NETZSCH

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DSC 204 *F1*

Phoenix®

The DSC 204 *F1 Phoenix*®

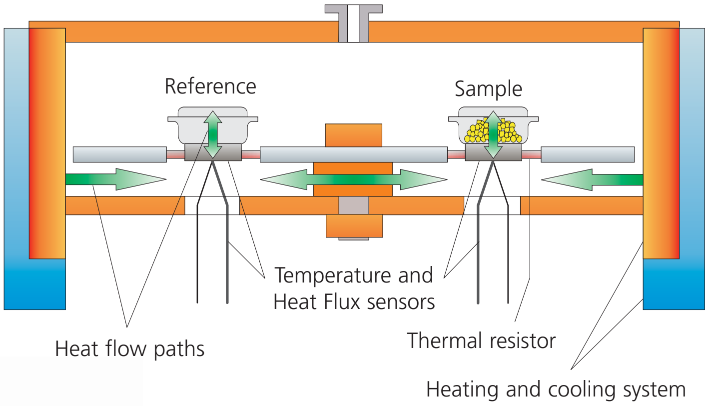
Differential Scanning Calorimetry (DSC) determines transition temperatures and enthalpy changes in solids and liquids under controlled temperature change.

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*®
The new DSC platform
from –180 to 700°C

Principle of the Heat Flow DSC



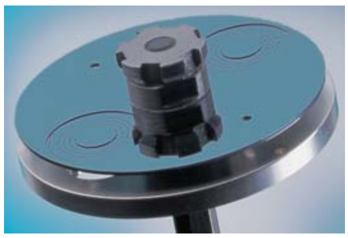
Based on a homogeneous temperature field in the furnace of the DSC, equal heat flows along the disk-shaped sensor are directed to the sample and reference sides. If the heat capacities on the sample and reference sides differ, or if the sample shows a changed heat absorption or loss due to transitions or reactions, the subsequent different heat flows cause temperature gradients at the thermal resistances of the sensor, which is otherwise a good conductor. Sensitive temperature sensors record these gradients and measure thereby every difference in the heat flows very 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.

Different Sensors:

The new 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, thus leading to top performance through the separation of overlapping effects in the DSC curve.

τ-Sensor

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.



μ-Sensor

Proteus® Software

DSC applications without computer-aided support are unthinkable these days. *Proteus*®, our true 32-bit software based on MS® Windows®, takes over all sample preparation, measurement and evaluation tasks during a DSC run as well as during other thermoanalytical experiments. One of the most complete software solutions in Thermal Analysis, *Proteus*® was developed, tried and tested by our software specialists and application experts, and supports you with a practice-oriented structure, easy-to-understand user guidelines and an example-rich comprehensive help system. It gives you leeway in practicing your individual evaluation and documentation style.

Data security and unfalsifiability are prerequisites for fair GLP, GMP and 21 CFR, part 11 application.
The multipoint (6-12) temperature and enthalpy calibrations, as well as online base-line corrections, are permanently linked to the raw data.
Analysis routines conform to standardized procedures and can therefore be easily validated.

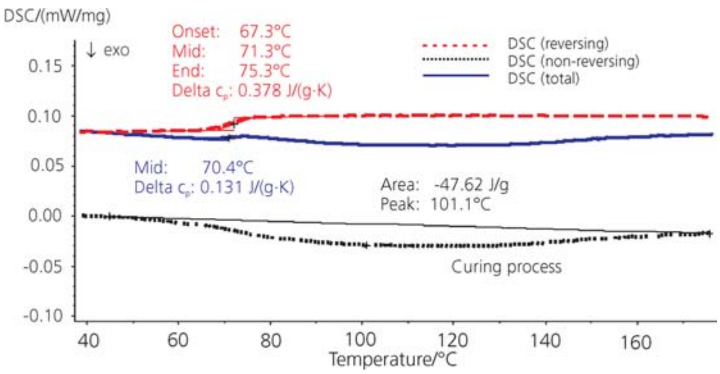
BeFlat® - The Revolution in Baseline Optimization

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

Temperature-modulated DSC - TM-DSC Software

In an experiment with temperature-modulated DSC, the underlying linear heating rate is superimposed by a sinusoidal temperature modulation. The sample to be investigated is analysed with a mostly low linear rate under nearly equilibrium conditions in order to accurately record thermodynamic properties. The sample is at the same time subjected to a relatively fast non-linear temperature change by means of temperature modulation for the detection of time-dependent (kinetic) processes in the sample with a sufficient sensitivity.

The good separation of thermodynamic transitions and kinetics processes offers a wide application field for the study of cross-linking systems where glass transition, relaxation, melting and thermally induced curing are often overlapped. Hardening of an epoxy resin shows how the overlapping of endothermal glass transition can be separated by the already beginning exothermal curing in a single TM-DSC test and how evaluation of the separated processes can be realized.



Advanced Software

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

- thermokinetics with multi-curve analysis by non-linear regression (NLR)
- thermal simulation for process forecasting on a production scale
- ChemRheo®, kinetics which link thermal and rheological data
- purity evaluation
- peak separation

Proteus®
flexible, intelligent, complete

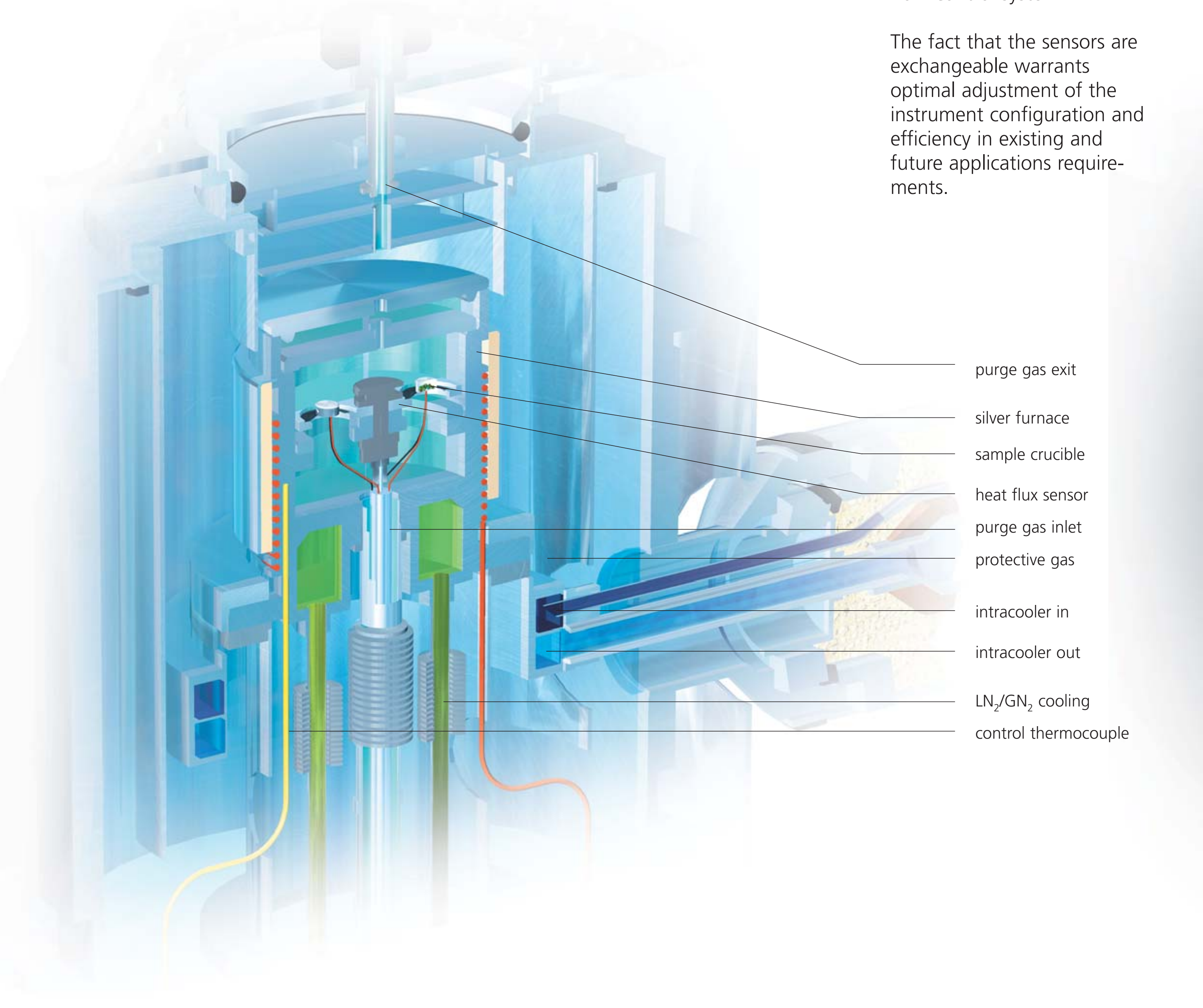
- editable experiment program
- repeat measurements with minimal parameter input
- ongoing analysis of the measurement underway
- comparison of up to 32 curves
- curve subtraction
- multi-method analysis (DSC, TG, DMA, TMA, etc.)
- zoom and picture-in-picture presentation
- 1st and 2nd derivative
- automatic evaluations for characteristic temperatures
- complex peak evaluations
- choice between 5 baseline types
- multipoint calibration for sample temperature
- multipoint calibration for enthalpy changes
- c_p calibration for heat flow
- storage and export of evaluation results
- data export and import (ASCII)
- direct data export to MS® Excel
- integral signal for solid-liquid presentations at melting curves
- signal-controlled measurement procedure (e.g. OIT, measurement deactivation at threshold value)
- automatic transmission of status messages or complete measurements by e-mail
- option for fully automatic macro evaluation
- option for TM-DSC

DSC 204 *F1 Phoenix*® - one fits all

The concept for the DSC 204 *F1* measuring cell is based on a homogeneous heating of the disc-sensor system for stable, reproducible baselines, on an effective and low-consumption cooling system, and on high mechanical and chemical resistance for long product life. 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 insulation 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 of the gases and the automatic change-over are due to the integrated, calibrated mass flow control system.

The fact that the sensors are exchangeable warrants optimal adjustment of the instrument configuration and efficiency in existing and future applications requirements.



DSC 204 *F1 Phoenix*® - one fits all

Alternative Cooling Techniques

Due to its broad temperature range and excellent cooling capacity, the DSC 204 *F1* with intracooler is optimally equipped for almost all applications. The instrument is therefore fully independent of any liquid coolant supply bottle-necks and is ready for use at any time.

Should the application require temperatures below -85°C, the DSC 204 *F1* with the configuration for liquid nitrogen cooling, with its start temperature of -180°C, offers the best solution for precise DSC tests in the low temperature range.

Should a working range beginning at room temperature suffice for the given application requirements, then the option to cool with compressed air (compressed air supply in the laboratory, gas bottle, membrane compressor) offers a low-priced solution for effective work. All cooling systems are software-controlled and linked to the respective temperature program of the DSC experiment.

DSC 204 *F1 Phoenix*® ASC
The automatic sample changer for 64 samples moves various crucible types to the sample or reference side securely and reliably and can also be transferred to the TG 209 *F1 Iris*®. An optional add-on punching device for aluminum crucibles is also available.

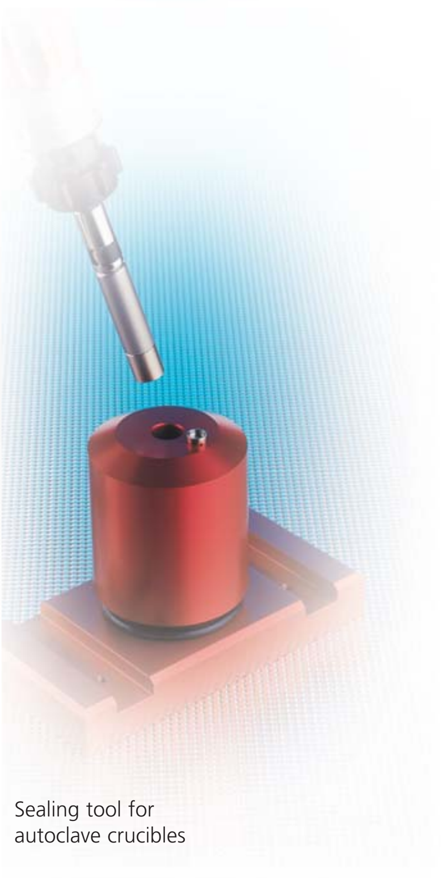
DSC 204 *F1 Phoenix*® - complete

The versatility of the DSC 204 *F1* is enhanced by the large assortment of sample crucibles. Choose the ideal crucible material, form and sealing method for your application and samples.

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.

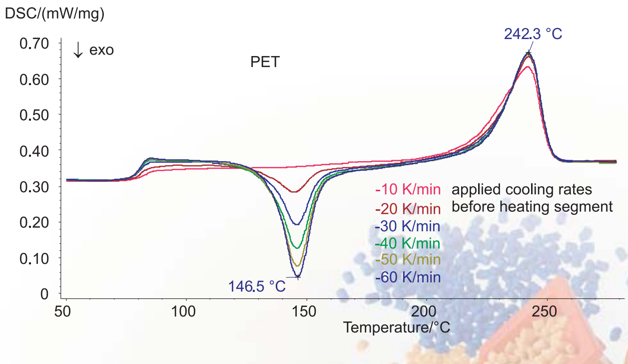
For measurements under increased pressure of up to 100 bar, reusable stainless steel and titanium autoclave crucibles do the job. Reproducible tightness of these crucibles, also under multiple use conditions, is assured by applying a special tool for closing crucibles at a fixed torque and by using interchangeable gaskets.

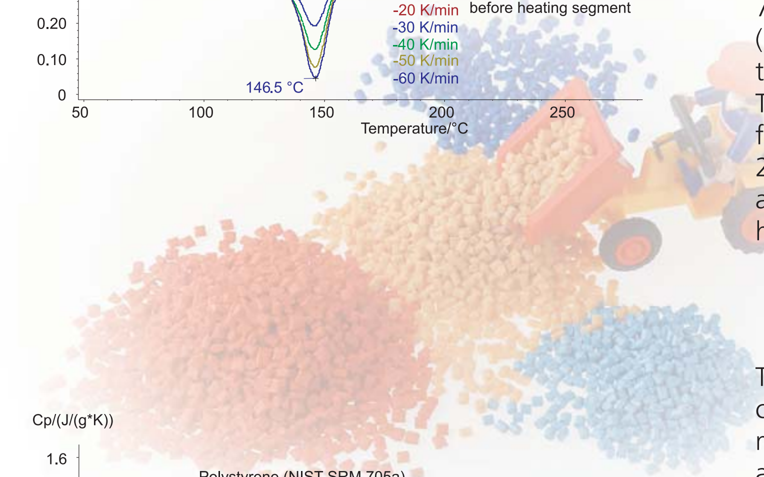
Sealing press for different crucibles



Sealing tool for autoclave crucibles

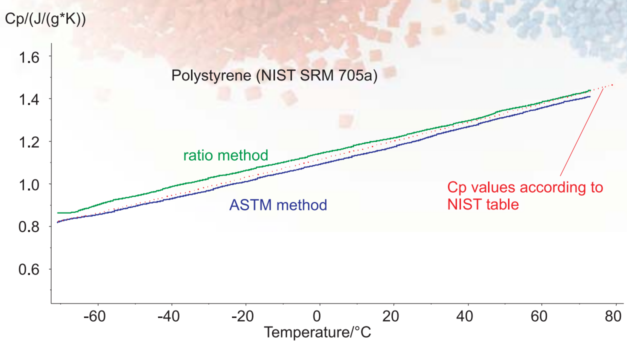
Applications



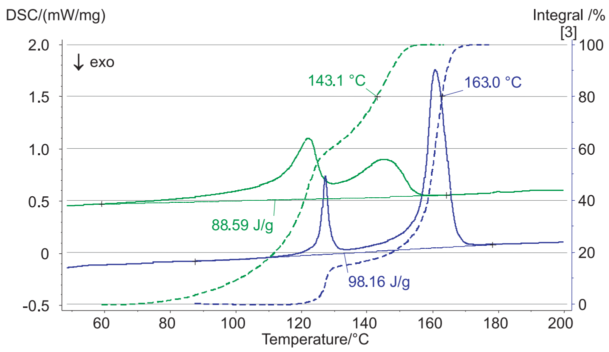


Polymers

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 75-85°C) and crystallinity (recrystallization 146°C, melting 242°C) are apparent. The samples were cooled from the melt in the DSC 204 *F1* with the intracooler at different rates prior to the heating shown.



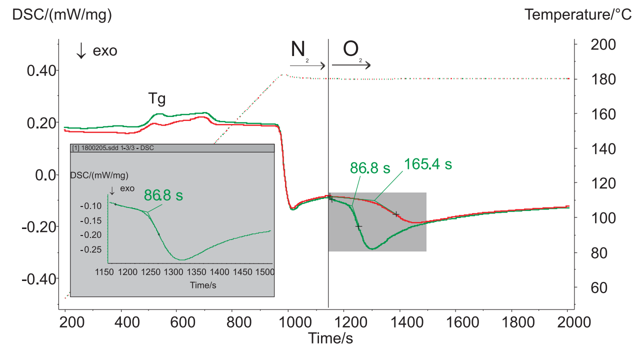
The ability to determine specific heat capacity for the most varied of materials is an important task for the DSC. A mean error of < 2% was attained on NIST Standard Reference Material 705a, a polystyrene with narrow molar mass distribution, by using a heating rate of 10 K/min and various analysis methods.



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.

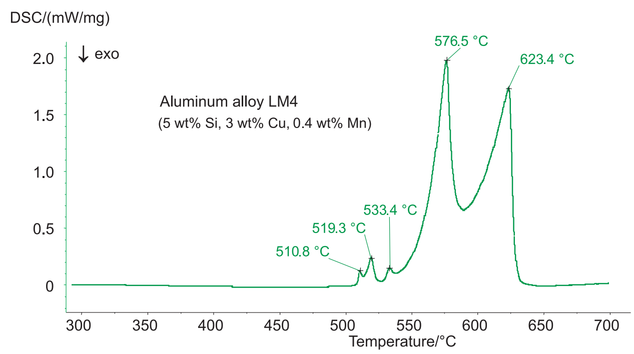
Applications

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 faultless reference sample (165.4 s).



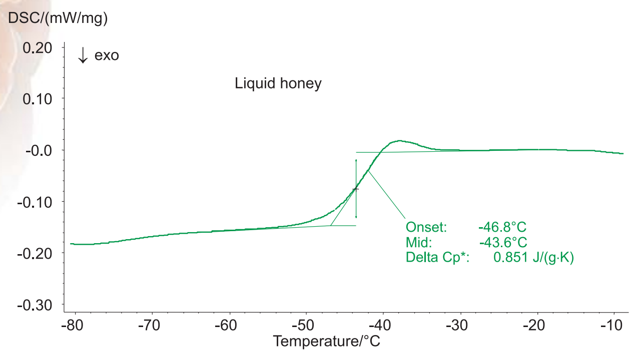
Metals

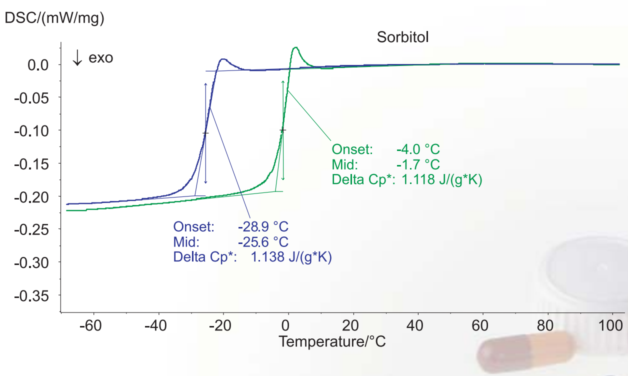
When analyzing modern metal alloys, it is important that there be a good separation of the melting peaks for the individual alloy components. The DSC 204 *F1* with τ-Sensor yields an excellent peak separation in the melting range from 510°C to 650°C for the aluminum alloy measurement shown here.

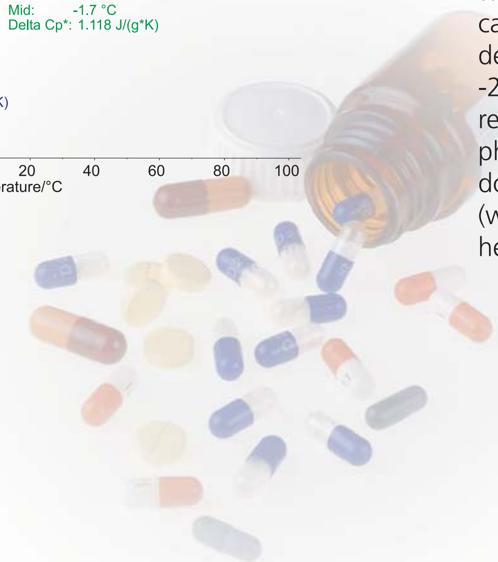


Foods

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).

Application literature

- "Focus on Thermal Analysis for Polymers"
- "Focus on Thermal Analysis for Paints"
- "TA for Polymer Engineering": NETZSCH Annuals for Science and Industry I, II, III
- NETZSCH Annual 2000 "Thermoanalytical Characterization of Pharmaceuticals"

DSC 204 *F1 Phoenix*® Technical Data

Temperature range:
25 ... 700°C
with air cooling
-85 ... 600°C
with mechanical cooling
-180 ... 700°C
with liquid nitrogen cooling

Heating rates: 0.001 to 200 K/min
Cooling rates: 0.001 to 200 K/min
(depending upon the cooling system and the temperature range)

Gas control and recording for two sample gases and protective gas by integrated mass flow control system and software

integrated controller with 24-bit AD converter

DSC limit of detection:
< 0.1 µW (depending upon the sensor)
System time constant: 0.6 to 3 s (depending upon the sensor)

Automatic sample changer for 64 sample and reference crucibles (on one carousel; variety in crucibles also possible)

Optional:
Coupling with mass spectrometry and/or FTIR for online gas analysis.

Perfect platform solution for DSC and TG

TG 209 *F1 Iris*® ASC

Same design, same control electronics, same automatic sample changer as
DSC 204 *F1 Phoenix*®

Thermal Analysis – NETZSCH-Gerätebau GmbH

- Complete instrument line for Thermal Analysis and Thermophysical Properties (DSC, DTA, STA, DMA, TMA, Dilatometer, DEA, MS/FTIR coupling, thermal conductivity, thermal diffusivity, etc.)
- Software
- Application service
- Contract testing laboratories
- Technical service worldwide

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