Thermal Analysis Premium

DSC 3+

STAR^e System

Innovative Technology

Versatile Modularity

Swiss Quality

Differential Scanning Calorimetry

for Unmatched Performance

METTLER TOLEDO

Unmatched DSC Performance
Tailored Exactly to Your Needs

Differential scanning calorimetry (DSC) is the most frequently used thermal analysis technique. DSC measures enthalpy changes in samples due to changes in their physical and chemical properties as a function of temperature or time.

Features and benefits of the METTLER TOLEDO DSC 3+:

- **Amazing sensitivity** – for the measurement of weak effects
- **Outstanding resolution** – allows measurement of rapid changes and close-lying effects
- **Robust endurance-tested sample robot** – operates efficiently and reliably around the clock
- **Small and large sample volumes** – for microgram or inhomogeneous samples
- **Modular concept** – tailor-made solutions for current and future needs
- **Flexible calibration and adjustment** – guarantees accurate results under all conditions
- **Wide temperature range** – from –150 to 700 °C in one measurement

The DSC utilizes an innovative DSC sensor with 120 thermocouples which guarantees unbeatable sensitivity and outstanding resolution.

Major Breakthrough in DSC Sensor Technology

Unsurpassed Sensitivity and Excellent Resolution

Don't make any compromises concerning the sensor, the heart of your DSC. The METTLER TOLEDO MultiSTAR® sensors successfully combine a number of important characteristics that are unattainable with conventional sensors and that until now have been impossible to achieve. These included high sensitivity, excellent temperature resolution, a perfectly flat baseline and robustness.

Sensitivity
A quantum jump in sensor technology enables us to offer the highest sensitivity sensors available in DSC instrumentation and allows you to detect the weakest thermal effects. The signal-to-noise ratio, an important instrument parameter, is determined by the number of thermocouples and their specific arrangement.

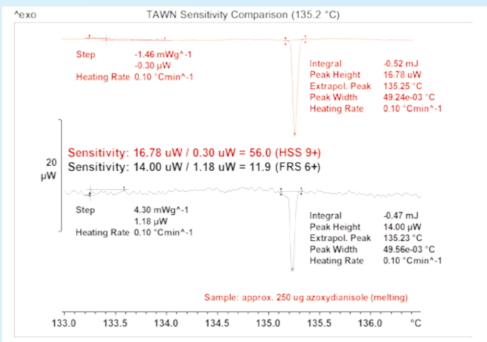
Temperature resolution
The signal time constant determines how well close-lying or overlapping thermal effects are separated from one another. We set unprecedented and unparalleled performance standards due to our high thermal conductivity ceramic sensor material with its low thermal mass.

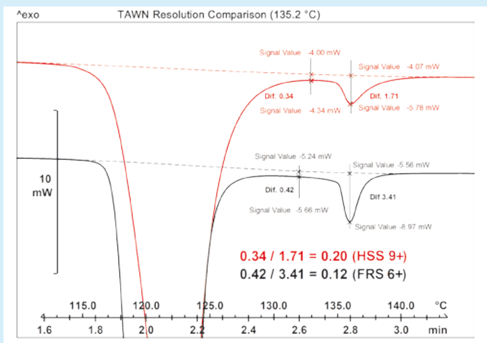
Baseline
Our revolutionary star-shaped arrangement of thermocouples around the sample and reference crucibles completely compensates any possible temperature gradients. This guarantees flat baselines and reproducible measurement results.

FRS 6+ sensor
With the new more robust FRS 6+ sensor, the reproducibility of results has again been increased. The FRS 6+ is clearly the right choice for standard applications, for high heating rates and difficult peak separations.

HSS 9+ sensor
The High Sensitivity HSS 9+ sensor measures very weak thermal effects and microgram sample amounts, even at low heating rates. The HSS 9+ has 120 thermocouples and provides excellent temperature resolution and previously unattained sensitivity.

TAWN test
The benchmark for DSC sensors is the widely used TAWN test. The test confirms the excellent sensitivity and high temperature resolution of the HSS 9+ and FRS 6+ sensors.



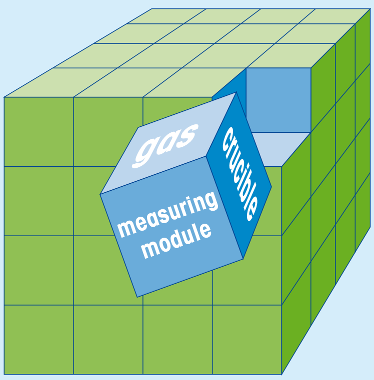


Swiss Quality

DSC 3+ from METTLER TOLEDO
the Right Decision

SmartSens terminal with One Click™ function
The terminal with the One Click™ function is clearly visible even at a distance and provides information on the status of the measurement. The One Click™ function allows you to easily and efficiently start a predefined method.
If the DSC is not installed next to a PC running the STAR® software, you can set up individual sequences directly at the instrument terminal. The adaptable and intuitive touchscreen or SmartSens allows you to switch screens or open the furnace handsfree.

Furnace chamber
The sensor is located in a corrosion-free silver furnace.



FlexCal® adjustment
The STAR® software stores a complete adjustment data record in the database for every crucible, gas and module combination. The module always uses the correct adjustment parameters, even if measurements are performed with different crucibles or if the gas is switched during the measurement.

Ergonomics in Perfection
We Care about You

Ergonomic design
If you insert samples manually, you can rest your hand on an ergonomically shaped support surface.

DSC

TGA

TMA

DMA

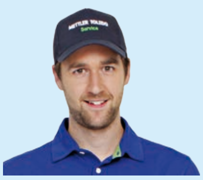
Complete thermal analysis system
A complete thermal analysis system comprises four different techniques. Each technique characterizes the sample in a particular way.

The combination of all the results simplifies interpretation. DSC measures the heat flow, TGA the weight curve, TMA the length change, and DMA the modulus.

The powerful STAR® software allows the user to control all the connected modules and provides unlimited evaluation possibilities.

Support and repair
Support and diagnosis in case of technical issues. Carrying out repairs at a customer's site or at one of our service centers.

Quality assurance and certification
Qualification, documentation, calibration with certificate.



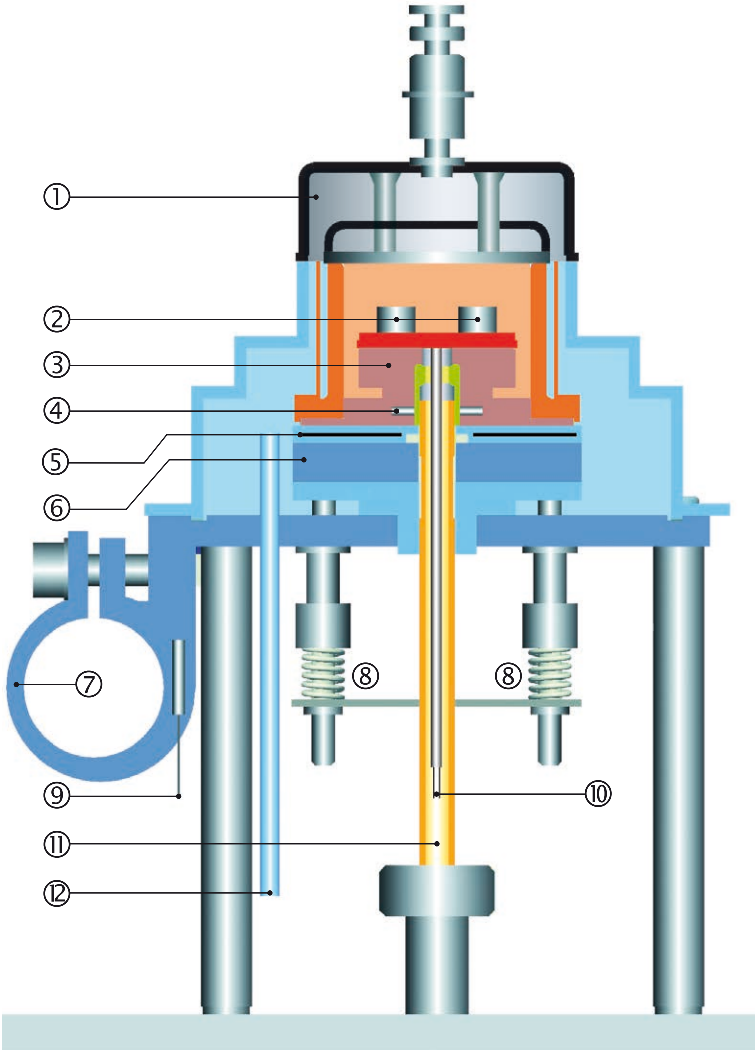
Performance services and preventive maintenance
Professional installation (IQ, OQ) and ensuring optimum performance during the life-time of the instrument (PQ and preventive maintenance).

Training and applications support
Professional applications support, basic and customized training courses, comprehensive applications literature.

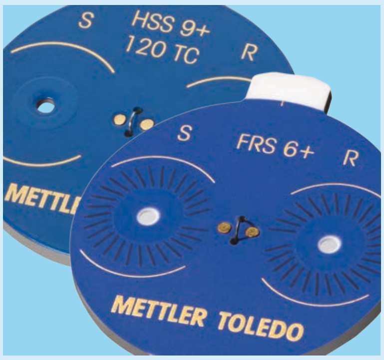
Unsurpassed Performance
over the Whole Temperature Range

Measurement principles
Differential scanning calorimetry (DSC) measures the difference between the heat flows from the sample and reference sides of a sensor as a function of temperature or time.

Physics of DSC
Differences in heat flow arise when a sample absorbs or releases heat due to thermal effects such as melting, crystallization, chemical reactions, polymorphic transitions, vaporization and many other processes. Specific heat capacities and changes in heat capacity, for example during a glass transition, can also be determined from the difference in heat flow.



Key
1. Furnace lid
2. Crucibles on the DSC sensor
3. Silver furnace
4. PT100 of furnace
5. Flat heater between two insulating disks
6. Thermal resistance for cooler
7. Cooling flange
8. Compression spring construction
9. Cooling flange PT100
10. DSC raw signal for amplifier
11. Purge gas inlet
12. Dry gas inlet



Robust sensor
The ceramic-coated surface protects the sensors against chemical attack and contamination. This ensures long lifetime and constant performance characteristics throughout the entire temperature range.

Reliable Automation
Saves Time

The sample robot is extremely robust and operates reliably 24 hours a day throughout the whole year.

Automatic and efficient
All DSC models can be automated. The sample robot can process up to 34 samples even if every sample requires a different method and a different crucible.

Features and benefits:

- **Up to 34 sample positions** – dramatically increases efficiency
- **Simple and rugged design** – guarantees reliable results
- **Unique "wasp" lid piercing accessory** – hermetically sealed crucibles are automatically opened prior to measurement
- **Universal gripper** – can handle all types of METTLER TOLEDO crucibles

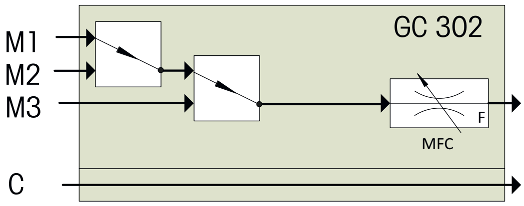
No sample reaction before measurement
The sample robot can remove the protective crucible lid from the crucible or can pierce the lid of hermetically sealed aluminum crucibles immediately before measurement. This unique feature prevents the sample from taking up or losing moisture between weighing-in and measurement. It also protects oxygen-sensitive samples from oxidation.

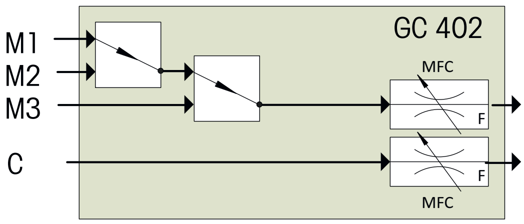
Modularity and Upgradeability
Unlimited Possibilities

Automatic furnace lid
The automatic furnace lid opens and closes the furnace chamber at a keystroke or when actuated by the SmartSens infrared sensors. Manual removal and replacement of the furnace lid is no longer necessary. The measurement cell is effectively isolated from the environment thanks to its optimized design with three superimposed silver lids and its heat shield.

Air cooling	RT to 500 °C / 700 °C
Cryostat cooling	−50 to 450 °C / 700 °C
IntraCoolers (several)	−35 to 450 °C / 700 °C −85 to 450 °C / 700 °C −100 to 450 °C / 550 °C
Liquid nitrogen cooling	−150 to 500 °C / 700 °C

Temperature range and cooling options
You can adapt the system to your requirements depending on the temperature range in which you want to measure.
The IntraCooler is a sealed system requiring only electrical power. It is therefore advantageous in locations where liquid nitrogen is undesirable or not available. Liquid nitrogen cooling offers greater flexibility because it allows you to measure over the entire temperature range.





Defined furnace atmosphere, programmable gas flow and gas switching
The furnace chamber can be purged with a defined gas flow. The software-controlled mass flow gas controller measures and regulates the gas flow between 0 and 200 mL/min and can automatically switch up to 4 gases. Regulate and switch gases such as air, nitrogen, oxygen, argon, CO_2, CO and inert hydrogen and expand your experimental possibilities.

Option → required option	FRS 6+	HSS 9+	Automatic furnace lid	SmartSens terminal	Peripheral control	Switched line socket	GC 402	Air cooling	Cryostat	Intra Cooler	Liquid nitrogen
DSC 3+ (500 °C)	•	•	optional	optional				•	•	•	•
DSC 3+ (700 °C)	•	•	optional	optional				•	•	•	•
Sample changer (34)			essential	essential							
Automatic furnace lid				essential							
Gas controller (GC 302)				recommended			optional				
Cryostat/IntraCooler						optional (recommended)					
Liquid nitrogen cooling					essential						

• = selectable

Innovative Accessories
Increase Measurement Power

DSC microscopy
DSC curves often exhibit effects that cannot immediately be explained. In such cases, it is often helpful to visualize the changes in the sample directly by means of microscopy.

The versatile optical accessory can be used with any METTLER TOLEDO DSC. It consists of an optical system, a CCD camera, and image capture and processing software.

DSC photocalorimetry
The photocalorimetry accessory for the DSC allows you to characterize UV curing systems. You can study photo-induced curing reactions and measure the effects of exposure time, UV light intensity and temperature on material properties.

Crucible sealing press

Enormous range of crucibles
We have the right crucible for every application. The crucibles are made of different materials with volumes ranging from 20 to 900 µL and for high pressures. All the different types can be used with the sample robot.

Crucible materials available include:

copper

aluminum

alumina

steel (gold-plated)

gold

platinum

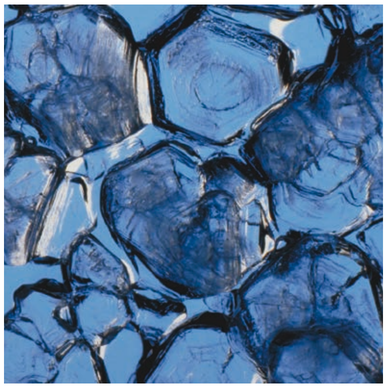
Extremely Wide Application Range

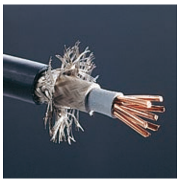
Differential Scanning Calorimetry measures the enthalpies associated with transitions and reactions and the temperatures at which these processes occur. The method is used for the identification and characterization of materials.

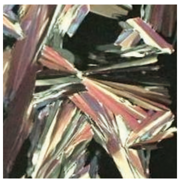
Differential scanning calorimetry (DSC) is fast and very sensitive. Sample preparation is easy and requires only small amounts of material. The technique is ideal for quality control, material development and material research.

DSC is the method of choice to determine thermal quantities, study thermal processes, and characterize or just simply compare materials. It yields valuable information relating to processing and application conditions, quality defects, identification, stability, reactivity, chemical safety and the purity of materials.

The method is used to analyze and study materials such as thermoplastics, thermosets, elastomers, composite materials, adhesives, foodstuffs, pharmaceuticals and chemicals.

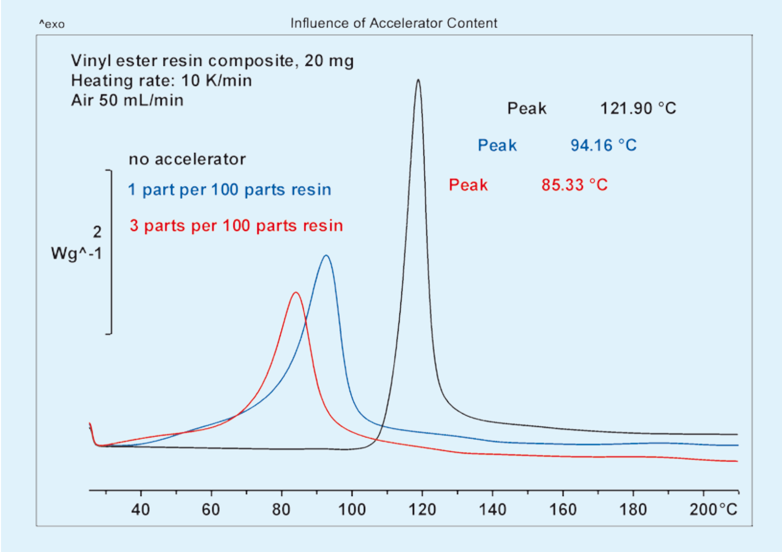






Examples of thermal events and processes that can be determined by DSC

- Melting behavior
- Crystallization and nucleation
- Polymorphism
- Liquid-crystalline transitions
- Phase diagrams and composition
- Glass transitions
- Reactivity
- Reaction kinetics
- Curing
- Stability
- Miscibility
- Effects of plasticizers
- Thermal history
- Heat capacity and heat capacity changes
- Reaction and transition enthalpies
- Purity

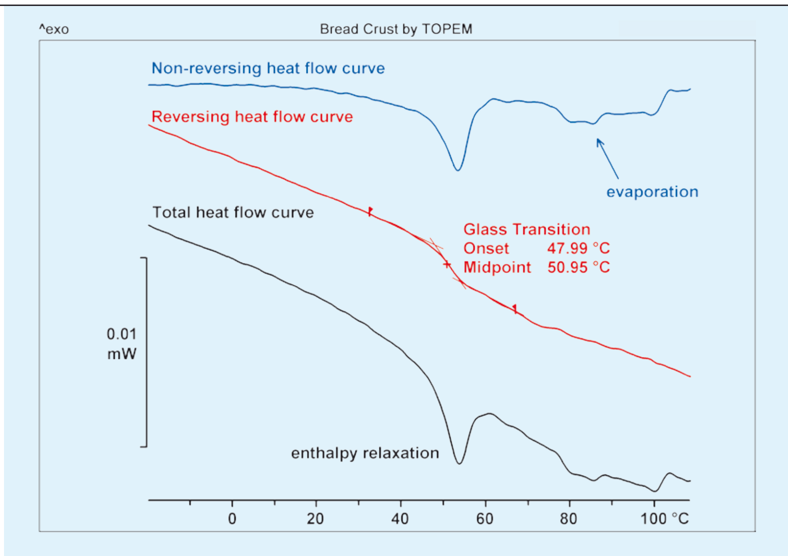


Influence of accelerator on curing

Curing measurements of samples of glass-fiber-reinforced vinyl ester resin composites containing different concentrations of an accelerator can be used to determine the influence of a particular accelerator on the curing process.

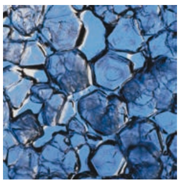
Each DSC curve exhibits an exothermic peak proportional to the heat produced during the exothermic curing reaction. Increased accelerator concentration speeds up the reaction and causes the reaction peak to shift to lower temperatures.

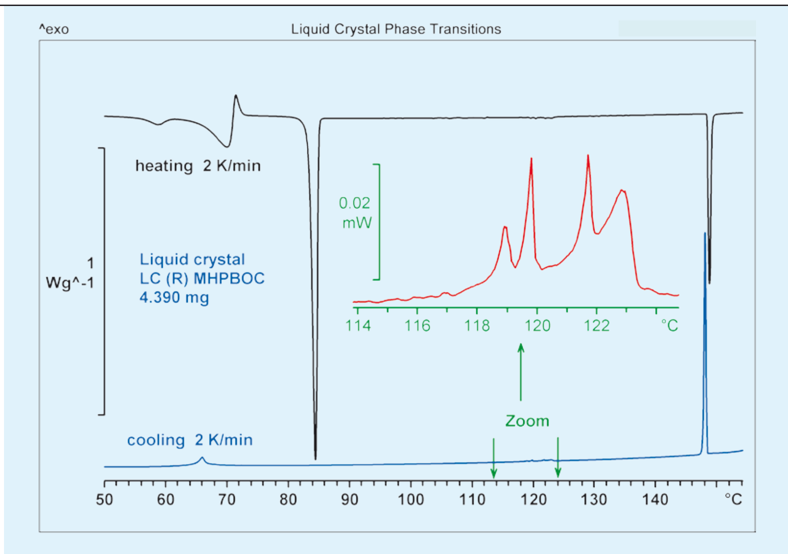
This type of DSC measurement allows production processes to be adapted and optimized by choosing the right accelerator, its concentration, and the curing temperature.



Bread crust

When complex materials are measured, different effects often overlap. The individual effects can be separated using **TOP**EM®, a temperature-modulated DSC technique. This is demonstrated using a sample taken from a bread crust. The total heat flow curve corresponds to the conventional DSC curve and a definite assignment of the measured effects is not possible. In contrast, the reversing heat flow curve clearly shows a glass transition at 51 °C. The non-reversing heat flow curve displays the peak due to enthalpy relaxation and the evaporation of moisture from about 70 °C onward.



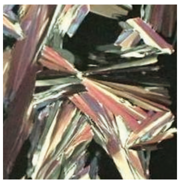


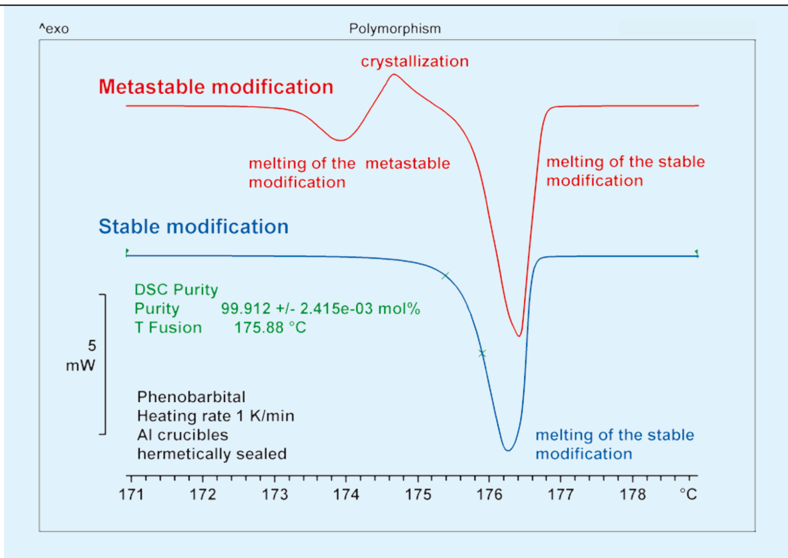
Liquid crystals

Materials consisting of relatively stiff molecules can form liquid-crystal phases. This behavior is demonstrated in the example showing DSC measurements of LC (R) MHPOBC. The substance exhibits several liquid-crystal transitions above the melting temperature at 85 °C.

The transitions that occur between 114 and 124 °C are very weak and are displayed in the zoomed region of the cooling curve.

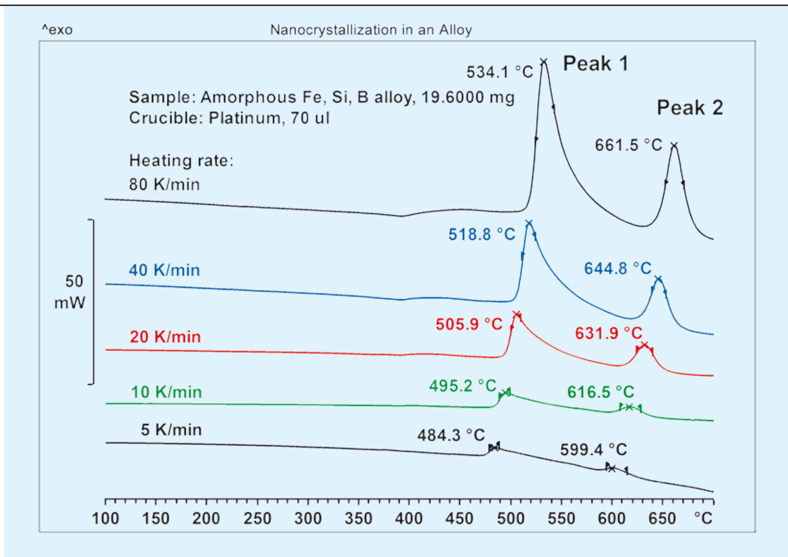
Since liquid-crystal transitions often produce only very small thermal effects, the DSC used to measure such transitions must have high resolution and low noise performance specifications.





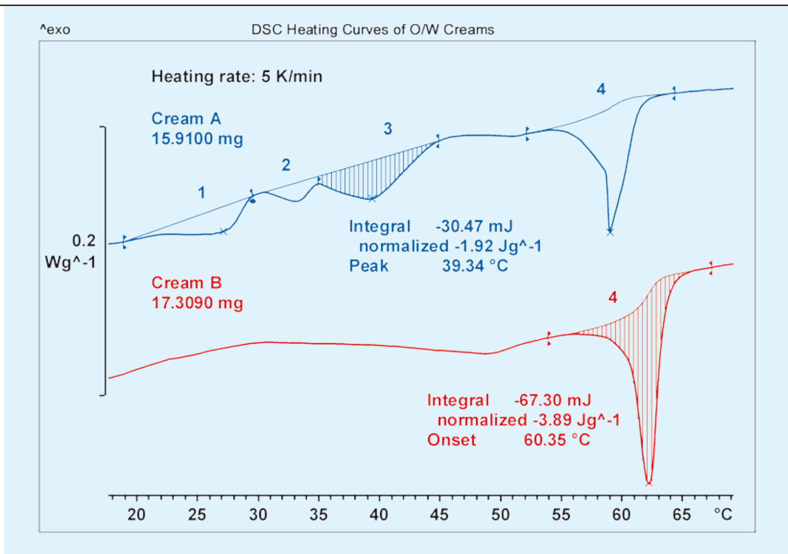
Polymorphism

The analysis of melting behavior is an important method for the quality control of pharmaceutical products. As the blue curve in the example shows, the melting curve of the stable form of phenobarbital can be used to determine the melting point and for purity determination.
DSC is also used to study polymorphic forms. The red curve shows that the metastable form first melts at a lower temperature. The melt then crystallizes to the stable form before this form also melts. Knowledge of the particular crystalline form present is very important for assessing the physical stability of substances.



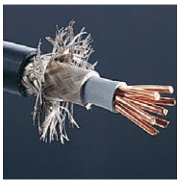
Nanocrystallization

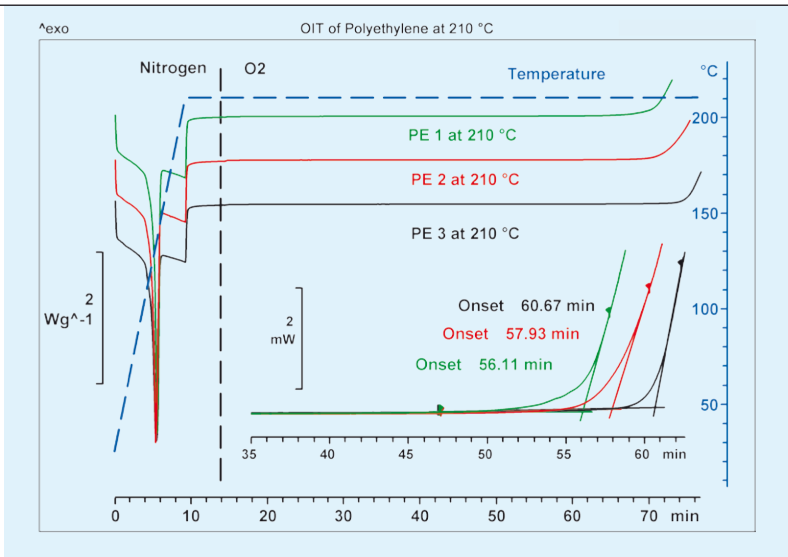
The crystallization behavior of an amorphous metal alloy prepared by rapid cooling in a melt-spinning production process was analyzed.
The diagram displays curves measured at different heating rates. The exothermic peak from 470 °C onward is due to the formation of the iron-silicon nanocrystallites. With increasing heating rates, the maximum of this peak shifts to higher temperatures. The relationship between the crystallization temperature and the heating rate yields information on the activation energy of the crystallization process. The highly asymmetric shape of the peak reveals additional information about the kinetics of crystallization.



O/W cream formulations

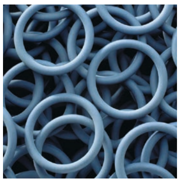
Creams are semi-solid emulsions and consist of mixtures of oil and water. Differences in the content of constituents can affect the consistency and quality of the finished cream formulation.
DSC curves of two different creams exhibit a large melting peak in the range 55 to 65 °C. This peak is due to the presence of glyceryl monostearate, which acts as a thickening agent and stabilizer.
Cream A, however, has three additional peaks in the region 25 to 45 °C, which are the typical melting points of mono-, di- and tri-glycerides. These inactive ingredients form three-dimensional structures of different types and strengths.

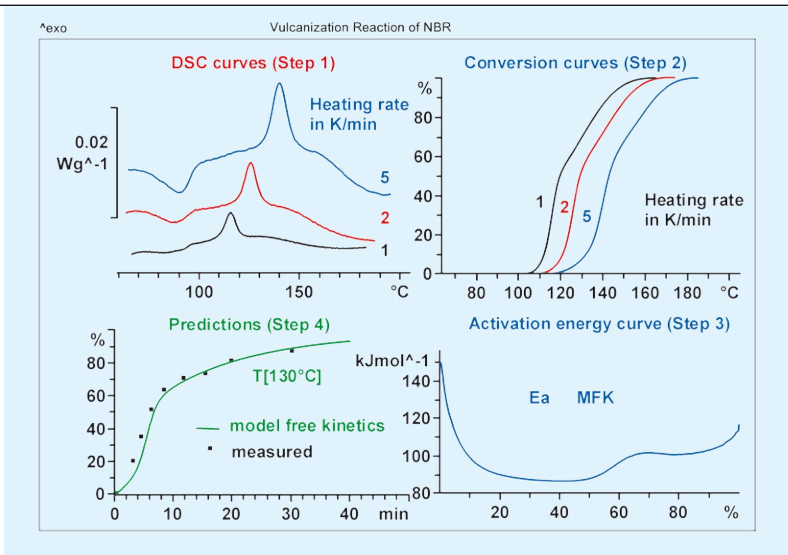




Oxidation stability

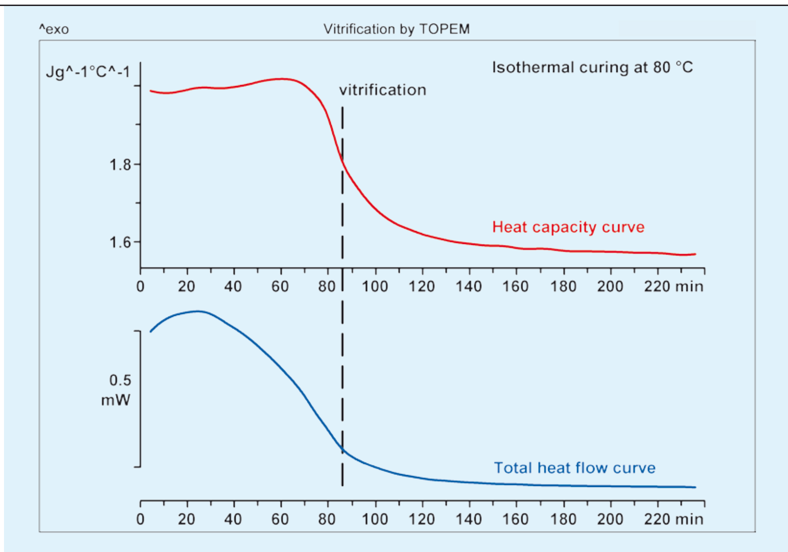
Information about the stability of materials can be obtained from the analysis of decomposition reactions. One widely used standard test method is the measurement of the oxidation induction time, OIT. This is the time up to the onset of oxidation when a sample is held isothermally at a certain temperature in an oxygen atmosphere.
In the example, the OITs of three polyethylene samples stabilized to different extents were measured at 210 °C. The differences in stability toward oxidation can be clearly seen. These measurements also allow thermally, mechanically or chemically stressed material to be distinguished from fresh material.





Vulcanization reaction

DSC results can be used to analyze and predict the kinetics of vulcanization reactions using Model Free Kinetics (MFK) software. In Step 1, three dynamic DSC curves of acrylonitrile-butadiene rubber (NBR) were measured at different heating rates. The results were then used to calculate the conversion curves shown in Step 2. From this data the activation energy curve of the reaction was calculated as a function of conversion as shown in Step 3.
The activation energy curve was then used to predict the isothermal kinetics at 130 °C. (green curve in Step 4). The prediction is in excellent agreement with data measured under the same conditions shown by the solid black squares.



Curing of adhesives

When an adhesive cures isothermally, the material changes from a liquid to a solid as a result of a chemical reaction. An amorphous polymeric glass is formed and the reaction practically stops. This process is known as vitrification and is of great practical importance because a vitrified adhesive is not fully cured and is therefore unstable. The properties of the material gradually change over a long period of time.
The example shows that measurement of the heat capacity during the curing reaction using **TOP**EM® (a temperature-modulated DSC technique) is a simple and reliable method to identify vitrification processes.

DSC 3+ Specifications

Temperature data			
Temperature range	air cooling	RT to 500 °C (200 W)	RT to 700 °C (400 W)
	cryostat cooling	–50 to 450 °C	–50 to 700 °C
	IntraCooler	–100 to 450 °C	–100 to 700 °C
	liquid nitrogen cooling	–150 to 500 °C	–150 to 700 °C
Temperature accuracy [1]		± 0.2 K	
Temperature precision [1]		± 0.02 K	
Furnace temperature resolution		± 0.00006 K	
Heating rate [2] RT to 700 °C		0.02 to 300 K/min	
Cooling rate [2]		0.02 to 50 K/min	
Cooling time	air cooling	8 min (500 to 100 °C)	9 min (700 to 100 °C)
	cryostat cooling	5 min (100 to 0 °C)	
	IntraCooler	5 min (100 to 0 °C)	
	liquid nitrogen cooling	15 min (100 to –100 °C)	

Calorimetric data			
Sensor type		FRS 6+	HSS 9+
Sensor material		Ceramic	
Number of thermocouples		56	120
Signal time constant		1.8 s	3.1 s
Indium peak (height to width)	raw data	19.5 [3]	6.9
	mathematically corrected	>155 [4]	>85 [4]
TAWN	resolution	0.12	0.20
	sensitivity	11.9	56.0
Measurement range	at 100 °C	± 350 mW	± 160 mW
	at 700 °C	± 200 mW	± 140 mW
Resolution		0.04 μW	0.02 μW
Digital resolution		16.8 million points	

Sampling	
Sampling rate	maximum 50 values/second

Special modes	
ADSC	standard
IsoStep®, **TOP**EM®	optional
Automation	
Microscopy	
Photocalorimetry	

Approvals

IEC/EN61010-1:2001, IEC/EN61010-2-010:2003
CAN/CSA C22.2 No. 61010-1-04
UL Std No. 61010A-1
EN61326-1:2006 (class B)
EN61326-1:2006 (Industrial environments)
FCC, Part 15, class A
AS/NZS CISPR 22, AS/NZS 61000.4.3
Conformity mark: CE

[1] based on metal standards
[2] depends on instrument configuration
[3] no mathematical treatment to the data or correction applied
[4] corrected according to B. Wunderlich, Thermal Analysis of Polymeric Materials, Springer (2005), page 346

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Environmental management system according to ISO 14001.

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