NETZSCH

Proven Excellence.

Investigation of Pharmaceuticals, Foods, Cosmetics and Organics

Analyzing & Testing

High-end DSC, TGA, STA, TMA equipment, rheometers and adiabatic calorimeters

ESSENTIAL ANALYTICAL INSTRUMENTATION

for Every Laboratory in the Chemical, Pharmaceutical, Cosmetics and Food Industries

These NETZSCH instrument lines are specifically designed to meet the needs of the chemical, pharmaceutical, cosmetics and food branches. They are therefore optimal for research and development, quality control and contract testing in these areas.

Thermal Analysis

Thermal Analysis is a set of techniques which has been used in countless laboratories around the globe to analyze and characterize organic and inorganic substances – whether solid or liquid. Classical Thermal Analysis assesses changes in material properties during heating or cooling with regard to mass loss, dimensional changes or phase transitions. The methods most frequently applied are:

- Differential scanning calorimetry (DSC)
- Thermogravimetric analysis (TGA)
- Simultaneous thermal analysis (STA, which is a combination of DSC and TGA)
- Thermomechanical analysis (TMA)

SEE PAGE 4

Rheology

Rheology provides information about the rheological properties of matter, mostly semi-solids and liquids. This comprises their flow behavior, their viscosity and their viscoelastic behavior, depending on the operation mode.

Two different types of rheometers are typically in use:

- Rotational rheometers (also for oscillatory tests)
- Capillary rheometers

Rotational rheometers are indispensible analytical tools for finding the suitable consistancy, texture or formulation of materials for final product application and – for example – for estimating the shelf life of dispersions. Capillary rheometers are best for simulating and optimizing processes under conditions of high force/high pressure and/or high shear rates.

SEE PAGE 20

Adiabatic Calorimetry

Every time a chemical process is scaled up to industrial scale or when production is to be moved from one site to another, assessment of thermal safety must be carried out. One of the key parameters in this context is T_{D24}, the temperature at which the time to maximum rate under adiabatic conditions is 24 hours.

ARC® (Accelerating Rate Calorimetry) instruments, often in combination with kinetics evaluation, can identify chemical hazards and simulate worst-case scenarios.

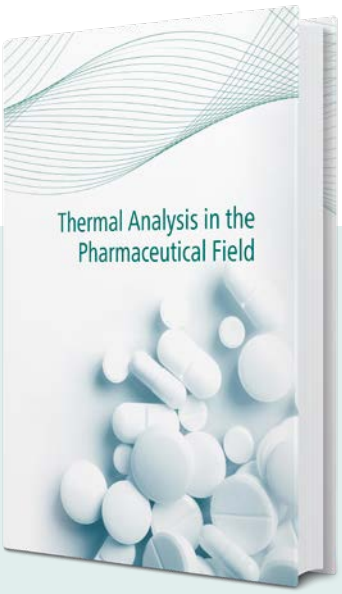
The MMC (Multiple Module Calorimeter) comes with several measuring modules. Along with the ARC module for thermal safety investigations, it can also be applied with a scanning module for performing DSC-like measurements.

SEE PAGE 26

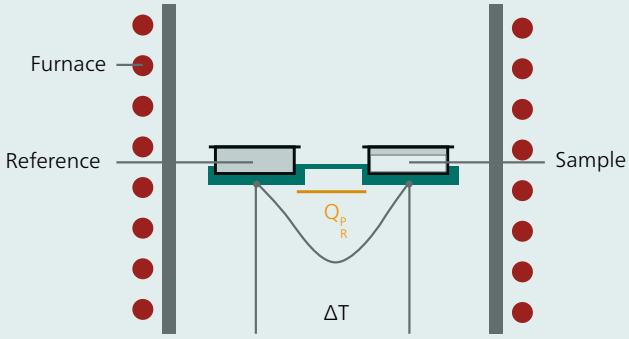
All NETZSCH devices are easy-to-operate, precise and reliable, and offer a lot of clever solutions, including a handful of ways to automate your day-to-day work.

A lot of application examples of DSC, TGA and TGA combined with a gas analyzing system can be found in the "Thermal Analysis handbook in the Pharmaceutical Field".

For more information, please check our website.



THERMAL ANALYSIS

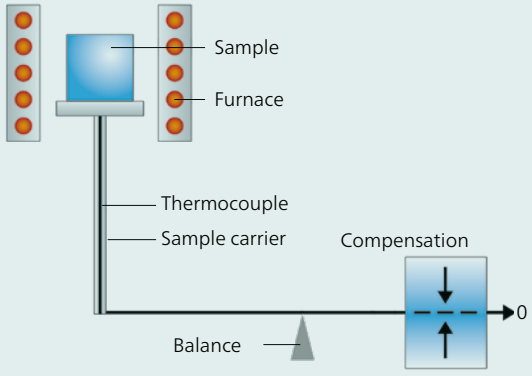


Cell of the DSC 300 *Caliris*®

DSC measures the change in heat-flow difference into a sample and a reference material while they are subjected to a controlled temperature program. In a heat-flux DSC, the difference in heat flow between a sample and a reference is derived from the temperature difference between the sample and the furnace and the reference and the furnace or between the sample and the reference.

DSC Results

- Melting temperatures and enthalpies (heats of fusion)
- Polymorphism
- Crystallization temperatures and enthalpies
- Glass transitions (e.g., amorphous content)
- Solid-solid transitions
- Compatibility
- Phase diagrams
- Eutectic purity
- Solid-fat content
- Reaction temperatures and enthalpies
- Cross-linking reactions (curing)
- Specific heat capacity (c_p)
- Oxidative-induction time and temperature (isothermal and dynamic OIT)
- Thermal kinetics (combined with Kinetics Neo)



Principle of the top-loading TG 309 *Libra*®

Thermogravimetric analysis, or TGA (also known as thermogravimetry, or TG) measures the mass change of a substance while the sample is subjected to a controlled temperature program. "Top-loading arrangement" simply means that the sample is located above the balance. This ensures safe and easy handling.

TGA Results

- Thermal stability
- Compositional analysis
- Mass changes
- Decomposition behavior
- Pyrolysis
- Water amount
- Amount of solvents (also intercalated ones)
- Oxidation
- Shelf life
- Thermal kinetics (combined with Kinetics Neo)
- Identification of evolved gases (coupled to a gas analyzing system such as FT-IR, MS or GC-MS)

Complementary Methods: DSC and TGA

The strength of DSC lies in its capability of detecting and monitoring phase transitions of materials. However, the reason for a DSC effect is not always obvious; it might be:

- a polymorphic transition,
- a melting effect, or
- an effect caused by a mass loss.

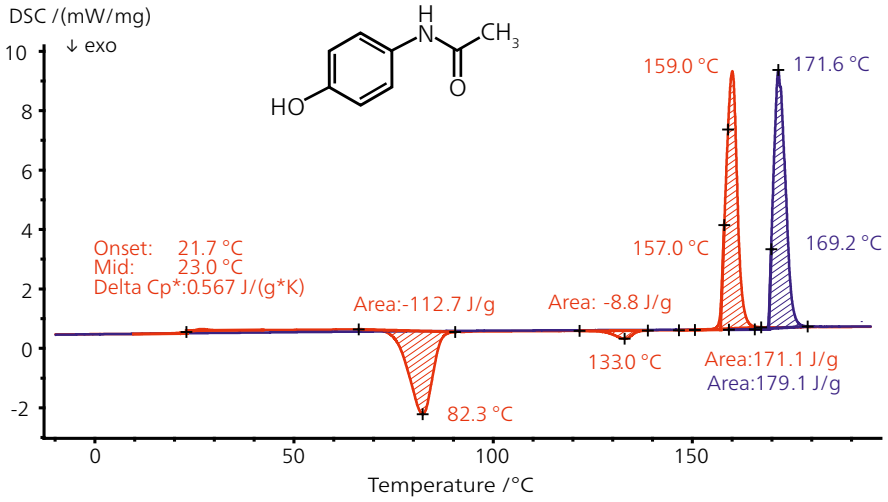
Therefore, DSC and TGA are very often used as complementary techniques. By applying the methods in combination, it is possible to distinguish whether an effect is related to structural changes and to determine in which temperature ranges mass changes occur. This information facilitates interpretation of the effects detected – especially when a sample's composition is unknown.

Improving Interpretation by Coupling Evolved Gas Analyzers to Thermal Analyzers

In order to gain a deeper understanding of what happens during evaporation and decomposition processes, it is possible to couple an evolved gas analyzer (e.g., a FT-infrared spectrometer (FT-IR), mass spectrometer (MS), or a gas chromatograph-mass spectrometer (GC-MS)) to the thermal analyzer.

Many laboratories already use FT-IR, and for them, coupling to a thermal analyzer is only logical. However, FT-IR is not capable of recognizing homonuclear diatomic molecules such as N_2 and O_2. In such cases, mass spectrometry is usually the method of choice – as it also is under other specific sets of circumstances. GC-MS is often coupled to thermal analyzers such as TGA and STA in situations where complex gas mixtures are present, such as the decomposition of large organic compounds.

Meaningful Conclusions Drawn from Thermal Analysis Curves



DSC measurement on paracetamol (4-acetaminophen); sample mass: 2.6 mg, Al crucibles with pierced lid, heating rate: 10 K/min, nitrogen atmosphere

Polymorphism of Paracetamol

Upon application of a heating-cooling cycle, this sample exhibits just one DSC peak within the 1st heating (blue). The extrapolated onset temperature at 169°C correlates well to the melting point of the crystalline form I.

No crystallization takes place during the subsequent cooling (not shown here), resulting in a glass transition at 23°C (mid point) and a post crystallisation (exothermal) at 82°C (peak temperature) within the 2nd heating (red). The formed modification III subsequently transforms into modification II (exothermal effect at 133°C peak temperature) which melts at 157°C (extrapolated onset temperature). The stuctural change can be verified by using PXRD.

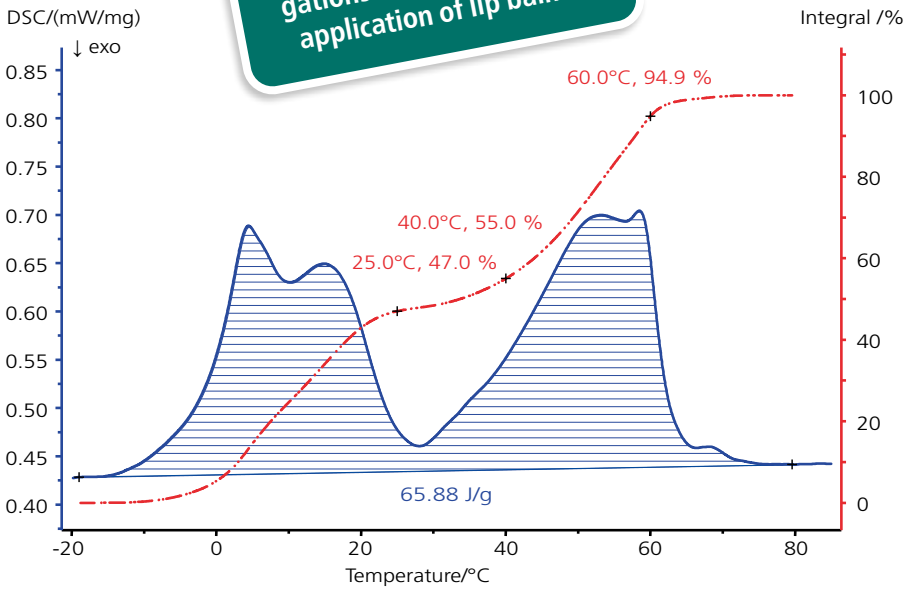
Maintaining the Feel and Spreadability of a Lip Balm over a Broad Temperature Range

Lip balms usually consist of various waxes or fats plus cosmetic additives which care for the lips. Shown here is the melting behavior of a commercial lip balm between -20°C and 85°C, recorded by DSC. Altogether, five superimposed peaks can be seen.

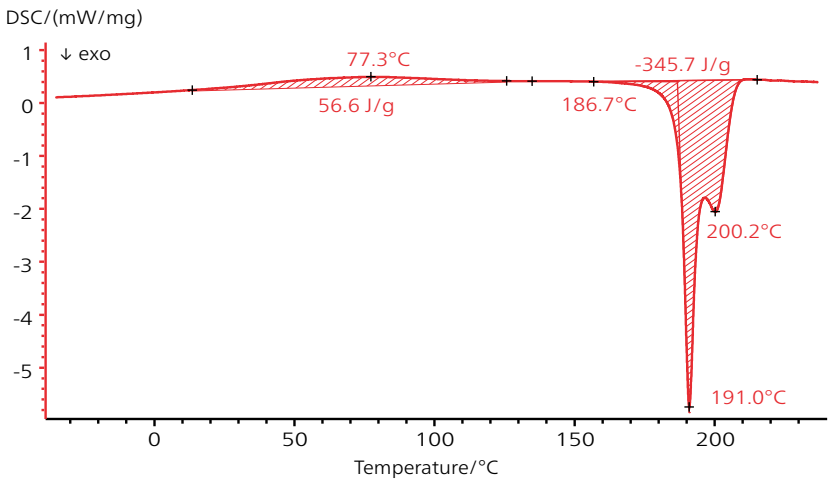
The melting progression is reflected by the integral curve (red). At 25°C, 47% of the mixture is already molten (liquid portion) and 53% (= 100% minus 47%) is still solid. Therefore, the amount of the "solid-fat content" in the present case is 53% at 25°C, 45% at 40°C and just 5.1% at 60°C.

The portion which is already molten at room temperature serves for creaminess and smooth application.

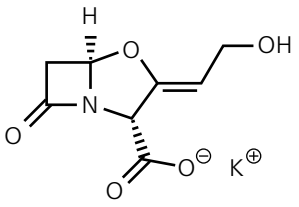
Solid-fat content investigations to ensure smooth application of lip balms



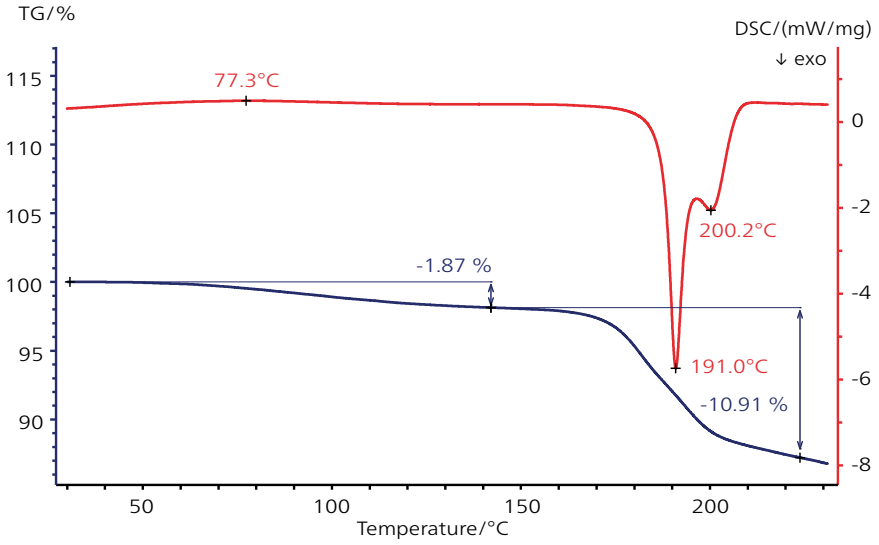
DSC measurement on a lip balm (blue curve); sample mass: 9.7 mg, Al crucibles with pierced lid, heating rate: 10 K/min, nitrogen atmosphere



DSC measurement on potassium clavulanate; sample mass: 2.3 mg, Al crucibles with pierced lid, heating rate: 10 K/min, nitrogen atmosphere



Structure of potassium clavulanate ($C_8H_8KNO_5$)



Joint presentation of DSC (red) and TGA (blue) results for potassium clavulanate; the TGA experiment was conducted under the same conditions as the DSC test.

Thermal Stability of Potassium Clavulanate

Hygroscopicity: A Challenge for Shelf Life

Potassium clavulanate is classified as hygroscopic. Therefore, the condition of the supplied material is crucial to its shelf life. Here, DSC and TGA-FT-IR measurements were carried out to elucidate the condition of this chemical.

DSC Measurement

During heating in a nitrogen atmosphere, the DSC curve (upper plot) shows two effects: a stretched endothermal one with a peak temperature at 77°C and a double exothermal peak starting at 187°C (extrapolated onset).

TGA-FT-IR Measurement

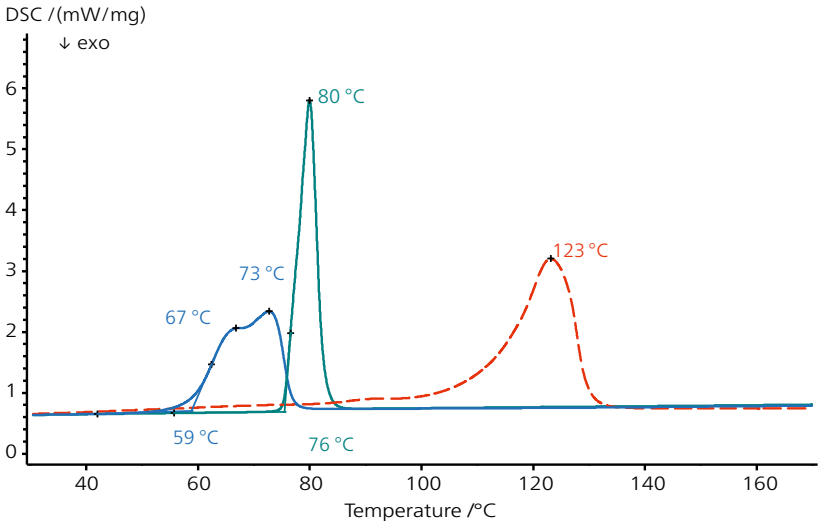
The TGA-FT-IR measurement (lower plot) reveals that the two effects are caused by mass losses with step heights of 1.9% and nearly 11%, respectively. The results of the coupled FT-IR measurement (not shown here) prove that the first mass loss is due to the release of water and the second (related to the double exothermal DSC peak) is due mainly to the release of CO_2. This suggests that decomposition already begins at just above 150°C.

Based on these findings, it can be concluded that the potassium clavulanate supplied has a water content of almost 2% (probably surface water). Since the water loss already begins at slightly above room temperature, a change in the material could be induced by warm and dry storage conditions. At higher temperatures, only decomposition but no melting can be detected.

Compatibility of Materials in a Physical Mixture – Ibuprofen

Here, the thermal behavior of pure ibuprofen (blue-green) is compared to that of pure magnesium stearate (red dashed line) and that of a 90:10 mixture of ibuprofen with magnesium stearate (blue). Magnesium stearate is often applied as an excipient in the production of tablets.

The pure ibuprofen exhibits one DSC peak with an extrapolated onset temperature of approx. 76°C. The main peak of the pure magnesium stearate occurs at 123°C (peak temperature). The DSC profile for the mixture (blue), however, does not reveal what would be expected if the substances were compatible; i.e., this peak plus an additional separate one. Instead, the DSC curve of the mixture reveals a double peak at 67°C and 73°C (peak temperatures). This indicates that there is an interaction between the two substances.

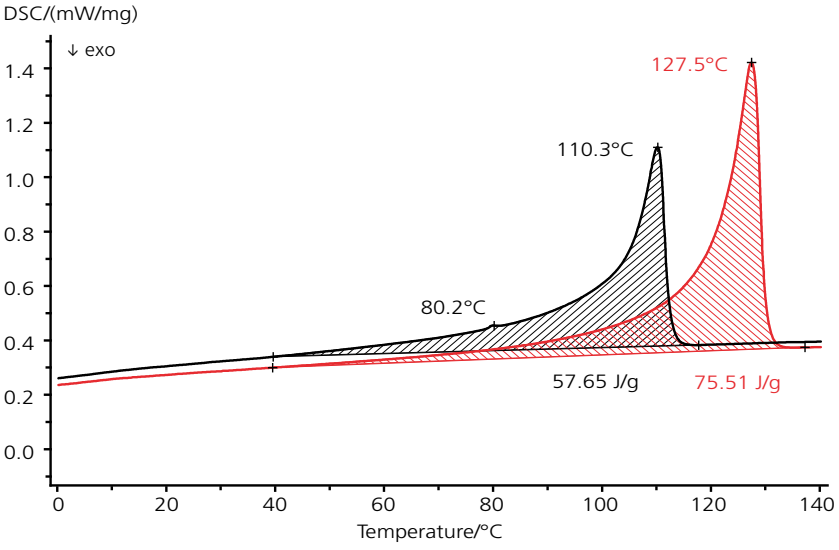


DSC measurement on ibuprofen (blue-green), magnesium stearate (red) and a mixture of both (blue) at a ratio of 90:10; sample masses: 5.2 to 5.8 mg, Al crucibles with pierced lid, heating rate: 10 K/min, nitrogen atmosphere

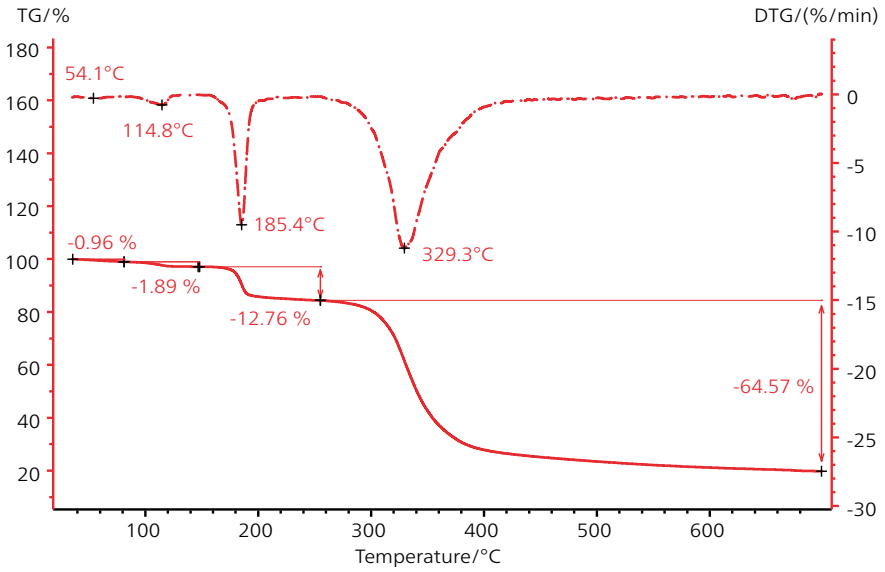
Characterization of Packaging Materials for Quality Control

Here, the DSC profiles of two different compound films based on polyethylene (PE) are depicted. The peak temperatures of the endothermal melting effects indicate that the red curve is correlated to PE-HD (high-density polyethylene), whereas the black curve is most probably related to low-density polyethylene (PE-LD).

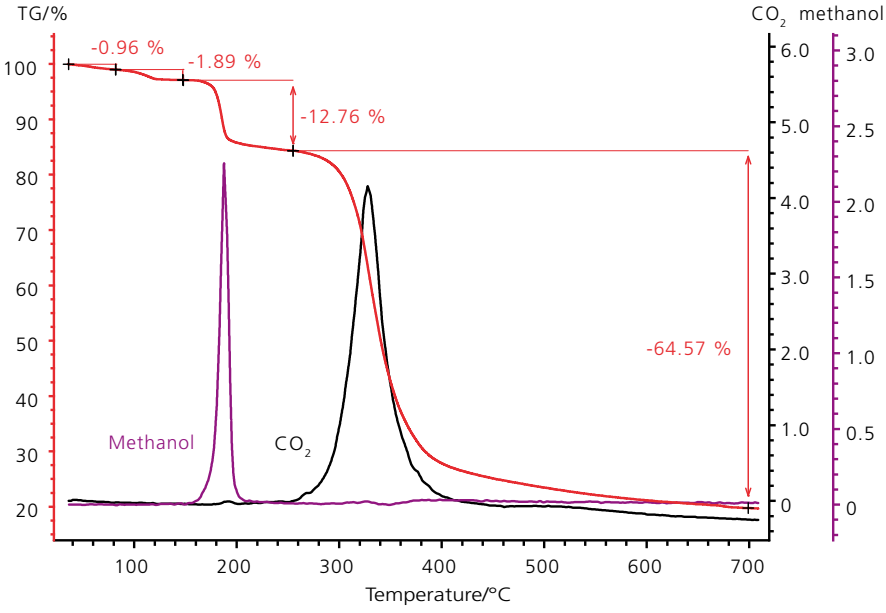
In the case of the black curve, an additional small effect can be seen at approx. 80°C which indicates the presence of further additives.



DSC measurements of two PE-based films, 2nd heating each; sample masses: 5.1 mg, Al crucibles with pierced lid, heating rate: 10 K/min, nitrogen atmosphere



TGA measurement on aspartame; sample mass: 7.5 mg, Al_2O_3 crucible, heating rate: 10 K/min, nitrogen atmosphere

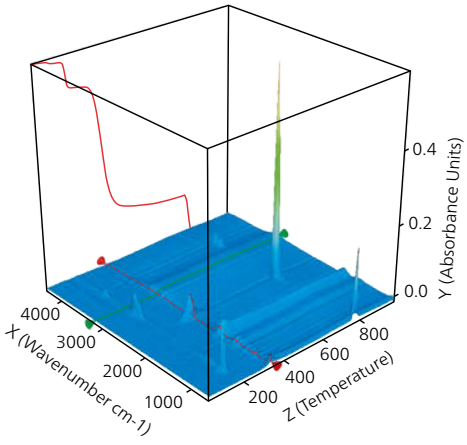


Presentation of the TGA (red) curve of aspartame and the corresponding traces of methanol and carbon dioxide (as examples), derived from the associated FT-IR measurement. A trace represents the course of the absorption intensity of a specific FT-IR band as a function of time or temperature.

Why Does Aspartame Lose Its Sweetening Power During Heating?

Aspartame is not suited for baking and cooking. The reason is that it already starts to decay before it even reaches 250°C.

Derived from the associated TGA-FT-IR experiment, there is a two-step mass change at 54°C and 115°C (DTG peaks), due to the evolvement of surface water (1st step) and dehydration. The mass step of 12.8% at 185°C (DTG peak) is associated with the release of methanol (see lower graph) and is an indicator for the beginning of degradation of the sweetener. In the subsequent step, CO_2 is among the substances which can be found in the gas phase, which suggests further decomposition of aspartame. By comparing the TGA curve with the particular FT-IR traces, you can see at a glance which gas evolves during which mass change.



3D presentation of all measured FT-IR spectra, incl. TGA curve and TGA sample temperature

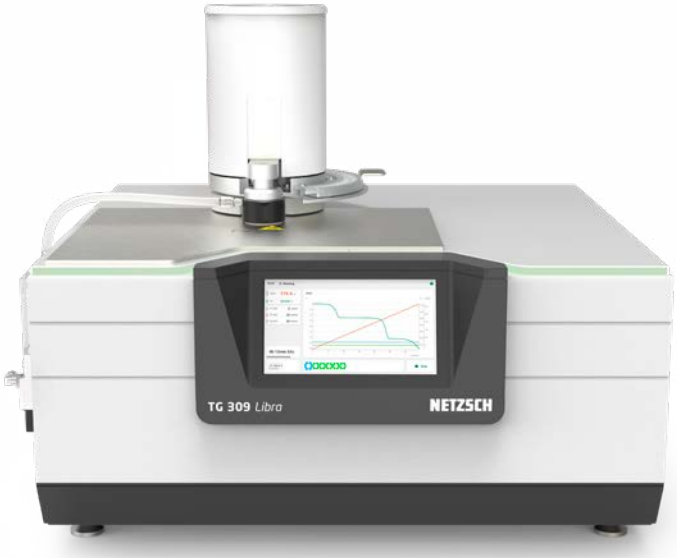
NETZSCH Thermal Analysis Instruments
OFFER EVERYTHING YOU NEED

The Workhorses

TG 309 *Libra® Classic*

The thermobalance for routine analysis, quality assurance and process optimization

- Robust and extremely reliable
- Variety of sample carriers for greatest flexibility
- c-*DTA®* for monitoring caloric effects
- Vacuum-tight design
- Corrosion-resistant ceramic furnace
- High sample throughput thanks to the automatic sample changer with 20 positions (optional)



DSC 300 *Caliris® Classic*

Heat-flux DSC ideal for routine analysis, quality control, at-line operation and teaching

- Space-saving design
- Easy-to-use, robust and precise
- High sample throughput thanks to the automatic sample changer with 20 positions (optional)

LabV®-primed

NETZSCH instruments are compatible with the LabV® data management platform, a user-friendly software that automates data collection, regardless of method or device, and provides a centralized view for organizing, analyzing, and exploring your data. LabV®'s AI-powered digital assistant simplifies data analysis, allowing labs to easily find insights with no effort. It uses natural language processing, similar to ChatGPT, making it easy for labs to create visualizations, spot trends, and uncover complex correlations with straightforward commands.

NETZSCH – MORE THAN 60 YEARS OF EXPERIENCE IN MANUFACTURING TOP-CLASS THERMAL ANALYSIS DEVICES.

	TG 309 *Libra*® *Classic*	DSC 300 *Caliris*® *Classic*
Temperature range (max.)	(10°C) RT to 1025°C	-170°C to 600°C
Cooling rate/heating rate (max.)	0.001 to 200 K/min	0.001 to 100 K/min
Measuring range/ weighing range (max.)	2000 mg*	± 750 mW
Enthalpy accuracy	n/a	< 1%**
TGA resolution	50 ng	n/a
Exchangeable sensors	Yes	n/a
Cooling options	n/a	▪ Compressed air: RT to 600°C ▪ Intracooler: -70°C to 600°C ▪ Liquid nitrogen: -170°C to 600°C
Gas atmospheres	Inert, oxidizing, static and dynamic	Inert, oxidizing, static and dynamic
Mass flow controller for purge/protective gas	Optional, up to 4 (0 to 250 ml/min)	3, optional (0 to 250 ml/min)
Automatic Sample Changer (ASC)	Optional	Optional
Display	Optional	Optional
UV extension	N/A	Optional
Proteus® software	▪ *SmartMode* ▪ *ExpertMode* ▪ *AutoEvaluation* ▪ *AutoCalibration* ▪ c-*DTA*® ▪ Report generator ▪ Eco Mode ▪ TGA-*BeFlat*® ▪ Predefined methods	▪ *SmartMode* ▪ *ExpertMode* ▪ *AutoCalibration* ▪ Oxidative-Induction Time (OIT) ▪ *BeFlat*®+ ▪ *TauR*®
Software extensions (optional)	▪ Temperature modulation ▪ *Proteus*® *Protect* ▪ *Identify* ▪ *Peak Separation* ▪ Kinetics Neo ▪ *Super-Res*® ▪ LIMS support ▪ *Proteus*® *Search Engine*	▪ Temperature modulation ▪ Specific heat capacity (c_p) ▪ *Proteus*® *Protect* ▪ *Purity Determination* ▪ *Peak Separation* ▪ Kinetics Neo ▪ *AutoEvaluation* ▪ *Identify* ▪ LIMS support ▪ *Proteus*® *Search Engine* ▪ KIMW database for polymers
Size (W x H x D) – incl. ASC, without physical connections	575 mm x 460 mm x 560 mm	415 mm x 480 mm x 570 mm

* minus weight of crucible
** for indium, adamantane, zinc

Flexibility at Its Finest

TG 309 *Libra® Select/Supreme*

High-quality ultra-microbalance for research and development

- Vacuum-tight design
- Corrosion-resistant ceramic furnace
- c-*DTA®* for monitoring caloric effects
- Large filter system for direct trapping of decomposition products
- Pre-configured for coupling to gas analyzing systems (FT-IR, MS or GC-MS)
- High sample throughput for measurements over night or during the weekend – automatic sample changer for up to 192 samples and automatic piercing device (optional)

DSC 300 *Caliris® Select/Supreme*

Premium differential scanning calorimeter

- Various exchangeable (*Select*) or interchangeable (*Supreme*) modules optimized for different applications
  - Standard module
  - Polymer module
  - High-performance module
- Extremely efficient – automatic sample changer for up to 192 samples and automatic piercing device (optional)
- Touch display and LED status bar

LabV®-primed

STA 449 ***F3*** *Nevio*

Specialized for demanding tasks

- Modular setup, adjustable to a variety of requirements, e.g., for measurements under relative humidity or for TGA experiments on large samples
- Vacuum-tight
- Pre-configured for coupling to gas analyzing systems (FT-IR, MS or GC-MS)
- Automatic sample changer for up to 20 samples (optional)

	TG 309 *Libra*® *Select/Supreme*	DSC 300 *Caliris*® *Select/Supreme*	STA 449 ***F3*** *Nevio*
Temperature range (max.)	(10°C) RT to 1025°C/1100°C	-180°C to 750°C (H-Module)	-120°C to 675°C[3]
Max. cooling rate/ max. heating rate	0.001 K/min to 200 K/min	500 K/min (P-Module)	50 K/min
Measuring range/ weighing range (max.)	2000 mg[1]	± 750 mW	35000 mg[1] /± 250 mW[4]
Enthalpy accuracy	n/a	< 1%[2]	± 1 ... 3%
TGA resolution	20ng/10ng	n/a	0.1 µg
Sensors	Interchangeable	▪ Exchangeable furnace-sensor module (*Select*) ▪ Interchangeable furnace-sensor module (*Supreme*)	Interchangeable
Cooling options	n/a	H-Module: ▪ Air compressor: RT to 750°C ▪ Compressed air: < 0°C to 750°C ▪ Intracooler: -90°C to 600°C ▪ Liquid nitrogen: -180°C to 750°C	▪ Compressed air: < 0°C to 675°C[3] ▪ Liquid nitrogen: -150°C to 1000°C[3]
Gas atmospheres	Inert, oxidizing, static and dynamic	Inert, oxidizing, static and dynamic	Inert, oxidizing, reducing (forming gas), humid, vacuum, static, dynamic
Gas-tight/vacuum-tight	<<10^{-1} mbar	Gas-tight	Vacuum-tight
Mass flow controller for purge/protective gas	*Select*: 4, optional *Supreme*: 4, included (0 to 250 ml/min)	*Select*: 3, included *Supreme*: 4, optional (0 to 250 ml/min)	3, optional (0 to 250 ml/min)
Automatic Sample Changer (ASC, optional)	192+12 positions	192+12 positions	20 positions
Proteus® software	▪ *SmartMode* ▪ *ExpertMode* ▪ *AutoCalibration* ▪ c-*DTA*® ▪ *AutoEvaluation* ▪ *Identify* ▪ Temperature modulation *(Supreme)* ▪ *Peak Separation (Supreme)*	▪ *SmartMode* ▪ *ExpertMode* ▪ *AutoCalibration* ▪ Specific heat capacity ▪ *AutoEvaluation* ▪ *Identify* ▪ Temperature modulation ▪ *Peak Separation* ▪ LIMS support ▪ *Proteus® Search Engine* ▪ *OIT*	▪ *AutoEvaluation*
Software extensions, (optional)	▪ Temperature modulation (*Select*) ▪ *Peak Separation (Select)* ▪ *Proteus® Protect* ▪ Kinetics Neo	▪ *Proteus® Protect* ▪ Kinetics Neo ▪ *c-DTA®* ▪ *Identify* ▪ Specific heat capacity ▪ *Purity Determination*	▪ Temperature modulation ▪ *Proteus® Protect* ▪ *Peak Separation* ▪ Kinetics Neo ▪ *c-DTA®* ▪ *Identify* ▪ Specific heat capacity

1) minus weight of crucible
2) for indium, adamantane, zinc
3) optimized temperature range for pharmacy, cosmetics and foodstuffs; depending on the selected furnace: total temperature range: -150°C up to 2400°C
4) for type E thermocouple
5) in Al_2O_3 crucibles

MEASUREMENTS UNDER HUMID ATMOSPHERES

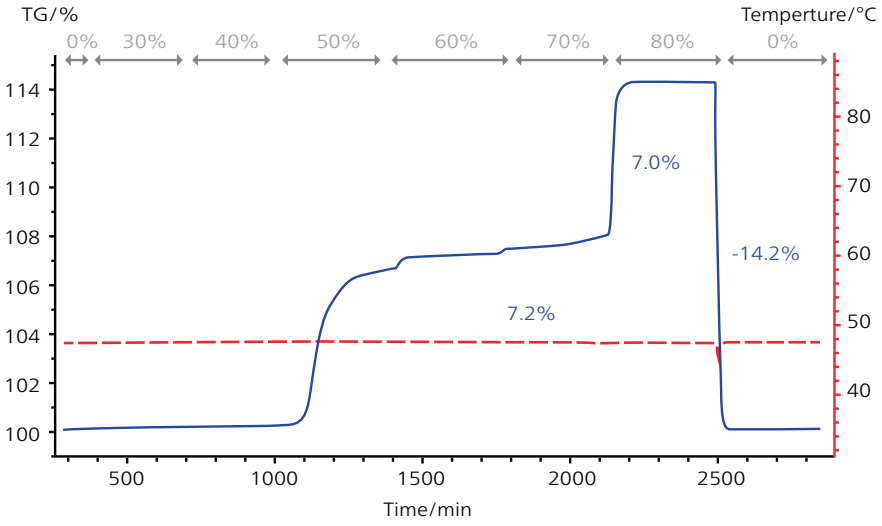
Moisture can be found almost everywhere in the surroundings – sometimes more, sometimes less, depending on the climate zone and the season. However, the presence of water may have an impact on the properties and the handling of powder materials in many ways. It can induce swelling, the formation of hydrades, hydrolysis or degradation reactions (even resulting in toxic degradation products), or it can affect a material's glass transition or degree of crystallinity – to name just a few consequences. Therefore, it is important to know how APIs, excipients, formulations, food stuff, etc., will behave in contact with moisture.

Versatile STA: One Instrument – Many Measurement Options

One way to quantify the interaction with water is to measure the mass change during water uptake or water loss using thermogravimetric analysis; i.e., when working under a controlled humid atmosphere, it is possible to study the sorption/desorption capability of a substance. The corresponding method is called Dynamic Vapor Sorption, or DVS.
A suitably configured Simultaneous Thermal Analyzer (STA) in combination with a humidity generator (see Figure 1) is capable of carrying out test runs under dry gas atmosphere as well as under humid conditions. To achieve the largest possible contact area between the sample and the surrounding atmosphere, typically TGA sample holders with plates or nets are applied (Figure 2).
To get a feel for the relationship between temperature, humidity level and associated dew point, a humidity calculator is included into the instrument software (*Proteus*®).

Fig. 1. STA 449 ***F3*** *Nevio* and humidity generator

Fig. 2. TGA sample holder with Al_2O_3 slip-on plate



STA investigation of 35.8 mg of sodium naproxen using an STA 449 ***F3*** instrument in combination with a humidity generator. TGA sample carrier with Al_2O_3 slip-on plate.

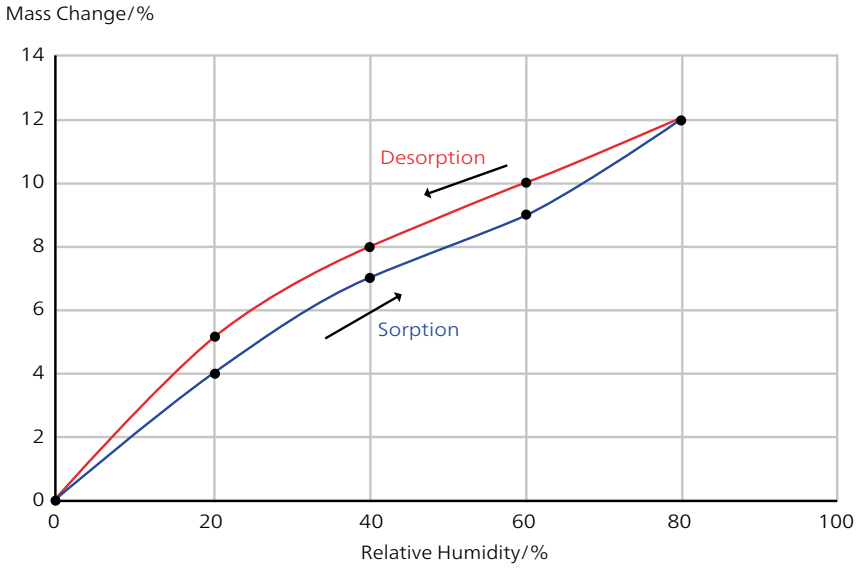
Sodium Naproxen

Sodium naproxen, a pain reliever, can occur as anhydrate, in two different dihydrate forms and as tetrahydrate.

After keeping commercial sodium naproxen at 90°C under dry nitrogen gas, the temperature was reduced to 48°C and the relative humidity level of the atmosphere inside the instrument stepwise increased: from 0% to 80% and then immediately back to 0%.

The detected mass gains of 7.2% (between 0% and 60% rel. humidity) and 7.0% (between 60% and 80% r.h.*) indicate that – with the anhydrate as starting point – first the monohydrate and then a dihydrate is formed. By reducing the relative humidity level again to 0%, all absorped water is released.

* r.h. = relative humidity



Sorption hysteresis; plot of the resulting mass change versus the applied relative humidity

Microcrystalline Cellulose

Microcrystalline cellulose (MCC) is commonly employed as a filler or binder during tablet formulation. In dietary foods, it serves, among other things, as an indigestible fiber.

The sorption hysteresis shown on the left is the result of measuring 41.2 mg of microcrystalline cellulose at 44°C while the relative humidity was changed (increased and subsequently decreased) in 5 equidistant steps between 0% and 80%.

For higher molar water concentrations (up to 100% water vapor), the STA instrument can be equipped with a water generator instead of a humidity generator.

EVOLVED GAS ANALYSIS

Unveiling the Processes Behind Mass Losses

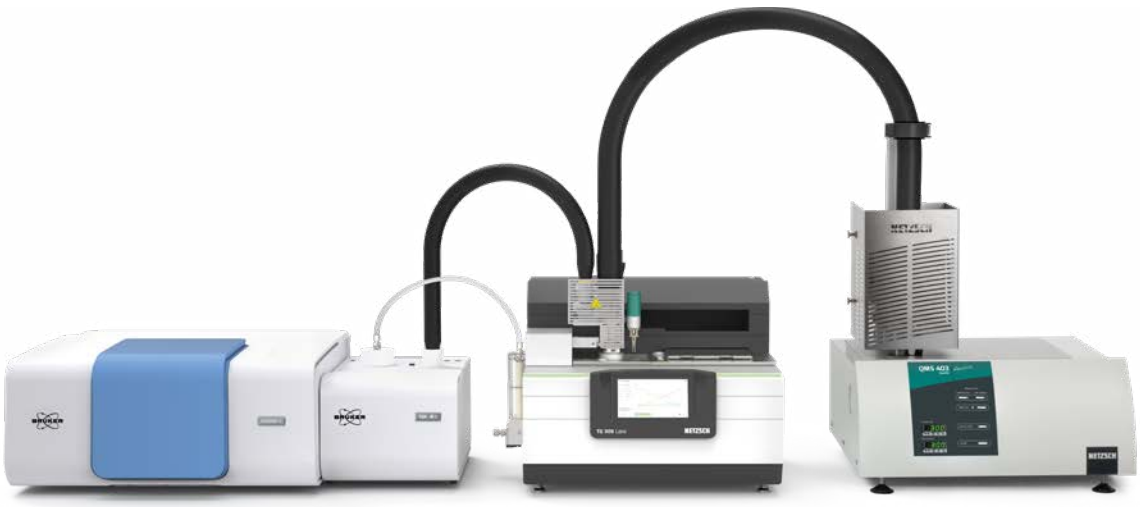
Coupling a gas analyzing system to the TG 309 *Libra*® *Select/Supreme*, the STA 449 ***F3*** *Nevio* or TMA 402 allows for deeper insight into a material's behavior and facilitates identification and quantification of the gases released.

The following coupling possibilities are available:

	PERSEUS®** FT-IR	*Aeolos* Quadro MS (via capillary)	FT-IR (via capillary)	GC-MS (via capillary)	PERSEUS®** FT-IR + MS	PERSEUS®** FT-IR + GC-MS	FT-IR (capillary) + MS
TG 309 *Libra*®	✓	✓	✓	✓	✓	✓	✓
STA 449 ***F3*** *Nevio**	✓	✓	✓	✓	✓	✓	✓
TMA 402***	-	✓	✓	-	-	-	✓

* temperature range of the instrument is depending on the selected furnace; total temperature range: -150°C to 2400°C
** PERSEUS® corresponds to direct coupling, without transfer line
*** not combinable to TMA 402 *Hyperion* with IC furnace

TGA and STA combinations can additionally be equipped with an automatic sample changer (ASC). In these cases, the *Proteus*® software is capable of operating and controlling the thermal analyzer and the gas analyzer simultaneously.



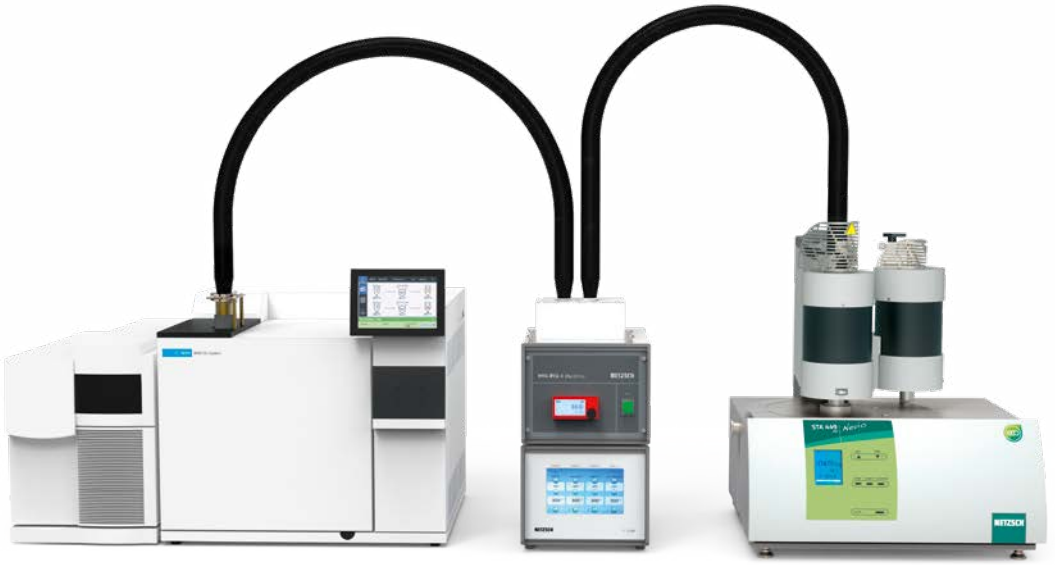
TG 309 *Libra*® simultaneously coupled to an FT-IR (Bruker INVENIO with external gas cell) and MS (QMS 403 *Aëolos Quadro*)

Top-Loading Arrangement of NETZSCH TGAs Ensures Optimum Protection of the Balance

Warm gases have a tendency to rise. This chimney effect causes the purge gas (which comes from below in top-loading balances) to pull the released products away from the sample and transport them to the gas outlet at the top of the furnace. The risk of contaminating the balance (which is located beneath the sample) is thus minimized; this makes top-loading balances ideal for coupling to evolved gas analyzers.

NETZSCH Systems Provide a Pure Inert Gas Atmosphere for Evolved Gas Analysis (EGA)

In order to detect minimal concentrations of evolved products in the gas phase, the background level in a gas analyzing system has to be as low as possible, especially with regard to H_2O and CO_2 coming from the environment. Additionally, mass spectrometers used as GC detectors are very sensitive to greater amounts of residual oxygen. Thus, a prerequisite for the successful investigation of released gases is vacuum tightness, which is the case for any NETZSCH TGA/STA/TMA instrument.



GC-MS coupled to a STA 449 ***F3*** *Nevio*

PERSEUS® TG 309 *Libra®*

Saving 50% of Benchtop Space with the PERSEUS® FT-IR-Coupling

PERSEUS® is the name of a unique TGA-FT-IR system including a compact Bruker Optics FT-IR spectrometer. The built-in heated gas cell is directly connected to the gas outlet of the TGA furnace. The low volume of the short transfer path guarantees fast transport and is advantageous for condensable gases.

THERMOMECHANICAL ANALYSIS

Precise Determination of Dimensional Changes on Solids, Powders, Pasty Materials and Liquids

TMA 402 *Hyperion*® with IC furnace[1] – its core task is to determine elongation or shrinkage of a sample material

Detection of Smallest Length Change Effects

Glass transitions sometimes show just small changes in specific heat capacity resulting in very tiny steps in DSC curves. This is the moment when mechanical methods, such as thermomechanical analysis, come into play. These techniques are often more sensitive to determine weak glass transition effects because they measure the change in elongation (in static mode) or the increasing compliance of the sample (in dynamic mode).

This can be applied to packaging, coating materials (e.g., for tablets) or amorphous (or partly amorphous) foodstuff.

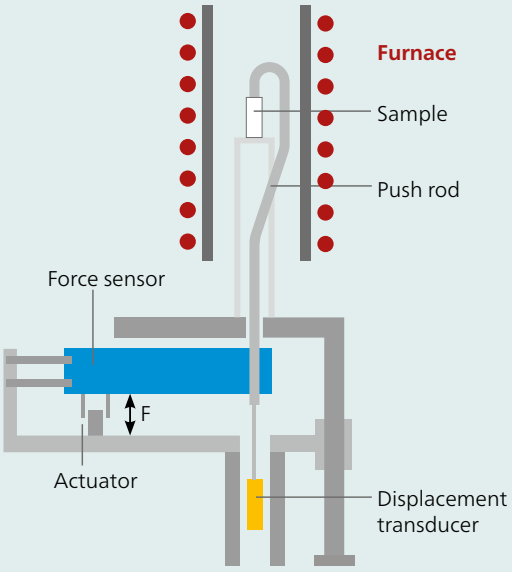
Maximum Flexibility

But TMA instruments are capable of detecting also length change phenomena caused by further effects. It may be due to phase transitions or melting (in special containers) or due to swelling in humid atmosphere. Thanks to a large selection of accessories, a wide range of application requirements can be met.

Coupled to a gas analyzing system such as MS (mass spectrometer) or FT-IR (Fourier Transform Infrared, see on the previous pages) it is even feasible to simultaneously identify if, for example, the occurring shrinkage is associated with water or solvent loss.

TMA 402 *Hyperion*®	
Force range	0.001 N to 3N, in steps of 0.02 mN without using additional weights
Measurement modes	Expansion, penetration, 3-point bending, tension; static and dynamic (applying a modulated force with a certain frequency)
Temperature range	-70°C to 450°C[1]
Cooling system	Intracooler or forced air[1]
Interchangeable sample holder systems	Fused silica
Special sample container	For tests on pastes, powders, liquids, waxes, molten metals, immersion
Sample dimensions	▪ Length: 30 mm max.; fused silica sample holder Ø 12 mm / 8 mm; ▪ Automatic sample length determination (precision: 0.01 mm)

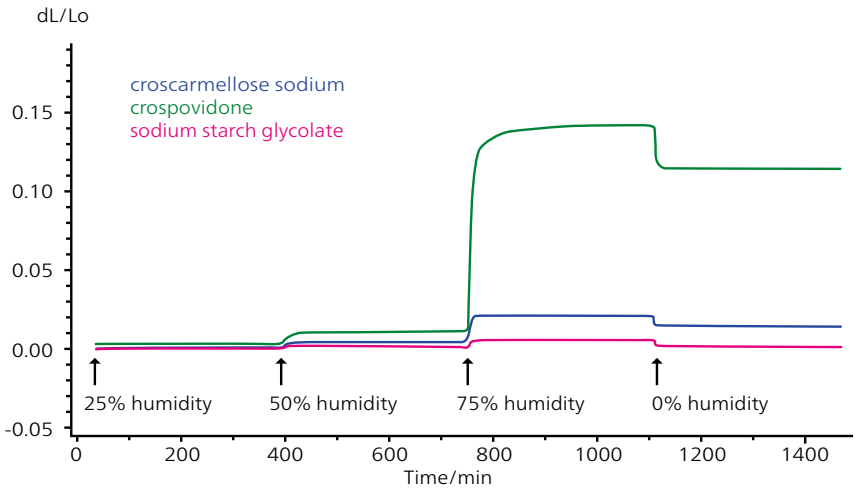
[1] optimized temperature range and cooling system for standard measurements on pharmaceuticals, cosmetics and food stuffs; IC means intracooler; further furnaces available – total temperature range: -150°C up to 1550°C; (e.g., special water-vapor furnace for measurements under humid conditions or furnace for coupling to evolved gas analysis available)



TMA Results (inter alia)

- Linear thermal expansion
- Coefficient of thermal expansion
- Volumetric expansion
- Shrinkage steps
- Density changes
- Phase transition temperatures
- Glass transition temperatures
- Decomposition temperatures
- Isostrain
- Creep
- Relaxation
- Stress/strain curve

During a TMA experiment (in expansion, tension, penetration or bending mode), the pushrod is always in direct contact with the specimen and – while a certain force is applied – detects any expansion or shrinkage the sample material may undergo during heating/cooling. The pushrod and corresponding sample holder are usually made of fused silica (in the temperature range up to 1100°C max.). The pushrod itself is connected to a sensitive displacement transducer (LVDT) which transforms its movement into an electrical signal.



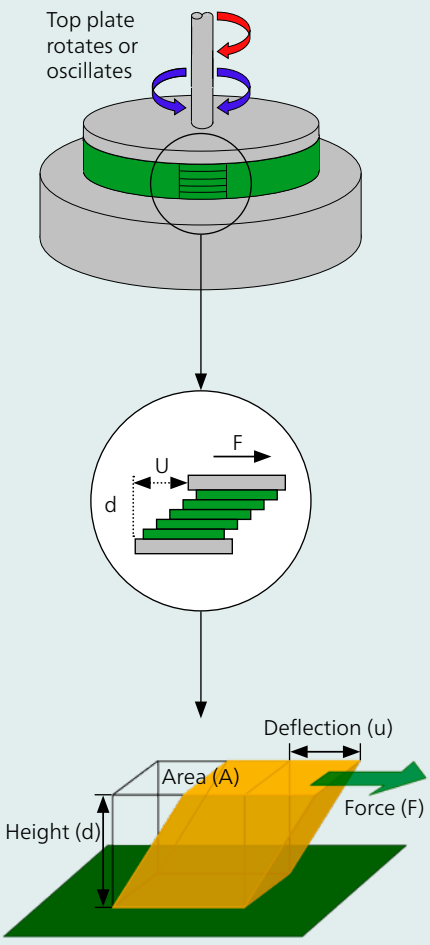
TMA measurements on tablets consisting of 95% filler (calcium hydrogenphosphate dihydrate), 1% lubricant (sodium stearyl fumarate) and 4% superdisintegrant (croscarmellose sodium, crospovidone or sodium starch glycolate). The test was run at 40°C with a 30 min preconditioning time at 0 % r.h.

Effectiveness of Disintegration of Tablets

Disintegrants help break a tablet apart and to release the included API by reacting with water. The main mechanisms of this hydration reactions are wicking, swelling and shape recovery. Most of these substances exhibit one dominating disintegration mechanism such as croscarmellose sodium (wicking) or sodium starch glycolate (swelling), but some of them, e.g., crospovidones, show swelling, wicking and shape recovery together.

RHEOLOGY

AS CLOSE AS POSSIBLE TO PRODUCTION AND FINAL APPLICATION



Rheology Results

- Dynamic viscosity
- Flow behavior (Newtonian, shear thinning, shear thickening)
- Yield stress
- Thixotropy (time-dependent structural recovery or rebuild)
- Melting/crystallization
- Temperature-dependent flow behavior and viscoelastic properties
- Creep test/creep recovery
- Stress relaxation
- Viscoelastic properties (moduli)
- Kinetics (combined with Kinetics Neo)

Rotational Measurement

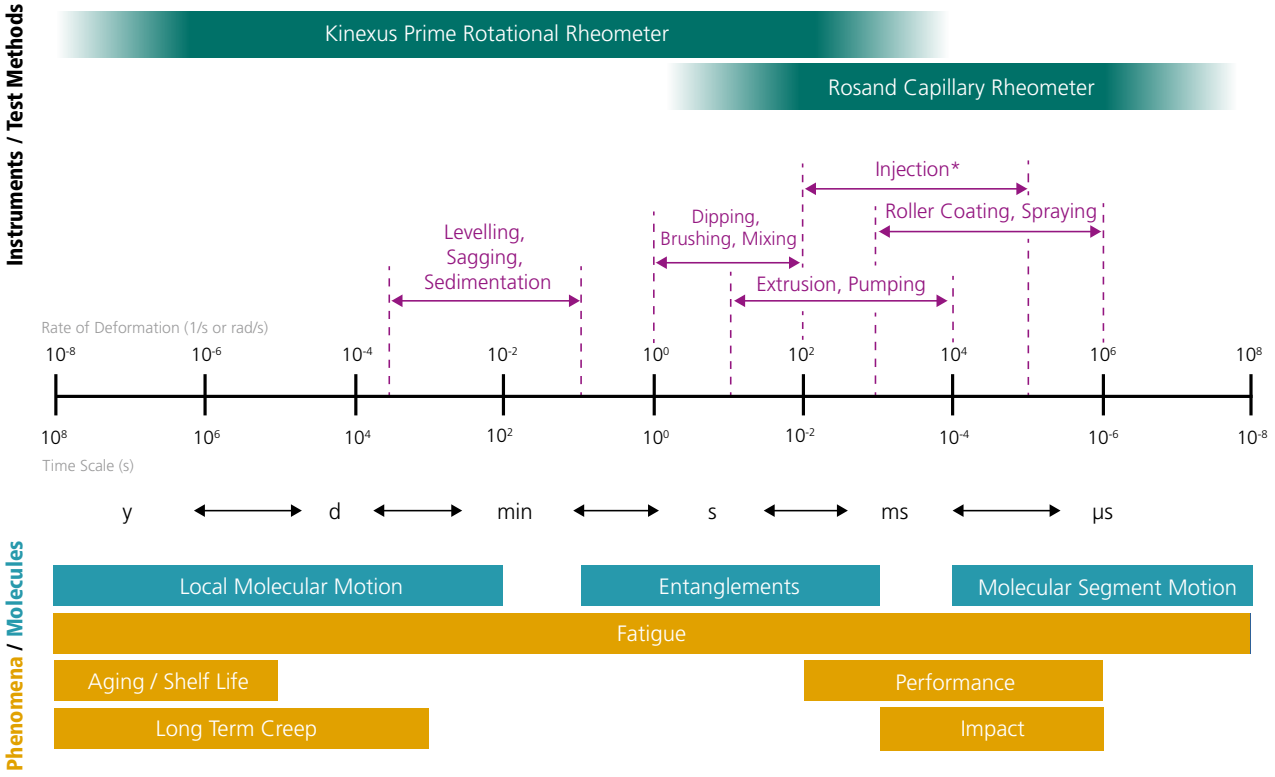
The upper plate rotates continuously under a controlled shear stress or strain.
The application of shear stress [F/A] induces deformation in the sample. The extend of that deformation [u] relative to the depth of the sample [d] is defined by the term shear strain;
The rate of change in strain with time yields the shear rate.
Viscosity quantifies a material's resistance to flow and is mathematically defined as shear stress divided by shear rate.

Oscillation Measurement

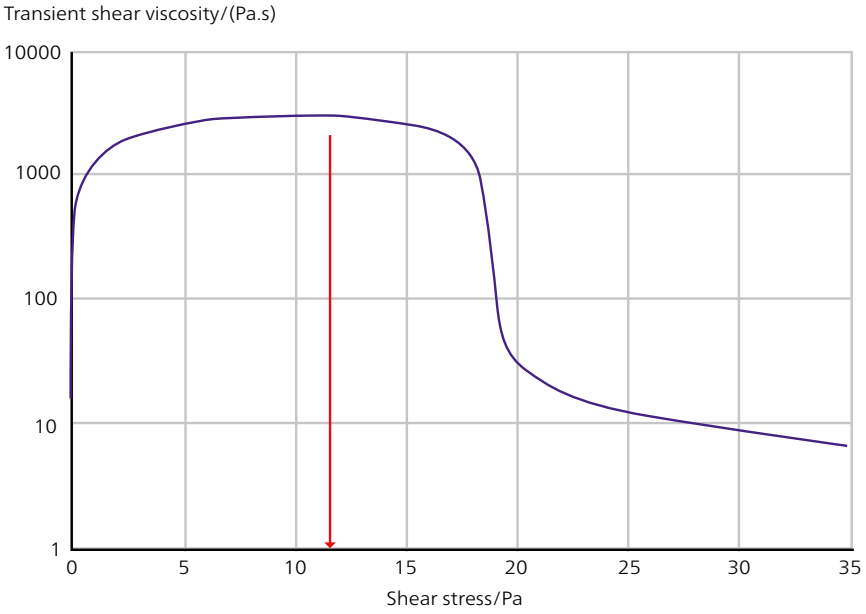
The upper plate oscillates with a defined frequency and amplitude. The shear stress required for this oscillation is measured and the viscoelastic properties of the sample are determined, in particular its complex shear modulus G*. The "in-phase" part of G* is related to the elastic properties (→ G', storage shear modulus), the "out-phase" part to the viscous properties (→ G'', loss shear modulus) of the viscoelastic material.

The dynamic viscosity of matter is not a single-point value but a function of environmental conditions such as shear rate, shear stress or temperature. As the shear rate $\dot{\gamma}$ (the rate of change in shear strain over time) is capable of simulating various application/administration situations, rheometry is an important tool in assessing the behavior of substances, formulations/mixtures or final products with regard to storage, processing or handling. Thus, it is possible to obtain information about material properties at near-rest by using low shear rates. On the other hand, measuring how a cream spreads on the skin requires a much higher shear rate.

Whereas low- and mid-shear rates are typical for rotational rheometers, capillary rheometers operate in the mid-to-high shear rate range. This allows for production processes such as hot melt extrusion to also be mapped. Classical viscometers cover only a relatively narrow range in the mid-shear rate region.



* 1 mL/10s; 27G needle (doi:10.1016/j.ejpb.2014.01.009)



Yield stress measurement – geometry: cone-plate 1/50; measurement gap: 0.03 mm; temperature: 35°C, shear stress ramp as quick check

Yield Stress Determination for a Hand Cream

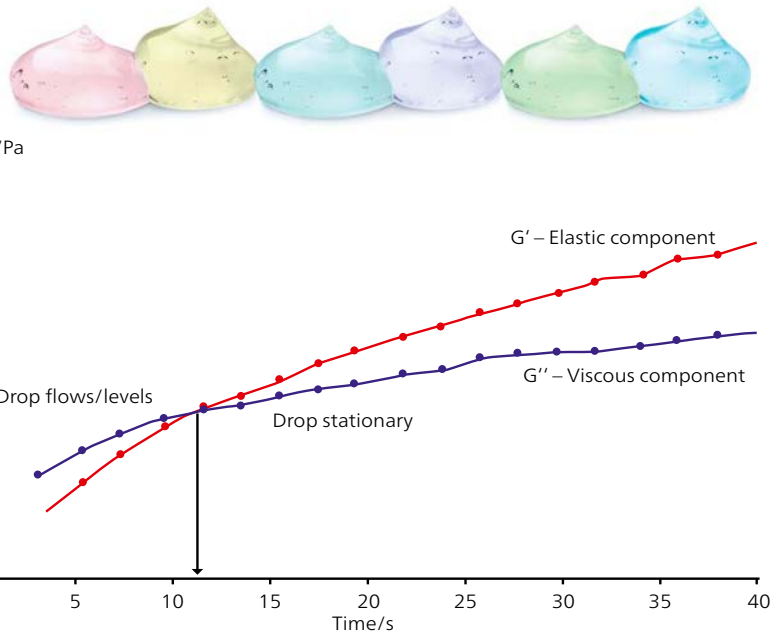
Creams should remain in the tube until they are squeezed out. This feature is reflected by the so-called yield stress, i.e., the minimum stress that must be applied to a material to induce its flowing.

If the shear viscosity (in Pas) is plotted versus the shear stress (in Pa), the yield stress can be determined from the curve's maximum. The hand cream in the present case starts to flow at a value of 11.7 Pa which corresponds to the peak in transient viscosity (see red arrow).

Thixotropy of an Eye Drop Gel

In order to reduce the drug loss by tear drainage and blinking action and thus to increase bioavailability of the pharmaceutical, so-called in-situ gels are often used. They are instilled as low-viscosity solutions into the conjunctival sac of the eye and gel after several seconds.

The behavior of such formulations can be simulated by a pre-sheared oscillation test. After a high-shear step to simulate the application process, the oscillation monitors the viscoelastic properties of the sample which is rebuilding after the shear. In such an experiment, the upper geometry of the rotational rheometer oscillates and applies small sinusoidal shear waves to the sample without breaking the structure. This type of operation results in modulus values, particularly in the storage modulus (elastic component), G´, and the loss modulus (viscous component), G". At the point where G´ exceeds the level of G", the drug product does no longer flow under the time scale of the applied frequency and remains in the same position.

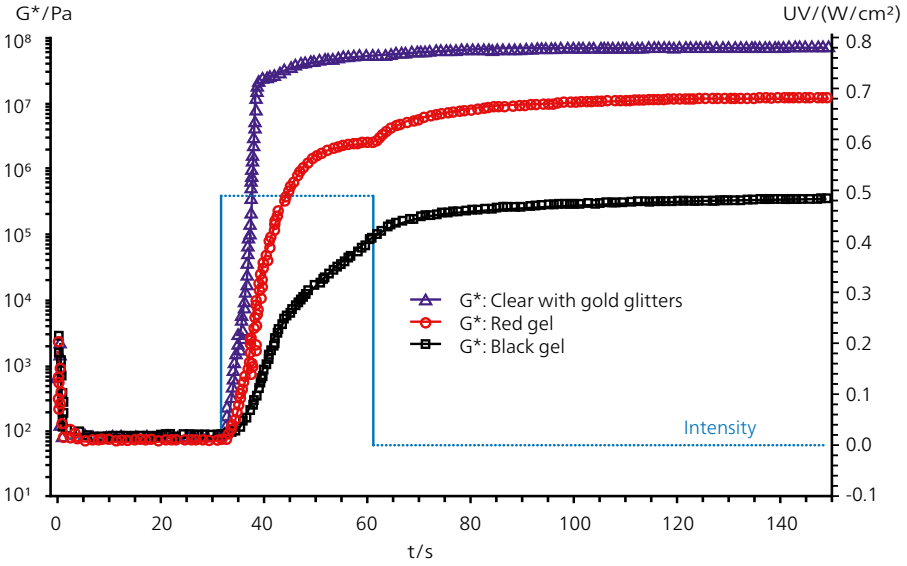


Measurement of the time-dependent property change of an eye drop gel

Nail Gel Curing

The perfect nail gel or polish should feel relatively liquid for an easy brush application but without flowing outside the nail. The drying or curing time should be as low as possible and lead to a smooth surface for a flawless appearance.

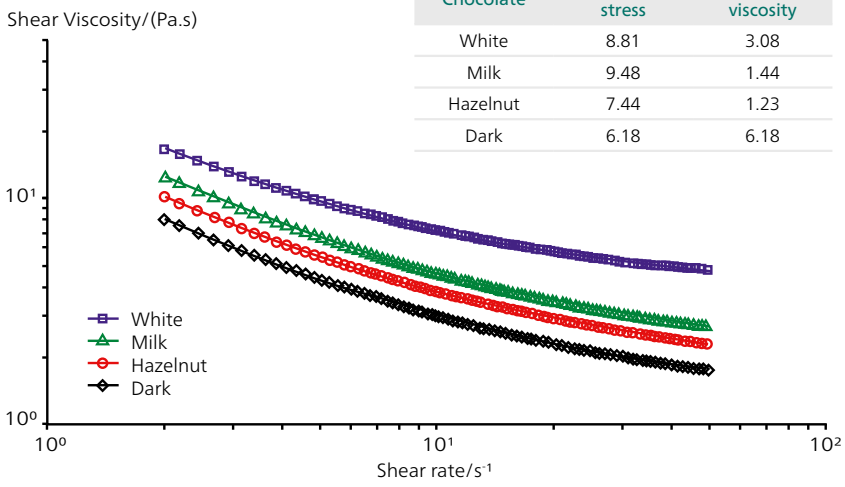
Some types of nail gels require a UV-lamp for curing. These products contain a photo-initiator that will initiate the curing reaction as soon as the gel is in contact with the suitable wavelengths emitted by the lamp. The sample colors were red, black and clear. The clear sample contained suspended glitters. Before curing, all samples possess a similar complex shear modulus of 70 - 80 Pa. The significant increase in modulus indicates curing has started and the slope of the curve is related to the reaction speed. The clear nail gel with glitters cures fastest and the black sample shows the slowest curing of the three samples. The samples differ also in their final modulus. The modulus of the clear gel with glitters increases by 6 decades during curing, vs. less than 4 decades for the black gel. This means that that the clear gel reveals the highest stiffness after curing. A faster cure is advantageous for the consumer. However, the final properties of the manicure after application are, of course, also important.



Complex shear modulus of three different nail gels

Characterization of Rheological Properties of Chocolate

The processing of melted chocolate in pipes, its mold filling and even the mouth feel of the consumer are closely related to its viscosity. The figure shows shear viscosity curves of different chocolate types from the same manufacturer: white, milk, hazelnut and dark. All these chocolates differ in their ingredients and in the concentration of each component. For instance, white chocolate contains no cocoa mass, while being the key ingredient of dark chocolate.
Shear viscosity and yield stress are the main rheological properties of interest in the chocolate industrial processes. The shear viscosity is simply calculated by dividing the shear stress by the shear rate. The yield stress is determined applying different model functions, like the Casson model, using the flow-curve diagram.



Chocolate	Yield stress	Casson viscosity
White	8.81	3.08
Milk	9.48	1.44
Hazelnut	7.44	1.23
Dark	6.18	6.18

Viscosity curves of 4 different chocolates at 40°C (rotational rheometer: Kinexus Prime ultra+, geometry: cup and bob 34 mm, temperature: 40°C)

Sample measured in accordance with the Analytical Method 46 issued by the International Office of Cocoa, Chocolate and Sugar Confectionary.

Selection of Accessories for Kinexus Prime Rheometer

Kinexus Rotational and Rosand Capillary Rheometers

EXEMPLIFYING VERSATILITY

Rotational rheometers are typically used for low-shear applications for determining structural information. Capillary rheometers, on the other hand, are preferred when measuring process-relevant flow properties.

Kinexus Prime Rotational Rheometer

Kinexus Prime rotational rheometers are available in three different versions: as lab+, pro+ and ultra+. All of them are robust, easy-to-use workhorses capable of operation in the direct strain control, the shear rate control and the shear stress control mode. The high-end device within this series is the ultra+, which offers the highest level of torque sensitivity available, ensuring reliable measurements on even highly fluid (low-viscosity) samples.

Kinexus Prime ultra+ rotational rheometer

Rosand Capillary Rheometer

Rosand capillary rheometers are available as table-top (RH2000) or floor-standing units (RH7 or RH10), depending on the drive force which is necessary to extrude the sample material.

Rosand capillary rheometer RH2000

Both series can be equipped with a variety of quick-connect accessories to realize a multitude of applications. This includes (among others):

For the Kinexus Prime series:

- Plug & play cartridge systems for temperature control
- Cone & plate as well as plate & plate geometries of various sizes, surface conditions or cone angles
- Cup & bob geometries with concentric cylinders of different diameters
- Broad range of accessories and customized solutions

For the Rosand series:

- Barrels made of different materials and with different bore sizes
- Option for measurement at sub-ambient temperature
- Alternative pressure transducers
- Slot die assembly

Each in the Top Class by Itself ....
Moreover Unbeatable in Pairs

Only the NETZSCH Rheometer Line Covers Both Low and High Shear Rates!

Suspensions intended for spraying can be found in a variety of cosmetics and pharmaceutical formulations. They consist of a heterogeneous mixture of a liquid and a solid finely dispersed therein. In order to optimize the composition of the product, its particle and droplet size distribution, and its sedimentation time, a wide range of shear rates needs to be applied. This is only feasible when rotational rheometers and capillary rheometers are combined.

Rotational Rheometer

	Kinexus Prime lab+	Kinexus Prime pro+	Kinexus Prime ultra+
Max. temperature range	-40°C to 450°C	-40°C to 450°C	-40°C to 450°C
Normal force range	0.001 N – 50N	0.001 N – 50N	0.001 N – 50N
Normal force response time	< 10 ms	< 10 ms	< 10 ms
Raw instruments variables	5 Hrz constant streaming data	5 Hrz constant streaming data	5 Hrz constant streaming data
Quick-connect cartidge system and geometries	Plug and play; auto-recognition and configuration by the software		
Torque range – viscometry	10 nNm – 200 mNm	5 nNm – 225 mNm	1 nNm – 250 mNm
Torque range – oscillation	5 nNm – 200 mNm	1 nNm – 225 mNm	0.5 nNm –250 mNm
Frequency range	1 µHz – 100 Hz	1 µHz – 150 Hz	1 µHz –150 Hz

Capillary Rheometer

	RH2000	RH7	RH10
Temperature range	Ambient to 400°C (500°C option) 5°C to 300°C (low-temperature cooling coil option)		
Max. force	12 kN (20 kN optional)	50 kN	100 kN
Max. speed	600 mm/min (1200 mm/min optional)	600 mm/min	1200 mm/min
Number of bores	Single/double bore	Double bore	Double bore
Bore material	Nitrided steel standard (Hastelloy or stainless steel options)		
Pressure transducer range	30000 down to 250 psi, accuracy ± 0.25 psi high-temperature pressure tranducer from 35 bar up to 2000 bar		
Dies	Tungsten carbide, precision ± 5µm		
Diameter of the dies	0.5 to 2 mm (in 0.5 mm increments) and 3 mm standard (other diameters, including fine bore dies, available to special order)		
Maximum shear rate	up to ≈ 10^8 s^{-1} (depending on die and barrel diameter, and piston speed)	up to ≈ $5*10^7$ s^{-1} (depending on die and barrel diameter)	up to ≈ 10^8 s^{-1} (depending on die and barrel diameter)

ADIABATIC CALORIMETRY

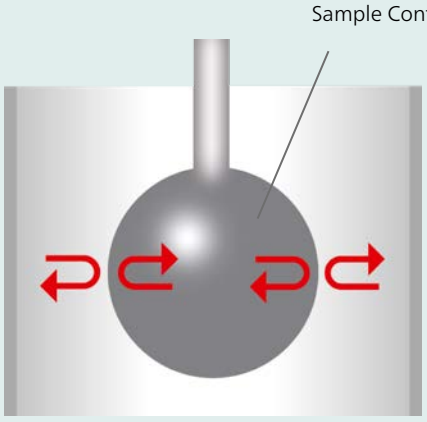
Clear Assessment of the Thermal Hazard Potential of Chemical Reactions

Thermal Process Safety

Estimation of the risk of a chemical reaction and the establishment of related preventive measures is usually based on worst-case scenarios. A worst-case scenario describes a situation in which temperature and/or pressure production caused by a reaction run out of control.

Adiabatic Calorimetry Results

- Temperature increase during thermal runaway
- Pressure increase
- Enthalpy of decomposition
- Time-to-maximum-rate (TMR) under adiabatic conditions
- Isothermal aging
- Kinetics (combined with Kinetics Neo)
- Determination of exothermal and endothermal events (scanning module)



Adiabatic System: No heat in – no heat out

Adiabatic means no heat exchange with the surroundings.

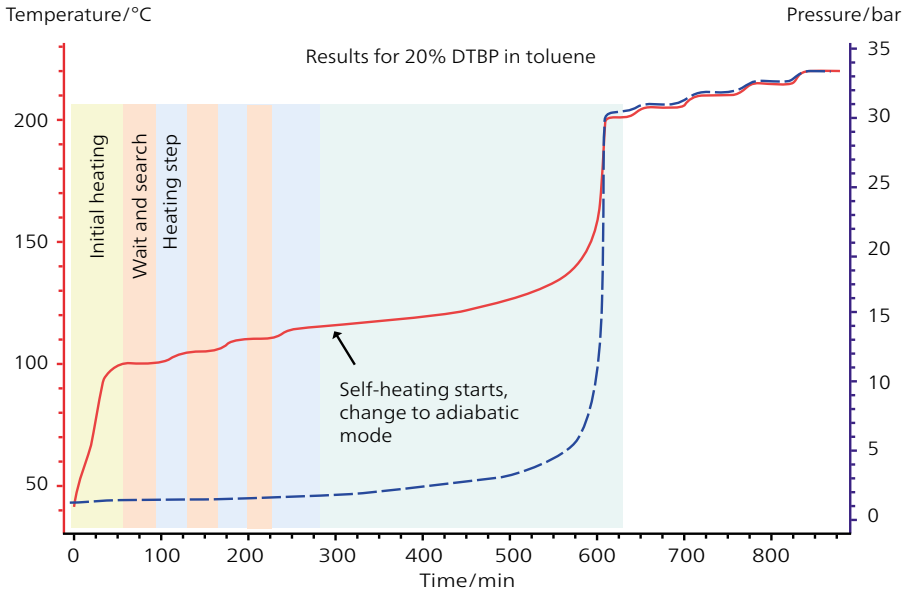
A sample (several grams) is placed in a spherical or cylindrical vessel which is surrounded by a sophisticated heating system. Depending on the working mode, the surroundings of the vessel are controlled to the same temperature as the sample. If there is no temperature difference between the heaters and the sample, then all the heat generated by the sample stays within the sample. This is the adiabatic condition.

Thermal Runaway

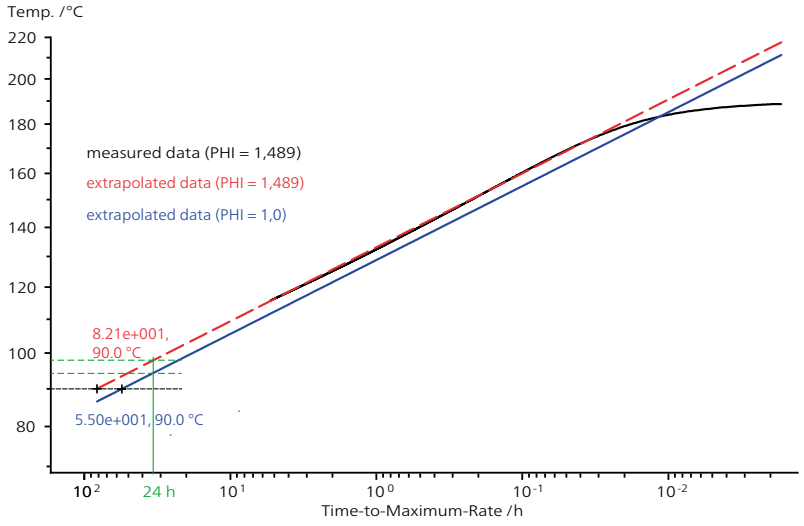
Thermal runaway starts when the rate of heat release from the reaction cannot be balanced by the cooling capacity.
In such a case, any heat generated by the material causes a further rise of the sample's temperature, fueling the reaction additionally (self-accelerating process). As a consequence, an explosion may occur.

Heat-Wait-Search Test (HWS)

The most popular operation mode for studying thermal runaway reactions is the Heat-Wait-Search. The measurement program starts by heating the sample material to a temperature slightly below the expected reaction temperature (→ heat). The second step is an isothermal waiting time of several minutes in which the sample temperature is monitored (→ wait and search). If the sample temperature remains constant within this period of time, the heaters will lift the sample temperature to the next temperature level. This will be continued step-by-step up to the point at which the sample temperature begins to change due to self heating of the sample; at this point, the temperature increase rate exceeds a certain threshold value (usually 0.02 K/min). If this happens, the calorimeter switches to the adiabatic mode and tracks the rising sample temperature.



Typical profile of a Heat-Wait-Search test on 20% DTBP in toluene (DTBP = Di-tert-butyl peroxide); temperature curve in red, pressure curve in blue.



Temperature plotted versus Time-to-Maximum-Rate (logarithmic scaling) for a measurement performed with 5.74 g of DTBP in toluene in a titanium sample vessel (ϕ factor = 1.489).
If the substance/mixture is stored in a vessel which exhibits heat loss in the order of ϕ = 1,489, then the maximum self-heating rate will be reached after 82 hours. However, if there is no heat loss during storage (ϕ = 1), the maximum self-heating rate will be already achieved after 55 hours.

Time-to-Maximum Rate (TMR)

The Time-to-Maximum Rate (TMR) under adiabatic conditions is often used for predicting the temperatures for safe processing and storage of chemicals. TMR is the time between the start of a thermal runaway reaction and the maximum reaction rate. As the experimentally observed TMR value using the ARC is influenced by the Φ-factor of the container, usually a correction to Φ = 1 is carried out. This reflects the situation when the sample mass is much higher than the container mass, as happens in practice during material storage in bigger tanks or industrial production in large reactors. TMR 24 or T_{D24} is the temperature at which it takes 24 hours to reach the maximum reaction rate. These values can be derived directly from the graph on the left (green lines).

Accelerated Rate Calorimeters by NETZSCH for Reliable Qualification of Risks and Hazards

Thermal safety is crucial in many areas where chemicals are involved, in particular:

- in production
- during transport
- in use
- in storage
- in disposal

and whenever a newly developed process is transferred from the laboratory to production or a production process is relocated from one site to another.

Along with DSCs, especially ARCs (accelerated rate calorimeters) are used to determine key parameters for thermal safety such as TMR (Time-to-Maximum Rate) or T_{D24} (see both on the previous page).

To obtain this information, ARC devices are utilized for measuring thermal runaway reactions as worst-case scenarios. To this end, the instruments have to be extremely robust and must be capable of withstanding (and measuring) the pressure generated by decomposition reactions.

ARC® 244 and ARC® 305

The floor-standing ARC® 305 is the premium instrument of this series. As a highly versatile, miniature chemical reactor, samples can be stirred, materials injected, and vent studies carried out. It was designed for use with the traditional 10-ml ARC spherical vessel, but the larger 130-ml container can also be used for low Phi or vent testing. The cost-effective floor-standing ARC® 244 is an option for those cutomers interested in keeping the platform developed by DOW.

MMC 274 *Nexus*® Multiple-Module Calorimeter

The MMC 274 *Nexus*® is a table-top device which can be equipped with different interchangeable calorimeter modules. This way, it is possible to perform classical ARC tests for thermal safety investigations as well as to determine exothermal and endothermal effects using the Scanning Module or, alternatively, the ARC Module plus *VariPhi*® with the same base unit.

	ARC® 244	ARC® 305	MMC 274 *Nexus*®
Temperature range	RT to 500°C	RT to 500°C	RT to 500°C
Pressure range (Standard)	0 bar to 150 bar	0 bar to 150 bar	0 bar to 150 bar
Lift mechanism	motorized	motorized	manual
Typical sample volume*	0.5 ml to 7 ml	0.5 ml to 8.5 ml	5 ml to 8.5 ml (ARC Module) 2.6 ml (Scanning Module)
Max. tracking rate	20 K/min	200 K/min	50 K/min
Temperature reproducibility	0.1 K	0.1 K	n/a***
VariPhi®	Optional	Optional	Optional
Operating mode	▪ Heat-Wait-Search ▪ Constant heating rate ▪ Isothermal	▪ Heat-Wait-Search ▪ Constant heating rate ▪ Isothermal	▪ Heat-Wait-Search ▪ Constant heating rate ▪ Isothermal ▪ Segment haeting (like DSC)
Stirring	Optional	Optional	No
Injection	Optional	Optional	No
Venting	Optional	Optional	Optional
Low Φ factor	Yes, with *VariPhi*® **	Yes, with *VariPhi*® **	Yes, with *VariPhi*® **
Kinetics software	Optional	Optional	Yes
Applications	▪ Process safety ▪ Comparison to legacy data	▪ Process Safety ▪ Energetic materials	▪ Process safety ▪ DSC-like investigations

* Sample volumes shown are typical and not limits. The choice of sample vessels can influence test results. See product manual or spare parts list for more details.
** Dependent on the sample pressure built-up
*** Temperature readability: 0.01 K

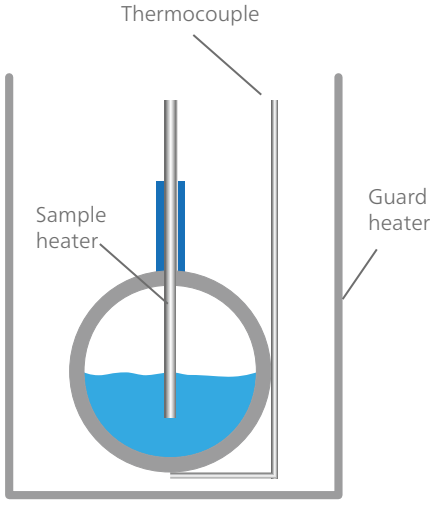


Diagram of *VariPhi*®

ARC® *VariPhi*® – Patented Solution for Low Phi Tests

So-called low Phi tests are important to simulate industrial scale processing or storage conditions.

In addition to the ability to account mathematically for the influence of the sample container (ϕ = 1), there is also an experimental solution for reducing the Phi factor – not only to 1, but to any desired value.

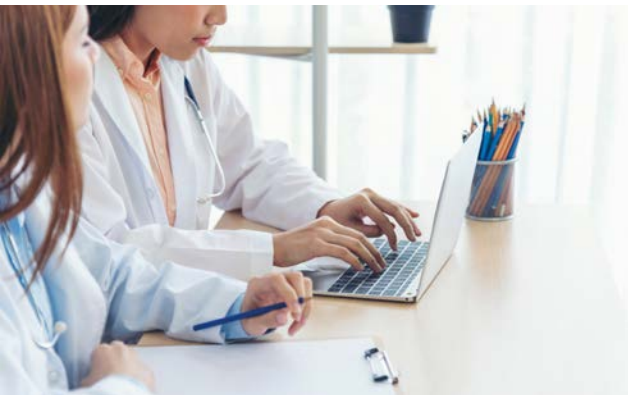
VariPhi® is an additional controlled heater which can be inserted into the liquid sample. It thus enables compensation for any heat loss from the sample to the container.

With this tool, the user can quickly and easily run low and high thermal inertia (Phi) tests in the same calorimeter using a small and inherently safer sample size.

VariPhi® is compatible with both ARC types and the MMC.

SOFTWARE

Tailor-Made for Every Measurement Technique



Smart and Easy-to-Use

NETZSCH instrument software offers everything the user needs to successfully define, perform and evaluate test runs. Pre-defined measurement methods for thermal analyzers, rotational rheometers and adiabatic calorimeters considerably facilitate entry into the examination of unknown samples. The automated, self-acting evaluation routines for thermal analyzers can serve as a second opinion, where necessary.

At the same time, the software is fully customizable for advanced research and development purposes. It features the right tools for almost any application, thus providing the flexibility necessary for all manner of tasks, including the more demanding ones.

- *Proteus*® for DSC, TGA, TMA, coupled systems, ARC and MMC
- rSpace for rotational rheometers
- FlowMaster for capillary rheometers

Details about the command set of the individual software packages can be found on the upcoming pages.

KINETICS NEO

If You Want to Get Even More out of Your Measurements

NETZSCH Kinetics Neo is a formal kinetic software for analyzing different kinds of temperature-dependent chemical processes. Potential data sources include studies employing thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), themomechanical analysis (TMA), Fourier-Transform infrared spectroscopy (FT-IR), mass spectrometry (MS) and rheometry, as well as temperature analysis during adiabatic measurements.

The result of such evaluation is a kinetics model correctly describing the experimental data. Based on this model, predictions of a chemical system's behavior under user-defined temperature conditions are feasible. This includes temperature profiles which cannot be realized experimentally or can only be realized with difficulty, for example, long-term predictions for shelf life issues. Alternatively, such models can be used for process optimization

Proteus®

Proteus® is the name of the thermal analysis and adiabatic calorimetry software by NETZSCH. It is intuitive and easy-to-handle, supports the operator with a variety of clever and useful features (e.g., the unique *AutoEvaluation* for thermal analysis), and contains an expert system called *Identify* (also referring to thermal analysis curves).

In addition, users may select from two user interfaces for DSC and TGA experiments: the simplified *SmartMode*, which is particularly advantageous if a touch monitor is preferred, and the *ExpertMode*, for anyone who favors a more classical interface. The operator can switch between the two presentation formats, even if an automatic sample changer (ASC) is being used.

For all other systems, the *ExpertMode* is standard.

Instrument-Independent Methods

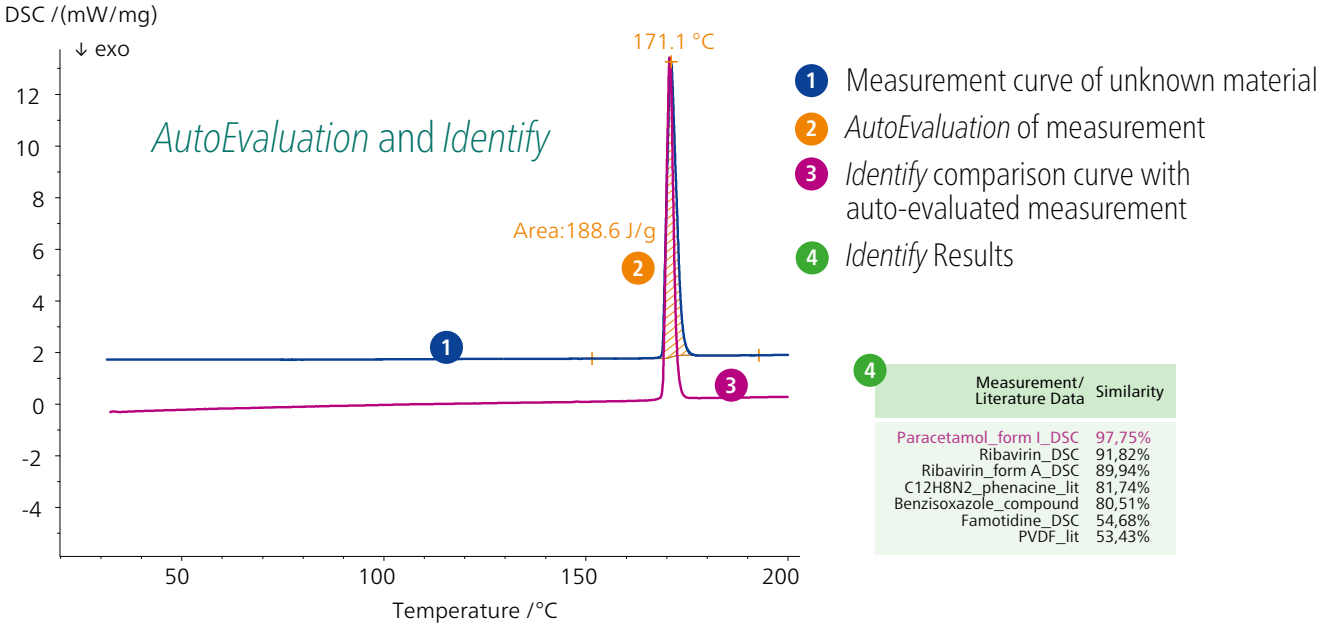
The *Proteus*® software is based on measurement methods which can define and operate the required temperature program as well as evaluate the resulting curve, if desired. The methods can be used for any device of the same type (for example, any DSC of the *Caliris*® series) and of comparable configuration. Thus, it is possible to transfer such methods from one location to another in a simple but safe way – ideal for labs located at different sites or projects that are worked on by different collaborators.

Assure data integrity with Proteus® Protect*

AutoEvaluation and *Identify* – Still Unrivaled

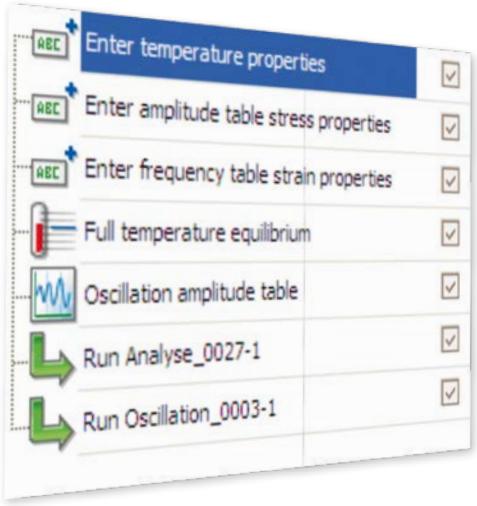
On the one hand, the *AutoEvaluation* function detects and evaluates thermal effects – i.e., peaks or mass changes – without any user intervention. Intelligent algorithms are capable of handling DSC and TGA curves fully automatically and thus of generating completely objective results.

Identify, on the other hand, allows for the identification and classification of materials via database comparison with just one click. The result is a hit list where the database entry with the highest similarity is on top. To refine the database search, DSC and TGA data can also be used in combination.



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

* *Proteus*® *Protect* is an option for DSC, TGA and STA devices (incl. coupling) and meets the technical requirements of 21 CFR part 11



rSpace

The rSpace software is designated specifically to handle rotational rheometers. With this software, instruments of the Kinexus line can combine the requirements of quality assurance in accordance with standardized test procedures (SOPs, see a sequence as an example on the left) and the requirements for university and industrial research, to achieve completely open programming and access to raw data.

Based on Sequences

Sequences represent a logical flowchart of fundamental rheological actions that can be linked together with other test actions, such as user feedback and choices, calculated values, loops and triggers. All actions can be included very easily via Drag and Drop functionality.

Rheology Toolkit

A lot of pre-configured rheological tests for different applications are already available and can be started simply by a mouse click.

For more sophisticated experiments, it is possible to access existing sequences and use them as templates for inserting current specific sample properties.

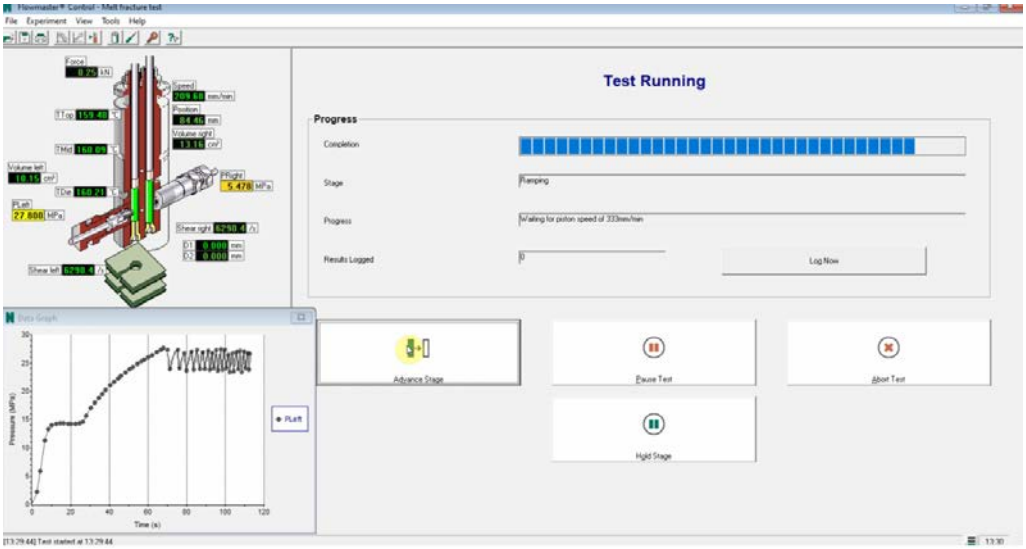
Standard Operating Procedures (SOP)-Driven Tests for Reliable Rheological Measurements

Fully configured SOP-driven tests with associated test description stand for:

- Continuous feedback and user guidance
- Available for company-wide use
- Consistent and reliable measurements
- Supporting application and support notes

Flowmaster

The Flowmaster software is best adapted to the operational principle of capillary rheometers and offers comprehensive measurement and evaluation possibilities in multiple languages.



All Information at a Glance

During a measurement, all of the relevant parameters are continuously monitored (left part of the window). This comprises temperatures, pressures, volumes and piston speed.

On the right side of the measurement window, the situation during a melt fracture test (optional) for investigating flow instabilities is shown. To this end, an accelerated shear rate ramp is applied to the material and the pressure is continuously recorded. Instabilities in the pressure readings indicate instabilities in flow properties which may occur as melt fracture while the sample is flowing through the capillary die.

A Wide Range of Measurement Options, Indlucing:

- Constant shear
- Extensional tests
- Manual control
- Die swell (option)
- Melt fracture/molt instability (option)
- Wall slip analysis (Mooney, option)


NETZSCH Thermokinetics
Curing of Epoxy Resin
Heat flow rate/(W/g)
0
-0.5
-1.0
-1.5
-2.0
-2.5
20.4 K/min
10.1 K/min
5.0 K/min
150
Temperature/°C

Expertise in Service

Our Expertise – Service

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.

Our Expertise – Applications Laboratories

The NETZSCH Thermal Analysis applications laboratories are a proficient partner for nearly any Thermal Analysis issue. Our involvement in your projects begins with proper sample preparation and continues through meticulous examination and interpretation of the measurement results. Our diverse methods and over 30 different state-of-the-art measuring stations will provide ready-made solutions for all your thermal needs.

Within the realm of thermal analysis and the measurement of thermophysical properties, we offer you a comprehensive line of the most diverse analysis techniques for materials characterization.

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

TECHNICAL SERVICE

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- Moving Service

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- NETZSCH Online Academy
- Comprehensive Instrument and Method Training

LABORATORY

- Application Service and Contract Testing

* for DSC, TGA, STA devices and rotational rheometers

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. 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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