Diamond Differential Scanning Calorimeter (DSC)

high **sensitivity** thermal analysis

PerkinElmer
precisely.
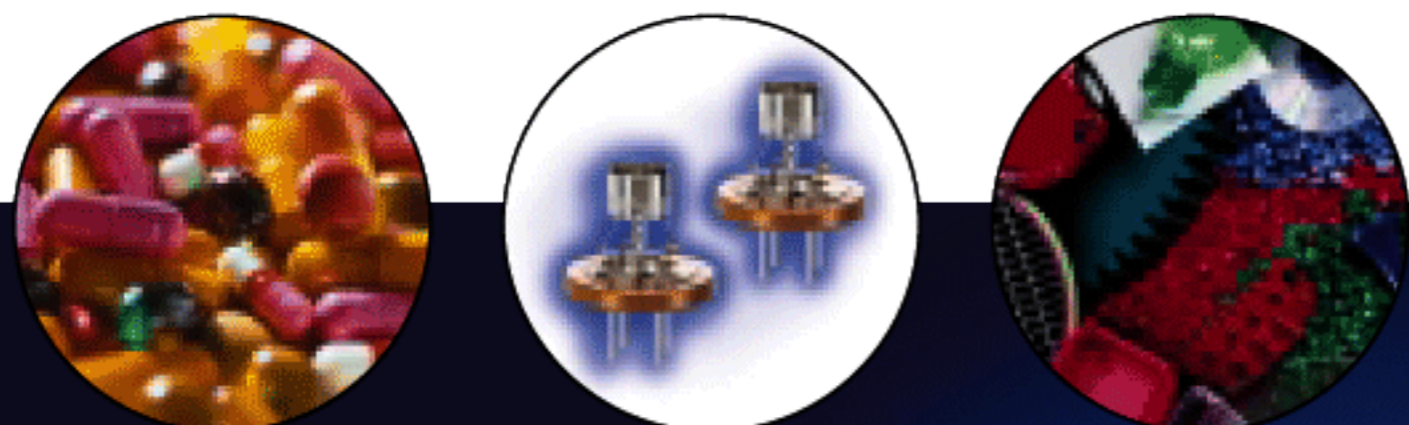
Diamond DSC

specifications

DSC Type	Power-compensation temperature null principle. Measures temperature and energy directly, rather than differential temperature (DT).
DSC Cell	Independent dual furnaces constructed of platinum-iridium alloy with independent platinum resistance heaters and temperature sensors with furnace mass less than 1g.
Temperature Sensors	Distributed, Platinum Resistance Thermometers for best linearity.
Atmosphere	Static or dynamic, including nitrogen, argon, helium, carbon dioxide, air, oxygen or other inert or active gases, over full temperature range. Oxygen can be used up to 730 °C which allows easy cleaning.
Temperature	Range -170 °C to 730 °C Accuracy / Precision ±0.1 °C / ±0.01 °C
Calorimetry	Accuracy / Precision < ±1% / < ±0.1% Sensitivity 0.2 µW Dynamic Range 0.2 µW to 800 mW
Signal Response	(1 mg Indium, 10 °C/min, nitrogen purge) Peak Height 7.44 mW ± 0.15 mW Width at half height 0.42 ± 0.10 °C H/W Ratio 17.6 mW/°C ± 1 mW/°C
Isothermal Drift (10 min)	-150 °C / 100 °C < 15 µW / < 10 µW
Scanning Rates	Heating/Cooling 0.01 °C to 500 °C/min
Temperature Overshoot	100 °C/min < 0.1 °C

Controlled Cooling	Ambient Coolant - nitrogen purge
	                                10 °C/min to 50 °C
	                                20 °C/min to 65 °C
	                                50 °C/min to 100 °C
	                                100 °C/min to 170 °C
	Liquid N_2 Coolant - helium purge
	                                10 °C/min to -170 °C
	                                50 °C/min to -165 °C
	                                100 °C/min to -135 °C
	                                200 °C/min to -85 °C
	                                300 °C/min to -80 °C
	                                400 °C/min to -10 °C

Cooling Times	Ambient Coolant 725 °C to 100 °C (under 4 minutes) Liquid N_2 Coolant 200 °C to -150 °C (under 2 minutes)
Cooling Options	Ice Water 25 °C to 730 °C Circulating Liquid -10 °C to 730 °C Refrigerator (Intracooler) -70 °C to 730 °C Automatic Liquid N_2 (CryoFill) -170 °C to 300 °C
Autosampler	The Diamond DSC Autosampler can run up to 44 sample positions unattended. It has the ability to be customized through Pyris Player to meet your analysis needs and increase productivity.
High Pressure Cell	Extends the capabilities of the power-compensation Diamond DSC design to elevated pressure measurements. Pressure range is up to 42 bar (600 psi).
Quality Assurance	Developed under ISO 9001
Dimensions (HxWxD)	34 x 40 x 67 cm (14 x 16 x 27 in)
Weight	20 kg (44 lbs)
Power Requirements	100-240 Volt, 50/60 Hz

power-compensation technique yields
# confidence-building results

Effective competitive analysis requires high resolution and sensitivity

Liquid crystalline materials can exhibit a wide range of liquid crystalline phases with varying degrees of molecular order. These changes can be seen by DSC studies but the energies involved in these transitions can be very small, making their identification difficult. In the past, most of these small energy transitions have been identified by optical microscopy alone. In this example, we show that it is possible to see these small energy transitions plainly with the Diamond DSC.

If the DSC trace is zoomed into the area of 80-95 °C, it is possible to identify some small endothermic events that occur in this range. The energies involved in these transitions are very small (0.024J/g) and demonstrate the extremely high sensitivity of the Diamond DSC.
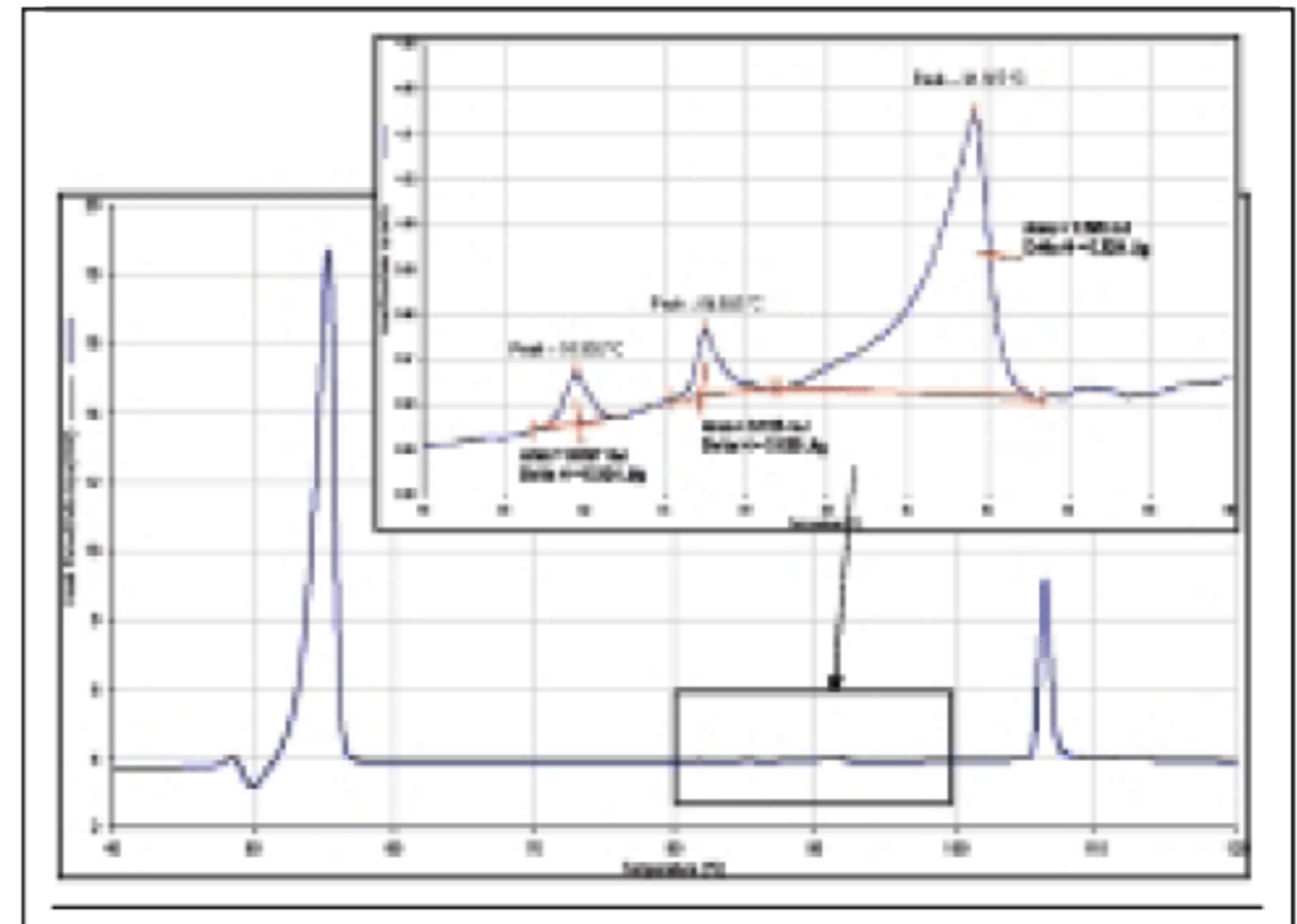

Liquid crystal phase change transitions.

Oftentimes, instrument companies provide specifications as to sensitivity and resolution without describing how they were generated

The use of 4, 4' azoxyanisole has been recommended as both a resolution and sensitivity standard for assessing DSC performance by the Netherlands Society for Thermal Analysis (TAWN). This substance has two closely occurring endothermic transitions at 117 °C and 134 °C. The resolution of the DSC instrument can be defined as how well the heat flow response returns to a linear baseline in the region between the two transitions when the sample (5 mg) is heated at a rate of 20 °C/min using a nitrogen purge gas. The better the resolution of the instrument, the better the heat flow response returns to the baseline. An instrument with a high inherent resolution such as the Diamond DSC will yield a linear heat flow response between the two transitions and a better return to the baseline.

The resolution index, or R value, can be used to compare DSC performance. This is done by taking the heat flow value at the minimum point between the two peaks and then dividing this by the value of the heat flow at the smaller peak maximum.
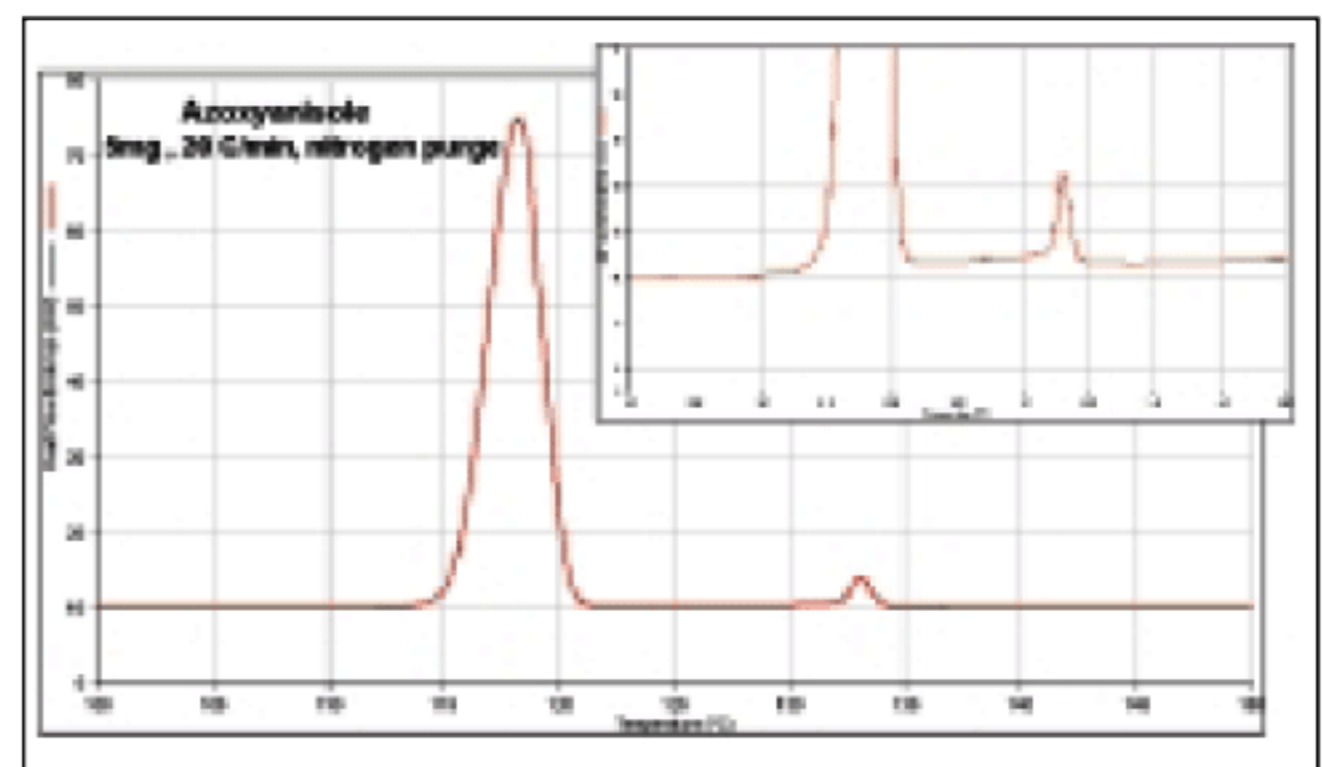
DSC results on azoxyanisole test.

HyperDSC in polymers

HyperDSC helps mimic process conditions

The plastics industry has a high interest in having a clear understanding of how materials behave under process conditions. In injection molding, blow-molding and extrusion processes, high cooling and heating rates play the dominant role. Product development requires lab-scale experiments in which these conditions can be simulated. HyperDSC provides that additional information that you don't capture with standard DSC. In addition, high heating and cooling rates minimize noise, thereby enabling highly quantitative measurements.

The analysis of a PET sample demonstrates the influence of the scanning rate on the results.

The slow heating at 10 °C/min after cooling the PET at 100 °C/min shows extensive cold crystallization followed by a melting of the formed crystals (dashed line). This heating run performed at 10 °C/min is definitely not representative of the thermal history (blue line) in which no crystallization event occurs. Heating at 100 °C/min (red curve) after cooling at 100 °C/min shows hardly any cold crystallization on the time scale of the experiment. With

an increasing heating rate, recrystallization is suppressed and the resulting heating curve reflects the characteristic of the material at room temperature obtained by a fast cooling.

Note: Acknowledgement to Vincent Mathot, Thijs Pijpers, Eric van der Vegte, DSM Research, The Netherlands for their initiative and work in this application field.
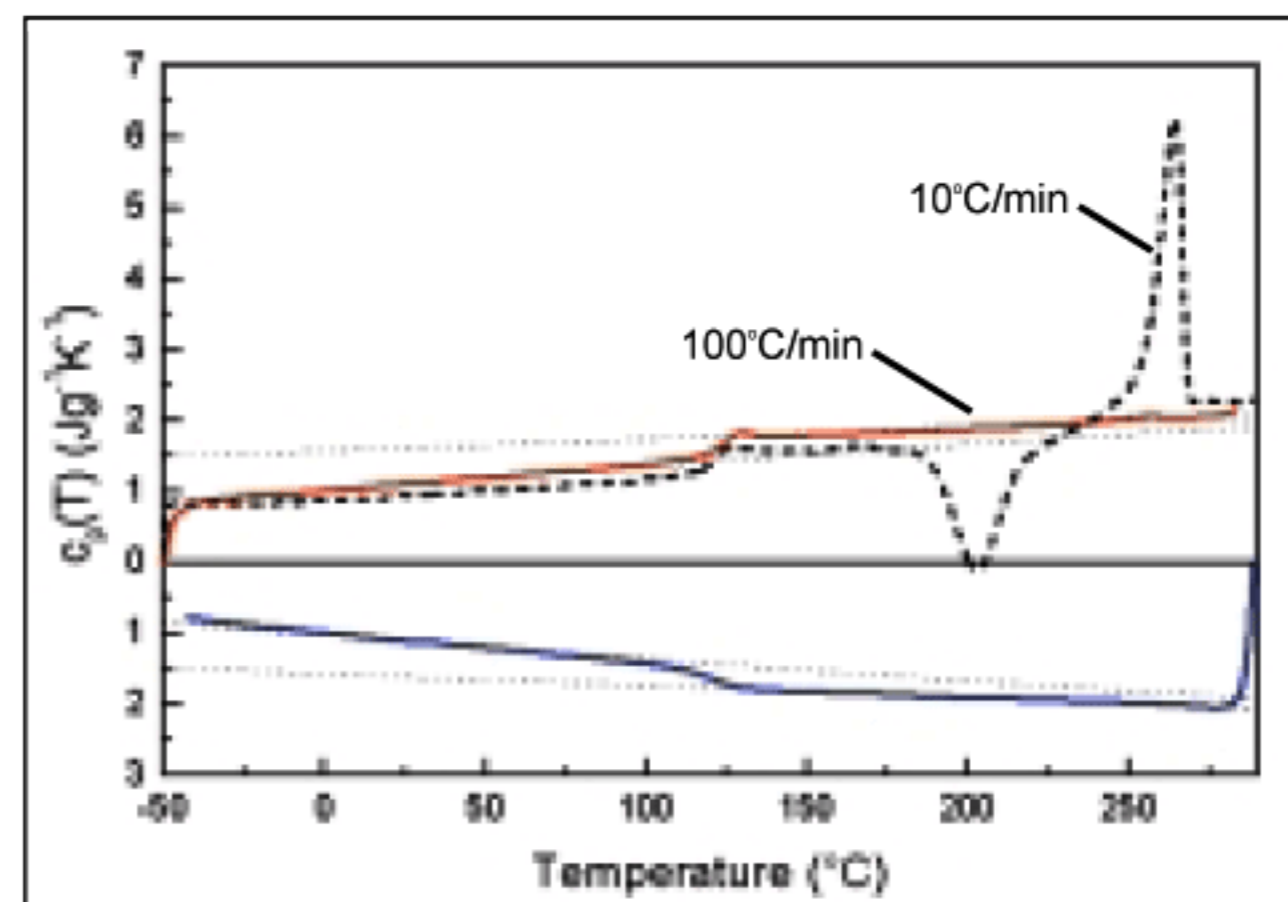

PET Analysis by HyperDSC: No cold crystallization during fast scanning.

HyperDSC in polymers

HyperDSC rapidly characterizes materials with weak transitions

Conventional DSC analysis often does not provide the high sensitivity required for the determination of weak glass transitions in highly crystalline materials or filled polymers. Techniques such as dynamic mechanical analysis (DMA) or modulated temperature DSC are often used to analyze the transition temperature in these cases. Unfortunately, both approaches are very time consuming and may require highly experienced technicians or scientists to run and analyze the data. HyperDSC provides a way to use DSC analysis for these applications, simplifying the experimental method and analysis of data. This example shows the measurement of an epoxy which is highly filled with glass fiber analyzed with a HyperDSC method (scanning rate of 250 °C/min). The increased sensitivity with HyperDSC provides an unambiguous glass transition event

in less than one minute as shown here. To demonstrate the reproducibility of the Diamond DSC in the HyperDSC mode, a second sample was analyzed. The results show a nearly identical curve for both sample runs.
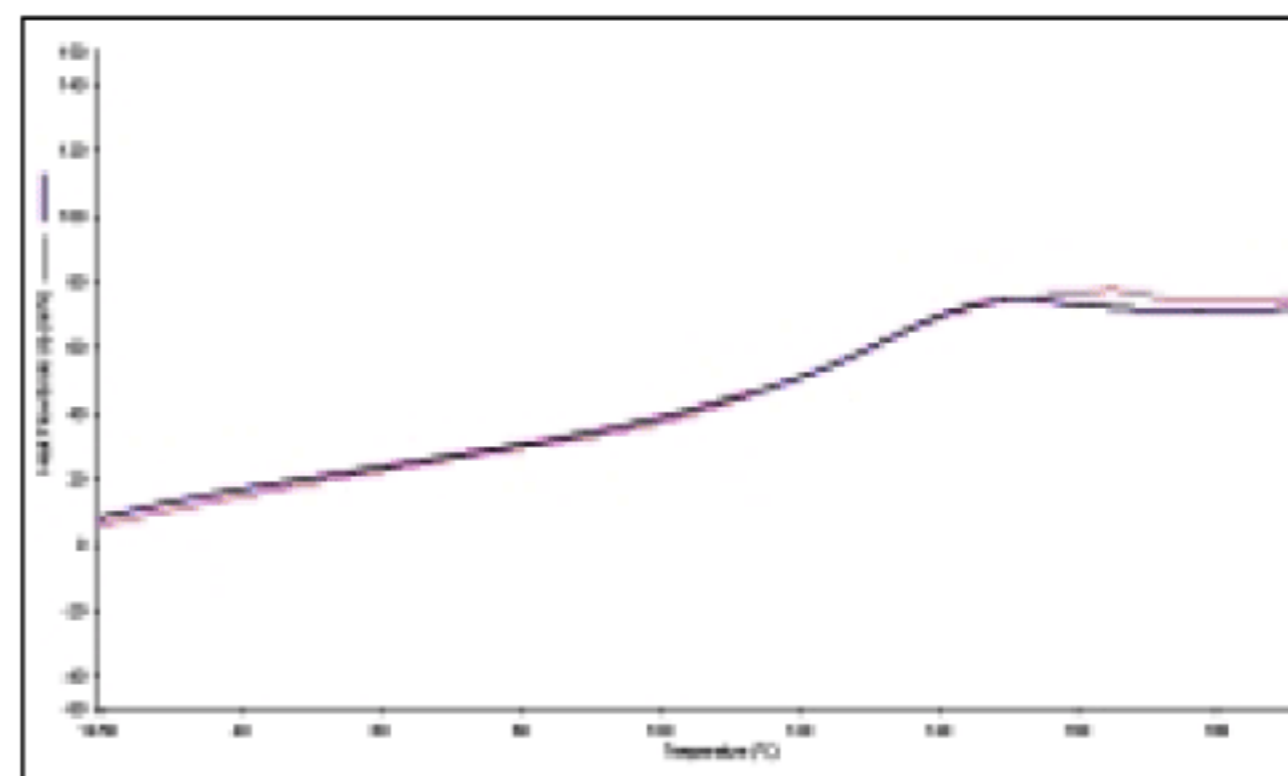
Glass transition in a highly-filled epoxy analyzed by HyperDSC.

HyperDSC significantly increases
# sensitivity and throughput

HyperDSC in pharmaceuticals

HyperDSC simplifies identification of the glass transition in amorphous materials

There has been great interest over the years in the study of the Glass Transition (Tg) of amorphous lactose. Lactose is a very important excipient for pharmaceuticals and is used widely as a diluent in the formulation of tablets. A spray-dried lactose that approached 100% amorphous content as determined by solution calorimetry was analyzed with the Diamond DSC. Data were collected with conventional scanning rates of 20 and 100 °C/min, and the HyperDSC data are collected at the scanning rates of 250, 400, and 500 °C/min. All the scans were performed on the same analyzer. The Tg of lactose is normally seen in the temperature range of 100-120 °C and is difficult to identify using conventional DSC scanning rates. HyperDSC increases the sensitivity and shows the transition clearly. The Tg found with HyperDSC was in the temperature range of 80-100 °C. This lower Tg temperature is believed to be caused by the plastization of the lactose by water, which is not lost during the fast scan. After the glass transition, an exotherm associated with re-crystallization is observed, and this is followed by two melting

events. The two peaks are associated with the two forms of lactose that re-crystallize from the post Tg material. The first peak is the melting of anhydrous α lactose, and the second peak is associated with β lactose.

Note: Acknowledgement to Paul Gabbott, Paul Clarke, Tim Mann, Paul Royall and Sukhraj Shergill for their initiative and work in this field of application.
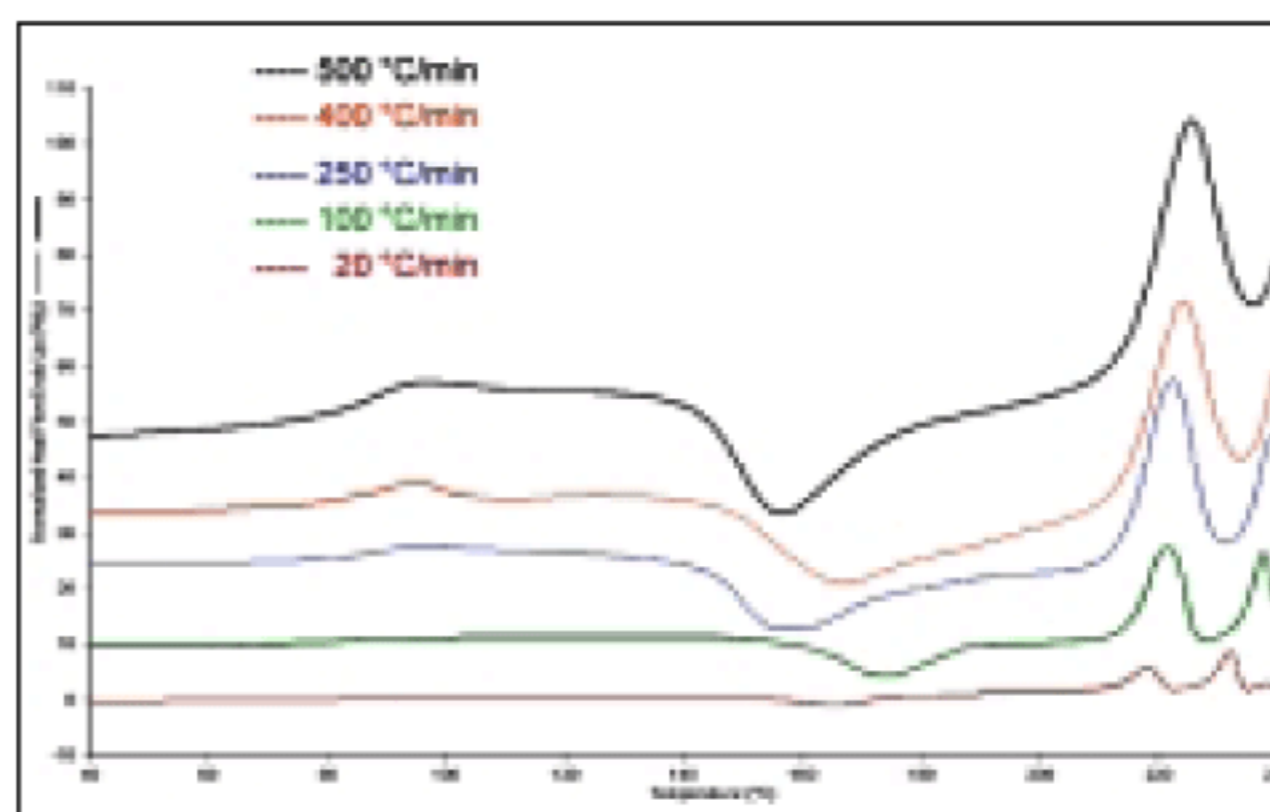

Analysis of amorphous lactose at HyperDSC and conventional DSC scanning rates.

HyperDSC in pharmaceuticals

HyperDSC as a screening tool

An unfounded concern using HyperDSC is the loss of resolution. This example shows the run of Dotriacontane with 250 °C/min and the second derivative, a very valuable tool for the use of HyperDSC — it clearly shows several transitions which are not resolved in the high scanning rate heat flow curve. The information can be generated in less than two minutes and helps in the selection of samples which may provide additional information at lower scan rates.
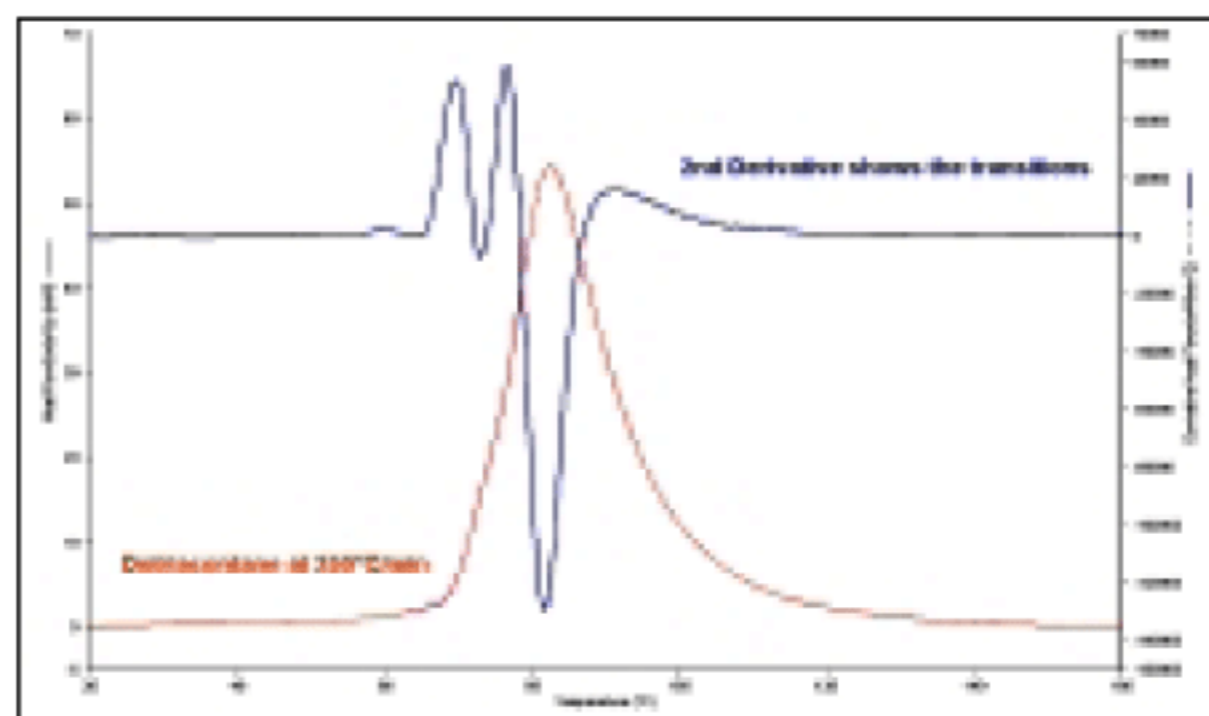
Heat flow of Dotriacontane, scanned with 250 °C/min.

StepScan DSC

StepScan DSC is a modulated temperature DSC technique that operates in conjunction with power-compensation DSC. The approach applies a series of short interval-heating and isothermal-hold steps to cover the temperature range of interest. This approach requires a DSC with very fast responsiveness to achieve short-interval linear heating and isothermal steps. The use of the ultra-low mass furnaces (1 g) with the power-compensation DSC ensures the fastest response time of any DSC instrument. StepScan DSC offers many advantages over conventional DSC and other MTDSC heat-flux DSC approaches:

- Straightforward — pure linear-heating ramps and isothermal steps

- No sine waves and possibilities of distortions and other experimental artifacts

- No mathematical deconvolution (Fourier transforms) required with associated complexities

- Highly quantitative

- Eliminates need to perform heat-cool-reheat experiments

- Greatly helps in data interpretation since reversible and irreversible effects are separated out

- Provides clearer identification of the glass transition event (Tg)

- Yields more accurate heat-capacity results since Cp measurements are generated over short-interval temperature segments

StepScan: A Pure Approach to MTDSC Applications

With the StepScan DSC approach, two signals are obtained. Thermodynamic Cp signal represents the reversible aspects of the material, while the Iso K signal reflects the irreversible nature of the sample during heating. The following basic equation mathematically describes the StepScan DSC approach:

Heat Flow = Cp(dT/dt) + f(T,t)

In this equation, Cp is the sample's heat capacity, dT/dt is the applied heating rate and f(T,t) is the kinetic response. The first Cp term represents the reversible aspects of the sample and the power-compensation DSC applies a purely linear heating ramp for the best results rather than a sine wave where the first item is continuously varying. When the sample is held under isothermal conditions, the heating rate becomes 0 and the sample's heat flow is purely described by the kinetic term.

Because the sample is either linearly heated or held isothermally (true isothermal), the StepScan DSC approach is straightforward and provides the purest and fastest approach to MTDSC measurements.
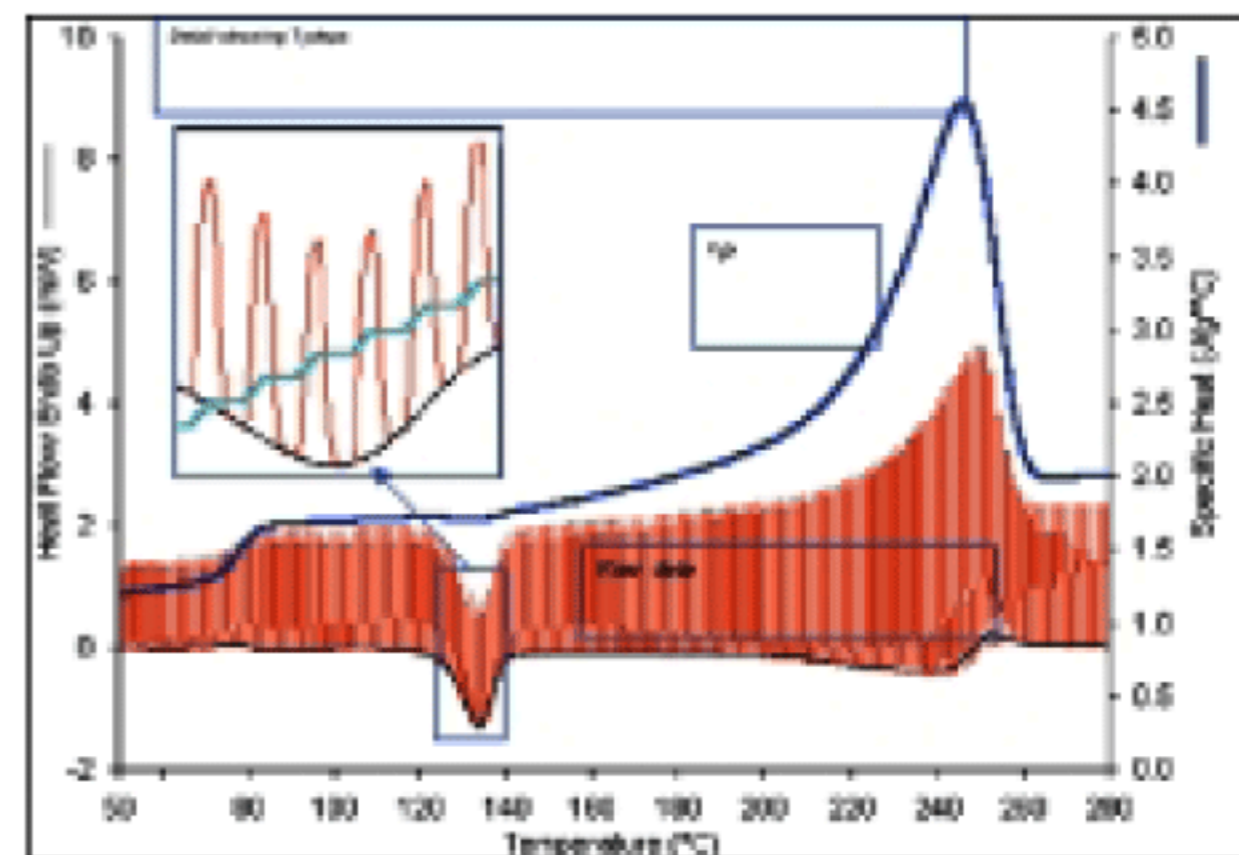
StepScan DSC Results for PET.

Pyris software improves lab productivity

Pyris software is the benchmark application for thermal analysis. In combination with the Microsoft Windows® operating system, it provides new functionality essential for productive operation of all thermal analysis techniques. Whether in a totally automated research laboratory, an automated QA/QC lab, or a stand-alone instrument, you can be sure that the proven Pyris software meets your operating requirements.

Report Manager

The Pyris Report Manager gives you the capability of exporting a Pyris data file to a document in Microsoft® Word or HTML (Hypertext Markup Language) format. The software provides user control of the report and of the content of the report. This report template can be re-used to conveniently facilitate the creation of new reports.

Calibration Wizard

The integrated Calibration Wizard offers two levels of calibration control. E-Z Cal, based on predefined conditions, allows fast calibration with minimal user interaction. If greater flexibility or accuracy is required, Advanced Cal provides the ability to calibrate using multiple temperature and heat flow standards, enabling the calibration to be customized for the temperature range of interest. Advanced Cal provides an algorithm which allows the use of a certified Sapphire standard in accordance with ASTM to calibrate heat flow. In addition to offering two levels of control, the Calibration Wizard utilizes Autotune for automatic baseline optimization.

Valet

The convenient Valet feature allows creation of pre-set start-up and shut-down events. For example, valet can be used to automatically condition and equilibrate the Diamond DSC prior to running experiments, or it can be used to automatically shut down the system after completion of runs.

Pyris Enhanced Security for regulatory compliance

Pyris Enhanced Security, an add-on to Pyris software, helps users in both research and quality control to comply with stringent data security requirements, including the 21 CFR Part 11 mandates of the U.S. Food and Drug Administration.

With Pyris Enhanced Security, users in regulated industries will be confident in their ability to provide the whole story about the generation of data from their thermal analyzers. It provides all of the required 21 CFR Part 11 technical compliance features to ensure that data integrity is always maintained:

• User Level Management & Security

• File Protection

• Audit Trails

• Electronic Signatures
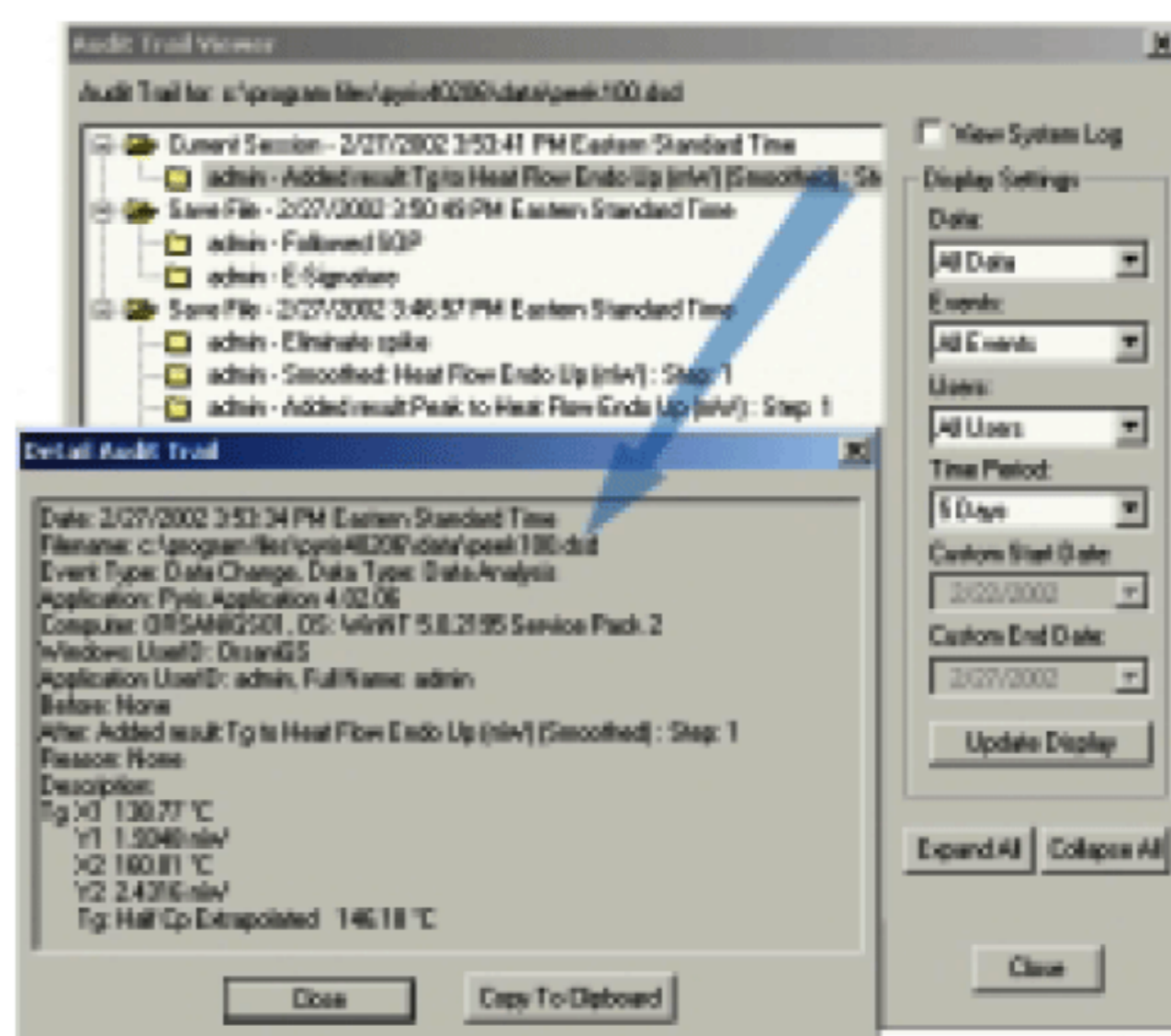
Audit Trail records all significant events pertaining to data and system changes which are displayed in a Viewer.

accessories provide limitless

flexibility

The Diamond DSC accommodates a wide range of accessories including an Autosampler, automatic gas control and switching accessories, several cooling devices and the widest variety of sample pans.

Autosampler

The Diamond DSC Autosampler allows automated DSC testing of multiple samples without operator intervention. Whether running many samples using "traditional" scanning rates, or very fast HyperDSC methods on many different samples, the Diamond DSC Autosampler finds use in a wide range of laboratory settings.

The Diamond DSC Autosampler uses a pneumatic sample arm with refined electronics for precise position control. A unique carousel holds multiple sample capsules and is designed to allow convenient addition and removal of sample materials before, during or after measurement. An integral enclosure isolates the Autosampler from mechanical interference and contaminants. This third-generation Autosampler design, used in laboratories throughout the world, provides the reliability you expect when performing unattended operation.

Convenience and ease-of-use are also important considerations when automating thermal analysis experiments. The Diamond DSC Autosampler utilizes the benchmark Pyris™ software for experiment set up, control of sample exchanges, and programmed data analysis at the conclusion of an experiment. The "PlayList", a standard feature of Pyris software, is used to operate the Diamond DSC Autosampler. With this software, a list of samples can be automatically loaded/unloaded, run and the results analyzed and printed. Complete sample lists and their methods can be saved and conveniently recalled and modified later for re-use. New samples or emergency "rush" samples can be added at any time, offering the ultimate in flexibility.

The Diamond DSC Autosampler and Pyris software do more than increase sample throughput and run your Diamond DSC continuously. They make running thermal analysis faster and easier for operators at all levels of experience, while providing accurate and reproducible results.

Enhanced cooling capabilities

A variety of cooling options are available for the Diamond DSC, including liquid circulation, a refrigerated cooling system and an automated liquid nitrogen cooling accessory, allowing experiments that require subambient analysis temperatures or fast controlled cooling steps.

Sample pans

A wide assortment of sample pans are available from PerkinElmer to address the range of thermal analysis applications. You can choose from different pan materials (aluminum, gold, platinum, graphite, aluminum oxide, copper, silver, stainless steel), capacities (a few µL to 50µL) and pressure ratings (ambient to 150 atmosphere) to optimize your analysis.
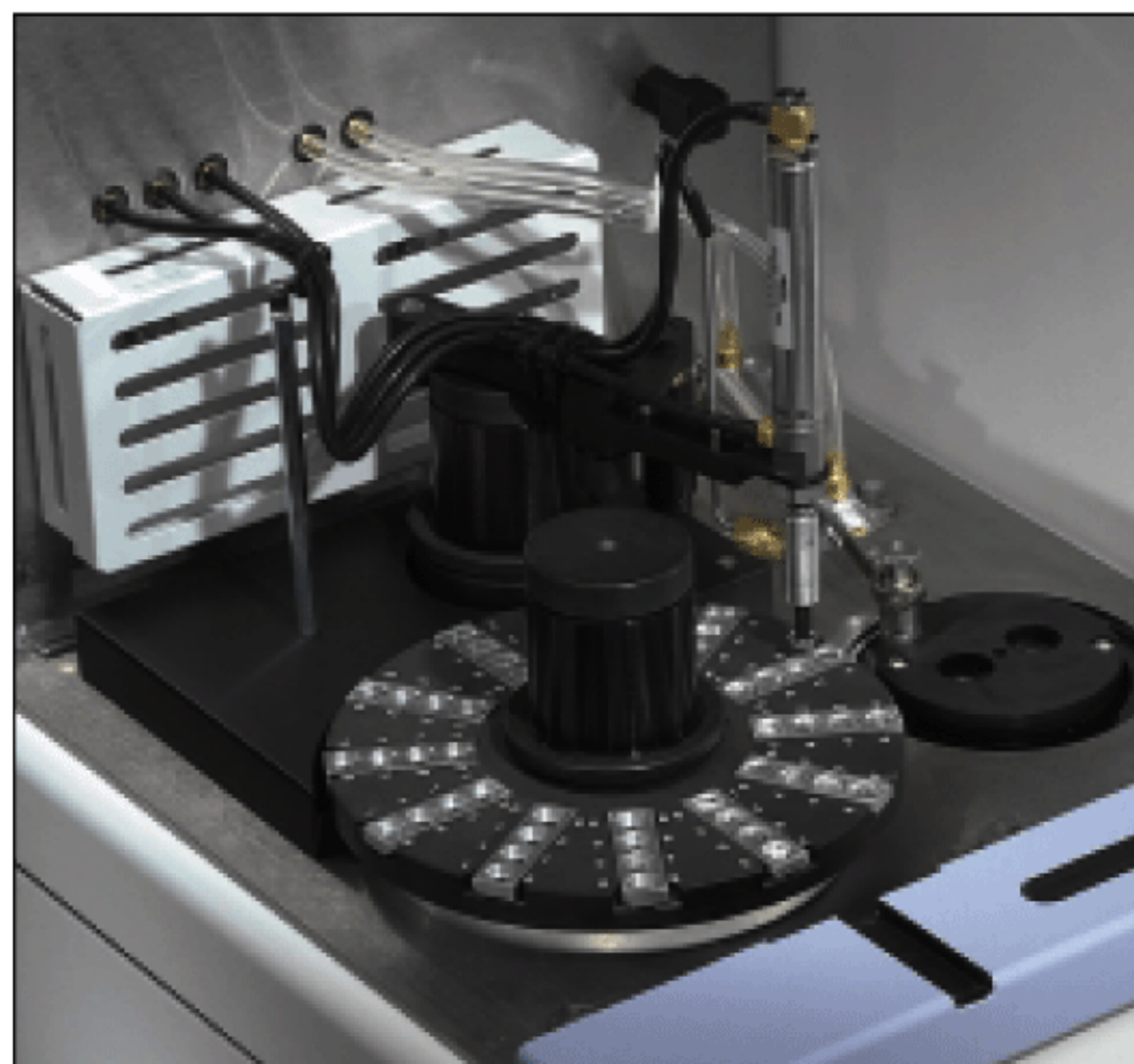
44-position Diamond DSC Autosampler.

unique capabilities ensure
clear identification (continued)

True isothermal measurements

Power-compensation DSC improves analysis of isothermal crystallization processes and curing reactions. By comparison, a conventional DSC performs these measurements only with limitations, since the temperature is not kept constant. During crystallization, the temperature can easily change, which influences the data obtained and results of any kinetic studies. The Diamond DSC maintains the temperature difference at zero and thereby ensures high quality results.

Cover design maximizes system baseline stability

The Diamond DSC system includes a unique cover design that improves both ease-of-use and performance. An innovative rotating sample head cover effortlessly slides in and out of position. The cover is in constant contact with the furnace block, reducing equilibrium times and ensuring baseline stability.

Rotating sample head cover.

High calorimetric accuracy

The power-compensation principle determines the most accurate calorimetric data of all available DSC's. The PerkinElmer power-compensation DSC is the benchmark for direct quantitative measurement of specific heat in a wide variety of materials. With a conventional DSC, the results vary with the sample preparation and instrument operating conditions such as contact with the sensor.

With the Diamond DSC you minimize the risk of wrong or inaccurate determination – the influence of inconsistent sample preparation on the results is negligible. You know the generated data is correct and feel secure using it as a basis for your decisions.

High pressure DSC

Designed for convenient operation with a standard DSC, the optional DSC High Pressure Cell extends the capabilities of the unique power-compensation Diamond DSC design to elevated pressure measurements. While most DSC experiments are performed at atmospheric pressure, there are some applications that must be carried out at elevated pressure and the DSC High Pressure Cell allows for such applications.

Typical applications include:
• Oxidation testing of oils, fats, foods and plastics
• Curing and crosslinking reactions
• Suppression of volatiles vaporization
• Analysis of pressure dependent chemical reactions

HyperDSC - a breakthrough method

The PerkinElmer-exclusive HyperDSC method delivers unparalleled sensitivity and new insights into materials processes that cannot be obtained with existing DSC methods. By providing sample information within seconds, HyperDSC significantly increases throughput in the polymer and pharmaceutical industries.

The HyperDSC method is only possible with the power-compensation Diamond DSC because it allows measurements with controlled scanning rates from 0.01 °C to 500 °C/minute. Unlike other DSC methods, HyperDSC offers true materials analysis while either eliminating or reducing changes such as re-crystallization, melting, and decomposition, which may be induced when utilizing slow scanning.

Fastest heating and cooling rates

Unlike conventional DSC systems utilizing large furnaces that are 30 grams or more, the Diamond DSC system features two small 1-gram furnaces. This design enables the system to achieve the fastest heating and cooling rates in the industry – up to 500 °C/min. Faster heating and cooling rates translate into higher sensitivity and throughput. Small furnaces also deliver better temperature control, providing linearity for better results.

Heating and cooling flexibility allows the Diamond DSC to more accurately mimic production conditions, facilitating process and property optimization, saving time and reducing production problems.

Better resolution means you see more

Resolution refers to the ability of the DSC to separate or resolve closely occurring thermal transitions. In thermal analysis, small differences can have a huge impact on your success. Ensuring access to all the information is critical to researchers in both the pharmaceutical and polymer industries. In pharmaceutical labs, researchers need to ensure that there are no unknown transitions, which could not only cause undesirable outcomes, but may potentially limit patent protection. In polymer work, a small shoulder peak that goes undetected can produce unwanted physical properties, resulting in product failure. Due to the low-mass furnace design of the Diamond DSC, the signal returns to baseline faster, providing outstanding resolution. The Diamond DSC delivers industry-leading performance to ensure all transitions are identified.

Higher sensitivity

Power-compensation yields higher sensitivity, which is the ability of a DSC to detect a weak transition from the background noise. You can even detect phase transitions of low energy, such as in biological or liquid crystalline samples. The Diamond DSC has the capability to analyze small amounts of material if sample availability is limited. Because the signal is directly proportional to the heating rate, the additional capability of fast scanning (HyperDSC) increases sensitivity.

Size matters

Heat-flux systems use one large furnace that is between 30 and 200 grams while the Diamond DSC system features two 1-gram furnaces. This size difference results in faster heating and cooling rates and allows direct heat flow determination, eliminating the need for complex mathematics.

Conventional DSC furnace

Diamond DSC furnace
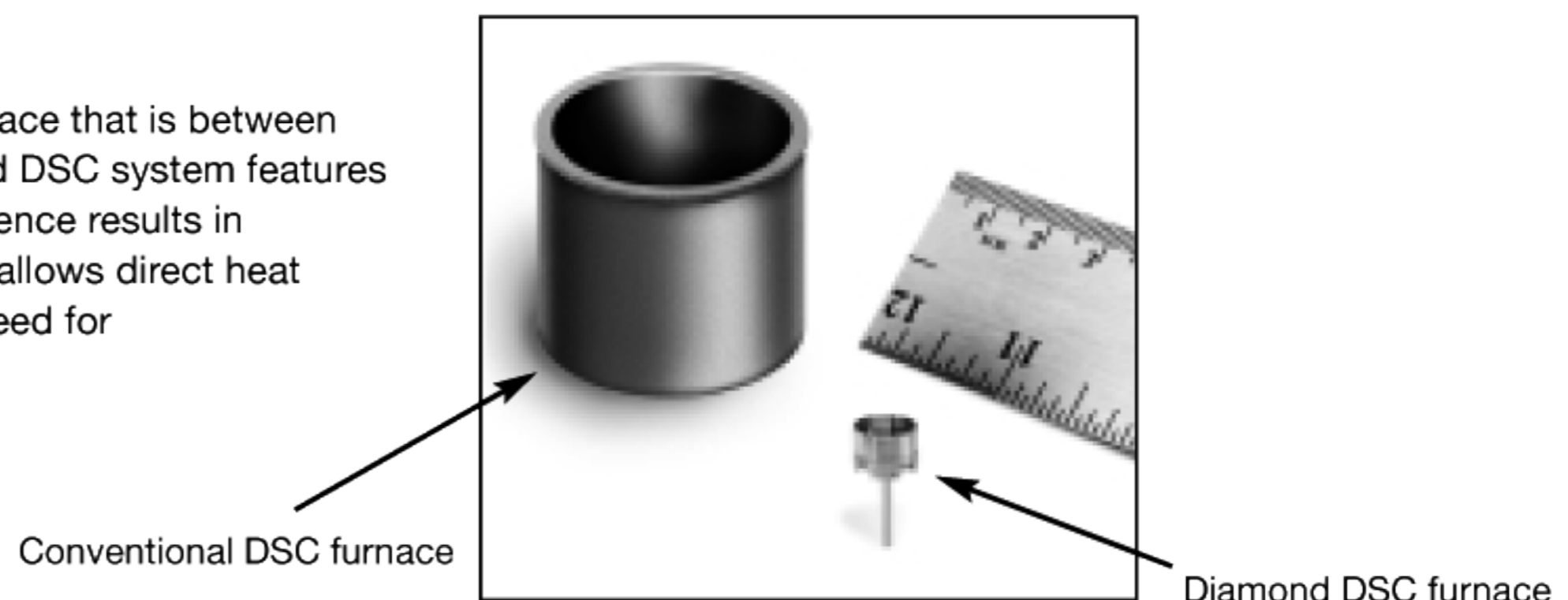
unique capabilities ensure
clear identification

When you can't afford to miss something in your DSC analyses, you need the Diamond DSC (Differential Scanning Calorimeter). Missing important information can make a big difference to your organization's success. It can lead to product failures, additional manufacturing expense and wasted time. With the PerkinElmer-exclusive power-compensation technique, you will be confident to achieve fast, accurate, reproducible results.

PerkinElmer, the **leader** in high sensitivity thermal analysis instrumentation, pioneered the proven power-compensation approach to DSC nearly 40 years ago. Two small, low-mass furnaces heat and cool rapidly, providing better resolution and higher sensitivity, enabling detection of transitions that are missed in conventional DSC systems. And, since the design measures heat flow (energy), you get direct results instead of having to derive them from a temperature difference (delta T) calculation as in other DSC's.
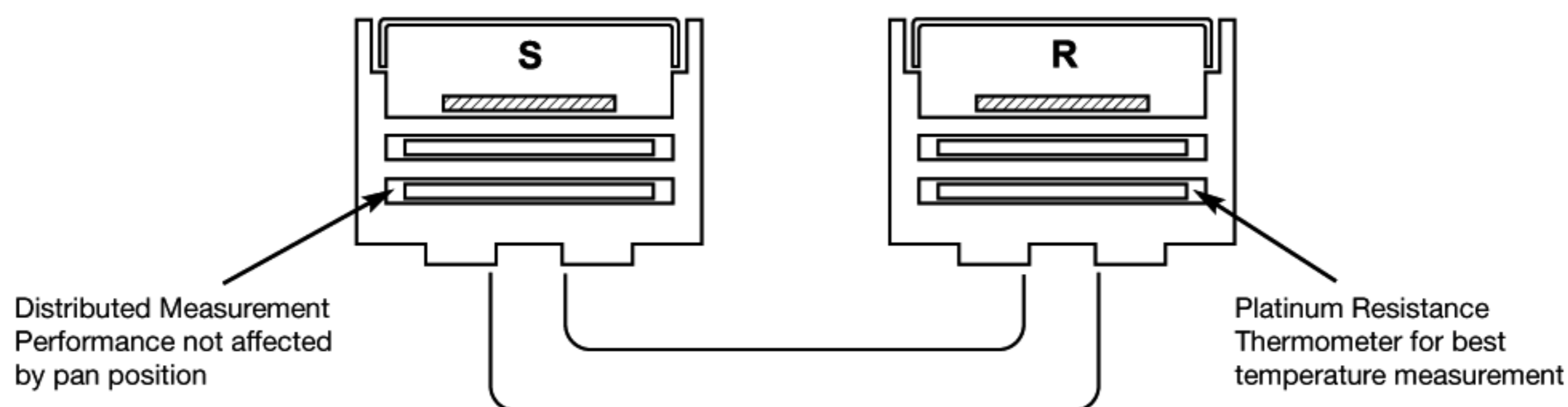
What does DSC measure?

DSC measures the amount of energy (heat) absorbed or released by a sample as it is heated, cooled or held at constant temperature. DSC also performs precise temperature measurements.

QUICK GLANCE

- HyperDSC™ for unmatched sensitivity
- Low-mass furnaces for reduced analysis time and high throughput
- Superior signal resolution so you don't miss transitions
- Closed-loop operation for true isothermal measurements
- Unsurpassed calorimetry for accurate specific heat analysis

The power-compensation principle

With power-compensation DSC, the sample and the reference material are placed in independent furnaces. When the temperature rises or falls in the sample material, power (energy) is applied to or removed from the calorimeter to compensate for the sample energy. As a result, the system is maintained at a "thermal null" state at all times. The amount of power required to maintain system equilibrium is directly proportional to the energy changes occurring in the sample. No complex heat-flux equations are necessary with a power-compensation DSC because the system directly measures energy flow to and from the sample.

S   R

Distributed Measurement Performance not affected by pan position

Platinum Resistance Thermometer for best temperature measurement

thermal analysis solutions

for material property analysis

PerkinElmer is the **leader** in high sensitivity thermal analysis instrumentation, providing you the confidence to achieve fast, accurate, reproducible results.

Differential Scanning Calorimetry (DSC)

DSC measures the amount of energy absorbed or released by a sample as it is heated, cooled or held at a constant temperature. This technique is used for polymer and pharmaceutical applications. PerkinElmer offers the best of both worlds – the Diamond DSC for highest resolution and sensitivity, and the Sapphire DSC and Pyris 6 DSC for ease-of-use and robustness.

Thermogravimetric Analysis (TGA)

TGA measures the change in weight of a sample as it is heated, cooled or held at a constant temperature. The PerkinElmer Pyris TGA instruments provide robustness and reliability for quality control and the answers researchers need to solve even the toughest problems.

Thermogravimetric/Differential Thermal Analysis (TG/DTA)

TG/DTA is a simultaneous technique that determines the weight change of a sample (TG) and measures the change in temperature between a sample and the reference as a function of temperature and/or time (DTA). The Diamond TG/DTA combines the high flexibility of the differential analysis feature (DTA, DSC) with the proven capabilities of the thermogravimetric (TG) measurement technology to provide highly reliable characterization information.

Dynamic Mechanical Analysis (DMA)

DMA measures changes in mechanical behavior, such as modulus and dampening, as a function of temperature, time, frequency, stress or a combination of these parameters. The Diamond DMA, with its more than 20 patents, provides state-of-the-art measurement to a wide variety of materials and applications.

Thermomechanical Analysis (TMA)

TMA determines dimensional changes in materials as a function of temperature or time. It is used to measure changes in length, width, thickness and linear expansion of materials. The Diamond TMA is an advanced thermomechanical analyzer which allows for samples to be analyzed using dynamic force, providing basic DMA data besides many TMA features, continuing the tradition of high sensitivity TMA.

OneSource℠ Laboratory Services — comprehensive service and support for today's results-driven lab

With over 60 years of experience, and as a world leader in analytical instrumentation, PerkinElmer is the right partner for your application needs. In concert with global distribution of instruments, turnkey systems, and consumables, we provide factory-trained global service and support. PerkinElmer's OneSource Laboratory Services provides you with a comprehensive worldwide service offering that allows you to take care of business and set your sights on what matters most — results. With over 1,000 professionals serving more than 125 countries worldwide, PerkinElmer is your single source for instrument care and repair, validation services, software and hardware upgrades, education, and more.

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precisely.