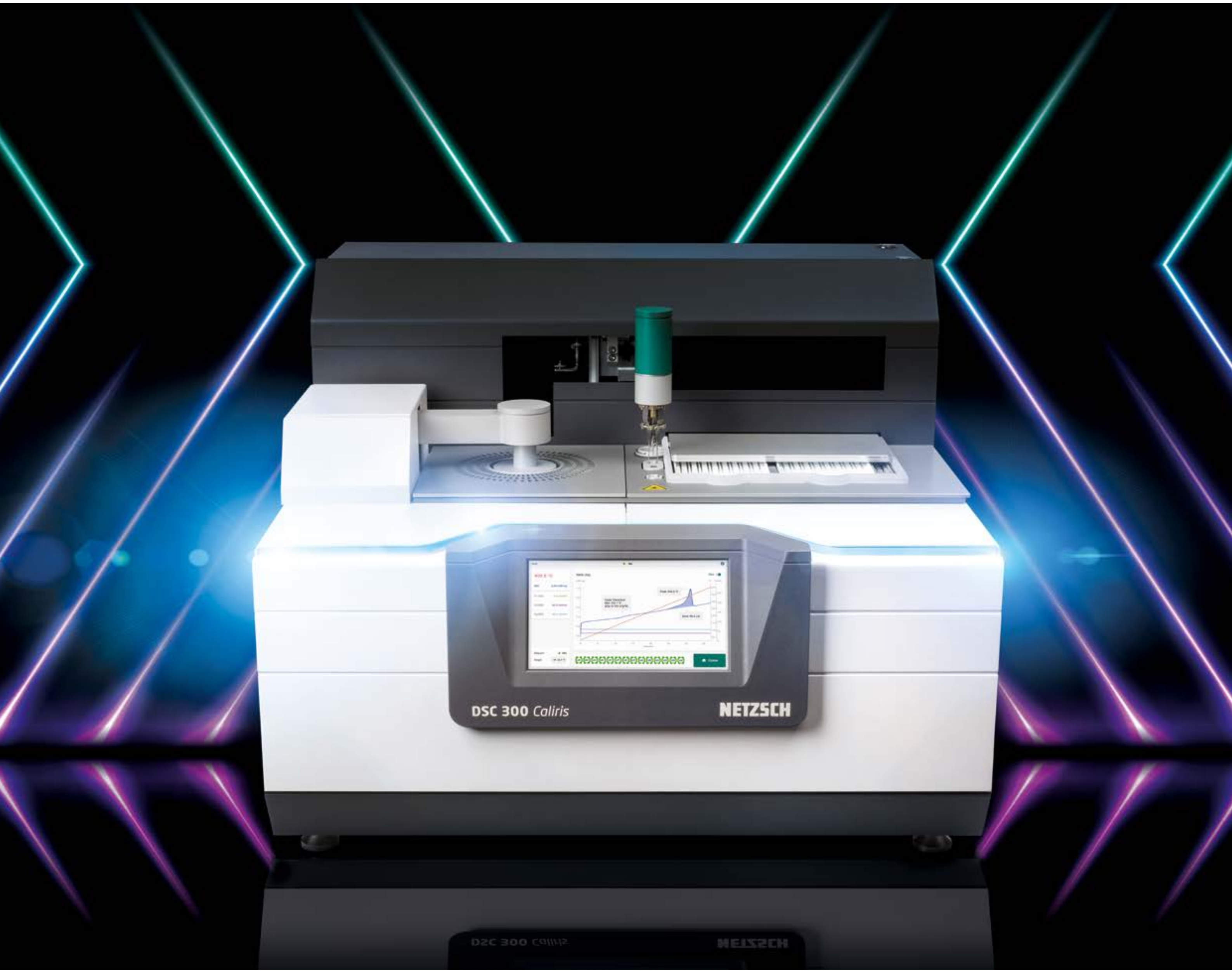
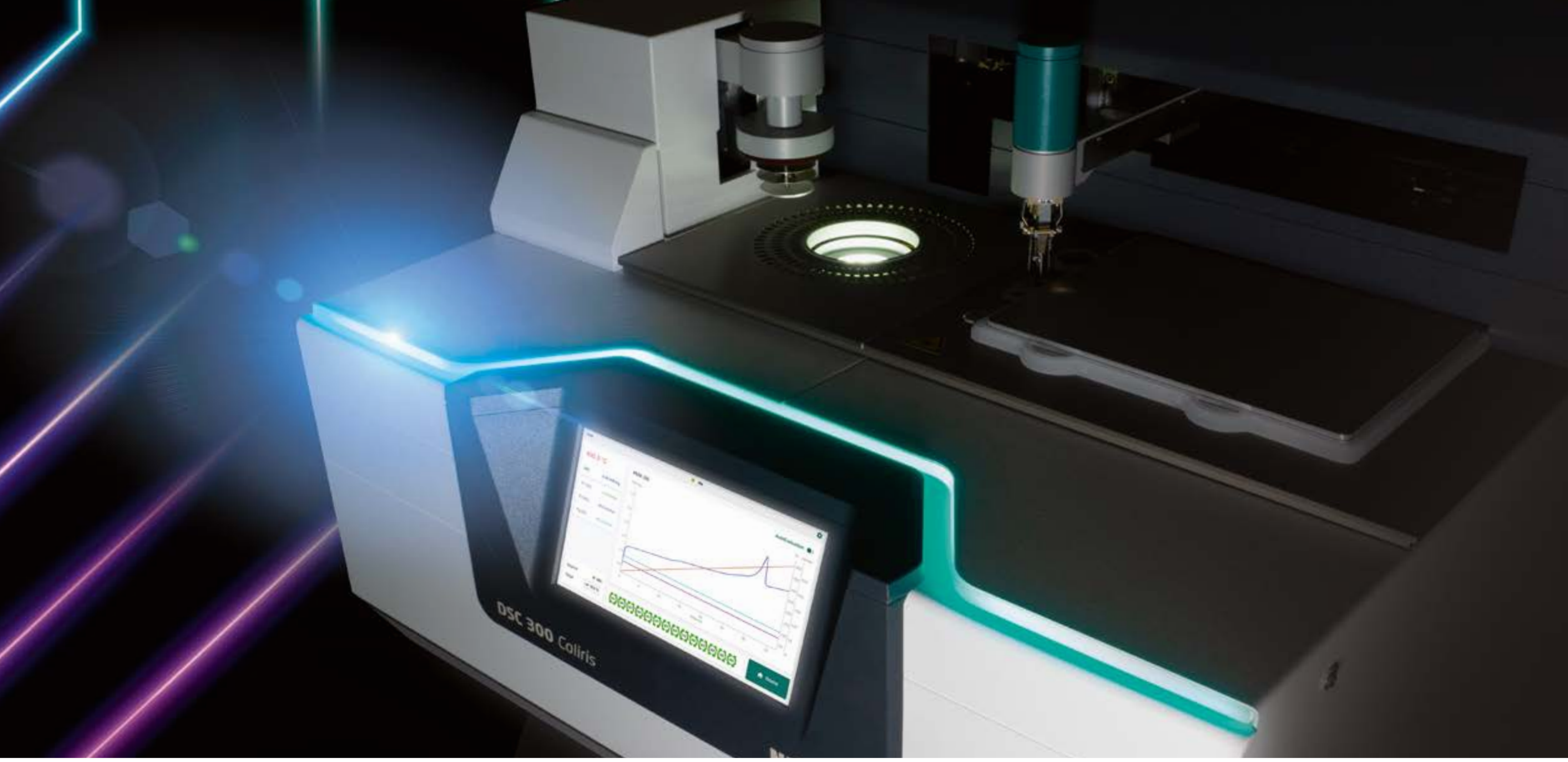
NETZSCH
Proven Excellence.



Differential Scanning Calorimetry – DSC 300 *Caliris*® *Supreme* and *Select*

Method, Technique, Applications

Analyzing & Testing



DSC 300 *Caliris*® *Supreme* and *Select*

Whether you are working in research & development, quality control, contract testing or the specification of materials for applications, information about a material's behavior under changing temperature and different atmospheres is important.

The DSC 300 *Caliris*® can support:

- Material identification
- Process optimization
- Quality control
- Phase diagrams
- Kinetic analysis
- Compatibility
- Failure Analysis

Typical DSC Results

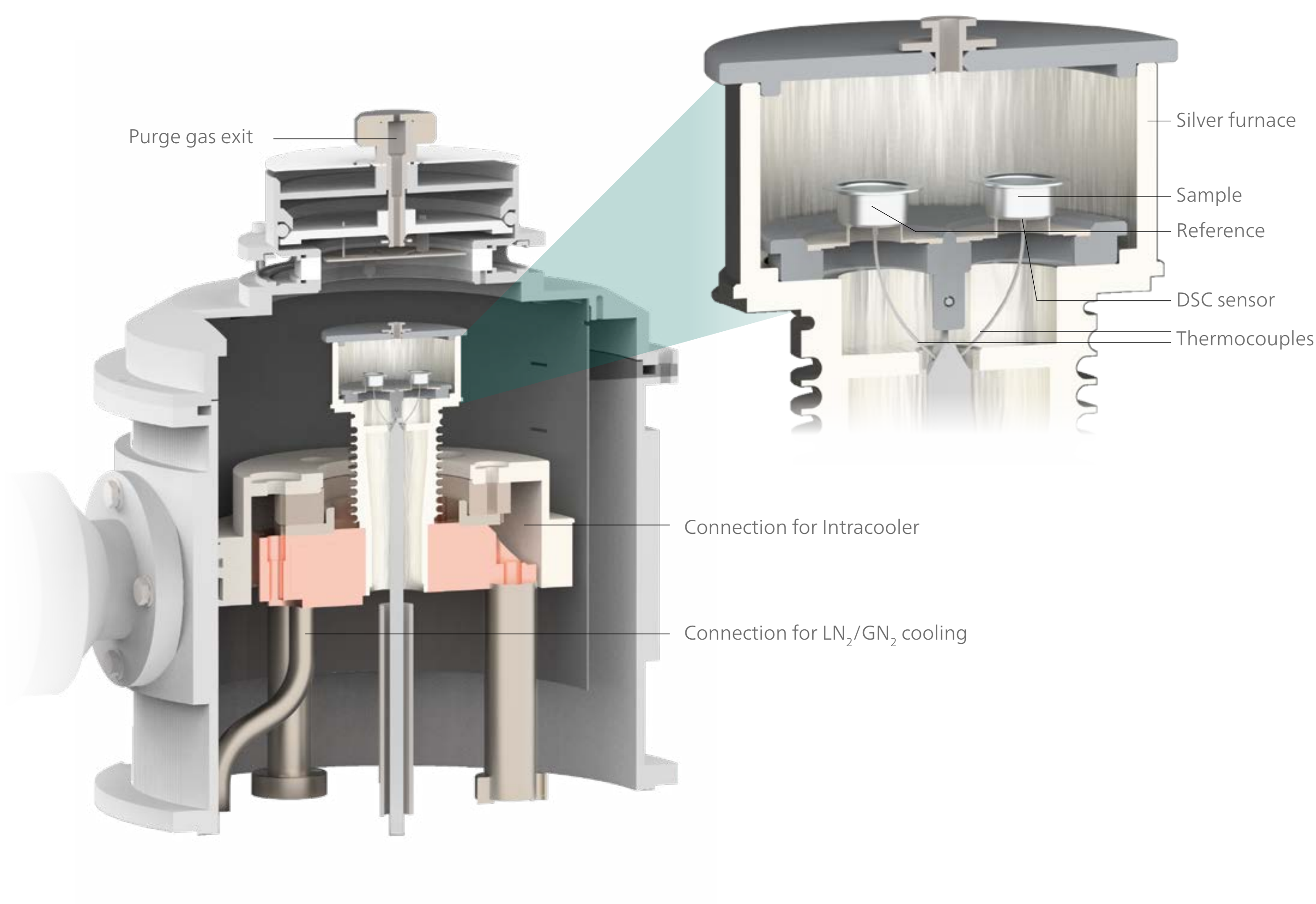
- Melting temperature and enthalpy
- Crystallization temperature and enthalpy
- Specific heat capacity
- Solid-liquid ratio (solid-fat content)
- Polymorphism
- Solid-solid transitions
- Liquid crystal transition
- Degree of crystallinity
- Curing, degree of cure
- Glass transition
- Oxidative stability
- Aging
- Purity
- Decomposition onset

The DSC 300 Caliris® is the Most Comprehensive, Most Reliable and Versatile DSC for Material Characterization on the Market!

DIFFERENTIAL SCANNING CALORIMETRY (DSC) –
The Most Widely Used Thermal Analysis Technique

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

The DSC 300 *Caliris*® is in line with all relevant DSC standards, such as ASTM E793, ASTM E967, ASTM E968, ASTM E794, ASTM E1356, DIN 51007, etc.



DSC Provides Quick, Reliable Measurement Results on a Sample's Endothermic and Exothermic Caloric Effects!

Modular Design – Change the Setup if You Need to

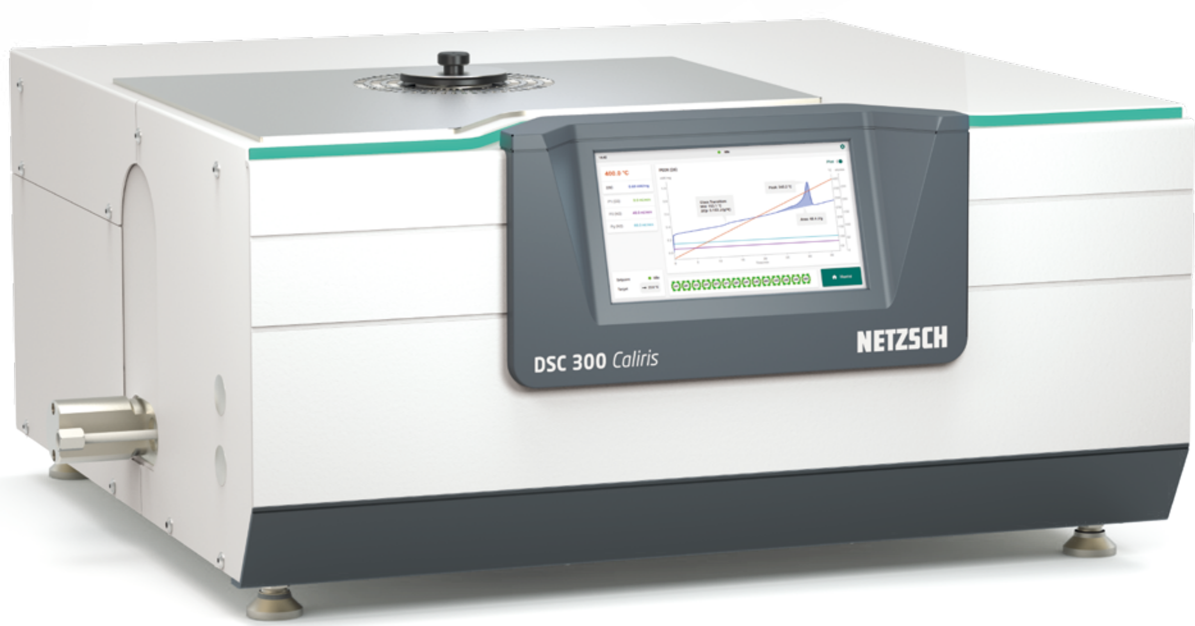
Ever-accelerating new material developments, triggered by fast-moving mobility and technical trends, require continuous adaptability. The new generation of NETZSCH DSCs is based on a modular concept. The DSC 300 *Caliris*® the only instrument of its kind with interchangeable and/or exchangeable sensor-furnace modules.

The *Supreme* version of the DSC 300 *Caliris*® allows the exchange of modules, to be able to adapt to current and future needs. Just choose between broad temperature range, fast heating rates and high sensitivity as needed, whereas the *Select* version lets you determine your module at time of purchase.

DSC 300 *Caliris*® *Supreme* and *Select*

Next Generation NETZSCH DSCs – Two Premium Instruments to Suit Every Budget and Demand

LabV®-primed



The DSC 300 *Caliris® Supreme* – The Future-Proof Choice

The only multi-module instrument on the market – making your investment truly future-proof. This instrument offers a choice of three modules and can be configured to achieve an unrivaled maximum temperature range of -180°C to 750°C. New modules to fit the DSC 300 *Caliris® Supreme* will be launched in the future and will be compatible with the current base unit. It is possible to update your device at any time to take advantage of the latest technological developments or to change your application. The choice of module you use remains unrestricted.

The DSC 300 *Caliris® Select* – Tailor-Made for Your Applications

With the *Select* version of the DSC 300 *Caliris®* initially there is a choice between modules. The maximum temperature range available in the *Select* is -170°C to 650°C. Modules of the same type can get exchanged, for example during service, to avoid down time.

Improved Status Information and Control – Even from Afar

The DSC 300 *Caliris®* offers complete information about your current measurement. You can check the general instrument status at a glance via the LED status bar. The integrated color touch display shows the important information and enables you to:

- Start measurements with the tap of a finger
- See the progress of your measurement and time remaining
- Check gases, idle states and the current temperature
- Check the execution list on recently finished and newly added measurements

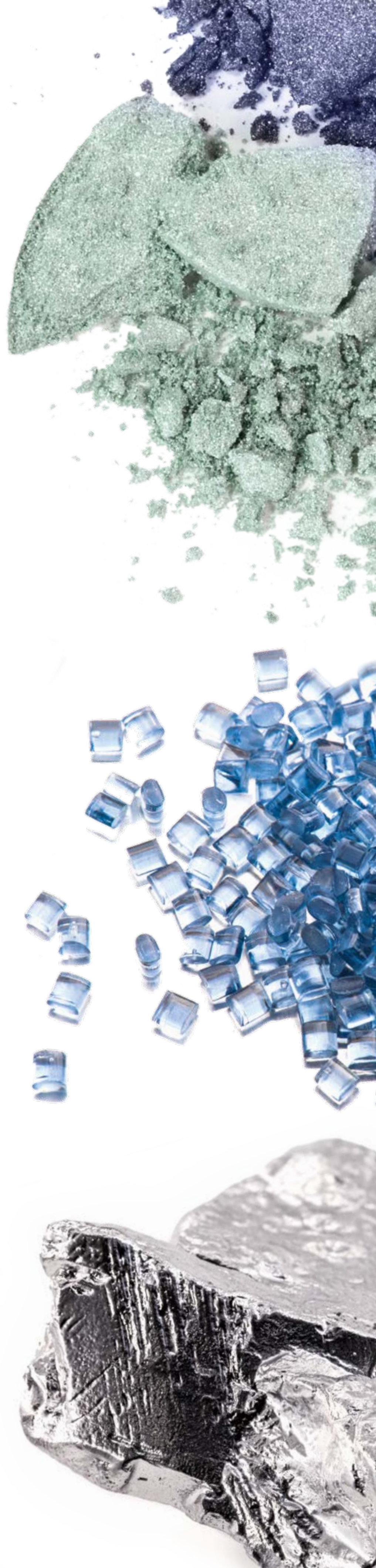
Both versions of the instrument come with integrated color touch display. They also have an LED status bar and have the option of an automatic sample changer.

Improving Your Lab Work Flow

The volume of data recorded in analytical laboratories is growing continuously. Keeping track of, and organizing, collected data to be available for future experiments or final reports is key for a smooth laboratory workflow. Also, evaluation and comparison of measurement plots can be tricky. NETZSCH offers powerful software evaluation algorithms and data-based comparison tools, making your workflows more efficient. Furthermore, the DSC 300 *Caliris®* comes with perfect connectivity and is LabV®-primed for sharing data between multiple locations and over different methods.

LabV®-Primed® – Taking Advantage of the Digital Lab

Using the LabV® cloud solution keeps all your lab and test data in one place, connects all your devices, irrespective of manufacturer, and creates the basis for analyses and forecasts. Keeping track of, and intelligently organizing the volume of large data, is key for optimized workflows. Predictions based on gathered data improve material quality and part performance. LabV® connects all the analysis methods, making projects more flexible and product quality predictable. The NETZSCH DSC 300 *Caliris®* comes LabV®-ready, allowing for effortless implementation into the LabV® environment.



Now as Easy as Slipping on a Different Pair of Shoes

CHANGING YOUR DSC'S CAPABILITIES

The NETZSCH DSC 300 *Caliris*® currently offers a choice of three modules. Modules are furnace/sensor combinations and are compatible with the DSC 300 *Caliris*® *Supreme* and *Select* alike. The different modules change the instrument's performance.

The *Supreme* version of the instrument is fully flexible. Modules can be exchanged freely by the user in a matter of minutes. New modules are going to be available in the future, so you can make sure that the DSC 300 *Caliris*® is, and stays, the most current DSC on the market. Furthermore, with the high performance module the temperature range of the *Supreme* version is the broadest on the market at -180°C to 750°C.

The *Select* version of the *Caliris*® requires the selection of one of the modules at time of order. The maximum temperature range available is -170°C to 650°C.

Modules can be exchanged by the user in a matter of minutes.

Three Modules for Different Needs

H-Module

The High-Performance Module

Supreme: -180°C to 750°C
Select: - 170°C to 650°C

The premium module impresses with a perfect baseline and outstanding reproducibility. The very small peak-to-peak noise ratio allows the detection even of the smallest peaks. It is the gold standard for most DSC applications. In combination with the *Supreme* version, this module offers an short time constant with a simultaneously high sensitivity and, on top of that, covers the entire available temperature range from -180°C to 750°C. The H-modue also offers an illuminated measuring cell for easy placement of crucibles and to ensure the sensor is clean.

The H-Module and its sensitive H-sensor is an ideal complement for advanced materials research and development in both industry and academia.

P-Module

The Polymer Module

-170°C to 600°C

This module is perfect for all tasks in the polymer field. Its optimized low-mass furnace allows for heating rates of up to 500 K/min over a wide measurement range. Temperature profiles simulating real processing conditions can be realized. Additionally, one can speed up the measurements and thus save valuable time.

The P-Module is perfect for research and development or quality control in the polymer processing industry.

S-Module

The Standard Module

-170°C to 600°C

This module combines high stability and optimized resolution of thermal effects. Laser-guided welding processes for the sensor disks and thermocouple wires yield true sensitivity and robustness. The monolithic DSC sensor features high metrological stability and optimal resolution.

The easy to handle S-Module is the module of choice for industry and contract laboratories when routine measurements are the main task.

Measurement Update in Passing – LED Status Bar

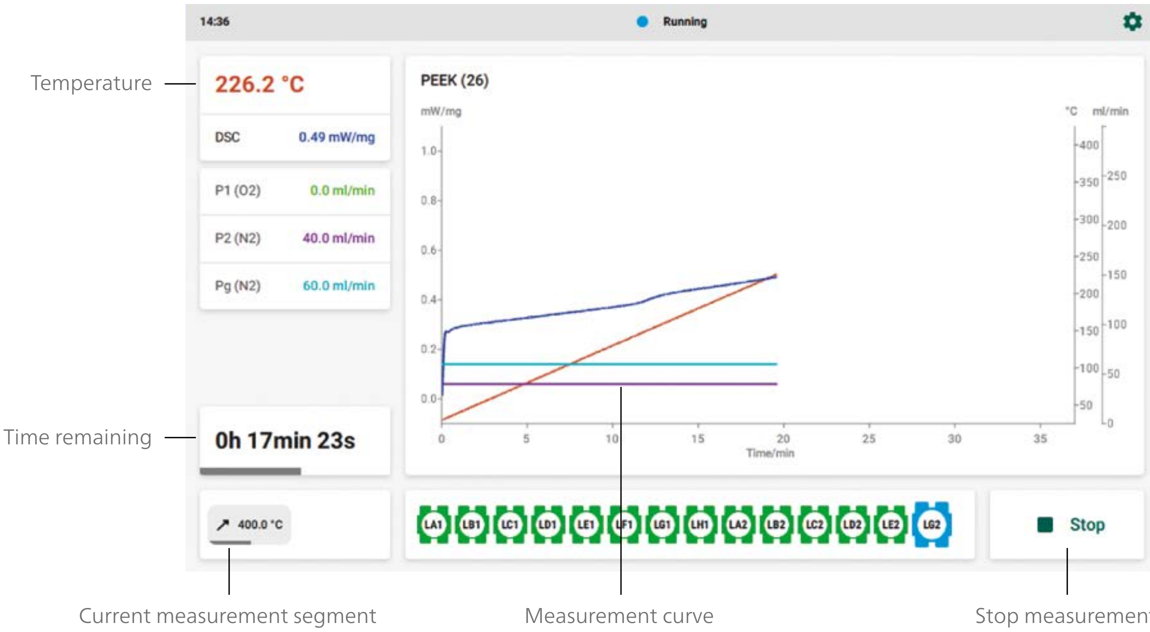
The DSC 300 *Caliris*® provides an LED light bar that allows you to check the status of your instrument as you walk by, with different colors representing different statuses. It is reassuring to see from afar, without having to log into your PC, that your measurement is running smoothly and to be able to read instrument status notifications such as:

- Instrument is ready
- Measurement is running
- Measurement progress
- Heating/Cooling to setpoint
- User interaction needed
- A problem occurred

Improving Your Productivity and Workflow Using the New User Interface

The integrated color display allows you to start a measurement that was previously prepared in the NETZSCH *Proteus*® software. Just touch the prepared measurement button on the display and you will be informed about the setup of the measurement. This moves the final check before you start a new measurement directly onto the instrument. The color touch display offers:

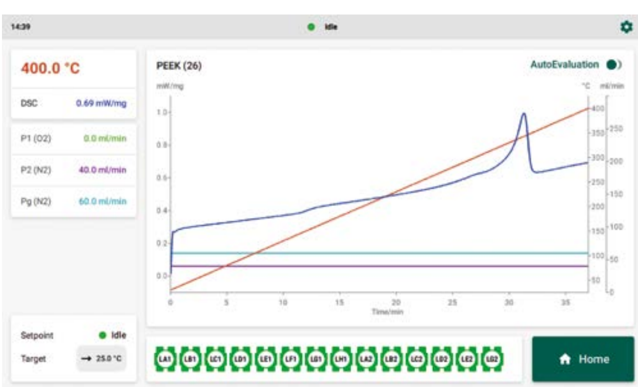
- Start measurements by the touch of a finger
- Follow measurement progress
- Check recently finished measurements
- See the progress of your measurement and time remaining
- Check gases, idle state and current temperature
- Get an immediate overview of the evaluated measurement



Control over your measurements without logging into a PC

AutoEvaluation: Objective Results Available After the Measurement Has Finished

If *AutoEvaluation* has been activated in the measurement setup, the measurement data will be evaluated immediately and objectively within the blink of an eye. An objective evaluation of the measurement curve will be available in an analysis window after the measurement has finished. The original plot will still be accessible.

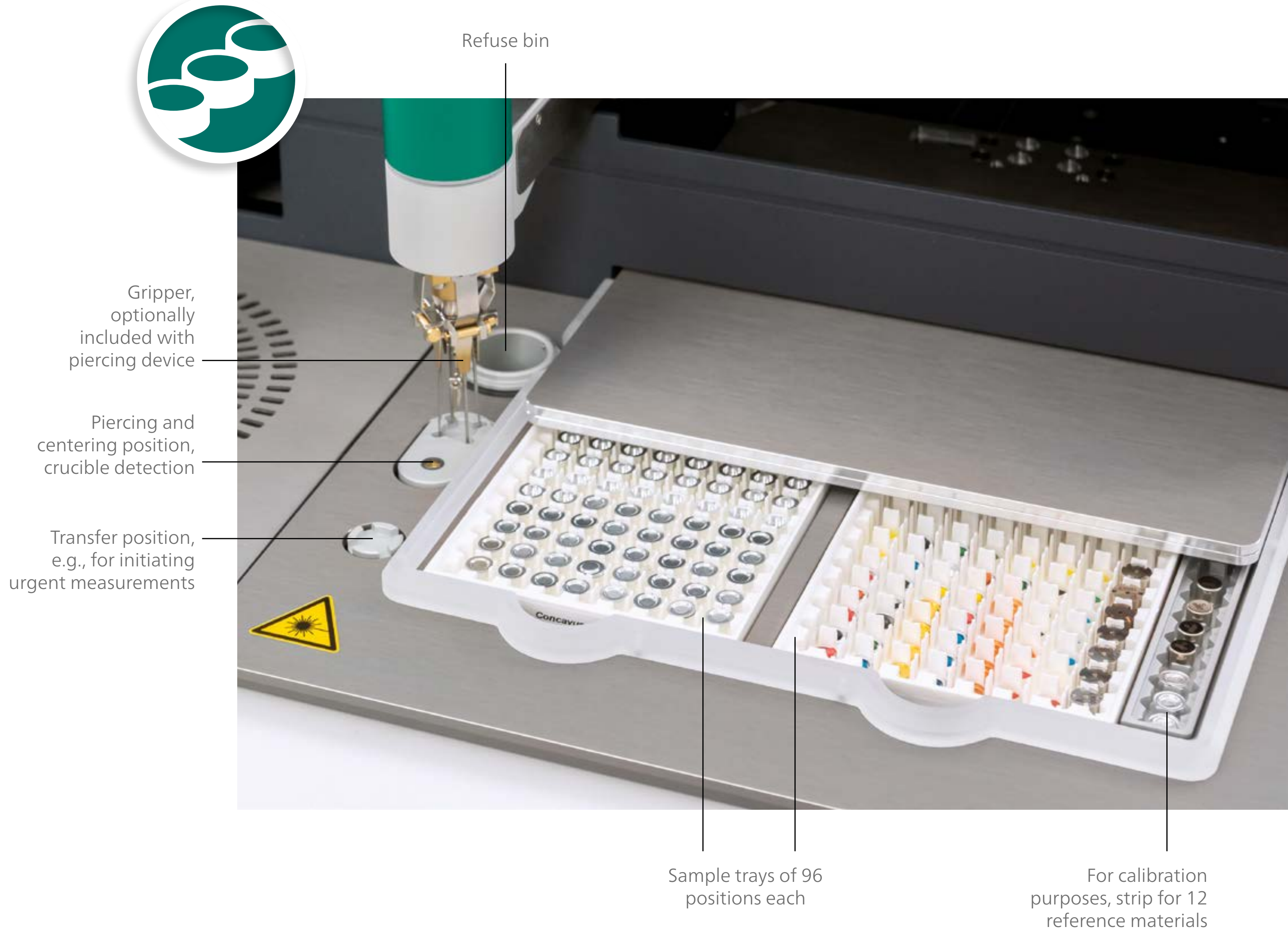


Measurement plot, current temperature and gases are displayed during measurement

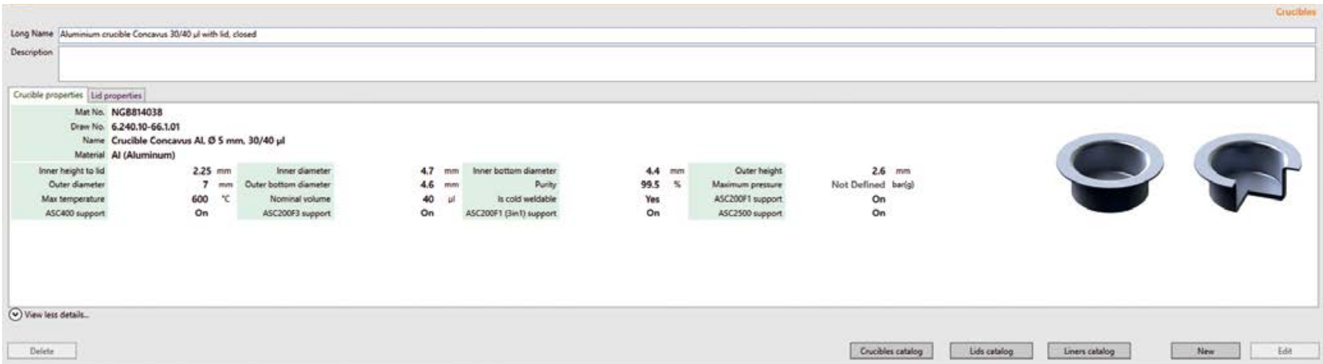
When *AutoEvaluation* is defined, the autonomous evaluation is available on the display after the measurement.

The DSC 300 *Caliris®* – Designed to Keep the User Thoroughly Informed and in Full Control

Unique Automatic Sample Changer, ASC, Available with the DSC 300 *Caliris*® *Supreme* and *Select*



Improving Your Efficiency with the Support of a Dependable Sample Robot



Crucible database defining the contact force for the gripper. Customer-specific crucibles can be added.

Removable Sample Trays Making Preparation and Storage Easy

The DSC 300 *Caliris*® with ASC is designed to hold two interchangeable sample trays in microplate format, each holding 96 samples. This allows for the clear assignment of the samples when they are prepared away from the instrument. On one side of each standardized sample tray, a 2-D code is printed which identifies the tray. This is especially helpful when several people are using the same DSC but have separate sample trays in use.

SafeTouch ASC Gripper – Always in Good Hands

The *SafeTouch* functionality ensures that every crucible type is treated with the ideal contact force. The appropriate contact force is automatically deduced from a comprehensive database containing all crucibles and their properties (dimensions, material, cold-welded, open, etc.). Thus, the selected contact force is always the lowest possible force required for the circumstances. Even thin-walled metal crucibles can be handled gently without the risk of deformation. The ASC gripper is able to handle every type of crucible defined in the crucible database. For unstable samples or samples with volatile components, an automatic piercing device attached to the gripper is available, as an option, which opens the crucible lids shortly before the measurement starts.

Reducing Environmental Influences while Waiting

In order to prevent sample materials from being affected by the surrounding conditions, whilst waiting in the queue – such as humidity – the ASC is equipped with a tray cover. The interspace between the sample trays and the cover is purged with gas to stop contact with unwanted atmospheres.

Great Variety of Sample Crucibles

The sample 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 crucibles in various dimensions and materials; 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 measurements on films as well as a reshaping tool for preparing SFI* crucibles, which are used for liquids (e.g., oil or wax measurements into the melt) with a high wettability.

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

* SFI = Solid Fat Index

Sealing press for different crucible types made of aluminum

Concavus® crucibles and lids, cold-weldable

Slide-in lid for *Concavus*® crucible; for demonstration purposes, the covers are colored

Medium-pressure crucible

High-pressure crucible

Crucible and lid made of platinum

Crucible made of Al_2O_3

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 LN_2 (liquid nitrogen) as well as in the GN_2 (gaseous nitrogen) mode. This helps to save coolant.

If desired, the liquid nitrogen cooling device can be connected to the DSC simulta-neously with the intracooler. This reduces liquid nitrogen consumption even further, since it is only activated at temperatures below -90°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. Many measurements using the ASC can easily run without interruption for a refill.

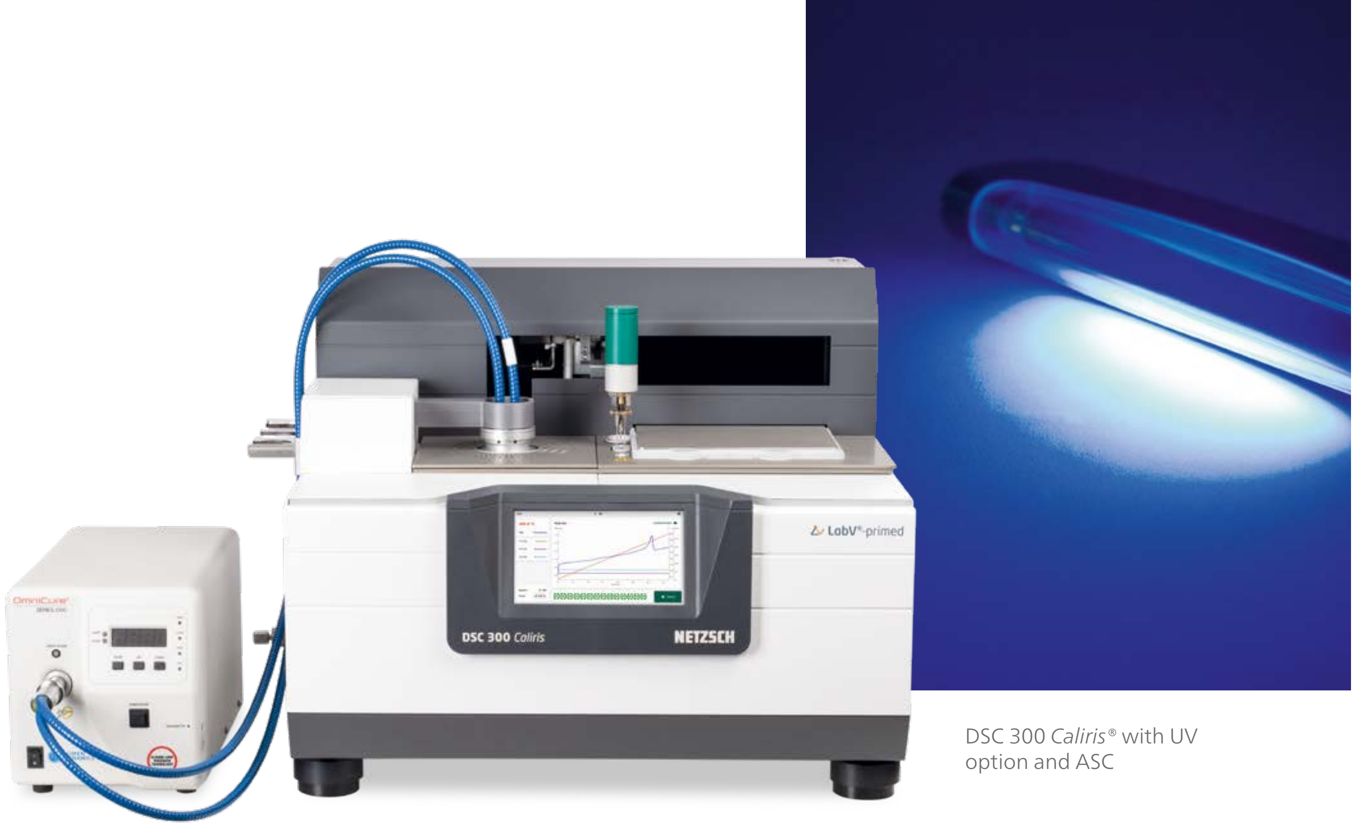
Mechanical cooling allows for a maximum temperature range of -90°C to 600°C. Liquid nitrogen cooling offers the widest temperature range of -180°C to 750°C without changing the instrument setup (e.g., furnace, lid, etc.) in the premium specification of the DSC 300 *Caliris® Supreme*.

The *AutoCooling* function of the NETZSCH *Proteus®* software automatically detects which cooling unit is present. It activates the cooling only when it is actually needed for the defined temperature program.

Temperature Range of the Modules DSC 300 *Caliris® Supreme* and *Select*

Module	LN_2/GN_2* Cooling		Intracooler	
	Supreme	Select	Supreme	Select
H	-180°C to 750°C	-170°C to 650°C	-90°C to 600°C	-90°C to 600°C
P	-170°C to 600°C	-170°C to 600°C	-70°C/-40°C** to 600°C	-70°C/-40°C** to 600°C
S	-170°C to 600°C	-170°C to 600°C	-70°C/-40°C** to 600°C	-70°C/-40°C** to 600°C

* cooling by GN_x restricted
** depending on the intracooler version



DSC 300 *Caliris*® with UV option and ASC

Photo-Calorimetry with Automatic Sample Changer – 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 DSC 300 *Caliris*® equipped with the UV accessory, the light guides are permanently installed in the automatically moving furnace lid; this allows the DSC to be immediately ready for UV measurements. It is easy to exchange the lid to switch back to conventional DSC measurements, thus covering the entire temperature range. The photo-DSC system allows for the selection of temperature, atmosphere, light intensity, and exposure time.

Recommended UV lamps*	Wave length range
OmniCure® S2000	320 nm to 500 nm
LX500	365 nm, 385 nm, 395 nm, 405 nm

* It is also possible to adapt other commercial lamps

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
- Only photo-DSC that offers an Automatic Sample Changer (ASC)

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. In such cases, evolved gas analysis can be of great help in identifying the gaseous products. FT-IR (Fourier Transform Infrared Spectroscopy) and QMS (Quadrupole Mass Spectrometry) *Aëolos* are ideal for detecting moisture, released solvents or other volatiles.

In general, DSC measurements are employed to detect thermal effects in a temperature range before the start of the decomposition of a substance. Therefore, the coupling of DSC is a particularly suitable technique for the investigation and detection of evolved gases that may already occur before decomposition (e.g., release of solvents during melting). In particular, this DSC 300 *Caliris®* coupling can be employed for samples which release substances with a boiling point below 120°C.

If the objective is to investigate gases released beyond the melting range and into the decomposition range, it is particularly suitable to couple thermogravimetry to an FT-IR or QMS *Aëolos*.

In the 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.

Coupling to FT-IR to Detect and Identify Evolved Gases

DSC 300 *Caliris®* with ASC, coupled to the Bruker FT-IR spectrometer

ACCESSORIES

Expanding the Application Range

Proteus® Software

ADAPTING TO THE USER'S NEEDS

DIFFERENT MEASUREMENT TASKS – DIFFERENT APPROACH

To allow for individual preferences, NETZSCH offers different configurations for the software interface.

SmartMode for Routine Tasks – No More, No Less

An intuitive interface for fast measurement setup, the *SmartMode* is designed especially for routine measurements as are often needed in quality control. It allows for the quick and easy preparation and start of measurements for tasks using clearly defined measurement procedures. Wizards (quick-start routines), user-defined measurement methods and pre-defined measurement methods are helpful assistants.

ExpertMode – the Sky is the Limit

This mode is designed for users preferring to access the full scope of *Proteus*® possibilities. It is perfect for advanced measurement tasks and offers infinite potential.

Workspaces – the Full Scope of *Proteus*® Analysis, but the Way You Prefer It

When working regularly with *Proteus*® Analysis, it can be overwhelming to deal with all of the available functionalities. Take advantage of *Proteus*® *Workspaces* to tailor the *Proteus*® Analysis menu and tool bar icons to your daily routine. Move all your frequently used items to the front, hide options you rarely or never use and save your preferred setup as your personal workspace. This is especially useful for workstations used by more than one person. Users can easily switch between custom and joint workspaces.

Proteus® Search Engine – Database Capability without the Need to Own a Database License

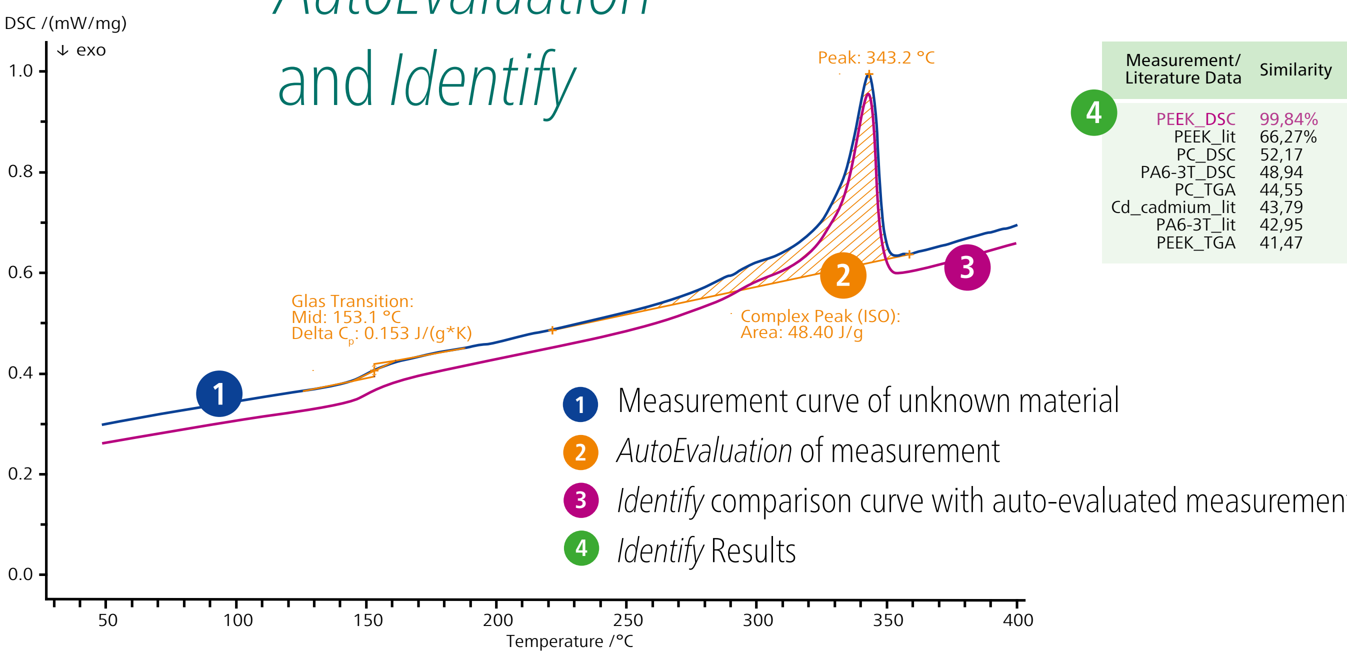
When working with measurement and evaluation data for different materials and from different measurement setups, it is enormously helpful to be able to filter by certain criteria. The *Proteus® Search Engine* filters your measurement data in a matter of seconds. It is a very powerful data mining tool.

After selection, previews of measurement curves or analysis statuses are displayed automatically. Folders in the file system can be opened simply with one click. Users can create individual searches, for example "MyPolymers", and switch easily between different existing searches.

Filters can be set by:

- File and sample name
- Remark, operator, method
- Instrument name
- File and signal type
- Date of measurement
- Measurement conditions
- Evaluated effects (glass transitions, peaks, onsets, enthalpies)

AutoEvaluation and *Identify*



Points 1 to 4 show the results of *AutoEvaluation* and *Identify* applied on a PEEK sample.

AutoEvaluation

Objective Results Seconds After the Measurement End

AutoEvaluation is the first self-acting evaluation routine for DSC curves on the market. Fully autonomously and without user intervention, it evaluates all effects such as glass transition temperatures, melting temperatures, and melting enthalpies of unknown substances. Oxidative Induction Time/Temperature (OIT) also gets evaluated for isothermal and dynamic tests, using the Tangent and Offset method in accordance with standards.

Experienced users can take the automatic evaluation result as a second opinion – and, of course, recalculate values if desired. When *AutoEvaluation* is selected in the chosen method, the evaluated curve will be shown automatically after the measurement has ended.

Report Generator

Each operator can easily create personal report templates – including logos, tables, description fields and plots. Several report examples are already included as templates within the *Proteus*® software.

Identify

Material Identification and Comparison Database

Identify is a unique software tool within the thermal analysis field for the identification and classification of measurements via database comparison. In addition to allowing one-on-one comparisons with individual curves and literature data, it can also check whether a particular curve belongs to a certain class. These classes can consist of curves of the same material type (material identification) or of reference curves for Pass/Fail testing (quality control).

The provided NETZSCH libraries contain about 1300 entries related to different application areas such as polymers, organics, pharmaceuticals, inorganics, metals/alloys or ceramics. The additionally available KIMW* database includes the DSC curves of another 1150 commercially available polymer products. Users can expand *Identify* as desired, adding unlimited amounts of their own data. In general, all database entries serve as a pool of results and useful measurement conditions.

* KIMW = Kunststoff-Institut Lüdenscheid, Germany

ADDITIONAL SOFTWARE CAPABILITIES

Temperature-Modulated DSC

In TM-DSC, the underlying linear heating rate is superimposed by a sinusoidal temperature variation. The benefit of this procedure is the ability to separate overlapping 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 time-dependent phenomena such as curing, dehydration and relaxation.

Peak Separation

Peak Separation serves for the more precise determination of individual peak areas and the temperatures of overlapping caloric effects based on selectable mathematical algorithms. This program allows for the separation of overlapping peaks, using the profiles from the following peak types: Gaussian, Cauchy, Pseudo-Voigt (additive mixture of Gaussian and Cauchy), Fraser-Suzuki (asymmetric Gaussian), modified Laplace (double-sided rounded) and Pearson.

Purity Determination

For crystalline substances with known molar mass, *Purity Determination* serves to determine the percentage of eutectic impurities on the basis of the Van't Hoff equation. The DSC melting peak is evaluated for this purpose.

Kinetics NEO

The NETZSCH Kinetics Neo software is used to analyze temperature-dependent processes. The result of such analysis is a kinetics model correctly describing experimental data under different temperature conditions. The model allows for the prediction of a chemical system's behavior under user-defined temperature conditions. Alternatively, such models can be used for process optimization.

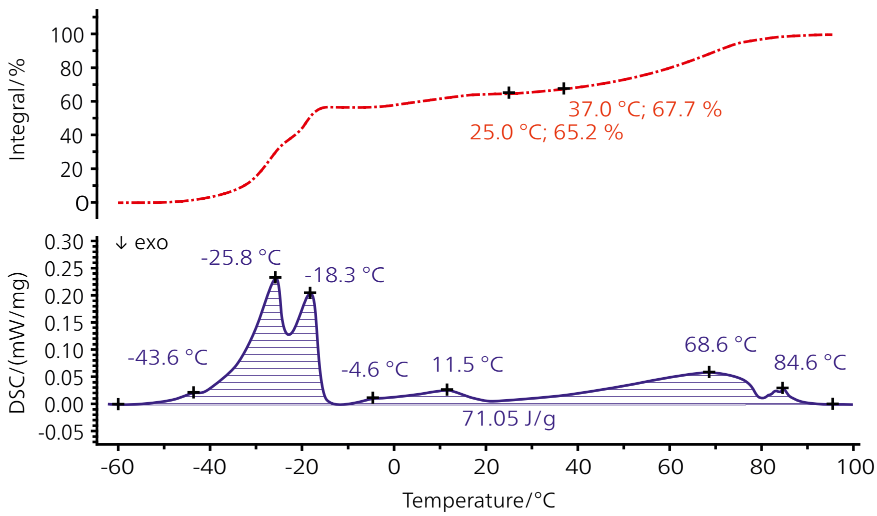
What Makes *Proteus*® Software Special

- ■ *AutoCooling*
- ■ *AutoCalibration*
- ■ Report Generator
- ■ *AutoEvaluation*
- ■ *Identify*
- □ External Polymer Database
- ■ OIT/OOT
- ◪ Temperature-modulated DSC (TM-DSC)
- ◪ Specific heat capacity (c_p)
- ◪ *Peak Separation*
- ◪ *Search Engine*
- ◪ LIMS support
- □ Purity
- □ Kinetics Neo

■ included
◪ included (DSC 300 *Caliris® Supreme* only)
□ optional

APPLICATIONS

DSC Measurements on Commercial Lipstick



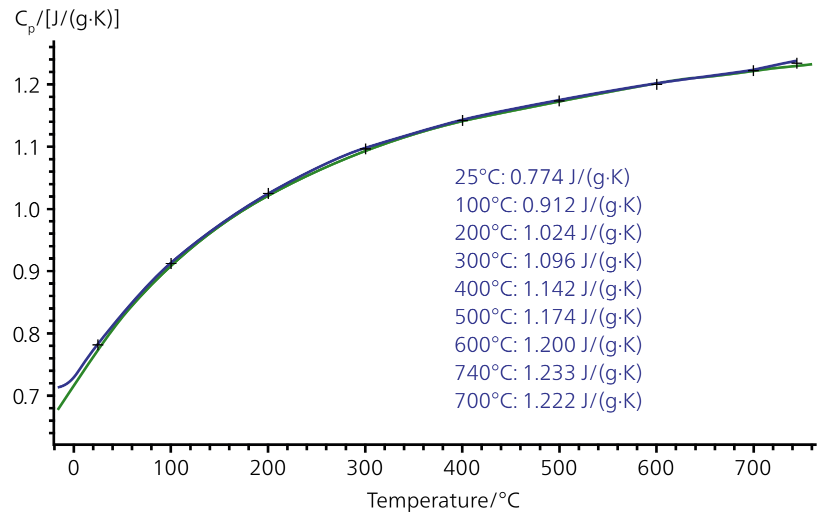
DSC measurements on a commercial lipsticks using the S-Module
Sample masses: 10.28 mg; heating rate: 5 K/min; Al crucibles, closed; nitrogen atmosphere; displayed is the 2nd heating step (blue) together with the integral of the DSC curve (red)

Lipsticks consist of various fats, waxes and oils, such as castor oil, coconut oil, carnauba wax or bee wax as well as of some cosmetic additives including emollients or color pigments. Ingredients with high melting ranges, e.g., carnauba wax which melts above 80°C, are responsible for the long-lasting property of the lipstick. Ingredients with lower melting ranges serve for smoothness and an even application.

Here, the thermal behavior of a commercial lipstick between -60°C and 100°C during its second heating step is shown. Altogether, minimum seven superimposed endothermal effects can be seen which reflect the complex formulation of the lipstick.

The melting progression is described by the additionally displayed integral curve (red). At 25°C, 65% of the mixture is already molten (liquid fraction) and 35% (= 100% minus 65%) is still solid. This corresponds in the present case to a "solid-fat-content" of 35% at 25°C and approx. 42% at 37°C (body temperature) – related to the total amount of oils, fats and waxes which are melting in the displayed temperature range.

C_p Determination of Sapphire



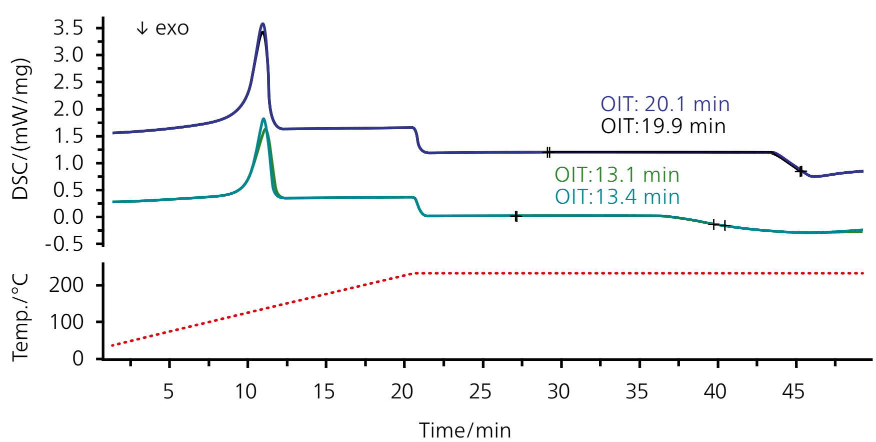
Specific heat capacity of a sapphire disk (84 mg); measurements at heating rates of 20 K/min in N_2 atmosphere (20 ml/min); measurement was carried out with H-Module

Specific heat capacity is a decisive parameter within the thermophysical properties of a material. By knowing the specific heat capacity, one can calculate the amount of energy needed to heat up a material. It is an essential parameter for many thermal simulations.

This example shows the specific heat capacity of sapphire up to 740°C. The determination was carried out in accordance with DIN EN ISO 11357-4. The blue curve represents the measured data; the green curve, literature values. The data shows a good correlation between the measurement and the literature data. Even at the highest temperature, the deviation is less than 1%.

Oxidative-Induction Time of PE

The oxidative-induction time can be determined by means of measurements. The sample is heated at a constant rate in an inert gas atmosphere above the melt. When the defined temperature is reached, the nitrogen atmosphere is switched to an oxygen or air atmosphere at the same flow rate. The sample is then held at a constant temperature until the oxidative reaction is indicated by the exothermal deviation of the DSC heat flow curve. The isothermal OIT is the time interval between the start of the oxygen or airflow and the beginning of the oxidation reaction (e.g., DIN EN ISO 11357-6).



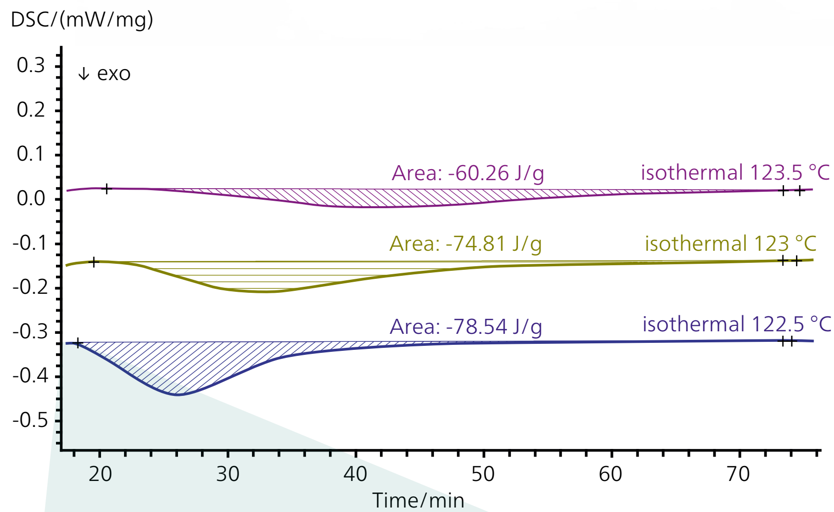
Measurement of two different PE samples conducted using the S-Module of the DSC 300 *Caliris*®. Sample mass: approx. 13 mg; aluminum crucibles: *Concavus*, open; Atmosphere: N_2/air

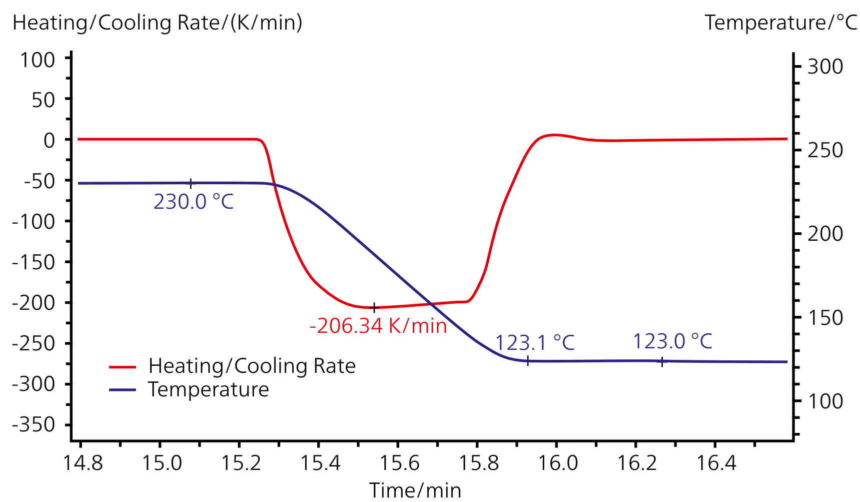
The figure shows two measurements each (for reproducibility) of two different PE-HD samples. The measurements performed here under air atmosphere indicate significant differences in the stability against oxidation. It can be concluded that the blue sample has the higher oxidative stability. This data can be used for quality control of materials and products.

Isothermal Crystallization of PE-HD

Isothermal crystallization measurements deliver deep insights into the crystallization behavior of thermoplastic materials. This information can be used to determine appropriate processing conditions.

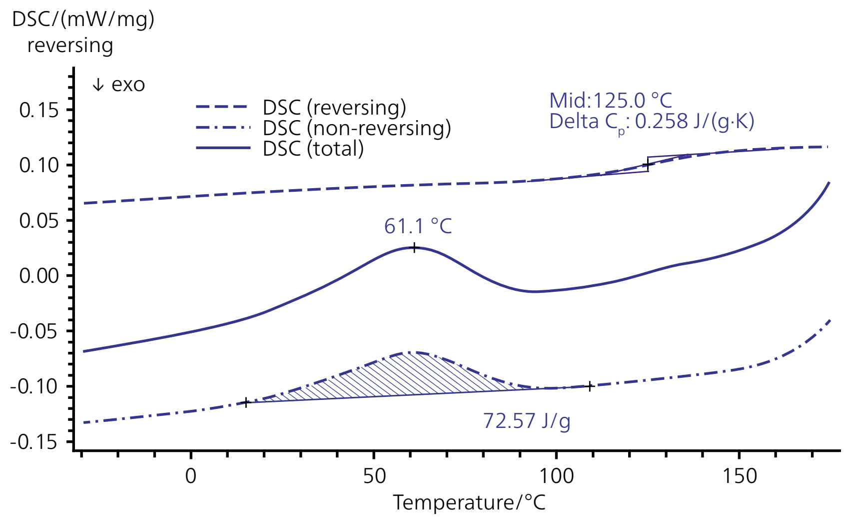
As expected, the slope of the crystallization peak is steeper with decreasing isothermal temperature, so the peak minimum is reached faster. This signifies a faster crystallization. Also, the crystallization enthalpy (peak area) increases as the temperature of the isothermal segment decreases, indicating a higher degree of crystallinity in the final product. Such measurements require a DSC, which allows for very fast cooling (see image below). This can be achieved with the DSC 300 *Caliris*® with P-Module.





Crystallization at different temperatures measured with the P-Module. Sample mass: approx. 5.5 mg,; aluminum sample pans: *Concavus* with pierced lid; atmosphere N_2.

Temperature-Modulated DSC Measurement on Eudragit® L100-55



Sample mass: 3.02 mg, underlying heating rate: 3 K/min, amplitude: ± 0.5 K, period: 60 s, crucibles: Al crucibles with pierced lid and measurement done with S-Module

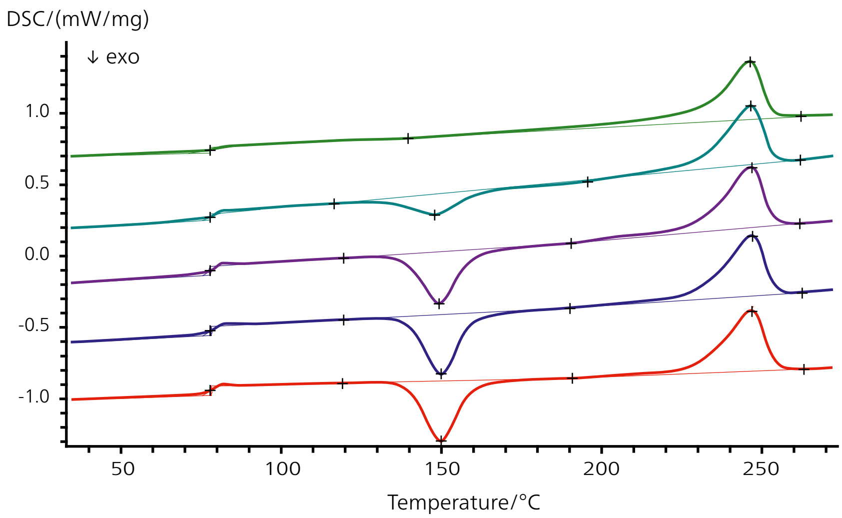
Eudragit® is the brand name for amorphous copolymers based on esters or derivates of acrylic and methacrylic acid. They are mainly used as coatings for oral dosage forms such as tablets, capsules or granules, but also as binders or scaffold creators. The functional and physical properties of the copolymers depend essentially on the monomers selected and their proportions in the polymer. This also affects the position of their glass transition temperature. The sample used in the present case is Eudragit® L100-55, which is applied in practice as an enteric coating.

During heating, several effects occur. TGA-FT-IR investigations (not shown here) revealed that the broad endothermal effect with a peak temperature of 61°C is caused by the release of water, whereas the material starts to decompose above 150°C. The question remains as to whether the effect in between represents the glass transition of the polymer.

In order to answer this, a temperature-modulated measurement was carried out. By applying a sinusoidal modulation overlaying the linear heating ramp, the total DSC signal (which corresponds to a standard DSC curve) can be split into a reversing DSC signal (dashed line) and a non-reversing DSC signal (dash-dotted line).

Time-dependent processes such as the release of water are detected in the non-reversing DSC curve. The reversing signal contains only information which is related to the change in heat capacity – i.e., to the glass transition of Eudragit® – and allows in this way for its accurate evaluation.

Influence of Cooling on the Crystallinity of PET



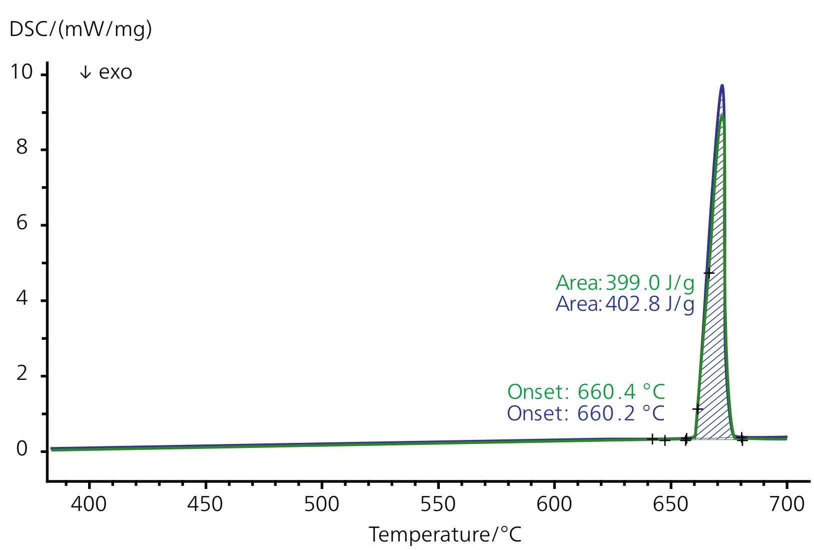
PET measurements with the P-Module. Sample mass: approx. 5.5 mg; aluminum pans: *Concavus* with pierced lid; atmopshere: N_2; 2nd heating with 10 K/min

Cooling Rate (prior to heating)	Glass Transition		Post-Crystallization		Melting		Crystallinity
[K/min]	Δc_p [J/(g·K)]	Midpoint [°C]	Enthalpy [J/g]	Temper-ature [°C]	Enthalpy [J/g]	Temper-ature [°C]	[%]
10	0.240	77.7			42.49	246.4	30.35
20	0.253	77.8	-18.11	147.7	38.44	246.7	14.35
50	0.368	77.9	-32.68	149.5	38.61	246.8	4.24
100	0.379	78.1	-34.15	150.1	38.42	247.0	3.05
200	0.394	78.2	-34.48	150.0	38.38	246.9	2.79

PET is a commonly used semi-crystalline thermoplastic polymer. The crystallinity of PET is influenced by the crystallization rate. This means that if cooling occurred fast enough, post-crystallization will show up in the subsequent heating.

In the DSC experiments shown here, various effects are evident: endothermic DSC steps representing the glass transition (around 80°C), exothermic effects for post-crystallization (peak temperature at around 150°C) and endothermic melting effects (peak temperature around 247°C). The crystallinity of the material is determined from the enthalpy of melting and post-crystallization. The amorphous portion of the material is represented by the glass transition. At the glass transition of the sample, the specific heat capacity changes: the greater the change, the greater the amorphous fraction.

Melting of Aluminum



DSC measurements on aluminum with the H-Module. Sample mass: approx.12 mg; atmosphere: N_2.

Measurements above 600°C require crucible materials other than aluminum, which melts at 660°C.

In this example, a metallic sample was measured in a platinum crucible. To avoid any reaction between the two metals, an Al_2O_3 liner was used in the Pt crucible. Despite the influence on the time constant and the caloric sensitivity, the two measurements exhibit very good reproducibility – below 1% with respect to onset and enthalpy of fusion.

Curing of UV Ink Measured at Different Temperatures

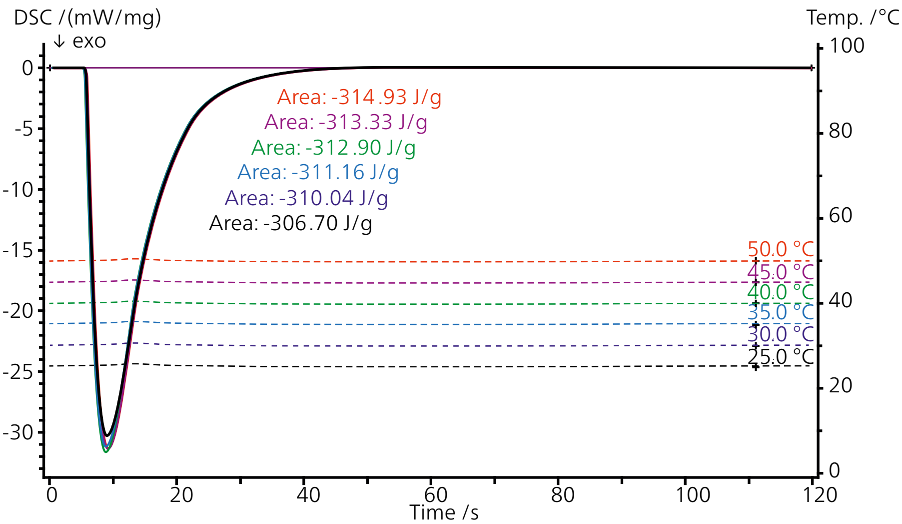


Fig. 1: UV ink exposed to UV light source for 10 seconds at different isothermal temperatures

Unlike conventional inks, solvent-free UV ink cross-links when exposed to ultraviolet light, quickly transforming from a liquid to a solid coating.

In fig. 1, the sample and reference were irradiated with UV light at different isothermal temperatures until the sample was cured. In this case, the curing is almost unaffected by different temperatures. Thus, the reactivity of this sample just depends on the irradiation. Furthermore, it is possible to carry out such photo-DSC experiments with different irradiation intensities.

Curing and Post-Curing of a UV Adhesive

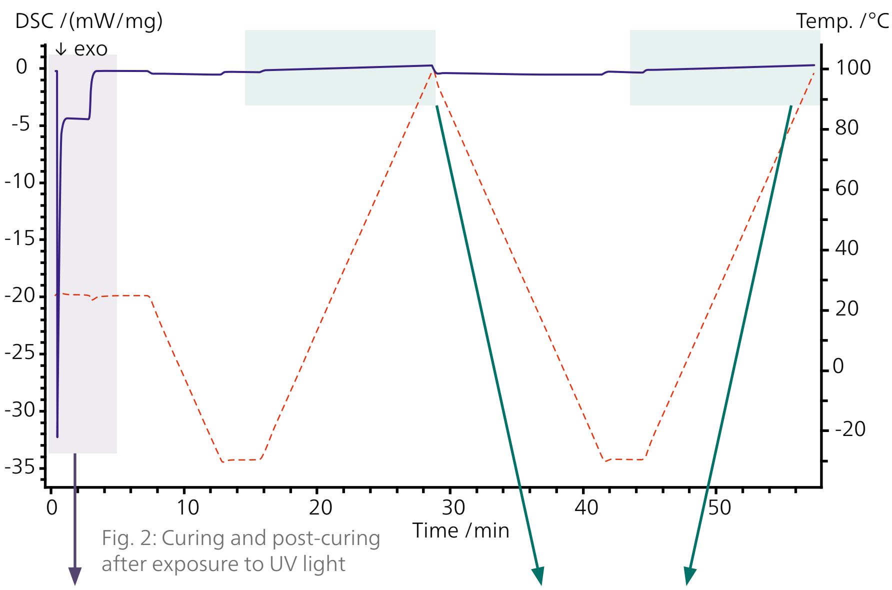


Fig. 2: Curing and post-curing after exposure to UV light

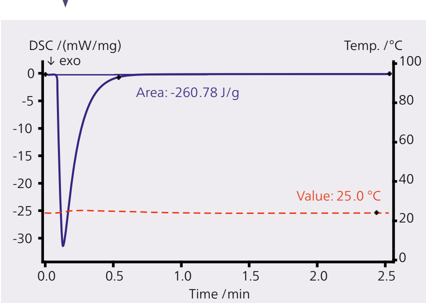


Fig. 3: Exposure of sample to UV light at room temperature

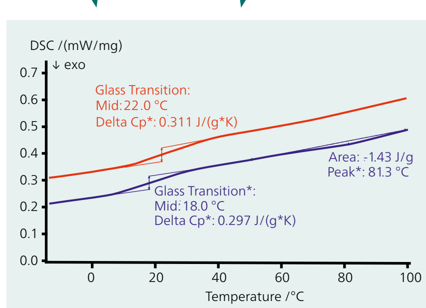


Fig. 4: Glass transition and post curing effect during first heating (blue curve) and final glass transition (determined in the second heating curve, red curve)

UV adhesives are acrylate- or epoxy-based resins often used in the medical and electronics industries. They polymerize and cure by irradiation with a special UV light source. Additionally, quite often a thermal post-curing process is required to achieve the best material properties. This allows them to offer functionalities that areimportant in manufacturing processes, like superior resistance to chemicals (e.g., solvents), a wide operating temperature range, low shrinkage, and strong, tack-free finishes.

With the NETZSCH photo-DSC 300 *Caliris*®, it is possible to monitor such curing processes within a single measurement. Firstly, the sample was cured for 2 ½ minutes at room temperature (see fig. 3). The subsequent first heating up to 100°C (blue curve in fig. 4) exhibits a glass transition at 18°C and post-curing at 60°C. In the second heating (red curve in fig. 4), no more post-curing occurs and the glass transition can be finally determined at 22°C.

	DSC 300 Caliris®					
	Supreme			*Select*		
Color touch display	■			■		
Modules	freely selectable and upgradeable			fixed selection		
Module type	H	P	S	H	P	S
Max. T/°C	750	600	600	650	600	600
Temperature accuracy/K	± 0.05 (indium)	± 0.1 (indium)	± 0.1 (indium)	± 0.05 (indium)	± 0.1 (indium)	± 0.1 (indium)
Heating/cooling rates K/min*	0.001 to 200	0.001 to 500	0.001 to 100	0.001 to 200	0.001 to 500	0.001 to 100
Cooling with LN_2, min. T/°C	-180	-170	-170	-180	-170	-170
Cooling with intra-cooler, min. T/°C	-90	-70/-40	-70/-40	-90	-70/-40	-70/-40
Cooling with pressurized air, min. T/°C	<0	<0	<0	<0	<0	<0
Gas-tight design	■	■	■	■	■	■
Gas atmospheres	inert/oxidizing, static/dynamic					
Integrated 3-fold MFC	■	■	■	■	■	■
4th MFC**	□	□	□	-	-	-
192+12-position ASC	□	□	□	□	□	□
Piercing device	□	□	□	□	□	□
100 Hz data acquisition	■	■	■	□	□	□
Enthalpy accuracy/%	< 1 for adamantan, indium, zinc; < 2 for most materials					
Measuring range/mW	± 750	± 750	± 650	± 750	± 750	± 650
Evolved Gas Analysis***	Yes	Yes	Yes	Yes	Yes	Yes

* depending on the cooling device
** for gas mixtures
*** FT-IR Coupling to DSC is recommended only for samples for which the released substances boil at temperatures below 120°C.

■ included
□ optional

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