



# Differential Scanning Calorimetry – DSC 300 *Caliris* <sup>®</sup> *Classic*

Method, Technique, Applications

Analyzing & Testing

## DIFFERENTIAL SCANNING CALORIMETRY (DSC)

The DSC 300 Caliris<sup>®</sup> operates according to the heat-flux principle. With this method, a sample and a reference are subjected to a controlled temperature program (heating, cooling or isothermal).

A crucible containing the sample is placed on the sensor inside the measuring cell (furnace) of the DSC system along with a reference pan (typically empty).

Sample and reference are subjected to the same controlled temperature program and atmosphere. Due to the heat capacity of the sample, the reference temperature rises slightly faster than the sample temperature.





Signal generation in a heat-flux DSC

The two temperature curves run in parallel as long as the heating rate is constant until a reaction occurs in the sample. At time  $t_1$ , the sample begins to melt. During melting, the sample temperature remains constant, while the reference temperature continues to increase linearly. Once the melting process is complete, the sample temperature begins to rise again, following a linear pattern at time  $t_2$ .

The measured properties are the sample temperature and the temperature difference between the sample and reference. From the raw data signals, the heat flow difference between sample and reference is determined, which represents the caloric changes of the sample.

The DSC 300 *Caliris*<sup>®</sup> is compliant with virtually all relevant instrument and application standards, including ISO 11357, ASTM E793, ASTM D3895, ASTM D3418, DIN 51007.



### DSC 300 Caliris<sup>®</sup> Classic Slim in the Lab, Big in Quality Control

Knowledge about a material's behavior under changing temperature and different atmospheres is of fundamental importance for many application fields.

#### **Typical DSC Results**

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

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

### DSC 300 Caliris<sup>®</sup> Classic Ideal for Quality Control and Teaching

#### Perfect Measurement Conditions, Even in Less Than Perfect Environments

The gas-tight measurement cell provides optimal atmospheric conditions for precise measurements. The gas flow is regulated by three magnetic valves that can be turned on or off programmatically. Mass flow controllers are also available as an option. This is of particular advantage for measuring the oxidative induction time/temperature (OIT).

Moreover, the gas-tightness of the cell ensures that environmental humidity does not affect the DSC system. This is particularly useful in regions with high humidity, as it largely minimizes problems caused by condensation.

#### Compact Design for More Space in Your Lab

The DSC 300 *Caliris*<sup>®</sup> *Classic* boasts a slim design that requires minimal space. As laboratory space is often limited, the compact design of the DSC 300 *Caliris*<sup>®</sup> *Classic* with a cooling accessory, such as the intracooler, makes it an excellent choice. It is perfect for at-line investigations and can be easily set up in a production environment for QA/QC purposes.

#### **Excellent Performance and Competitively Priced**

The monolithic DSC sensor is stable even under harsh environmental conditions and offers optimal resolution. The sensor disks and thermocouple wires are laserwelded, resulting in high sensitivity and robustness.

In the event of unwanted contamination of the cell or sensor, the temperature range of up to 600°C makes it easy to clean by baking out the contaminants. In addition, cleverly designed connectors allow for quick and easy installation of various cooling systems.

ROBUST INSTRUMENT WITH ATTRACTIVE PRICE-PERFORMANCE RATIO



# *More Than a DSC – A Smart, Easy-to-Use Quality Control System*

#### The Quick-Start System for DSC Measurements

The DSC 300 *Caliris® Classic* offers a quick way to introduce Differential Scanning Calorimetry to your lab. After the instrument has been set up and calibrated, the simplified and intuitive *SmartMode* user interface will guide you through defining your measurement parameters.

After the measurement is complete, the *AutoEvaluation* and *Identify* software features will take care of the timeconsuming task of comparing your results to known references or literature values. These routines offer support in the evaluation of measurement curves and serve as a second opinion for assessing unknown samples. The *Identify* database system is capable of verifying materials and allows for quality assurance tests.

#### Making Routine Easy

The gas-tight DSC 300 *Caliris® Classic* is the preferred choice for routine measurements in industry and contract laboratories, and is ideal for projects within the scope of education. It combines the advantages of a highly sensitive, high-tech analysis device with a rugged, easy-to-use workhorse.



### Smart Lab

#### *Proteus*<sup>®</sup> *Search Engine* and LabV<sup>®</sup> Are Keeping Track of Material Quality – Even in Decentralized Quality Control Labs

Keeping track of, and organizing, collected data to be available for future experiments or final reports is key for a smooth laboratory workflow. Using the LabV<sup>®</sup> cloud solution keeps all your test data in one place, connects all the data of all your devices in use, irrespective of manufacturer, and creates the basis for analyses and forecasts.

*Proteus*<sup>®</sup> Search Engine synchronizes and quickly filters measurement data against prefedefined directories. It provides one-click access to preview of curves and analysis status.

The NETZSCH DSC 300 *Caliris*<sup>®</sup> *Classic* comes LabV<sup>®</sup>primed, making data sharing between multiple locations easy and allowing for effortless implementation into the LabV<sup>®</sup> environment.

### The DSC 300 Caliris® Classic Keep Track of Your Measurements



#### Measurement Update in Passing – LED Status Bar

The DSC 300 *Caliris® Classic* features an LED light bar that allows you to check the status of your instrument as you walk by, with different colors indicating different statuses. It is reassuring to be able to see from a distance, without having to log on to your PC, that your measurement is running smoothly and to be able to read instrument status messages such as:

- Instrument is ready
- Measurement is running
- Measurement progress
- Heating/cooling to setpoint
- User interaction needed
- Incident occurrence

### Improving Your Productivity and Workflow Using the Display

The DSC 300 *Caliris* <sup>®</sup> *Classic* offers an optional integrated touch screen display that provides detailed instrument and measurement information. Once a measurement has been prepared using the *Proteus*<sup>®</sup> software, it can be started directly from the display. By allowing you to double-check the measurement setup on the display, the final check before starting a new measurement is performed directly on the instrument. Your touch display functions:

- Start measurements by the touch of a finger
- Follow measurement progress
- Choose gas type and set gas flow
- Check idle state and current temperature
- See the progress of your measurement and time remaining
- Check recently finished measurements and get an immediate evaluation by activating AutoEvaluation



#### Live Measurement Information to Keep You Informed and in Control

The built-in display provides up-to-the-minute measurement information without the need to access your PC.



Measurement plot, current temperature and gases are displayed during the measurement

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

#### AutoEvaluation: Objective Results Available after the Measurement

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

# Accessories

# Easy-to-Adjust Cooling Options for Economic Cooling



Compressed air cooling



Closed-loop intracooler



Closed-loop intracooler







For the DSC 300 *Caliris® Classic*, different cooling options are available, each tailored to specific temperature ranges; these include air cooling devices and liquid nitrogen cooling. The liquid nitrogen cooling option can be operated in either  $LN_2$  (liquid nitrogen) or  $GN_2$  (gaseous nitrogen) mode, which helps conserve the cooling agent.

Connecting the standard 60-liter Dewar to a large  $LN_2$  tank (e.g., containing 300 liters), automatic refilling is enabled during long measurement series or even during a running measurement. This allows many measurements using the Automatic Sample Changer (ASC) to run without interruption.

The AutoCooling function of the NETZSCH Proteus<sup>®</sup> software detects the cooling unit present and automatically selects the optimum cooling parameters, e.g.,  $LN_2$  or  $GN_2$  in case the CC300 cooling device is used.

The mechanical cooling devices operate in the temperature range of  $-70^{\circ}$ C or  $-40^{\circ}$ C to 600°C, while the liquid nitrogen cooling operates in a wider temperature range between  $-170^{\circ}$ C and 600°C without any adjustments to the instrument setup (e.g., furnace, lid, etc.).

The liquid nitrogen cooling device can be connected to the DSC at the same time as the intracooler. Since this cooling is only required at temperatures below -70°C or -40°C, depending on the intracooler device, this results in a reduction in liquid nitrogen consumption.

### UV-Curing of Reactive Polymers Using UV Add-On

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*<sup>®</sup> *Classic* 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 <sup>®</sup> S2000	320 nm to 500 nm
LX500	365 nm, 385 nm, 395 nm, 405 nm

\* It is also possible to adapt other commercial lamps

#### Advantages of Photo-DSC Tests

- Extending the DSC technique with light radiation capability
- Analyzing photo-induced reactions in a broad variety of materials
- Measuring light curing of polymer resins, paints, coatings and adhesives (degree of cross-linking)
- Studying the influence of UV stabilizers in coatings of pharmaceuticals, cosmetics and food packaging (aging effects)
- Analysis of curing behavior of dual-curing systems, e.g., UV curing and thermal post-curing in one measurement
- Determining the reactivity and curing time of such materials as inks, adhesives, coatings, dental composites, nail polish, etc.

### Making Sample Preparation Easier



#### The SampleCutter – Perfect for Polymers

One prerequisite for reproducible and reliable DSC results is to have good thermal contact between the sample and crucible bottom, which postulates a flat sample area. With the *SampleCutter*, it is easy and comfortable to cut plane sample faces, irrespective of whether the sample is soft, hard or brittle.

#### Wide Selection of Crucibles



*Concavus*<sup>®</sup> crucibles and lids, cold-weldable, 30/40 μl



Slide-in lid for *Concavus*<sup>®</sup> crucible, especially for films or foils; colored lides for demonstration purposes



Medium pressure-tight crucible up to 20 bar



100 µl aluminum crucible, cold-weldable



Highly pressure-tight crucible up to 100 bar



#### Sample Preparation Kit

This set contains a variety of small tools including a cutting board, scissors, tweezers, spatula, etc., and is designed to make sample preparation as easy and convenient as possible.



### Automatic Sample Changer – Improve the Efficiency of Your Daily Measeurement Tasks



For high throughput applications and routine work, an automatic sample changer (ASC) is available for up to 20 samples and references. The gripper safely removes the crucible from the magazine and gently places it in position on the sensor. The reference crucible can also be changed as often as the application requires.

The ASC is easily programmed using the *SmartMode* of the *Proteus*<sup>®</sup> software. A specific measurement program (method) can be assigned to each sample on the tray. Different crucible types, different gas atmospheres and individual calibration curves can be handled within the same carousel run. Used samples are automatically disposed of in the integrated waste bin. For 24/7 operation, previously measured samples can be continuously replaced by adding new crucibles to the carousel in combination with new measurement methods.

### Proteus<sup>®</sup> Software GET STARTED QUICKLY WITH SMARTMODE

NETZSCH SmartMode					- 🗆 X
NETZSCH	DSC 300 Caliris Classic E				× 🍫
Favorites User Methods Enew Wizards	ABS	SAN	Acrylonitrile-styrene-acrylate copolymer 1-120.0. 180.0 °C	Styrens/Polybutadiene copolymer 1.158.0., 200.0 °C	PS         Image: Compare the second sec
Predefined Methods Measurement Setup & Control Setpoint 104 30 °C	PE-LD	Polyethylene linear low density 8 -30.0 - 200.0 °C	Polyethylene high density 8 -30.0 - 200.0 °C	Pe-UHMW	EVA     Since       Polyethylene-co-vinyl acetate     128.0 - 200.0 °C
www.netzich.com	Pt (isostatic)	● PB         ☆           Polybutene         8 - 76.0 = 188.0 °C	PVC-P	PVC-U	Polyvinylidene chloride 9 -568 - 1768 °C
	PVAL Polyvinyl alcohol B 0,0–250,0 °C	Polylactide	PA11     Polyamide 11     20.0 - 250.0 *C	PA12     Polyamide 12     0 - 50,0 - 230,0 °C	PA46            Pobyamide 46         0.0
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Pre-defined methods in the Proteus® software

### SmartMode User Interface – Getting Started Easily

SmartMode provides a clearly structured interface for fast measurement setup and is designed specifically for the type of routine measurements often required in quality control.

t makes preparing and starting measurements quick and easy. Even if you are new to the method, you will be creating the parameters and getting your measurement up and running in no time.

#### Wizards and Methods - Improving Workflows

The *SmartMode* interface of the system is designed with a clear and consistent structure, making it easy to navigate and user-friendly even for the beginner. Its intuitive interface enables users to quickly find what they are looking for.

The Wizards menu contains a collection of predefined measurement templates that require minimal input and lead to immediate measurement at the touch of a button. The Predefined Methods include all materials featured on the "Thermal Properties of Polymers" poster by NETZSCH, along with corresponding methods that can be used for measurement experiments.

In addition, the User Methods menu allows users to save their previously conducted methods as templates for subsequent measurements, eliminating the need for redefining the measurement parameters.

### AutoEvaluation and Identify – Speed Up Results



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

#### **AutoEvaluation**

#### Objectively Obtained Results Immediately after the End of the Measurement

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/OOT) also gets evaluated for isothermal and dynamic tests, using the Tangent and Offset method in accordance with standards (ISO 11357-6 and ASTM D3895).

Experienced users can take the autonomously obtained 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 own report templates – including logos, tables, description fields and plots. Several report examples are already included as templates in the *Proteus*<sup>®</sup> software.



# *Identify* – Ideal Support for Quality Control MATERIAL COMPARISON AND IDENTIFICATION DATABASE

#### What Is Identify?

*Identify* is a software tool by NETZSCH, unique within the thermal analysis field, for identifying and classifying measurements by comparing curves. There is no need to compare values (e.g., peak temperature); *Identify* does it automatically. Simply load the curve, run *AutoEvaluation* and *Identify*, and get the result.

In addition to allowing for 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 and ceramics.

## Quality Control of a Polypropylene Copolymer Using *Identify*

#### Comparison of different material batches

Figures 1 to 6 show DSC measurements on polypropylene copolymer (PPC) for quality control purposes. The second heating curves were used for comparison. The samples were measured at 10 K/min (heating and cooling) with a sample mass of  $10.0 \pm 0.1$  mg.

#### Create classes in *Identify* to stay within the preset quality threshold

Five out of six measurements agree with the "PASS" criteria created, which was specified to be in agreement to 98%. Batch 1 in Figure 1 shows an average similarity of 99.29% compared to the entire class ranging from 98.6 to 99.8%. This declares the batch to be a "PASS" sample.

#### Using *Identify* to specify unknown materials

However, for batch 6 in Figure 6, a similarity of less than 98% compared to the quality class is detected, which is outside the pass criteria and thus causes a "FAIL" error message.

*Identify* determined that the quality was inadequate. In addition. the comparion with the help of the database also identifies the failed material as PP-H, with a similarity of 99.31%.

#### STEP 1 Create classes Results: Database Entry Similarity Class PPC-04 99.84 % PP-C QC PPC-03 99.71 % POM PPC-02 99.13 % PA1x\_semi-cryst.



#### STEP 2 Set quality threshold

Similarity threshold:

STEP 3

18:50

SC /(r [6,6] exc 1,44 1,2 1.0 0.8

0,6

0,4 0.2 0.0 Glass Turnstion: Mrd 68.3 °C Delta Cp\* 9.050 J/(g%)

-50

Gass Tonstron\* Mid -6.6 °C Deta Op\* 0.046 Jhg\*R0

ò

50 Temperature /"C



Complex Peak Area 97,2 Jig Peak\*: 167,1 °C

150

200

Fig 1.: Batch 1 PPC

100

#### Proteus® Search Engine - Smart Data Management

When working with measurement and evaluation data for different materials and different measurement setups, it is enormously helpful to be able to directly access and sort data by certain criteria. *Proteus® Search Engine* automatically synchronizes your measurement data with pre-defined directories and filters it in a matter of seconds. Previews of measurement curves or analysis status are available with just one click.

Users are able to create individual searches, for example "MyPolymers", and switch easily between different existing searches. This makes *Proteus*® *Search Engine* a very powerful data management tool.



#### Advantages of Proteus® Search Engine

- Efficient data management
- Directly access and sort data by criteria
- Preview your stored data
- Quickly view measurement and analysis previews without opening files
- Retrieve data quickly and easily
- Search, e.g., by instrument name, method, operator, file and signal type, date, measurement conditions or evaluated effects

### Proteus® Search Engine and LabV®

🕹 LabV®-primed

#### LabV<sup>®</sup> – Taking Advantage of the Digital Lab

Using the LabV<sup>®</sup> 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. Data security is ensured at the highest standards. It seamlessly integrates LabV<sup>®</sup> with various AWS<sup>\*</sup> services, guaranteeing full compliance with EU GDPR<sup>\*\*</sup> guidelines. Organize your projects and analyze your data, trends, and reports. Discover the value of historical data and compare gathered data to improve material quality and part performance. LabV<sup>®</sup> connects all the analysis methods, making projects more flexible and product quality predictable.

The NETZSCH DSC 300 *Caliris*<sup>®</sup> *Classic* is prepepared for an effortless implementation into the LabV<sup>®</sup> environment.

\* Amazon Web Services \*\* General Data Protection Regulation

#### Advantages of LabV®

- Laboratory Automation Streamline your testing process and connect all your testing devices
- Cloud Solution
- Improved Quality Control Improve the quality of your materials with insights, intelligent alerts and intuitive data management
- Faster Development Leverage your lab data to accelerate material development

## APPLICATIONS

#### OIT Measurement on Two Grades of PE-HD



High-density polyethylene (PE-HD) is much harder, stiffer and less flexible than low-density polyethylene (PE-LD), but more abrasion-resistant. It is highly resistant to aggressive substances and external stresses, even under extreme climatic conditions.

Oxidative-Induction Time (OIT) is a relative measure of the resistance of a (stabilized) material to oxidative decomposition, determined by calorimetric measurement. 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 maintained at a constant temperature until the oxidative reaction is indicated by the exothermic deviation of the DSC heat-flow curve.

The OIT is the time interval between the start of the oxygen or air flow and the start of the oxidation reaction (in accordance with DIN EN ISO 11357-6 or ASTM D 3895).

The figure shows the OIT results for two samples. Under an air atmosphere at 220°C, the measurements indicate significant differences in the stability against oxidation. It can be concluded that the blue sample has the higher oxidative stability. This information is very useful when assessing the quality of organic materials or polymers, such as PE pipes.

OIT measurement on two different PE-HD grades indicating significant differences in the stability against oxidation. Sample mass:  $10.5 \text{ mg} \pm 0.2 \text{ mg}$ ; heating rate: 20 K/min; open Al crucible; nitrogen atmosphere switched to synthetic air at 220°C.







Glass transition temperature of the 2<sup>nd</sup> heating, measured by DSC; curves shifted along the y-axis for clarity; sample weight: app. 10 mg; heating rate: 10 K/min.

Polymers from left to right:

Q (Šilicone), BR (Butadiene Rubber), PIB (Polyisobutene), NBR (Nitrile Butadiene Rubber), PVDC (Polyvinylidenchloride), PLA (Polylactide), PET (Polyethylenterephthalate), PS (Polystyrene), PC (Polycarbonate), PEI (Polyetherimide), PESU (Polyethersulfone)

The glass transition temperature  $(T_g)$  of a polymer is the temperature range in which it changes from a rigid "glassy" state to a flexible "rubbery" state. For example, the glass transition limits the temperature range in which an elastomer can be used. Understanding the glass transition temperature is important for quality control as it helps to optimize processing conditions, ensure product performance and maintain consistency of material properties, thereby improving the overall quality and reliability of the final product.

Depending on the type of polymer (e.g., elastomer, thermoplastic or thermoset), the glass transition can be observed over a wide temperature range. Among other properties, the specific heat capacity changes at the glass transition temperature ( $T_g$ ), so that the  $T_g$  can be easily measured by DSC.

With the capability of connecting different cooling devices (even simultaneously), the DSC 300 *Caliris*<sup>®</sup> *Classic* can measure the T<sub>g</sub> of a wide range of polymers without any hardware changes.



#### Dehydration of Gypsum (Calcium Sulfate Dihydrate)

In the gypsum industry, a distinction is made between the four  $CaSO_4*xH_2O$  phases that are obtained depending on the firing process: Di-hydrate,  $CaSO_4*2H_2O$ , dehydrates to the so-called hemihydrate,  $CaSO_4*1/2H_2O$ , which finally dehydrates after a further dehydration stage to anhydrite III or anhydrite II.

The difficulty of the quantitative determination of the dihydrate or hemihydrate with the thermal analysis essentially consists in the separation of the two dehydration stages. If this is successful, the proportions can be determined by means of the enthalpy values of the DSC curves or weight loss stages in a TGA. By measuring under a higher water vapor partial pressure, the dehydration of the hemihydrate can be shifted to a higher temperature and thus the DSC peaks can be separated.

This example shows a DSC curve measured with a sealed crucible with a lid with a laser-pierced 50-µm hole. The two dehydration peaks can be easily separated. From comparing the enthalpy values with literature values, the sample has about 94% dihydrate, 5% hemihydrate and a small amount (1%) of impurity, probably anhydrite III.



DSC results for gypsum. Sample mass: 10.02 mg; heating from 25 to 300 °C at 10 K/min; atmosphere N,; Concavus® Al crucible with pierced lid

#### Polymorphism of Paracetamol



DSC results for Paracetamol (blue curve: 1<sup>st</sup> heating; green curve: 2<sup>nd</sup> heating). Sample mass: 1.54 mg; 1<sup>st</sup> heating from 25 to 190°C at 10 K/min, cooling to 25°C at 10 K/min, 2<sup>nd</sup> heating from 25 to 190°C at 10 K/min; atmosphere:  $N_2$ , *Concavus*<sup>®</sup> Al crucible with pierced lid.

Paracetamol, also known as acetaminophen, is a commonly used over-thecounter analgesic (pain reliever) and antipyretic (fever reducer). Paracetamol exhibits polymorph behavior. Polymorphism is the ability of a substance to exist in multiple crystal structures while maintaining the same chemical composition.

Paracetamol has three known polymorphic forms, referred to as Forms I, II, and III with Form I being the most stable and commonly encountered form, which exhibits good solubility and dissolution rates.

In the example above, paracetamol was heated twice and cooled in between at a controlled rate. In the first heating, an endothermal effect with an extrapolated onset temperature of 169°C can be seen. This temperature correlates well with the melting of Form I.

During the subsequent controlled cooling step (not shown here), no crystallization takes place. This means that paracetamol is still amorphous at the beginning of the  $2^{nd}$  heating step.

During the 2<sup>nd</sup> heating, first an exothermal effect (with a peak temperature of 82.7°C) related to a cold- or post-crystallization process occurs. Parallel XRD studies have shown that form III is formed here. This form III transforms into form II (exothermal effect at 132.7°C peak temperature) upon further heating (also confirmed by XRD investigations), which finally melts at 157°C (extrapolated onset temperature). The extrapolated onset temperature of 157°C is characteristic of Form II.



#### Finding the Right Storage Conditions for Spices

Curcuma is another name for turmeric, a spice that comes from the rhizome of a plant in the ginger family. It is used as food additive E 100, a yellow pigment that has anti-inflammatory and antioxidant properties.

Commercial turmeric powder as received has a glass transition temperature (green curve) of -2.1°C (midpoint), reflecting its amorphous nature. The glass transition temperature ( $T_g$ ) of a powder affects both its quality and shelf life. Above the  $T_g$ , the spice can become soft and sticky and the particles can cake. According to Roos and Karel\*, this sticky point is directly related to the glass transition temperature (constant temperature difference, e.g., about 20 K for dairy powders). Therefore, knowledge of the  $T_g$  is also important during processing (e.g., drying and grinding) of such spice powders in order to avoid clumping.

In addition, amorphous materials tend to absorb moisture from the environment, which can increase their susceptibility to microbial attack and affect the glass transition temperature. Water has a plasticizing effect and shifts the glass transition temperature to lower values. In this example, the glass transition moves to -39°C (blue curve) after 20 minutes of storage at 100% humidity. On the other hand, the glass transition temperature can be detected at 83°C (violet curve) if the spice was previously dried in a drying chamber.

\* Roos, Y. and Karel, M. 1991a, 1991b, 1993



DSC results of tumeric with different water content; sample weight: 10.71 mg, 10.05 mg and 11.03 mg; *Concavus* Al crucible, hermetically sealed: heating from -170 to 120 °C at 10 K/min; green curve: powder (as received); purple curve: dry powder (powder dried for 45 min at 80°C); blue curve: wet powder (stored for 20 min at RT at 100% rel. humidity)

## Technical Specifications

	DSC 300 Caliris <sup>®</sup> Classic
Temperature range	-170°C to 600°C
Heating/Cooling rate	0.001 K/min to 100 K/min*
Temperature accuracy	± 0.1 K (indium)**
Enthalpy precision	± 0.1% for indium
Enthalpy accuracy	< 1% for adamantan, indium, zinc; < 2% for most materials**
Cooling device options	<ul> <li>Compressed air cooling (RT to 600°C)</li> <li>IC40 (-40°C to 600°C)</li> <li>IC70 (-70°C to 600°C)</li> <li>LN<sub>2</sub> cooling, automatically controlled (-170°C to 600°C)</li> </ul>
Gas atmospheres	Inert, oxidizing, static and dynamic operation
Gas control	<ul> <li>Switches for 3 gases included</li> <li>MFC for 3 gases, optional</li> </ul>
ASC	Up to 20 samples and references, optional

\* Maximum rates depend upon the temperature

\*\* Deviation of measured value from "true value" (literature value)



#### **Software Features**

- SmartMode
- ExpertMode
- AutoCalibration
- AutoCooling
- Report Generator
- OIT
- Predefined Methods
- TauR
- $\hfill\square$  Specific heat capacity (c\_,) determination
- AutoEvaluation
- Identify
- TM-DSC (temperature-modulated DSC)
- ASC (Automatic Sample Changer) support
- Proteus<sup>®</sup> Search Engine

more features on request

■ included □ optional



All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, both 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. Choose your preferred training method: Online, on-site or at our NETZSCH training center.

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

## Expertise in SERVICE

#### **TECHNICAL SERVICE**



Maintenance

and Repair



Software

Updates





IQ/OQ Service Documents





Spare Part Service Assistance



Moving Service





Basic

NETZSCH Seminars Online





#### LABORATORY



**Application Service** and Contract Testing 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 4,000 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 60 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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