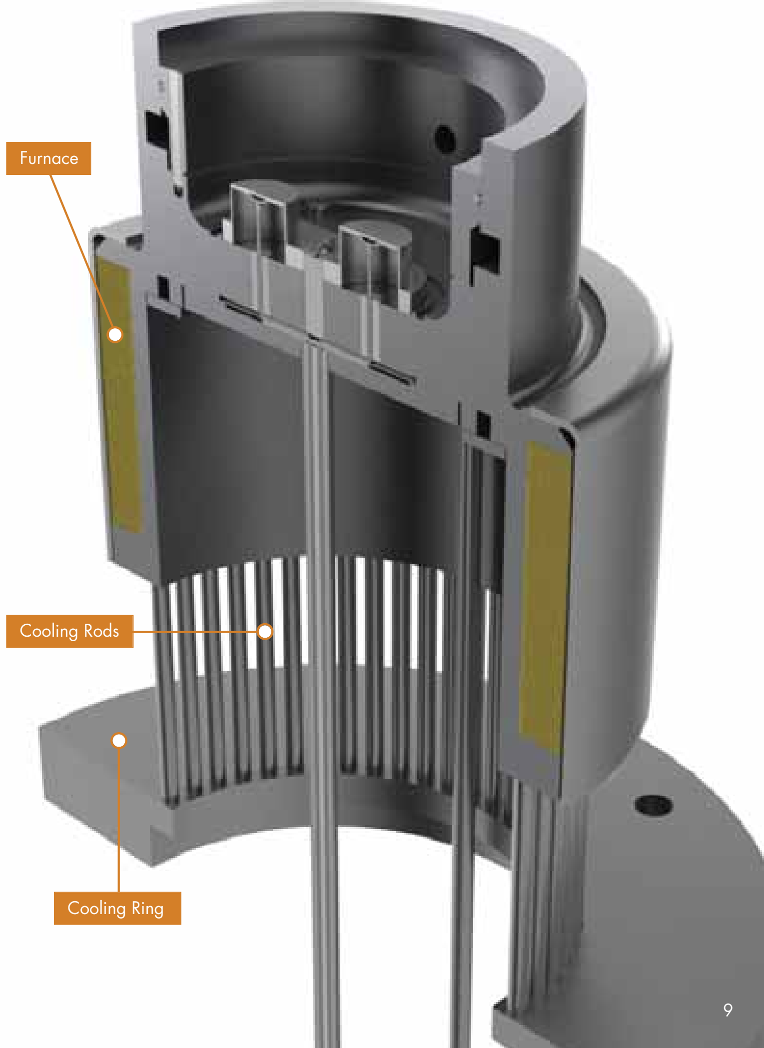
Cooling Rods & Ring

The unique design features an array of nickel cooling rods that connect the silver furnace with the cooling ring. This design produces superior cooling performance over a wide temperature range, higher cooling rates and better agility from heating to cooling operation. Lower sub-ambient temperatures and faster turnaround time can be obtained with our expanded range of cooling accessories in isothermal, programmed or ballistic cooling, and MDSC® experiments.

Furnace

The Tzero® transducer is enclosed in a high thermal conductivity, silver furnace, which uses rugged, long-life Platinel™ windings. Purge gases are accurately and precisely metered by digital mass flow controllers, and preheated prior to introduction to the sample chamber. Long furnace life and a highly uniform environment at the sample and reference sensors are ensured, as well as accurate isothermal temperatures, true linear heating rates, rapid temperature response, and the ability to heat at rates up to 200 °C / min.

Platinel™ is a trademark of the BASF Group



Q SERIES™ **ACCESSORIES**

Autosampler

The patented* DSC autosampler is a powerful performance and productivity enhancer for the Q Series DSC modules. It provides reliable, unattended operation of the AQ2000 and AQ20 modules. Unique in its ability to exchange up to 5 reference pans as well as 50 sample pans, the Q Series autosampler enables laboratories to reliably analyze samples "around-the-clock" in sequential order. An optical sensor guides the sample arm, ensuring precise pan placement and automatic calibration of the system. Maximum productivity from the DSC autosampler is achieved when paired with our intelligent Advantage™ software that permits pre-programmed analysis, comparison, and presentation of results.

*U.S. Patent No. 6,644,136; 6,652,015; 6,760,679; 6,823,278

Platinum™ Software

To further assure high-quality data, Q Series DSC modules equipped with the DSC autosampler (AQ20, AQ2000) can take full advantage of the Platinum features inherent in our Advantage software. These permit a user to automatically schedule a variety of calibration, verification and diagnostic tests to ensure that the DSC is constantly kept in optimum operating condition. Platinum software allows all Q Series DSC instruments to provide email notification of the completion of an analysis. Also included is the ability to view and download any new software versions that TA Instruments develops as upgrades to its standard Advantage software.

Tzero® DSC Sample Encapsulation Press

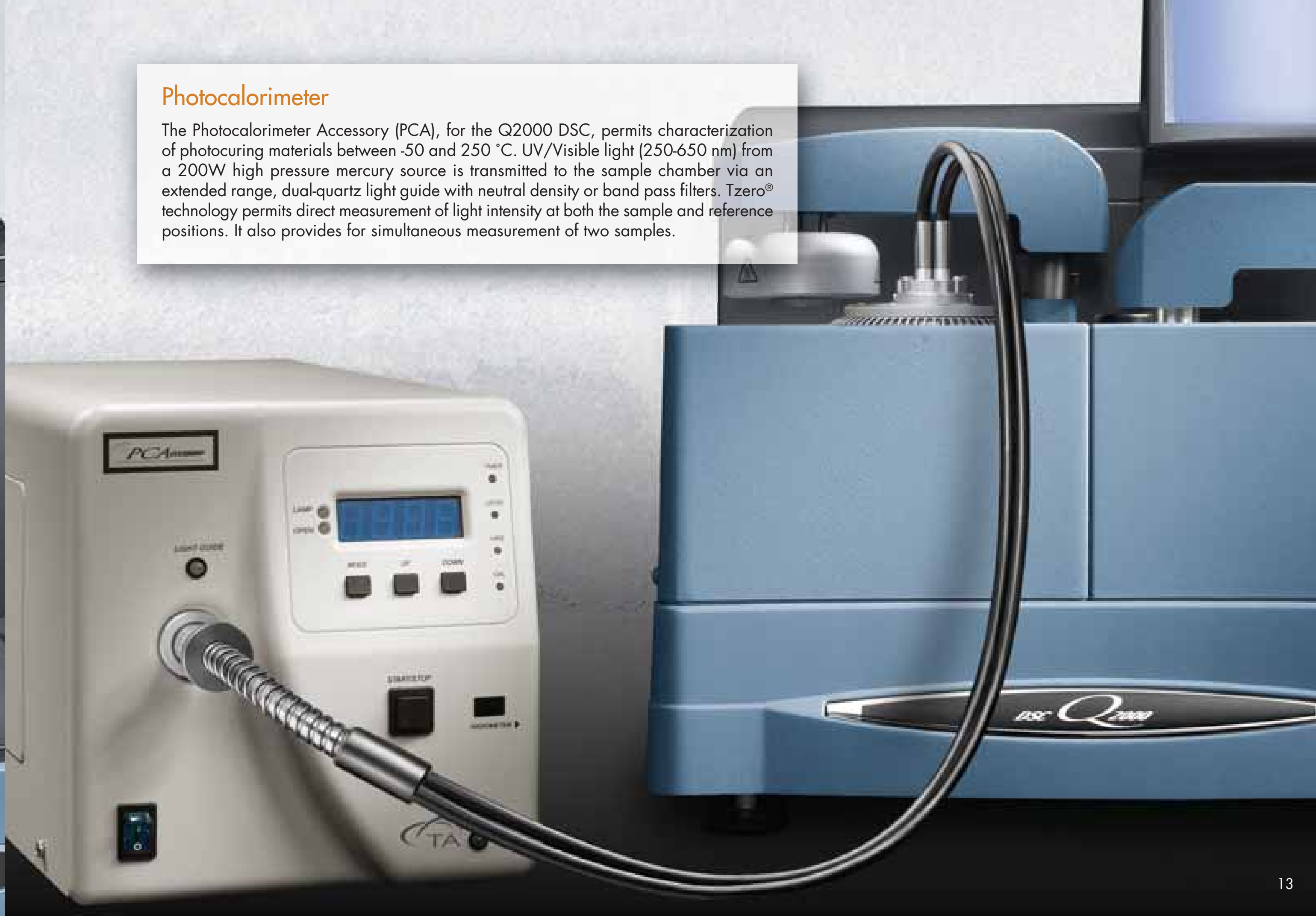
A key contributor to the quality of DSC results is the sample preparation. The new Tzero press takes sample encapsulation to a higher level of performance and convenience in crimp and hermetic sealing of a wide variety of materials. The press kit includes die sets (4) for the new Tzero aluminum and Tzero hermetic pans & lids and also for our upgraded standard and hermetic pans & lids (optional die sets are available for hi-volume DSC pans and Q5000 TGA sealed pans). The die sets are magnetically attached with no tools or user adjustments required. In addition, each die set is color-coded to the box containing the compatible Tzero or standard aluminum or hermetic pans and lids.

Pressure DSC

The Q20P is a dedicated pressure DSC system that provides heat flow measurements on pressure sensitive materials from -130 to 550 °C, at pressures from 1 Pa (0.01 torr) to 7 MPa (1,000 psi). The pressure cell employs standard heat flux DSC technology and incorporates pressure control valves, a pressure gauge, and over-pressure protection. This pressure DSC cell is also an accessory for the Q2000 DSC and can be used as a standard (ambient pressure) cell from -180 to 550 °C.

Photocalorimeter

The Photocalorimeter Accessory (PCA), for the Q2000 DSC, permits characterization of photocuring materials between -50 and 250 °C. UV/Visible light (250-650 nm) from a 200W high pressure mercury source is transmitted to the sample chamber via an extended range, dual-quartz light guide with neutral density or band pass filters. Tzero® technology permits direct measurement of light intensity at both the sample and reference positions. It also provides for simultaneous measurement of two samples.



TEMPERATURE CONTROL **OPTIONS**

Refrigerated Cooling Systems (RCS90 and RCS40)

The RCS is frequently selected as the preferred cooling device by thermal analysts for trouble-free, unattended DSC and MDSC® operation over a broad temperature range. Because it is a sealed system requiring only electrical power, the RCS has advantages for operation in areas where other refrigerants are difficult or expensive to obtain. TA Instruments offers two complementary models; the RCS90 and the RCS40. Both use the same cooling head, which fits snugly over the Q Series™ DSC Cell and completely eliminates frosting issues typical in competitive designs. Both controlled and ballistic cooling is achievable.

RCS90

The RCS90 employs a two-stage refrigeration system, which permits convenient DSC / MDSC operation over the temperature range from -90 °C to 550 °C. Typical RCS90 controlled cooling rates are detailed in the table below. Ballistic cooling from 500 °C to ambient is achieved in about 7 minutes. The RCS90 is compatible with all Q Series DSC Systems.

RCS40

The RCS40 employs a single-stage refrigeration system, which permits convenient DSC and MDSC® operation over the temperature range from -40 °C to 400 °C. Typical RCS40 controlled cooling rates are detailed in the table below. Ballistic cooling from 400 °C to ambient is achieved in about 7 minutes. The RCS40 is compatible with all Q2000 and Q20 models (except the Q20P).

RCS90 Controlled Cooling Rates, from 550 °C (upper limit)*

Controlled Rate	To Lower Temperature
100 °C/min	300 °C
50 °C/min	120 °C
20 °C/min	-20 °C
10 °C/min	-50 °C
5 °C/min	-75 °C
2 °C/min	-90 °C

RCS40 Controlled Cooling Rates, from 400 °C (upper limit)*

Controlled Rate	To Lower Temperature
65 °C/min	250 °C
50 °C/min	175 °C
20 °C/min	40 °C
10 °C/min	0 °C
5 °C/min	-15 °C
2 °C/min	-40 °C

* Performance may vary slightly, depending on laboratory conditions.

TEMPERATURE CONTROL **OPTIONS**

Liquid Nitrogen Cooling System

The Liquid Nitrogen Cooling System (LNCS) provides the highest performance and greatest flexibility in cooling. It has the lowest operational temperature (to -180 °C), greatest cooling rate capacity (to 140 °C/min), and an upper temperature limit of 550 °C. Typical LNCS controlled cooling rates are detailed in the table below. Ballistic cooling from 550 °C to ambient is achieved in about 5 minutes. Its autofill capability allows the LNCS to be automatically refilled from a larger liquid nitrogen source for continuous DSC operation. The LNCS is available for all Q Series™ DSC Systems (Except the Q20P).

LNCS Controlled Cooling Rates, from 550 °C (upper limit)*

Controlled Rate	To Lower Temperature
100 °C/min	200 °C
50 °C/min	0 °C
20 °C/min	-100 °C
10 °C/min	-150 °C
5 °C/min	-165 °C
2 °C/min	-180 °C

* Performance may vary slightly, depending on laboratory conditions.

Finned Air Cooling System

The Finned Air Cooling System (FACS) is an innovative cooling accessory for all the Q Series™ DSC modules that offers a cost-effective alternative to the RCS or LNCS cooling systems. The FACS can be used for controlled cooling experiments, thermal cycling studies, and to improve sample turnaround time. Stable baselines and linear heating and cooling rates can be achieved between ambient and 725 °C.

Quench Cooling Accessory

The Quench Cooling Accessory (QCA) is a manually operated cooling accessory, whose primary use is with the Q20 DSC to quench cool a sample to a sub-ambient temperature prior to heating to an upper limit. The recommended temperature of operation of the QCA is from –180 to 400 °C. The QCA reservoir is easily filled with ice water, liquid nitrogen, dry ice, or other cooling media.

Tzero® Technology Provides:

- Flat reproducible baselines with better than an order of magnitude improvement on competitive designs, especially in the sub-ambient temperature range
- Superior sensitivity due to flatter baselines and better signal-to-noise ratio
- Best available resolution (better than power compensation devices)
- Faster MDSC® experiments
- Direct measurement of heat capacity (Q2000)

Tzero technology represents a fundamentally more accurate system for measuring heat flow, by incorporating cell resistance and capacitance characteristics which were previously assumed to be negligible. The inclusion and compensation of these effects dramatically improves the baseline response and reproducibility. The heat flow resolution is also improved by directly measuring the heating rate difference between the sample and reference, and compensating for its effect on heat flow. This Tzero approach to measuring heat flow is a proprietary and patented* technology, only available on TA Instruments DSC instruments.

*U.S. Patent No. 6,431,747; 6,488,406; 6,523,998

MDSC® Technology Provides:

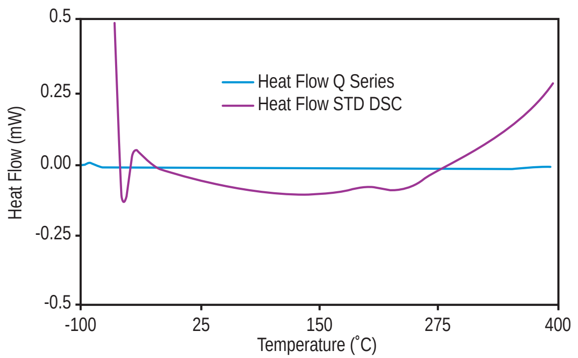
- Separation of complex transitions into more easily interpreted components
- Increased sensitivity for detecting weak transitions and melts
- Increased resolution without loss of sensitivity
- Direct measurement of heat capacity
- More accurate measurement of crystallinity

In MDSC*, a sinusoidal temperature oscillation is overlaid on the traditional linear ramp. The net effect is that heat flow can be measured simultaneously with changes in heat capacity. In MDSC, the DSC heat flow is called the Total Heat Flow, the heat capacity component is the Reversing Heat Flow, and the kinetic component is the Nonreversing Heat Flow. The Total Heat Flow signal contains the sum of all thermal transitions, just as in standard DSC. The Reversing Heat Flow contains glass transition and melting transitions, while the Nonreversing Heat Flow contains kinetic events like curing, volatilization, melting, and decomposition. The Q2000 uniquely permits increased MDSC productivity of high quality data by its ability to operate at standard DSC heating rates (e.g., 10 °C / min.).

*U.S. Patent Nos. 5,224,775; 5,248,199; 5,346,306
Canadian Patent No. 2,089,225 Japanese Patent No. 2,966,691

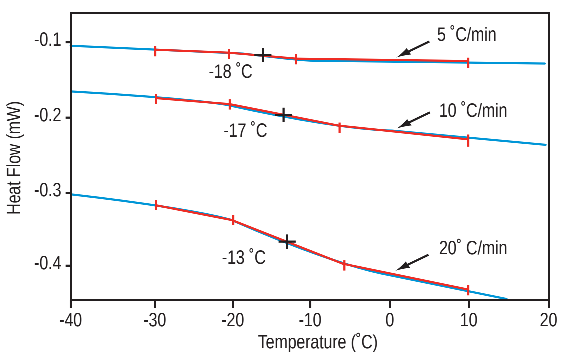
Baseline Stability (Flatness)

The figure to the right shows a comparison of a Q2000 empty cell baseline with that from a traditional heat flux DSC. The data shows that the Q2000 baseline is superior in every way. The start-up offset is much smaller, the baseline is dramatically straighter, and the slope is greatly reduced. This contrasts markedly with results from other DSC designs, where a baseline curvature 1 mW over the same temperature range is often considered acceptable.



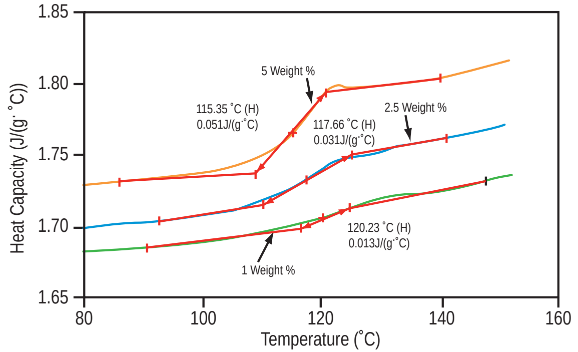
Sensitivity (Polymer Tg)

This figure shows a Q2000 high sensitivity glass transition (Tg) measurement, as a function of heating rate, for a very small (1 mg) sample of polypropylene, whose Tg is not easily measured by DSC due to its highly crystalline nature. The data shows that the Tg is easily detected even at a slow 5 °C/min heating rate. The excellent Q2000 baseline is the essential key for accurate measurements of glass transitions and heat capacity from materials that exhibit weak and broad transitions.



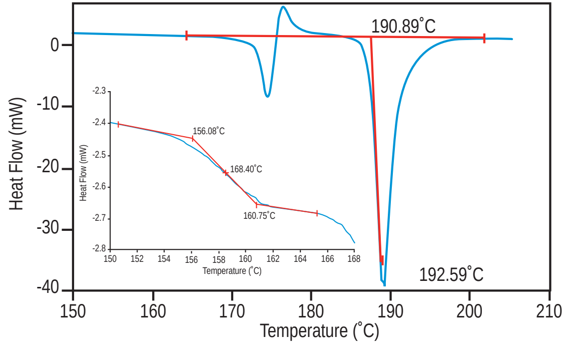
Sensitivity (Lactose Tg)

This figure illustrates the high level of sensitivity in a pharmaceutical application. The detection of small amounts of amorphous lactose is critical for drug development and is easily achieved on a 10 mg sample at 20 °C/min using the Q2000. The direct measurement of heat capacity allows the step change in Cp to be quantified, which is found to be directly proportional to the amount of amorphous material present in the sample.



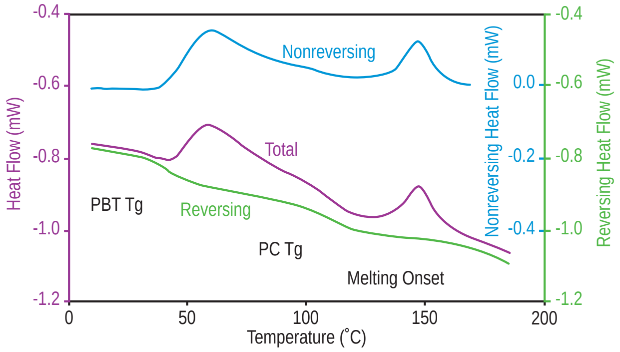
Resolution

The figure to the right shows the excellent resolution inherent in the Tzero® heat flow signal. This pharmaceutical compound contains a series of polymorphic transitions. Note that each peak is easily resolved, while simultaneously maintaining sensitivity for the subtle glass transition at lower temperature (inset).



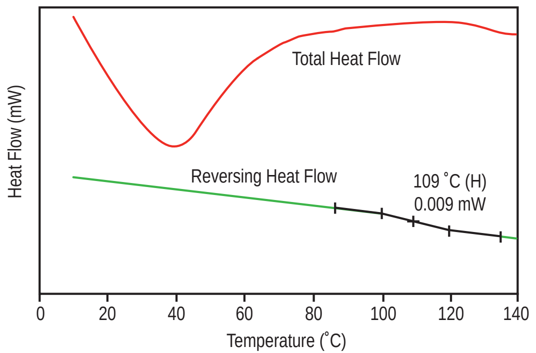
Separation of Complex Transitions

The figure to the right shows the MDSC results for a thermoplastic alloy blend of polycarbonate (PC) and polybutylene terephthalate (PBT). This material exhibits a variety of overlapping transitions, and interpretation of the Total Heat Flow is complicated. MDSC effectively separates the crystallization of the PBT component into the Nonreversing Heat Flow, thereby allowing for accurate determination of the glass transition temperatures of each polymer.



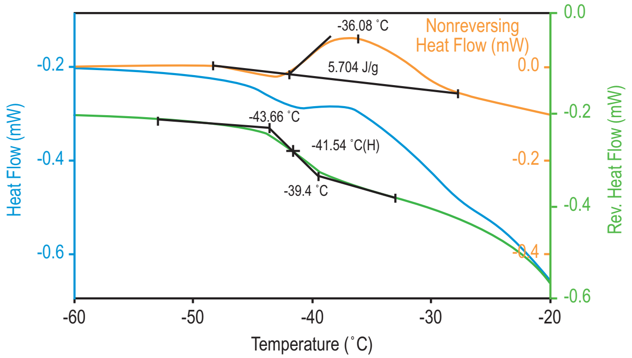
Improved Signal Sensitivity

MDSC provides improved sensitivity for measuring very broad and weak transitions, such as glass transitions in highly crystalline polymers or where the Tg is hidden beneath a second overlapping thermal event. This data was generated using a very small (2.2 mg) sample of a polymer coating. The total heat flow shows no transitions in the region where a Tg would be expected, though the large endotherm around 40 °C indicates solvent loss. The Reversing Heat Flow does indicate a very weak (8.5 μW) Tg around 109 °C, confirming the sensitivity of the MDSC technique.



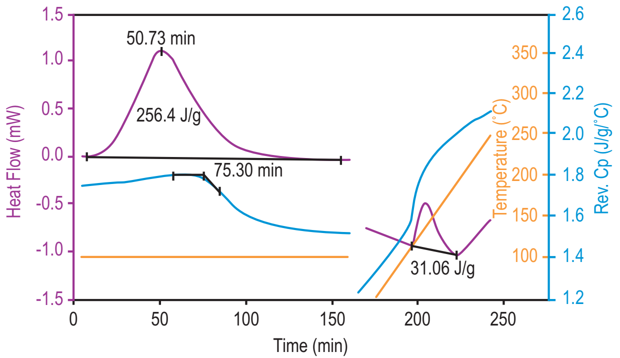
Improved Data Interpretation

The figure shows an application of interest in studies of foods or pharmaceuticals, in which the MDSC® total heat flow signal and its reversing and non-reversing components are displayed for a quenched 40% aqueous sucrose sample. The reversing signal clearly indicates a Tg for sucrose between -43.6 and -39.4 °C. The exothermic nonreversing signal relates to crystallization of free water that could not crystallize during quench cooling of the sample due to a significant increase in mobility and diffusion of the material at the glass transition.



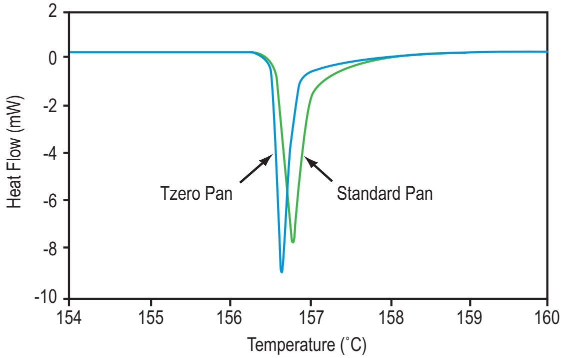
Quasi-Isothermal Heat Capacity

One of the major benefits of Modulated DSC® is the ability to measure heat capacity in a quasi-isothermal mode, i.e. isothermal with the exception of a small temperature modulation. Quasi-isothermal MDSC is particularly beneficial when studying curing systems. The figure below contains the quasi-isothermal analysis of thermosetting epoxy resin. In the first part of the experiment, the curing is monitored at 100 °C for 160 min, and is evident as a decrease in Cp and a large exotherm in the Total Heat Flow. The second stage involved heating the sample under MDSC conditions at 3 °C/min to measure the Tg of the cured system, as well as residual cure.

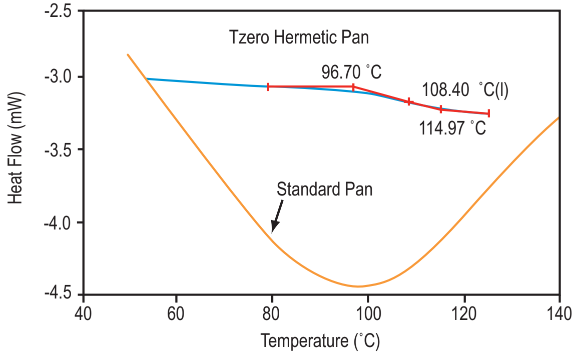


TZERO® PANS/LIDS **APPLICATIONS**

Fabricated using advanced technology and to extremely tight tooling specifications, the Tzero pans offer a significant improvement over previous generations, as well as competitors' technology, in standard performance tests on indium metal. In the figure to the right, note the improvement in signal response and peak quality when using the Tzero pan versus a standard pan.



The utility of the Tzero hermetic pan is shown here. In this example, DSC results for casein are shown using both a hermetic pan and a standard pan. Casein contains a significant amount of adsorbed moisture, which is evolved when heating in a standard pan. The resultant data only exhibits the large evaporation endotherm. However, when analyzed in a Tzero hermetic pan, the volatilization is suppressed, and the glass transition of the casein is clearly identified.



STANDARD PANS/LIDS **APPLICATIONS**

DSC pans & lids are available in aluminum, alodine-coated aluminum, gold, platinum, graphite, and stainless steel versions. They can be used under a variety of temperature and pressure conditions. Samples can be run in the standard DSC mode in open pans, crimped or hermetically sealed pans / lids or in pressure capsules. Samples in open pans can also be run at controlled pressures using the PDSC Cell. All aluminum standard pans have the same temperature and pressure rating. General details of the pans are shown here.

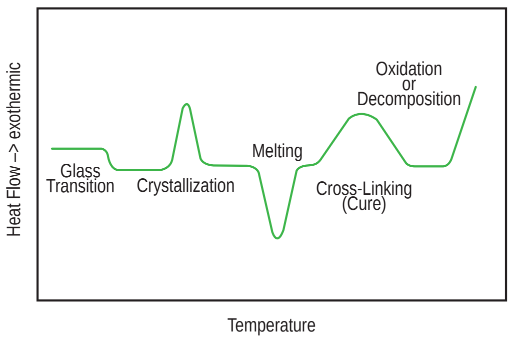
Standard	Temperature (°C)	Pressure
Aluminum	-180 to 600	100 kPa
Platinum	-180 to 725	100 kPa
Gold	-180 to 725	100 kPa
Graphite	-180 to 725	100 kPa
Hermetic	**Temperature (°C)**	**Pressure**
Aluminum	-180 to 600	300 kPa
Alodined Aluminum	-180 to 200	300 kPa
Gold	-180 to 725	600 kPa
High Volume	-100 to 250	3.7 MPa
Stainless Steel	Amb. to 250	10 MPa

Transition Temperatures

DSC provides rapid and precise determinations of transition temperatures using minimum amounts of a sample. Common temperature measurements include the following:

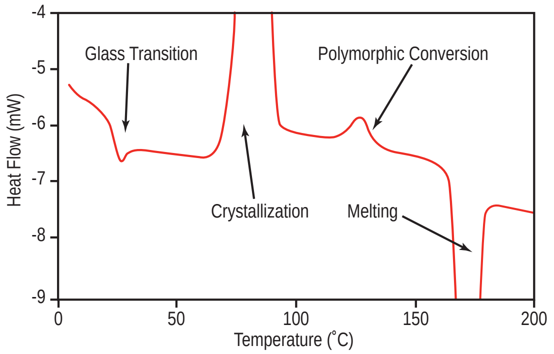
- Melting
- Glass Transition
- Thermal Stability
- Oxidation Onset
- Cure Onset
- Crystallization
- Polymorphic Transition
- Liquid Crystal
- Protein Denaturation
- Solid-Solid Transition

This composite shows typical shapes for the main transitions observed in DSC.



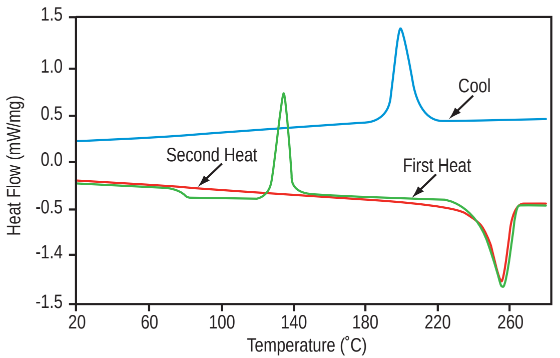
Heat Flow

Heat Flow is the universal detector, as all physical and chemical processes involve the exchange of heat. As such, the DSC Heat Flow signal is commonly used to measure and quantify a wide variety of transitions and events, often occurring in the same material as a function of temperature. This example shows a pharmaceutical material which is undergoing a variety of physical changes as it is heated from subambient through its melting temperature. DSC is sensitive to all of these events.



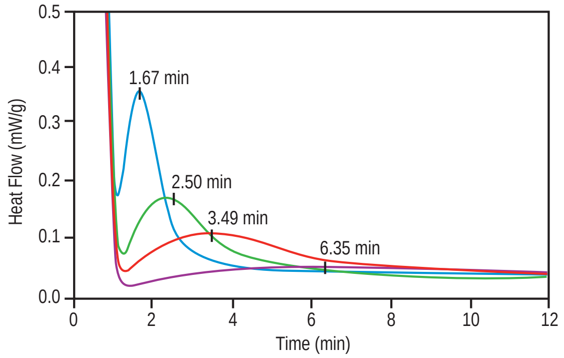
Thermal History

DSC is an excellent tool for determining the thermal history of a polymer sample. In this experiment, the sample is subjected to a "heat-cool-reheat" cycle and a comparison is made between the two heating cycles. This figure contains the heat-cool-reheat results for a polyester sample. By comparing the first heating cycle (unknown thermal history) to the second heating cycle (known thermal history), information can be derived concerning the original morphology of the material. This can be useful in troubleshooting problems in performance or processing conditions.



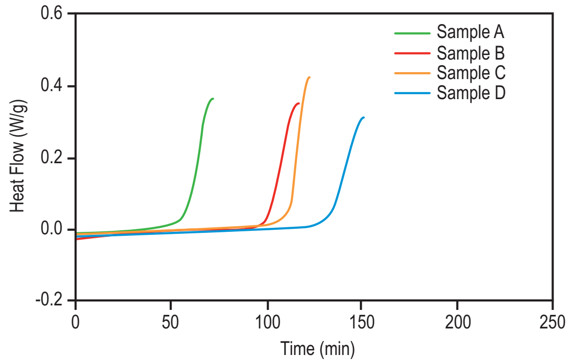
Kinetics

Kinetics is the study of the effects of time and temperature on a reaction. Isothermal crystallization is an example of an experiment in which kinetic information can be derived. This data shows the isothermal crystallization results for a polymer sample which is being crystallized at a variety of temperatures below the melting point. By analyzing the time to peak heat flow for each temperature, various kinetic factors can be calculated including activation energy, rate constant, and conversion percent.



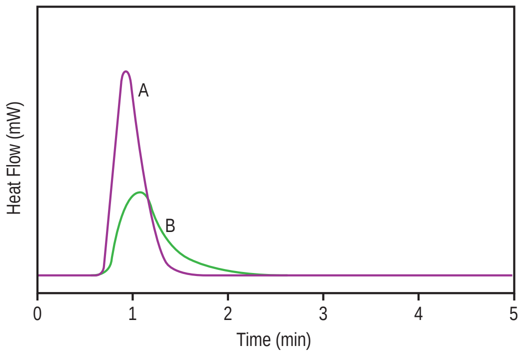
Pressure (and Time)

Pressure DSC accelerates OIT analyses and resolves the onset of the oxidation process. The figure to the right shows a comparative study of a series of two component polymer dispersions containing different levels of the same antioxidant. Clear performance differences are readily seen. The tests provided the same answer in under two days that took up to two months of traditional "field exposure" to obtain. Other common PDSC applications include a) thermoset resin cures, b) catalyst studies, and c) micro-scale simulations of chemical reactions.



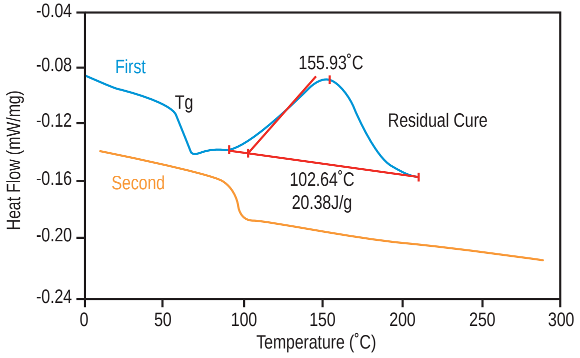
Photocuring

The Photocalorimeter Accessory (PCA) provides a convenient tool to assess reactions initiated with UV/Visible light. This figure compares two different acrylic formulations under the same conditions. The data shows that formulation A cures rapidly upon exposure to UV radiation, while formulation B reacts slower, and has both a longer time-to-peak and lower energy. In all PCA experiments, the peak shapes and transition energies are affected by the formulation chemistry, additives, initiators, and the purge gas used.



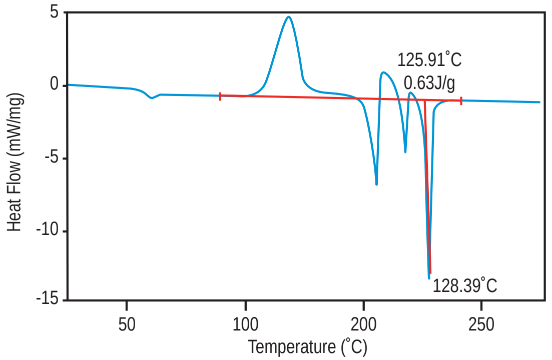
Degree of Cure

The degree of thermoset cure can dramatically affect the processing and end-use conditions. DSC is often used to investigate and quantify the degree of cure for epoxy and other thermosetting materials. This figure contains the data for the first and second heats of a thermoset material. The exotherm in the first heat indicates that the sample was not fully cured as received. By quantifying the residual cure, as well as comparing the glass transition temperatures of the two cycles, the degree of cure is easily determined.



Pharmaceutical Polymorph Analysis

Pharmaceutical materials often exist in multiple crystal forms called polymorphs. These have the same chemical structure but a different crystalline structure which can result in significant differences in physical properties such as solubility, bioavailability, and storage stability. DSC is the prevalent technique for the detection of pharmaceutical polymorphism. The DSC analysis of a pharmaceutical material in which three distinct polymorphs are detected on heating the amorphous compound is shown here.



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