NETZSCH
Proven Excellence.

Differential Scanning Calorimetry DSC 204 ***F1*** *Phoenix®*

Method, Technique, Applications

Analyzing & Testing

Are you looking for reliable instrumentation for thermal analysis?

An instrument which provides accurate results for cutting-edge research on sensitive samples?

A robust system, able to work continuously and without stillstands, to handle your workload for quality control?

A testing device which saves a lot of time through automated processing of routine tasks?

The DSC 204 ***F1*** *Phoenix*® is Your Solution!

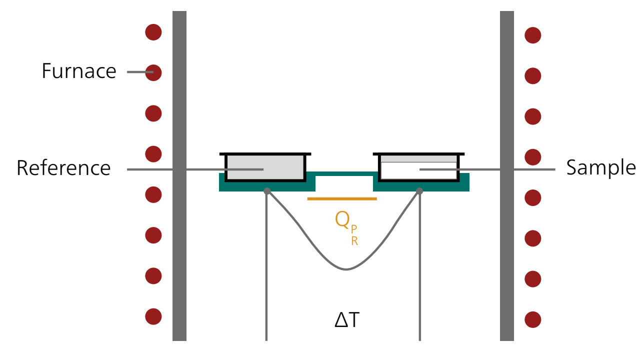
Best instrument for research and development, material characterization, quality control and contract testing!

Made by NETZSCH

DIFFERENTIAL SCANNING CALORIMETRY

Typical DSC Applications

- Melting, crystallization
- Glass transition
- Oxidative stability
- Crystallinity of semicrystalline materials
- Solid-liquid ratio
- Specific heat capacity
- Cross-linking reaction
- Decomposition onset
- Solid-solid transition
- Polymorphism
- Phase diagrams
- Liquid crystal transition
- Eutectic purity
- Compatibility



Scheme of a heat flow DSC cell

Differential Scanning Calorimetry (DSC) is the most widely used thermal analysis technique.

Based on ISO 11357-1, heat flow DSC is a technique in which the difference between the heat flow rate into a sample pan and that into a reference pan is determined as a function of temperature and/or time. During such measurement, the sample and reference are subjected to the same controlled temperature program and a specified atmosphere.

In practice, a sample is placed inside a pan and then positioned inside the measurement cell of the DSC instrument along with an empty reference pan. While heating or cooling at a constant rate or during isothermal steps, characteristic changes in sample properties can be determined – for example, with regard to glass transition, polymorphic transition, the occurrence of melting/crystallizing, or the start of oxidation.

DSC provides quick, reliable measurement results on caloric changes to the sample!

DSC 204 ***F1*** *Phoenix*®

Exceeding Your Expectations

Adaptable to Future Needs and Meeting a Wide Variety of Applications – Different Sensor Types

The τ-sensor is standard for most DSC applications. It offers a high level of calorimetric sensitivity along with an extremely short signal time constant and covers the complete temperature range of the DSC instrument. The µ-sensor, in contrast, delivers a much higher output signal and is therefore able to detect even very small effects.

No Need to Change Out the Whole Furnace

Both sensors are rugged and durable. For condition-based maintenance, however, each sensor can be replaced and there is no need to change out the whole furnace. This is especially beneficial if samples with unknown or corrosive properties have to be measured.

Highest Sample Through-put – The World's Largest Automatic Sample Changer

Two individual sample trays with 96 positions each for pans of any NETZSCH DSC type plus an additional strip for 12 calibration materials/reference pans: The new generation of automatic sample changers (optional) converts the DSC 204 ***F1*** *Phoenix*® into a fully automatic system, which is able to finish a complete measurement series over the weekend – even with reproducibility runs.

DSC 204 ***F1*** *Phoenix®* with Automatic Sample Changer; each conventional tablet running under Windows can be used as a mobile touch screen

GAS-TIGHT DESIGN

EVOLVED GAS ANALYSIS

FUTURE-PROOF DESIGN

SMARTMODE

RUGGED SENSOR

AUTOEVALUATION

AUTOMATED ROUTINES

EXCHANGEABLE SENSOR

EASY TO USE

IDENTIFY

ROBUST SYSTEM

AUTOSAMPLER WITH 204 POSITIONS

OPTIMIZED FOR COUPLING TO FT-IR OR MS

PHOTOCALORIMETRY

Proteus® Software – Trend-Setting in User Assistance and Intelligence

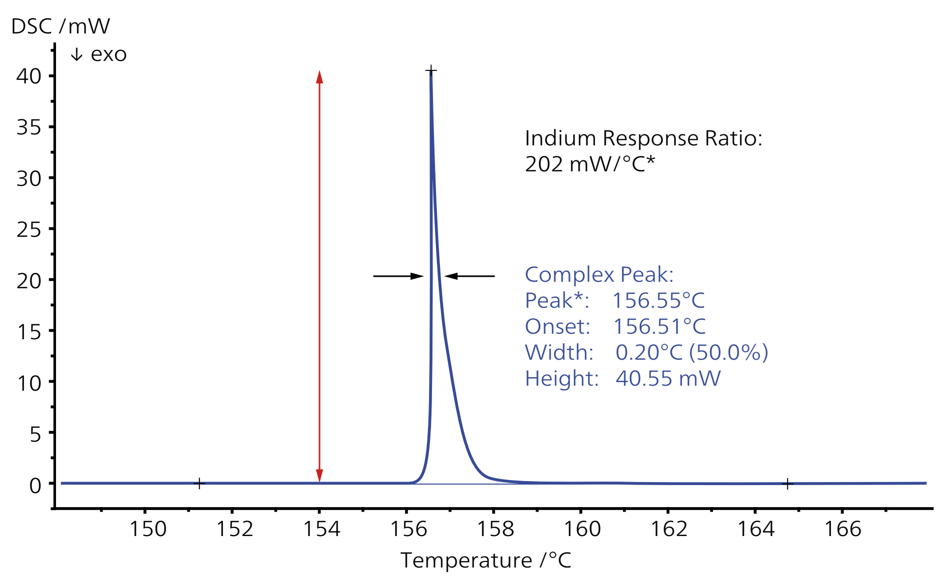
SmartMode, AutoEvaluation and *Identify* are the keys to making life in a laboratory much easier than ever before. A simplified and intuitively designed user interface; autonomous evaluation routines which can serve as a second opinion when assessing unknown samples; and a database system for identifying and verifying materials – all at your side at any time.
AutoCalibration performs calibration runs along the way and keeps your hands free for more important matters. Thanks to its flexibility, the *Proteus®* software can also be used without these little aids in *ExpertMode*, where more options are accessible.

Elaborate Technology for

τ-Sensor – Outstanding Sensitivity and High Resolution at the Same Time

The height-to-width ratio of an indium melting peak – or in other words, the indium response ratio – reflects both the sensitivity and the resolution of a sensor/pan pairing and is therefore an ideal criterion for evaluating sensor performance. The higher the value, the higher the ability to detect weak effects as well as to separate overlapped ones.
In the present case, the DSC 204 ***F1*** *Phoenix*® equipped with a τ-sensor generates an indium response ratio of 202 mW/°C, which is a clear sign of its excellence.

* Peak shape correction is based on the equation published by B. Wunderlich, Thermal Analysis of Polymeric Materials, Springer (2005), page 346



DSC measurement on indium, τ-sensor, sample mass: 13.6 mg, heating rate: 10 K/min, Al pans, N_2 atmosphere

τ-sensor

The sensor is the heart of a DSC system; however, the sensor alone does not constitute a complete DSC system. Rather, all relevant parts – primarily the sensor, the furnace, the cooling device and the pans used – have to be matched.

Optimum DSC Performance

Superior Cell Design

Each sensor of the DSC 204 ***F1*** *Phoenix* ® can be removed and replaced with another. This ensures that the system is also well prepared to handle any future changes in application requirements. The sensor is installed into a cylindrical silver furnace with embedded heating coils. The high thermal diffusivity of the silver provides excellent heat distribution inside and thus homogeneous heating of the sensor, which is a "must" for stable baselines.

The protective gas flow around the furnace effectively prevents frost or ice accumulation in the cell and allows for continuous work at low temperatures.

Lastly, the gas-tight construction of the cell enables measurements under a pure, defined gas atmosphere. The gas flows are controlled by three built-in and calibrated mass flow controllers.

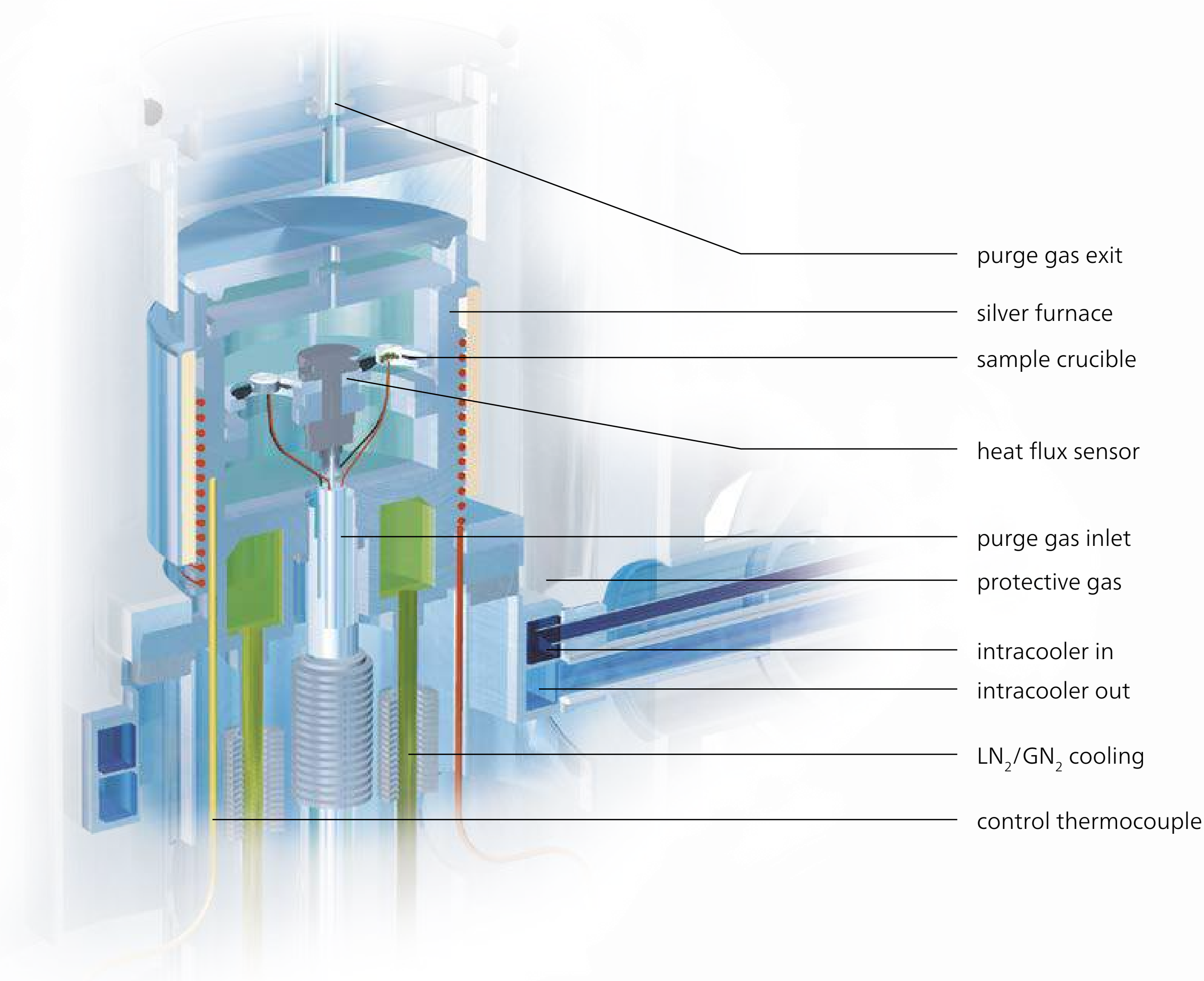


Diagram of the set-up of the DSC 204 ***F1*** *Phoenix*®

ACCESSORIES

Unsurpassed Versatility

Great Variety of Sample Pans

The sample pan (or crucible) can have a significant influence on the measurement result and therefore has to be appropriate for the application. Both the material and the shape are important. For this reason, NETZSCH offers a multitude of different pans in various dimensions; these may be made of metal, graphite, glass or oxide ceramic and may also be open (with a lid that can just be laid upon them) or hermetically sealed.

Additionally, in the NETZSCH portfolio are special lids for films and a reshaping tool for preparing SFI* crucibles, which are used for liquids with a high wettability.

For application in the polymer and the organics fields, the one-of-a-kind *Concavus*® pans (made of aluminum) are recommended. Their design allows for further increases in reproducibility.

* SFI stands for Solid Fat Index

Sealing press with several toolkits

High-pressure pans

Concavus® pans

Effective and Highly Economic Cooling Systems

Tailored to specific temperature ranges, four different cooling options are available, ranging from air cooling devices to liquid nitrogen cooling. The liquid nitrogen cooling option can be operated in the LN_2 (liquid nitrogen) as well as in the GN_2 (gaseous nitrogen) mode. This helps save cooling agent and provides better control.

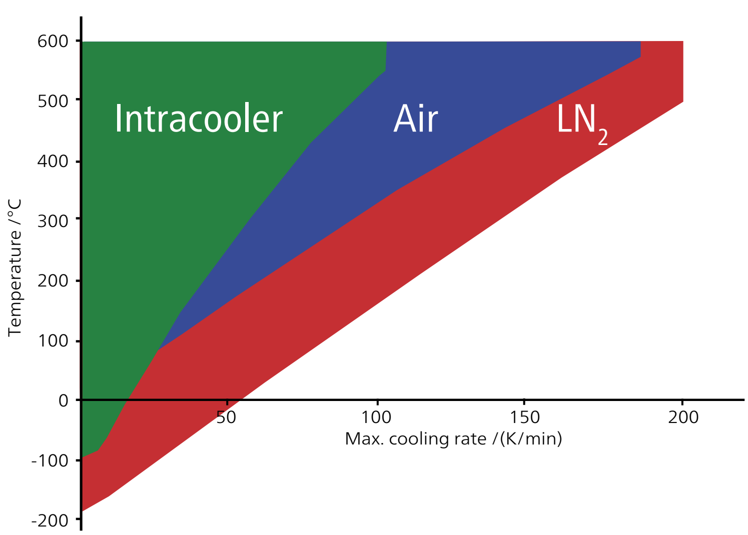
The *AutoCooling* function of the NETZSCH *Proteus®* software automatically recognizes which cooling device is present and activates cooling only if it is necessary for realizing the defined temperature program.

If desired, the liquid nitrogen cooling device can be connected to the DSC simultaneously with the intracooler. This reduces the liquid nitrogen consumption even more because in this case, it is only activated at temperatures below -85°C.

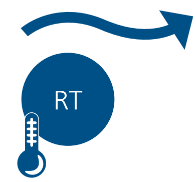
Connection of the standard 60 l DSC Dewar to a large LN_2 tank (e.g., containing 300 l) enables automatic refilling during a long measurement series or even during a running measurement.

All cooling devices operate up to 600/700°C.

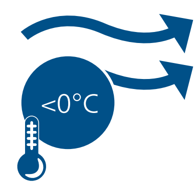
Significant Cooling Capabilities



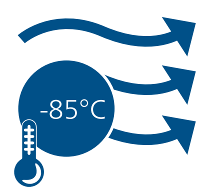
In order to realize the desired cooling speed within a given temperature range, various cooling systems are at your disposal. For example: Using liquid nitrogen cooling, a linear cooling rate of 100 K/min can be realized down to approx. 180°C.



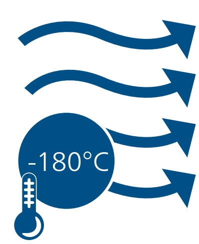
Air compressor
(RT to 700°C)



Compressed air cooling
(<0°C to 700°C)



Closed loop intracooler
(-85°C to 600°C)



Liquid nitrogen cooling
(-180°C to 700°C)

Automatic Sample Changer (ASC)

Efficiency at Its Best

Two interchangeable sample trays in microplate format with a total of 192 positions (96 each) can be quickly loaded and unloaded. Each sample tray can be filled with DSC pans of various types. The ASC gripper is able to manage them all. Its *SafeTouch* functionality ensures that every pan type is treated with the ideal contact force.

The appropriate contact force is automatically deduced from a comprehensive database containing all pans and their properties (dimensions, material, pierced vs. hermetically sealed lids, etc.). Thus, the selected contact force is always as high as necessary but, at the same time, as low as possible. Even thin-walled metal crucibles can be handled gently without the risk of deformation.

SafeTouch – The proper amount of contact force for each crucible type.

Gripper with piercing device and photo diode (left) for pan recognition "on the fly"

Two interchangeable pan trays plus one fixed strip yield a total of 204 positions for sample and reference pans; these are displayed here with the tray cover partially opened. Each tray can carry sample and reference pans of different types.

204 Positions for the DSC 204 ***F1*** *Phoenix*®-ASC

Calibration Materials and Reference Pans Always Ready for Operation

An additional fixed strip with another 12 positions is reserved for calibration materials or reference pans. Calibration materials can be preserved there, shielded until the next calibration runs are initiated.

Clear Sample Tray Identification

On one side of each standardized sample tray, a 2-D code is printed which encodes, for example, the serial number and facilitates identification of the tray. Additionally, the information about each position is stored in the respective code. This is especially helpful if several persons are using the same DSC but have separate sample trays in use.

Keeping Out Environmental Influences

In order to prevent sample materials from being affected by the surrounding conditions – such as humidity – if they have been in the queue for a longer time, the ASC is equipped with a tray cover. The interspace between the sample trays and the cover is purged with gas.

For unstable samples or samples with volatile components, an automatic piercing device is optionally available which opens the crucible lids shortly before the measurement starts.

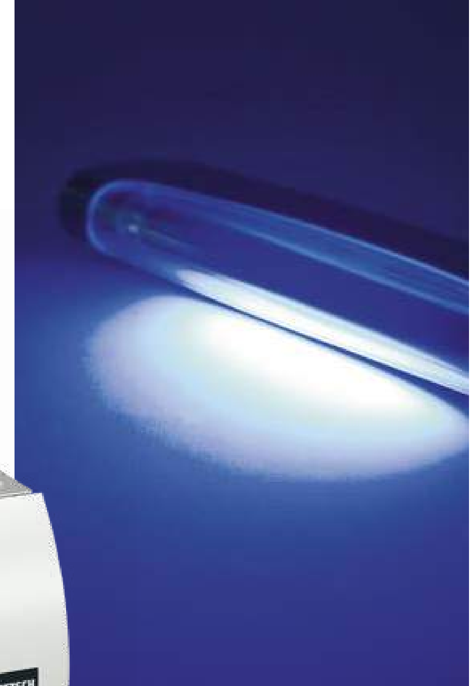


Photo-DSC 204 ***F1*** *Phoenix*® with ASC

Photo-Calorimetry – Perfect UV-Curing of Reactive Polymers

A photo-calorimeter or UV-DSC is the right instrument for investigating curing reactions which are initiated by irradiation (UV or light). In the Photo-DSC 204 ***F1*** *Phoenix*®, the light guides are mounted into a stationary position in the automatic furnace lid. This ensures that alignment of the light guides is always correct and that the distance to the sample and reference is always defined, thus providing for reproducible results.

Recommended as UV lamps are DELOLUX 04 or OmniCure® S 2000 (both are mercury lamps) with wavelength ranges between 315/320 nm and 500 nm. However, it is also possible to adapt other commercial lamps. The OmniCure lamp is fully software-controlled; the Photo-DSC system allows for the selection of temperature, atmosphere, light intensity, wave length and exposure time.

Thanks to the gas-tight construction of the DSC 204 ***F1*** *Phoenix*®, one can easily measure processes that may be sensitive to oxygen (which can act as an inhibitor), such as the cross-linking of paints.

The Benefits to You

- Analyzing photo-initiated reactions of a broad variety of materials
- Measuring the (UV) light-induced curing of polymer resins, paints, inks, coatings and adhesives
- Studying the influence of UV stabilizers in cosmetics, pharmaceuticals and foods (aging effect)

Please ask for your special copy of the Photo-DSC 204 ***F1*** *Phoenix*® brochure.

Coupling to FT-IR or MS to Detect and Identify Evolved Gases

The primary objective of DSC experiments is to investigate phase transitions or melting/crystallization phenomena, and rarely decompositions – except during oxidation tests. But in DSC measurements in which the sample remains stable, effects also occasionally occur which cannot be directly assigned, especially if the given material has not been previously investigated. In such cases, evolved gas analysis can be of great help in identifying the gaseous products. Both FT-IR (Fourier Transform Infrared Spectroscopy) and MS (Mass Spectrometry) are ideal for detecting moisture, released solvents or other volatiles.

A pre-requisite for these kinds of investigations is a gas-tight cell like the one in the DSC 204 ***F1*** *Phoenix*®. This ensures that the composition of the gas analyzed in the FT-IR or MS is identical to the gas evolving from the sample.

In case of FT-IR, the included interface (adapter plus transfer line) is optimized for Bruker FT-IR spectrometers, but not limited to them. Please ask your NETZSCH representative for more information.

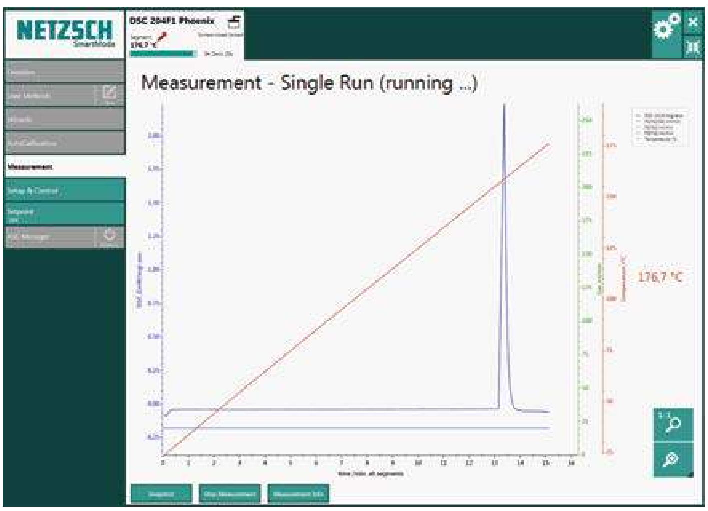
DSC 204 ***F1*** *Phoenix*® with ASC, coupled to the Bruker TENSOR II FT-IR spectrometer

ACCESSORIES

Expanding the Application Range

Proteus® Software

ALWAYS ONE STEP AHEAD



SmartMode interface during measurement

The User Comes First

The NETZSCH *Proteus*® software offers far more than ordinary measurement and analysis software for DSC instruments. Its many support features greatly assist operators in their day-to-day work. But users have full control at all times and can decide whether to go the traditional route, the software-aided route or a combination of the two.

SW-Features

- ■ *SmartMode*
- ■ *ExpertMode*
- ■ *AutoCalibration*
- ■ *AutoCooling*
- ■ Advanced DSC-*BeFlat*®
- ■ *AutoEvaluation* (see next pages)
- ■ *Identify* (see next pages)
- □ TM-DSC (temperature-modulated DSC)
- □ ASC (Automatic Sample Changer) support
- ■ Specific heat capacity (c_p) determination
- ■ Report Generator
- □ Purity
- □ *Peak Separation*
- □ *Thermokinetics*

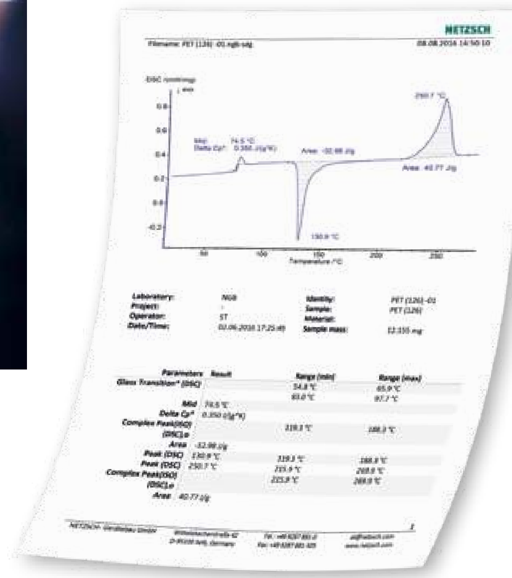
■ included □ optional

SmartMode and *ExpertMode* – Giving You the Flexibility You Need

Due to rapid development in electronics, the timelines of embedded display formats are often quite short. By using *SmartMode*, however, it is possible to have the same intuitive user interface on a tablet, a larger touch monitor or a regular PC – whatever you prefer.

For anyone who favors a more classical user interface or wants access to the entire range of functions afforded by the *Proteus*® software, *ExpertMode* is the solution.

Real-time operation of the software allows for the display of running curves in both modes and – if desired – presents the evaluation immediately when the measurement is finished. In order to start a test run, one may open either wizards (quick-start routines in *SmartMode*), predefined methods (e.g., related to polymer types in *SmartMode*) or user-defined methods (in both *SmartMode* and *ExpertMode*).



Report Generator to Meet Various Needs

Based on a Microsoft Word plug-in each operator can easily create his own report template – including logos, tables, description fields and plots. As an entry several report examples are already delivered together with the *Proteus*® software.

Ideal Flat Baselines Thanks to Advanced *BeFlat*®

Due to material and technical limitations, each DSC sensor has some imbalances which will have an impact on the shape of the corresponding DSC baseline.

Advanced *BeFlat*® is a method to compensate all these influences in a DSC 204 ***F1*** *Phoenix*® by performing just two measurements (one with a pan only on the reference side and a second one with two empty pans). The result are horizontal DSC baselines with minimal deviations in the µW range.

Temperature-Modulated DSC – TM-DSC

In TM-DSC the underlying linear heating rate is superimposed by a sinusoidal temperature variation. The benefit of this procedure is the chance to separate overlapped DSC effects by calculating the reversing and the non-reversing signals. The reversing heat flow is related to the changes in specific heat capacity (→ glass transition) while the non-reversing heat flow corresponds to temperature-dependent phenomena such as curing, dehydration or relaxation. An example is given on page 19.

ASC Interface: Instrument-Independent Database for Measurement Definitions

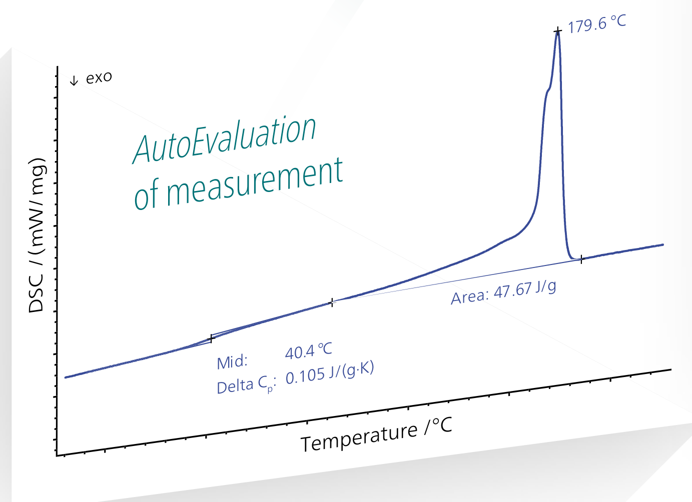
In the ASC mode, each sample definition is clearly assigned to the position of the particular sample pan inside the sample tray. As a result, it is possible to take this filled tray along and to apply it to any DSC 204 ***F1*** *Phoenix*® in the internal network which is currently not in use.

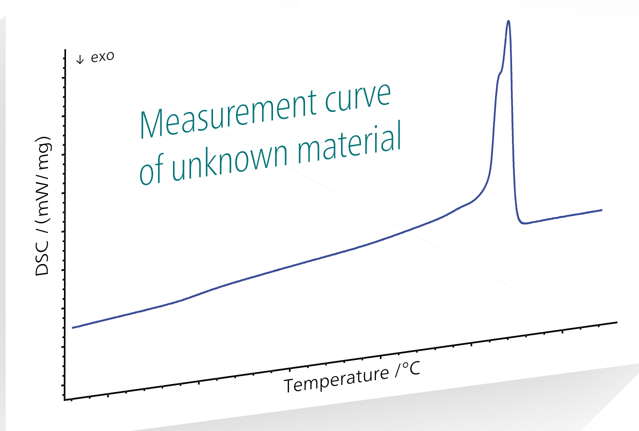
Just like Magic

AutoEvaluation

AutoEvaluation is the first self-acting evaluation routine for DSC curves on the market. Without user-intervention it is able to detect and to evaluate fully automatic glass transition temperatures, melting temperatures or melting enthalpies of unknown polymers or pure metals. Experienced users can take the automatic evaluation result as a second opinion – and, of course, recalculate values if desired.

one CLICK RESULTS



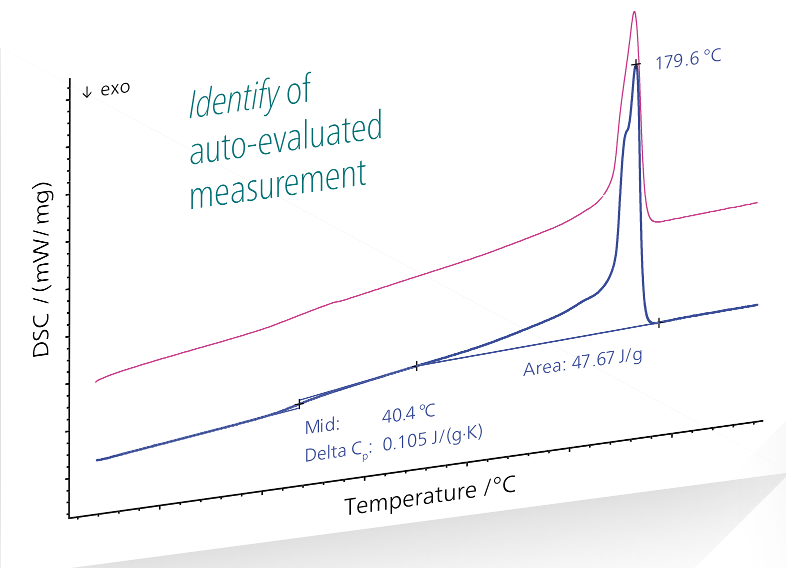


User-independent!

No evaluation macro, no selection of evaluation limits or thresholds!

Identify

- Is a unique curve recognition and interpretation system
- Includes a database with NETZSCH libraries and libraries that can be created by the user
- Manages measurements, literature data and classes



Results

Database Entry	Similarity
PA12_DSC	99.54 %
PA11_lit	94.27 %
PA11_DSC	94.00 %
PA12_lit	91.55 %

The Curve Comparison Database

Identify

Identify is a unique software package within the thermal analysis field for the identification and classification of materials via database comparison. Besides one-on-one comparisons with individual curves or literature data, it also checks if a particular curve belongs to a certain statistical class are feasible. These classes can consist of curves of the same material type (material identification) but also of reference curves for Pass/Fail testing (quality control).

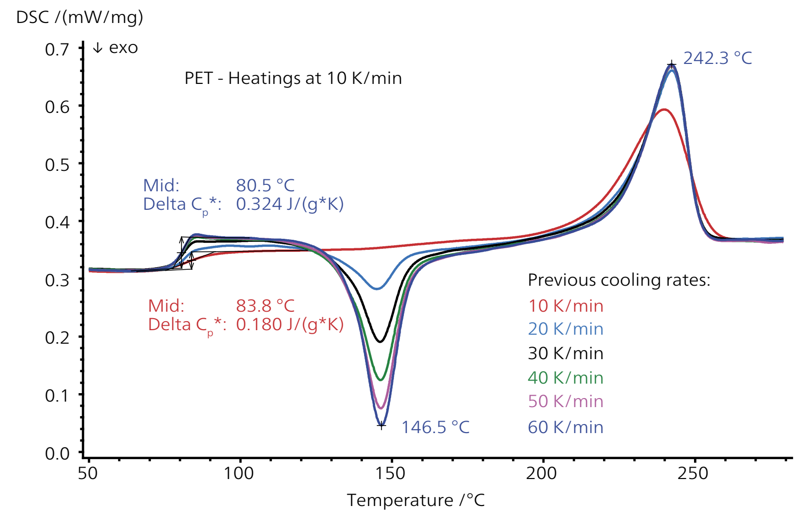
The provided NETZSCH libraries contain more than 1100 entries related to different application areas such as polymers, organics, inorganics, metals/alloys or ceramics.

The shown similarity hit list is the result of applying *Identify* to the measurement example displayed above. The highest similarity is attributed to PA12 (polyamide 12). The similarity value of 99.5% reflects the excellent conformity with the reference curve (pink).

APPLICATIONS

Discovering the Crystallinity and/or Amorphousness of Polyethylene Terephthalate (PET)

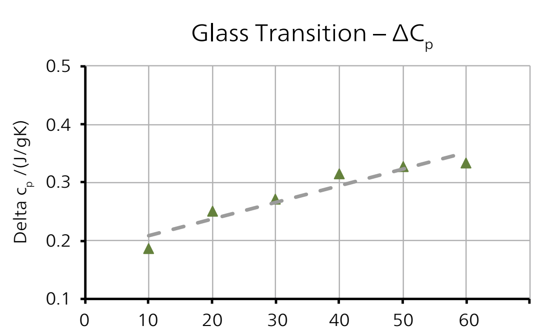
PET is a widely used semi-crystalline thermoplastic polymer. It shows post-crystallization during heating if the previous cooling was fast enough. In the DSC experiments displayed here, various glass transition steps with different heights and midpoints from 80°C to 84°C as well as various recrystallization peaks at 147°C and melting effects with peak temperatures of 242°C are apparent. The areas of the melting and post-crystallization peaks are related to the material's crystallinity; the glass transition steps are a measure for its amorphousness. Depicted are several reheating steps at 10 K/min after cooling down the sample with cooling rates between 10 K/min and 60 K/min .

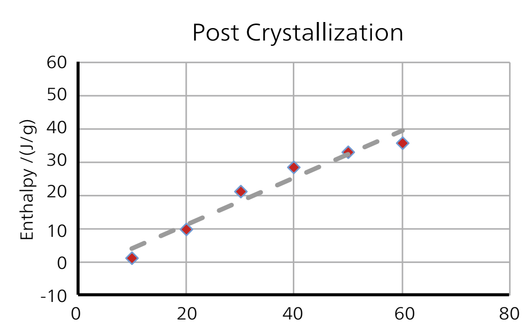


DSC measurement on PET – reheatings after cooling steps at various cooling rates; sample mass: 5.49 mg, heating rate: 10 K/min; nitrogen atmosphere

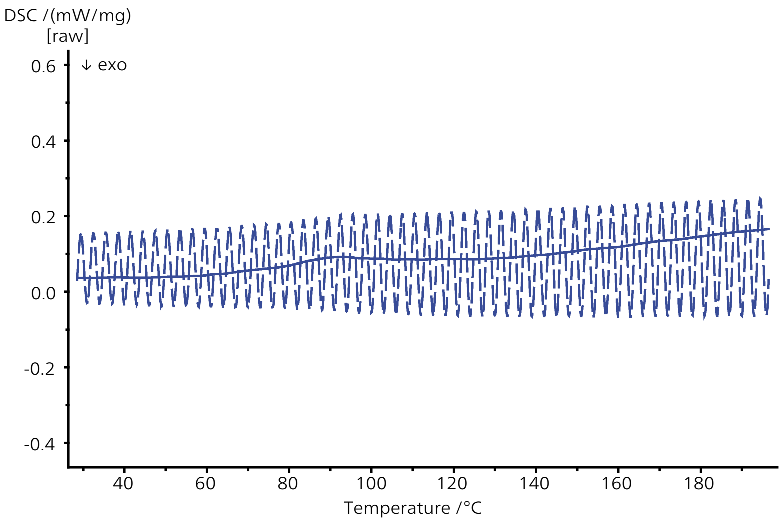
The cooling rate influences the opacity/transparency of PET bottles.

Plotting the step height (Δc_p) of the glass transition as well as the enthalpy of the post-crystallization peak versus the cooling rate yields in more or less linear relationships.





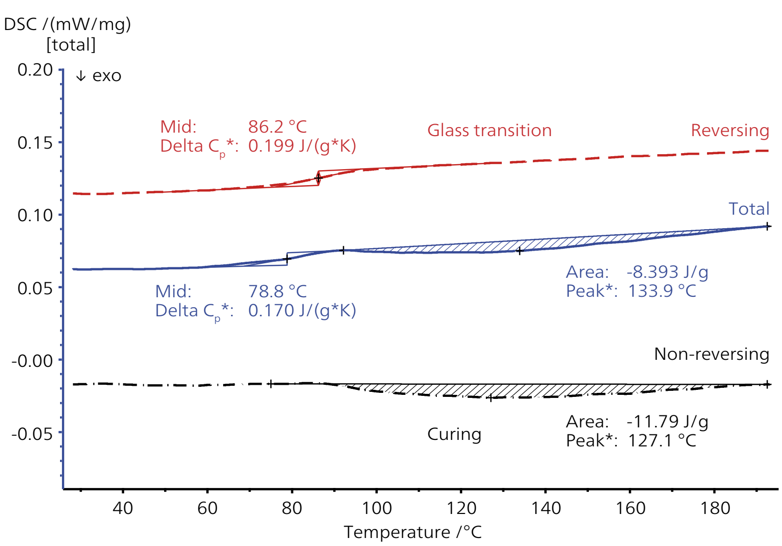
Separation between Glass Transition and Post-Curing of a Carbon-Fiber Reinforced Polymer Sample



Post-curing of a CFRP (carbon-fiber reinforced polymer) by using TM-DSC; resulting modulated DSC signal (dashed line) plus total DSC curve (average, correlates to a conventional DSC measurement, solid line)
Sample mass: 13.45 mg, underlying heating rate: 3 K/min, period: 60s, amplitude: 1 K, atmosphere: nitrogen

The total DSC curve (blue solid line in both graphs) illustrates an overlapping between the glass transition step and the broad exothermic post-curing peak as it can also be detected during a conventional DSC measurement. By using TM-DSC, however, both effects can be separated from each other. The glass transition is visible in the reversing DSC signal (graph below, red dashed line); the curing in the non-reversing DSC signal (black dashed-dotted line).

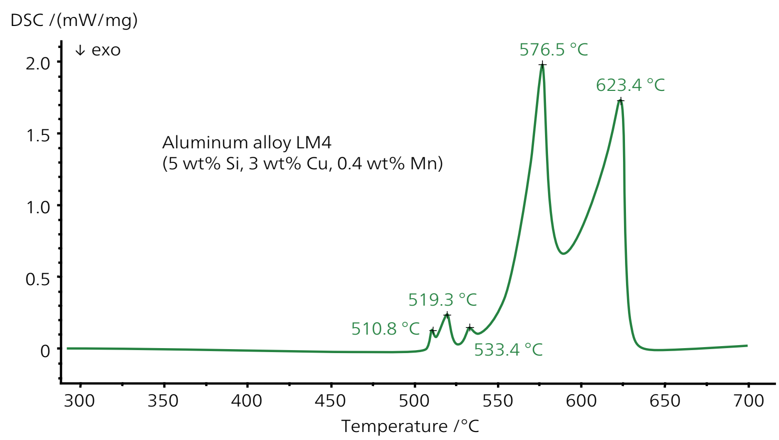
Additionally, the graphic below shows a clear difference between the results evaluated concerning glass transition and exothermic curing effect on the total DSC curve and on the reversing/non-reversing curves.



1st heating of a CFRP (carbon-fiber reinforced polymer) by using TM-DSC; same measurement parameters as above

Visualizing the Complex Melting Behavior of Alloys with High Resolution

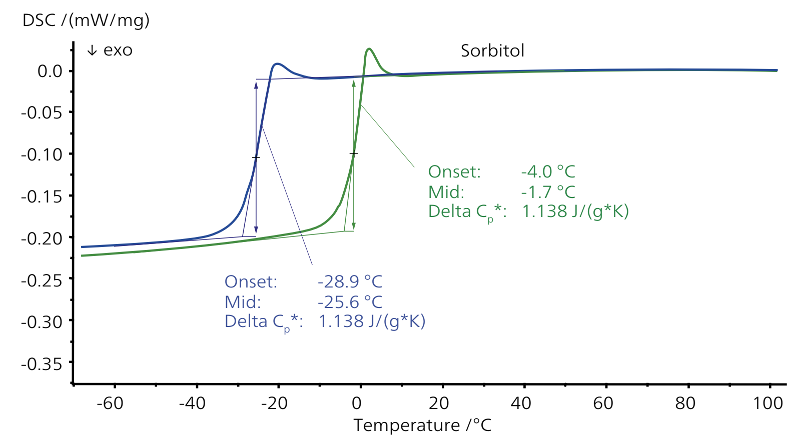
When analyzing metal alloys, it is important that there is a good separation of the melting peaks for the individual alloy components. The DSC 204 ***F1*** *Phoenix®* with τ-sensor yields an excellent peak separation in the melting range from 510°C to 650°C for the metal measurement shown here (peak temperatures at 511°C, 519°C, 533°C, 577°C and 623°C). For comparison: Pure aluminum exhibits a melting point at approx. 660°C.



DSC measurement on an aluminum alloy containing 5 wt% silicon, 3 wt% copper and 0.4 wt% manganese

Investigating the Influence of Moisture on the Glass Transitions Temperature of Sorbitol

Sorbitol is used as a sugar substitute in many sweets, diet products and medications. A proportion of 10% water in anhydrous sorbitol causes a decrease of the glass transition temperature of approx. 24 K (mid temperatures). Both samples remain completely amorphous after the rapid cooling from the molten state (which took place prior to the displayed heating step).

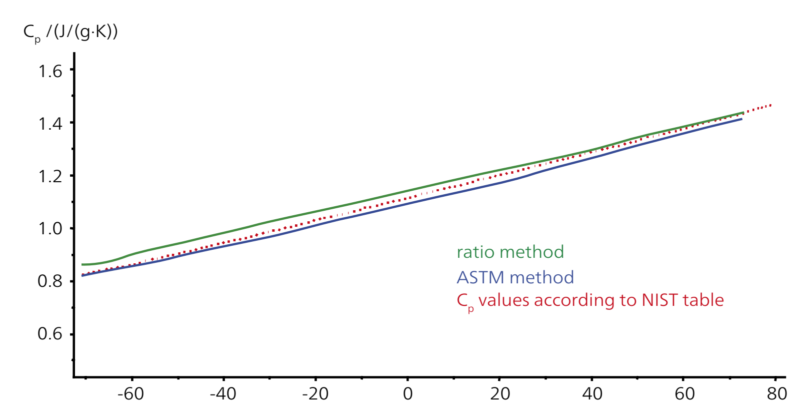


DSC measurement on sorbitol
Sample mass: 12.0 ± 1 mg, heating rate: 10 K/min, atmosphere: nitrogen

SPECIFIC HEAT CAPACITY – c_p
represents the amount of heat needed to increase 1 g of material by 1 K

Accurate Determination of the Specific Heat Capacity of Polystyrene

The ability to determine specific heat capacity is an important task for a DSC. Using the DSC 204 ***F1*** *Phoenix*® a mean error of < 2% was attained on NIST Standard Reference Material 705a, polystyrene (PS) with narrow molar mass distribution. The heating rate amounts to 10 K/min.



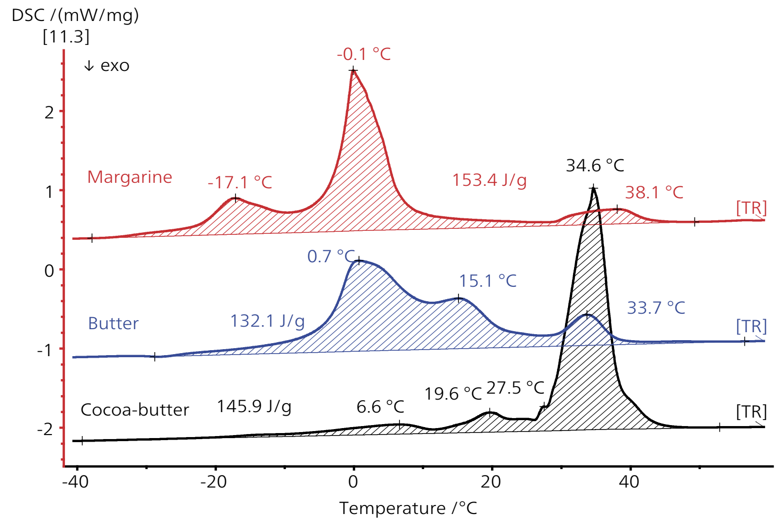
DSC measurement on polystyrene (NIST SRM 705a)

Verifying the Spreadability of Edible Fats

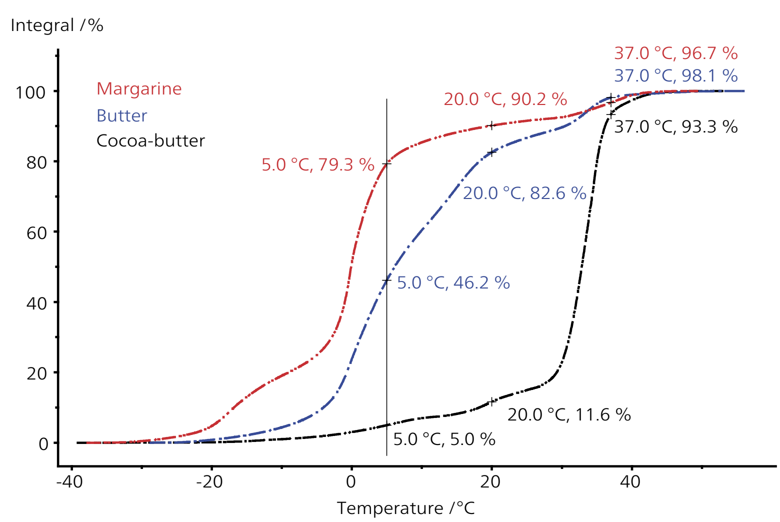
The plot on the right side depicts the melting behavior of margarine (red), butter (blue) and cocoa butter (black). The melting of margarine starts at lower temperatures compared to the two other samples. This is also reflected in the graphic below which presents the integral of the different melting areas versus temperature.

At approx. 5°C (refrigerator temperature, marked with a black line) the already molten part is 79% in case of margarine, 46% for butter and just 5% for cocoa butter. This explains why margarine is better spreadable if just taken out of the refrigerator than – for example – butter.

Hence, the so-called Solid Fat Index (SFI) at 5°C is 21% (100% - 79%) for margarine, 54% for butter and 95% for cocoa butter.



DSC measurements on margarine, butter and cocoa butter
Sample masses: 9.5 mg, 10.3 mg and 9.8 mg; heating rate: 10 K/min, nitrogen atmosphere, *Concavus* pans



Integral of the various melting areas (degree of conversion) from margarine, butter and cocoa butter (see graphic above) plotted as a function of temperature

DSC 204 ***F1*** *Phoenix*®	
Furnace Type	Silver Furnace
Temperature range (max.)	-180°C to 700°C
Cooling rate / heating rate	0.001 to 200 K/min
Measuring range (max.)	± 750 mW (τ-sensor)
Enthalpy accuracy	< 1%*
Precision of specific heat capacity determination	< 2 to 3% (for sapphire, RT to 500°C)
Exchangeable sensors	▪ τ-sensor (-180°C to 700°C) ▪ µ-sensor (-150°C to 400°C)
Cooling options	▪ Air compressor: RT to 700°C ▪ Compressed air: <0°C to 700°C (Vortex) ▪ 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 gas	3, integrated (0 … 250 ml/min)
Gas flow regulation	Software-controlled
Automatic sample changer (ASC)	Optional, for up 192 samples + up to 12 calibration samples
Evolved Gas Analysis	MS and/or FT-IR, possible with ASC
Photocalorimetry (optional)	UV extension for various commercial lamps, possible with ASC
Proteus® software (included features)	▪ *SmartMode* ▪ *ExpertMode* ▪ *AutoCalibration* ▪ *AutoCooling* ▪ *AutoEvaluation* ▪ *Identify* ▪ Advanced *BeFlat*®
Proteus® software extensions (optional)	▪ Temperature modulation, TM-DSC ▪ *Purity*
Advanced software extensions (optional)	▪ *Peak Separation* ▪ *Thermokinetics* ▪ *Thermal Simulations* ▪ *Component Kinetics*
Size (W x H x D) - incl. ASC and physical connections	approx. 62 cm x 42 cm x 58 cm

* for metals

The system works in line with all relevant DSC standards (DIN, ISO, ASTM).

Technical Specifications

The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 3,700 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

Our performance standards are high. We promise our customers Proven Excellence – exceptional performance in everything we do, proven time and again since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, 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.

Proven Excellence.

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