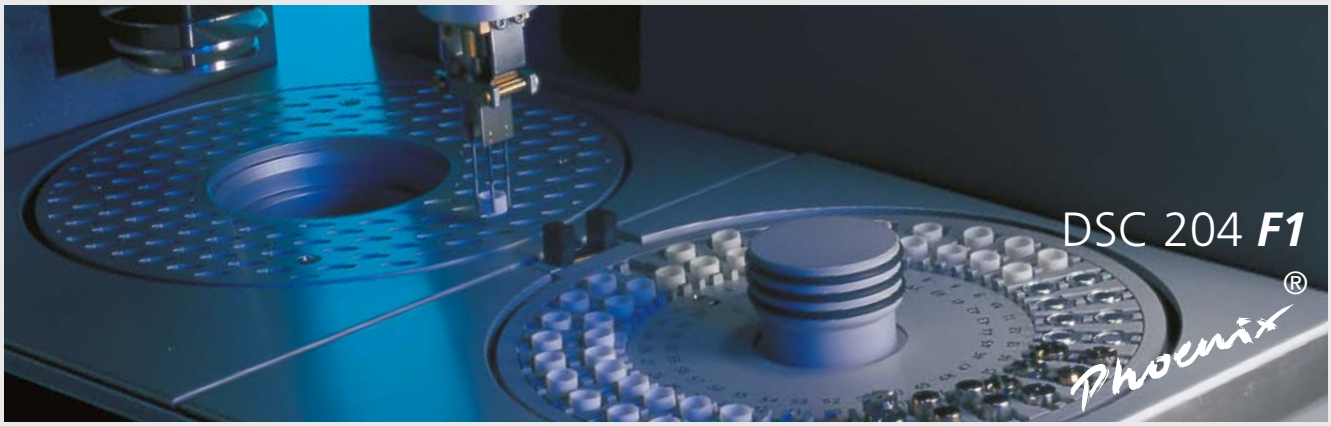
NETZSCH

Analyzing & Testing

Differential Scanning Calorimetry

Method, Technique, Applications



Leading Thermal Analysis

Method

Differential Scanning Calorimetry (DSC) determines transition temperatures and enthalpy changes in solids and liquids under controlled temperature change.

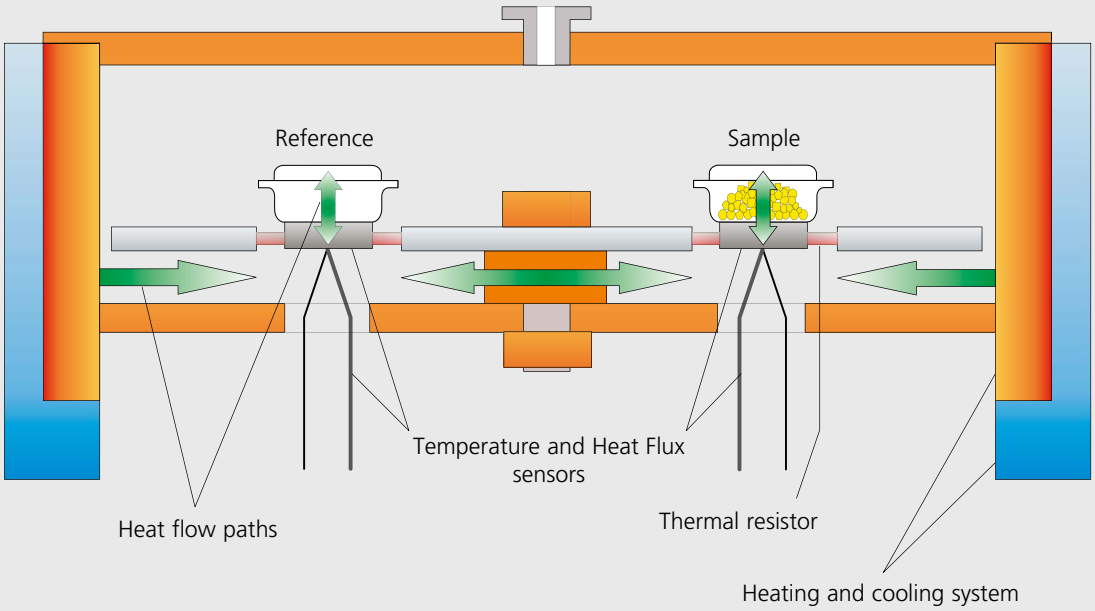
DSC is the most frequently used method in the field of Thermal Analysis. Rapid analyses, high significance for research and quality control tasks, and easy handling contribute to its versatility.

Many standards (ASTM, DIN, ISO,...) can be used as guidelines for the calibration of the instruments and for specific material-, product- and property-oriented applications, evaluations and interpretations.

Typical applications of DSC are:

- Melting-crystallization
- Polymorphism
- Phase diagrams
- Liquid crystal transitions
- Eutectic purity
- Crystallinity of semi-crystalline materials
- Solid-liquid ratio
- Solid-solid transitions
- Glass transitions
- Specific heat capacity
- Cross-linking reactions
- Oxidative stability
- Decomposition onset
- Compatibility

DSC 204 ***F1*** *Phoenix®*
can cover the temperature range from -180°C to 700°C

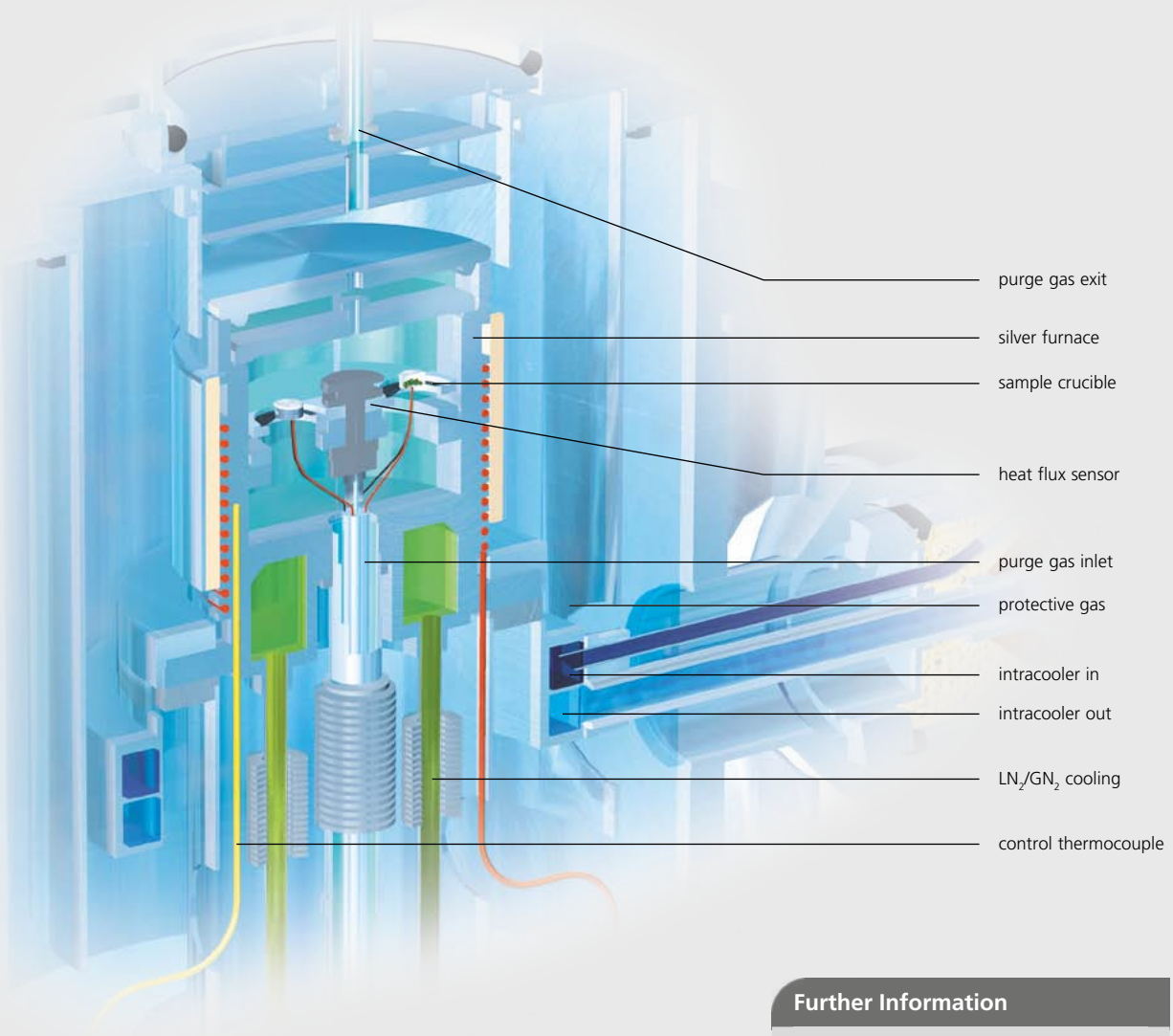


Principle of the Heat Flow DSC

Based on a homogeneous temperature field in the furnace of the DSC, equal heat flows along the disk-shaped sensor are directed to the sample and reference sides with crucibles. If the heat capacities on the sample and reference sides differ, or if the sample shows a changed heat absorption or loss due to transitions or reactions, the subsequent different heat flow causes temperature gradients at the thermal resistances of the sensor, which is otherwise a good conductor. Sensitive temperature sensors record these gradients and measure every difference in the heat flow quickly and accurately. The temporary deviations are shown as exothermal or endothermal peaks or as steps in the differential heat flow curves over a flat, horizontal DSC baseline.

DSC 204 ***F1*** *Phoenix*® - Trendsetting Technology

The concept for the DSC 204 ***F1*** *Phoenix*® measuring cell is based on a homogeneous heating of the disc-sensor system for stable and reproducible baselines. Top performance is achieved with the effective, low-consumption cooling system, and long product life is ensured with high mechanical and chemical resistance. This is achieved by incorporating the sensor into a cylindrical silver furnace with embedded heating coil and silver lids above and below the sensor, by ensuring a good coupling of the intracooler and the alternative cooling by liquid nitrogen or air, by reducing the inertia of the furnace, and by using resistant metals for furnace bodies and heat flow sensors. The perfect highly efficient and the protective gas flow allow continuous work at low temperatures without frost or ice accumulation in the main body of the measuring cell. The gas-tight construction of the DSC cell enables measurements under very pure, defined gas atmospheres. The precise control and the automated switching of gases is provided by the integrated and calibrated mass flow controller. The sensors are exchangeable allowing one to optimize around current needs, or future application requirements.



Further Information

www.dsc204.info

Various Cooling System

The DSC 204 ***F1*** *Phoenix*® is set up for various cooling system. The entire temperature range from -180°C to 700°C can be covered with the CC 300 (controlled cooling with liquid nitrogen (LN_2) and its gas phase (GN_2)).

Alternatively, an efficient, economical closed-loop intracooler is available, which operates between -85°C and 600°C. Compressed air cooling via compressor or central supply in the laboratory serves for tests above room temperature.

Different Sensors

The τ-Sensor is a constantan (CuNi) disk sensor. Due to the high conductivity of the silver carrier plate, the τ-Sensor greatly improves the response time for heat flow differences, and specializes in the separation of overlapping effects in the DSC curve.

The µ-Sensor's design is based on a specially doped silicon wafer used as a sensor disc. Along with the sensitive temperature sensors, the µ-Sensor offers a level of calorimetric sensitivity that had been considered impossible in DSC until now.

Technical Key Data for the DSC 204 *F1 Phoenix*®	
Temperature range	-180°C to 700°C
Cooling rate	0.001 to 200 K/min
Resolution (digital)	0.01 µW (µ-sensor)
Sensitivity	■ τ-sensor: 3.2 µV/mW ■ µ-sensor: 70 µV/mW
Sensor time constant	τ-sensor: < 0.6 s ; µ-sensor: < 3s
Enthalpy accuracy	< 1%
Exchangeable sensor	■ τ-sensor (-180°C to 700°C) (short time constant) ■ µ-sensor (-150°C to 400°C) (very high sensitivity)
Cooling device options	■ Compressed air: RT to 700°C, with cold air generator: 0°C to 700°C ■ Intracooler: -85°C to 600°C ■ Liquid nitrogen: -180°C to 700°C
Gas atmospheres	Inert, oxidizing, static, dynamic
Gas-tight	Yes
Mass flow controller for purge/protective gases	3
Automatic Sample Changer (ASC)	Optional for 64 samples
ASC reference sample changer	Yes
Temperature-modulated DSC (TM-DSC*)	Optional
Coupling to evolved gas analysis	MS and/or FT-IR, even with ASC

*not available in Japan

DSC 204 ***F1*** *Phoenix*® - Perfect Accessories

The versatility of the DSC 204 ***F1*** *Phoenix*® is enhanced by the large range of accessories.

Automatic Sample Changer (ASC)

An Automatic Sample Changer (ASC) can be installed, its carousel handles up to 64 samples and reference crucibles which can be run automatically in any order. The required gas atmosphere in the sample chamber is generated via an automatic, controlled process, as is the cooling. Of course, each sample can be assigned an individual measurement and evaluation macro.

For unstable samples or samples with volatile components, an automatic piercing device is available.

Coupling to Evolved Gas Analyzers FT-IR and/or MS - even with ASC

The gas-tight construction of the DSC 204 ***F1*** *Phoenix*® cell and the precise gas control allows for coupling to FT-IR or MS spectrometers. A unique heated adapter system even enables simultaneous coupling to both gas analyzers.

Further Information

www.dsc204.info

Crucible Variety and Sealing Press

Crucibles are available in various dimensions in metal, precious metal, graphite, glass and oxide ceramics. Should samples need to be shut off from the influence of the ambient atmosphere, or should gas separation from the samples need to be repressed, aluminum crucibles can be welded, gas-tight, in a handy sealing press.

Calibration Materials

For temperature and enthalpy calibration of the low-temperature DSC systems, various sets with different calibration materials are available for different crucible materials (including the high-pressure autoclaves) that cover the whole temperature range of the instruments.

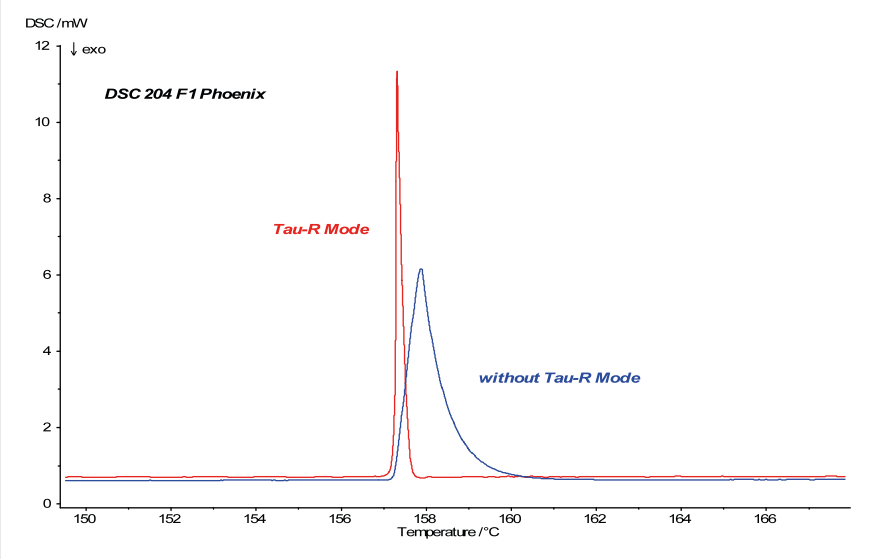
The calibration materials are selected and prepared for measurement according to the recommendations of the corresponding ASTM and CEI-IEC standards. A manufacturer certificate is included and detailed calibration instructions are given in each DSC's help system.

Proteus® Software for DSC systems

All differential scanning calorimeters run under *Proteus*® software on Windows® XP or Windows® 7. The *Proteus*® software includes everything you need to carry out a measurement and evaluate the resulting data. User-friendly menus combined with automated routines make *Proteus*® very easy to use while still providing sophistiaced analysis. The *Proteus*® software is licensed with the instrument and can of course be installed on other computer systems.

Tau-R Mode™ for Advanced DSC Analysis

On closer examination of the measured DSC data, it becomes clear that the curve shape does not exactly reflect the heat flow occurring during a phase transformation of the sample.
The onset temperature marks the beginning of the melting of the sample; when the peak temperature is reached, the sample is completely molten. The measured peak temperature does not correspond to the melting temperature of the sample, although the sample temperature should be the same as the melting temperature. Another discrepancy between the curve shape and reality is that after reaching the peak position, the signal does not immediately drop to zero. This, however, should be the case since the sample is already completely molten at this point and no longer needs any further energy.
Therefore, the DSC curve evidently incorporates information not only about the sample but also from the instrument. To overcome this discrepancy, the *Tau-R Mode*™ has been developed. This correction is based on two essential correction factors: the thermal resistance R and the time constant tau. The correction parameters were determined by measuring melting standards. Therefore this correction is based on real measurement results and can be applied also subsequently.



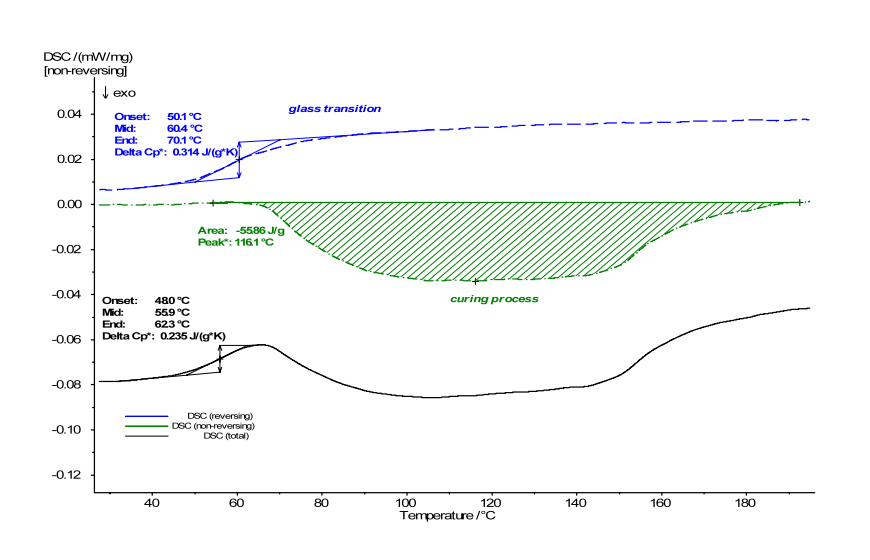
Tau-R Mode™ for Advanced DSC Analysis

BeFlat® - The Revolution in Baseline Optimization

Perfect thermal symmetry, which is often expected in a differential measurement set-up, is frequently not realized due to material and technical production limitations. The new, unique *BeFlat*® corrects DSC baseline discrepancies which are due to thermal asymmetry by using a multi-dimensional polynomial which is dependent upon temperature and heating rate. *BeFlat*® removes the baseline discrepancies from the DSC signal and yields perfect horizontal DSC baselines with minimal deviations in the µW range.

Temperature Modulated DSC

Temperature Modulated Differential Scanning Calorimetry (TM-DSC) is a DSC technique whereby a sample is subjected to a superposition of a linear and a periodic temperature program. With the temperature modulated DSC method it is possible to separate overlapped DSC effects by calculating the reversing and the non-reversing signals. Glass transitions can therefore be well separated from e.g. curing, decomposition, evaporation, relaxation or cold-crystallization processes. The following figure shows the curing of an unsaturated polyester resin and how the overlapping of an endothermal glass transition can be separated from exothermal curing in a single TM-DSC test.



Temperature Modulated DSC

Proteus®
Flexible, Intelligent, Complete

- Editable experiment program
- Repeat measurements with minimal parameter input
- Ongoing analysis of the measurement underway
- Comparison of up to 64 curves
- Simultaneously multi cure evaluation
- Curve subtraction, averaging
- Multi-method analysis (DSC, TG, DMA, TMA, etc.)
- Zoom and picture-in-picture presentation
- 1st and 2nd derivative
- Automatic evaluations for characteristic temperatures
- Complex peak evaluations
- Multipoint calibration for sample temperature
- Multipoint calibration for enthalpy changes
- c_p calibration for heat flow
- Data export and import (ASCII) and direct data export to Excel®
- Integral signal for solid-liquid presentations at melting curves
- Signal-controlled measurement procedure (e.g. O.I.T., measurement deactivation at threshold value)
- Automatic transmission of status messages or complete measurements by e-mail
- Option for fully automatic macro evaluation
- Option for c_p evaluation
- Optional TM-DSC*
- *Tau-R Mode*™
- *BeFlat*® for baseline optimization
- Option for *Purity Determination*

* not available in Japan

Advanced Software

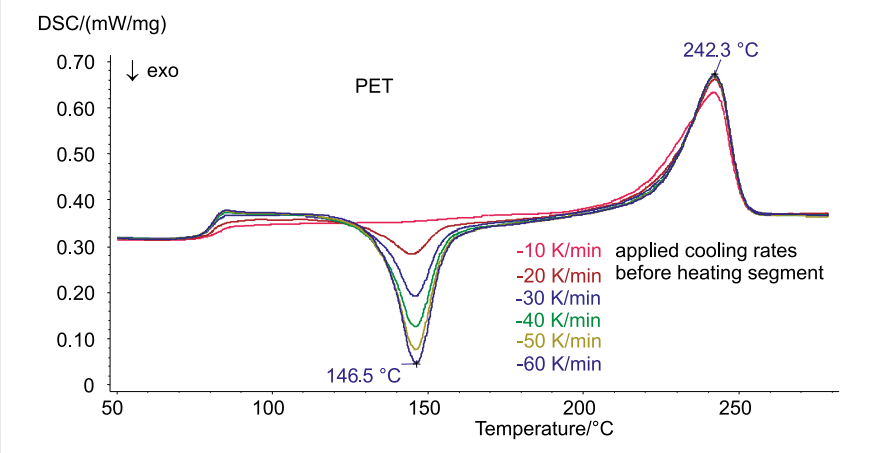
Increase the information yield of DSC measurements by using continuative software solutions with many unique features for:

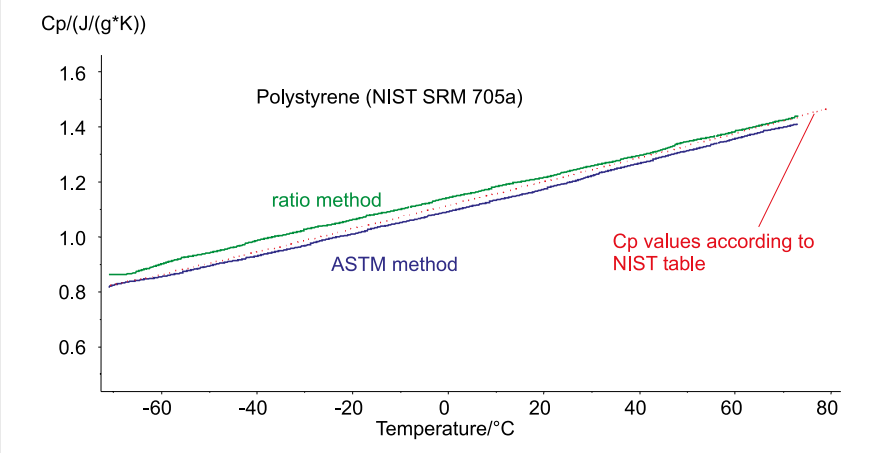
- *Thermokinetics* for kinetic analysis of several measurements by model-free and model based methods
- Component kinetics for reactions in homogeneous mixtures
- *Thermal Simulations* for process predictions on a production scale
- *Peak Separation* software for DSC curves with overlapped peaks

Applications

Polymers

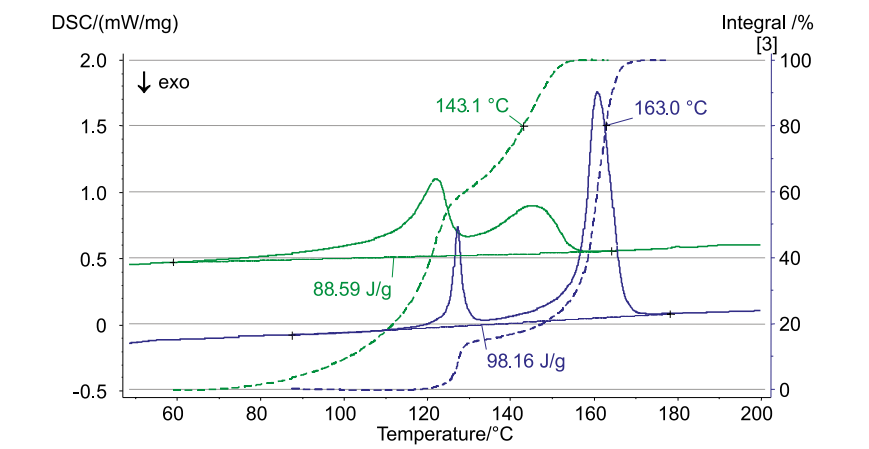
Polyethylene terephthalate (PET) is a semi-crystalline thermoplastic polymer with a relatively slow crystallization rate. In the DSC experiments, the various levels of amorphousness (Tg 75°C to 85°C) and crystallinity (recrystallization 146°C, melting 242°C) are apparent. The samples were cooled from the melt in the DSC 204 ***F1*** *Phoenix*® with the intracooler at different rates prior to the heating shown.

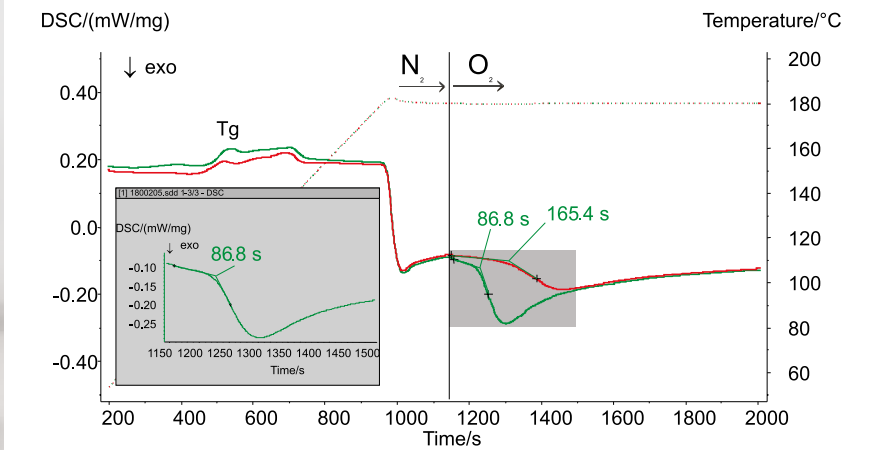




The ability to determine specific heat capacity for the most varied of materials is an important task for the DSC. A mean error of < 2% was attained on NIST Standard Reference Material 705a, a polystyrene with narrow molar mass distribution, by using a heating rate of 10 K/min and various analysis methods.

The information from a peak integration of DSC melting peaks is often not directly convertible for use in thermal processing. The *Proteus*® software's integral analysis provides an exact measurement for solid-liquid behavior at every temperature. The comparison of two PE-PP blends shows that one sample is 80% melted already at 143°C; the other only at 163°C.



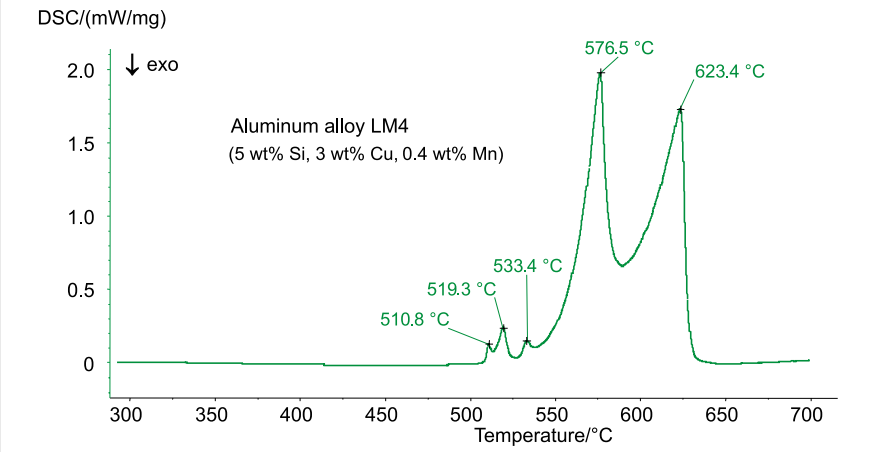


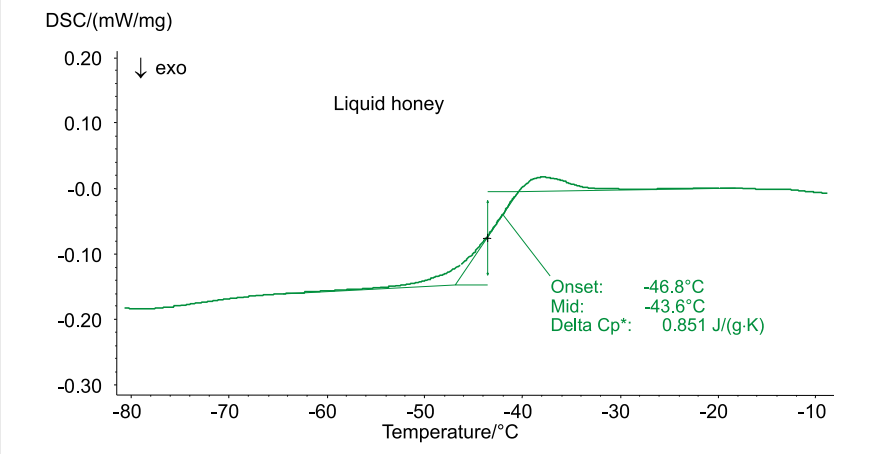
The aging stability of plastics can be calculated in the DSC by standardized analyses in oxygen atmospheres (O.I.T.). In the example shown of two ABS samples, the oxidation induction time of the defective sample, at 86.8 s, is only about half as large as in the non-defective reference sample (165.4 s).

Applications

Metals

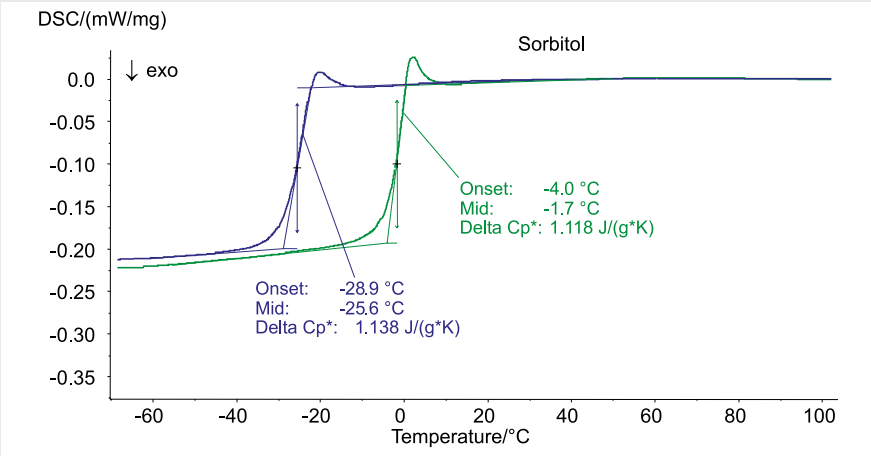
When analyzing modern metal alloys, it is important that there is a good separation of the melting peaks for the individual alloy components. The DSC 204 ***F1*** *Phoenix*® with **τ**-Sensor yields an excellent peak separation in the melting range from 510°C to 650°C for the aluminum alloy measurement shown here.





Foods

The sugar-water system is very important for the food and pharmaceutical industries. Besides its main constituent, consisting of various sugar types, natural honey also contains up to 17 % water. The amorphous structure of the sugar-water system is apparent in the low-temperature glass transition at -44°C.



Pharmaceuticals

Sorbitol is used as a substitute for sugar in many sweets, diet products, and medications. A proportion of 5.5 % water in anhydrous sorbitol causes the glass transition to defer from -1.7°C to -25.6°C. Both samples remain completely amorphous after the rapid cool-down following the melt (which took place before the heating mentioned above).

Expertise in Service

Our Expertise – Service

All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, before and after sale. Our qualified personnel from the technical service and application departments are always available for consultation.

In special training programs tailored for you and your employees, you will learn to tap the full potential of your instrument.

To maintain and protect your investment, you will be accompanied by our experienced service team over the entire life span of your instrument.

Summary of Our Services

- Installation and commissioning
- Hotline service
- Preventive maintenance
- Calibration service
- IQ /OQ/PQ
- On-site repairs with emergency service for NETZSCH components
- Moving/exchange service
- Technical information service
- Spare parts assistance

Our Expertise –
Applications Laboratories

The NETZSCH Analyzing & Testing applications laboratories are proficient partners for nearly any Thermal Analysis issue. Our diverse measuring methods and over 30 different state-of-the-art test stations will provide ready solutions for all your thermal needs.

Measurements can be carried out on samples of the most varied of geometries and configurations. You will receive high-precision measurement results and valuable interpretations from our expert team. This will enable you to precisely define new materials and components before actual deployment, minimize risks of failure, and gain decisive advantages over your competitors.

For production problems, we can work with you to analyze causal issues and work out solution concepts. The option of commercial testing is a relatively low investment which generally pays itself off in a few days or weeks and provides fast and reliable support for research on new materials or resolving any kinds of production issues.

NETZSCH

The NETZSCH Group is an owner-managed, internationally operating technology company headquartered in Germany.

The three Business Units – Analyzing & Testing, Grinding & Dispersing and Pumps & Systems – provide tailored solutions for highest-level needs. Over 2,500 employees at 130 sales and production centers in 23 countries across the globe guarantee that expert service is never far from our customers.

When it comes to Thermal Analysis, Adiabatic Reaction Calorimetry and the determination of Thermophysical Properties, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

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