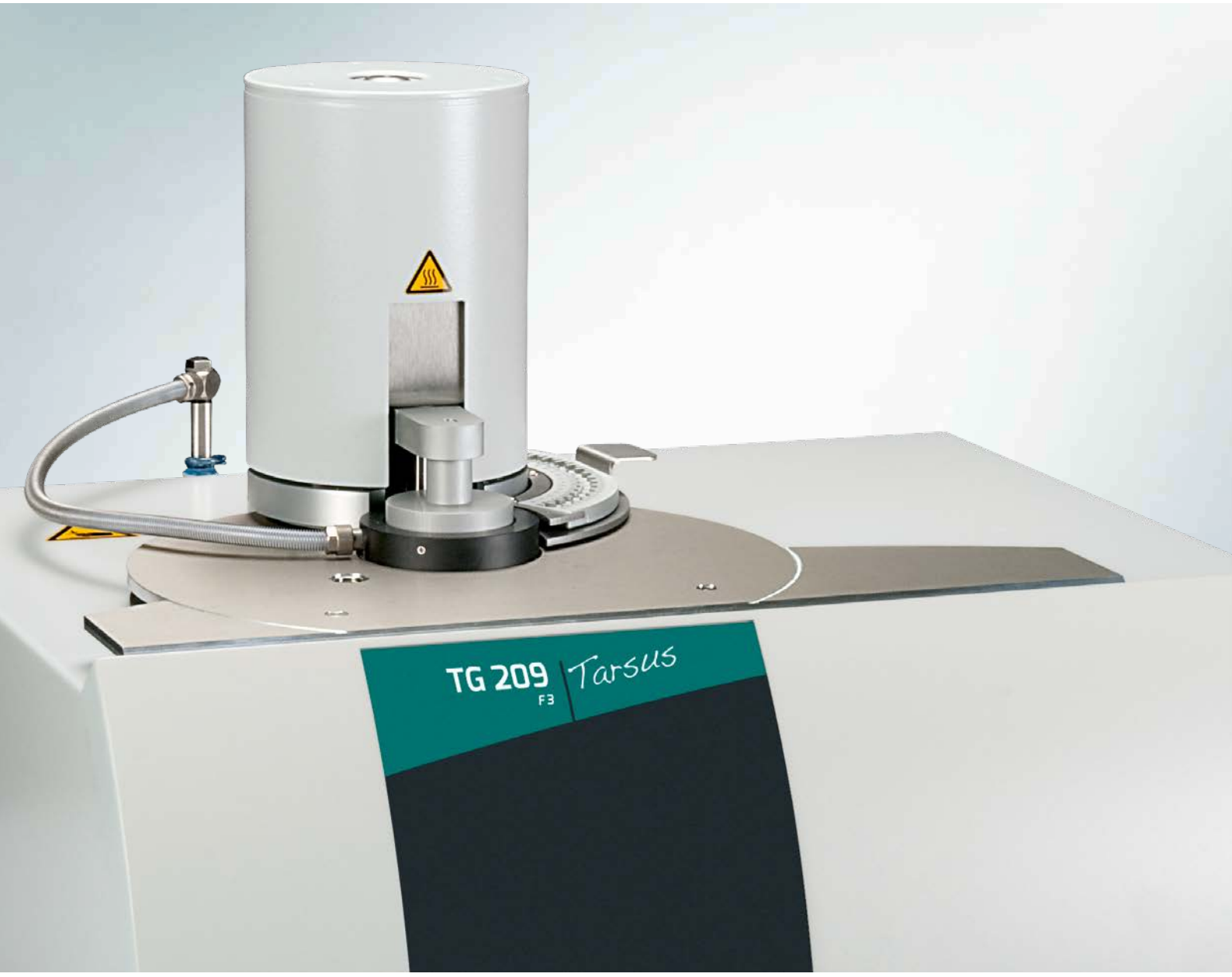
NETZSCH

Proven Excellence.



TG 209 *F3* *Tarsus*®

Thermogravimetric Analysis – TGA
Method, Technique and Applications

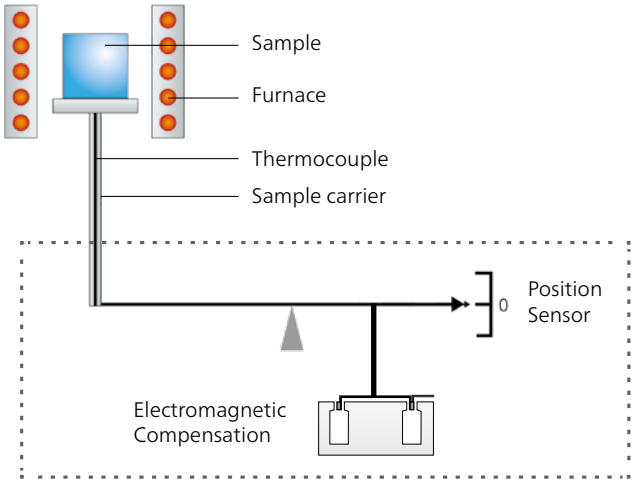
Analyzing & Testing

Thermogravimetry

- What is the thermal stability of the material?
- Is the sample really the specified filled granulate?
- Is the sample contaminated with an additional component?
- Is the filler homogeneously distributed in the part?
- Of how many individual components does the rubber seal consist?
- How much solvent or plasticizer is released of the rubber mixture?
- How high is the added carbon black content?
- How high is the ash content of the polymer mixture?
- How does the thermal stability change under the influence of an oxidizing atmosphere?
- Is the melting of the thermoplastic already superposed by the start of decomposition?
- How much residual solvent does the coating contain?
- How much is the loss on the drying amount?

Thermogravimetry (TG) or Thermogravimetric Analysis (TGA) is a well-proven method to answer questions like these. TGA is increasingly used for the quality control and assurance of raw materials and incoming goods as well as for failure analysis of finished parts, especially in the polymer processing industry.

NETZSCH Analyzing & Testing has been manufacturing thermo-microbalances for decades. Our vertical, top-loading design not only provides for easy operation and sample loading, but also allows gases to flow naturally in an upward direction – which is advantageous for direct coupling to evolved gas analyzers such as FT-IR spectrometers.



Principle of Operation

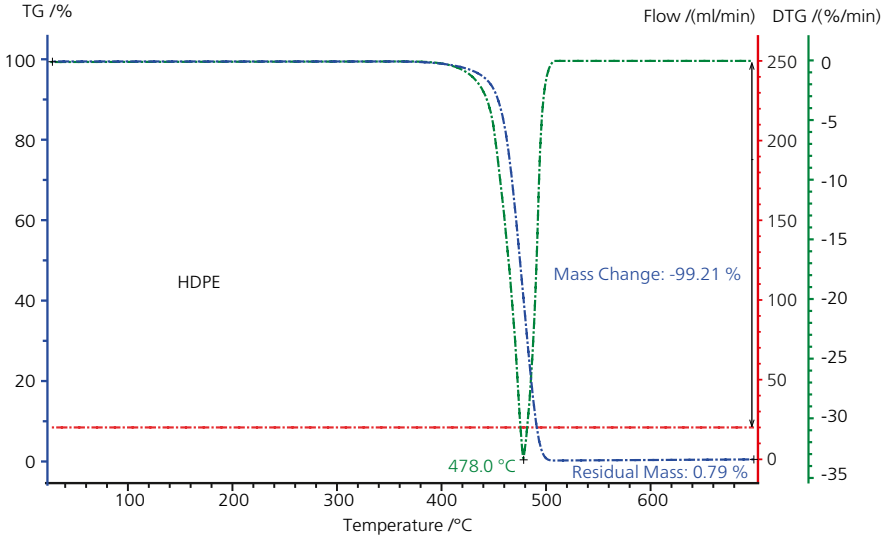
A thermobalance is used to measure the mass change of a sample as a function of temperature or time, under a defined and controlled environment with respect to heating rate, gas atmosphere, flow rate, crucible type, etc.

Various international standards describe the general principles of thermogravimetry for polymers (ISO 11358) or other specific applications, such as compositional analysis for rubber (ASTM D6370) and evaporation loss of lubricating oils (ASTM D6375).

Measurement Results

This TGA measurement shows the decomposition of a black high-density polyethylene (HDPE). In a nitrogen atmosphere, there is only a single mass-loss step detected and it represents a nearly complete decomposition. The residual mass of only 0.8% is evidently due to carbon black. The corresponding 1st derivative of the TGA curve, DTGA, provides the decomposition rate. The DTGA peak temperature (478°C) is usually used as a characteristic value to specify the appropriate step.

The decomposition products of PE are wax-like. However, by specially modifying the exhaust of the TG 209 ***F3*** *Tarsus*® (optional), PE can also be investigated on a routine basis.



Decomposition behavior of polyethylene in a nitrogen atmosphere, illustrated by the TGA, DTGA and gas flow curves; heating rate 10 K/min

TG 209 *F3 Tarsus*®

Convincing Technology
Controlled by Intelligent Software

Made by NETZSCH

Robust – Top-Loading Design

The unique top-loading balance ensures very low drift behavior under isothermal and dynamic measurement conditions across the entire temperature range. A thermostatic control maintains constant temperature and eliminates environmental influences to the balance's performance.

Flexible – Sample Carriers

Various sample carriers are available including corrosion-resistant sensors, high-sensitivity c-*DTA*® sensors for improved monitoring of endo- and exothermic effects, and special sensors for large sample masses.

Ready To Go – *SmartMode* and *ExpertMode*

The simplified *SmartMode* user interface applies an easy navigation concept allowing the operator to use the software without being an expert in thermogravimetry. *ExpertMode* offers access to the entire range of commands for enhanced option-setting or for method definition.

Intelligent – *AutoEvaluation* and *Identify*

The unique *AutoEvaluation* software feature offers autonomous detection and user-independent evaluation of all thermogravimetric effects. *Identify* can use these evaluated curves for material identification and classification of measurement curves.

VERTICAL, TOP-LOADING

PRECISE ULTRA-MICROBALANCE

GAS-TIGHT BY DESIGN

INTERCHANGEABLE SAMPLE CARRIERS

AUTOMATIC SAMPLE CHANGER FOR UP TO 20 SAMPLES

MICRO FURNACE

SMARTMODE

C-*DTA*®

AUTOEVALUATION

COUPLING TO FT-IR

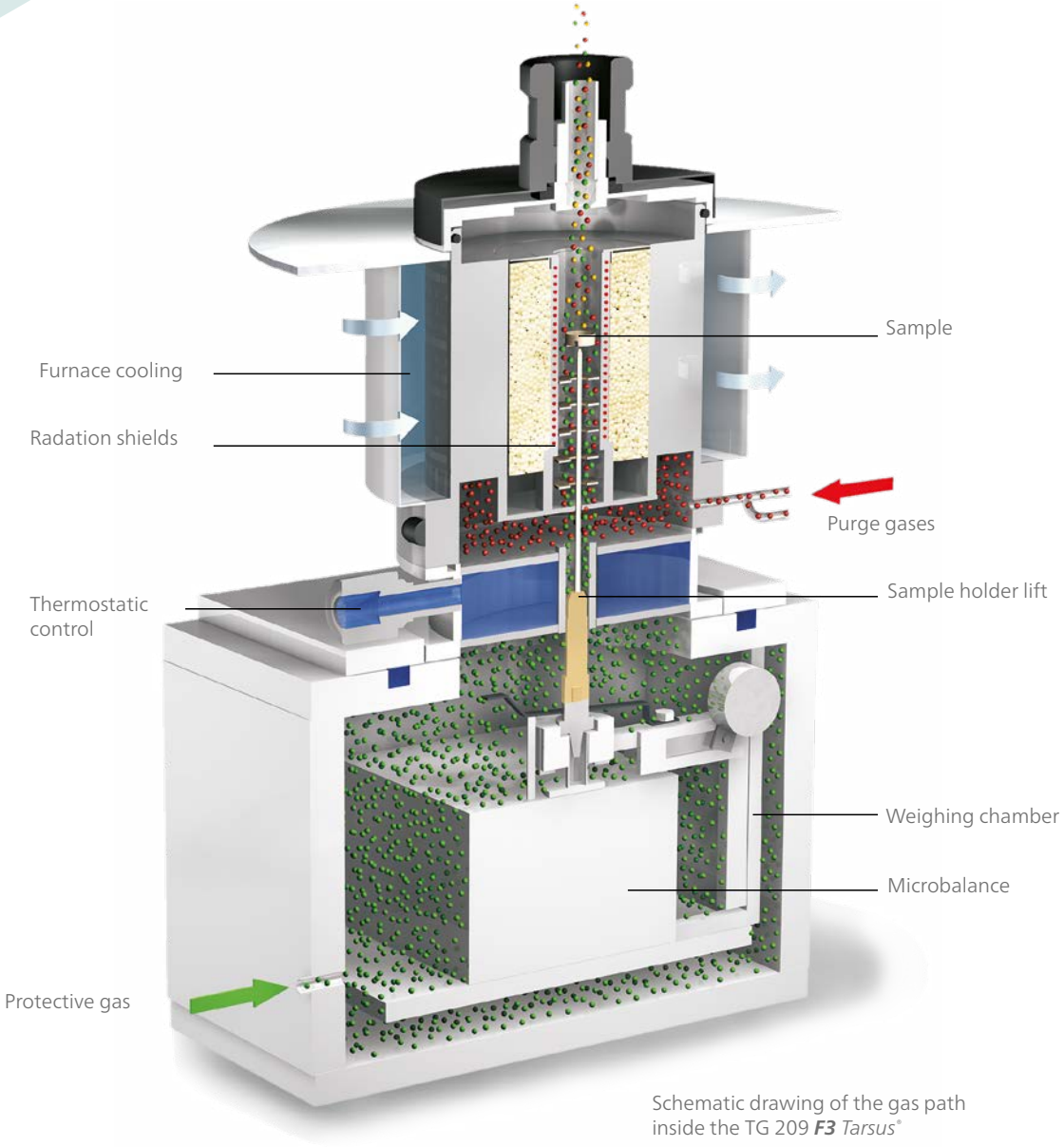
IDENTIFY

AUTO-CYCLE EVACUATION

TG 209 ***F3*** *Tarsus*®

More Than Just a Simple TGA!

For efficient laboratory work – everything at hand



Schematic drawing of the gas path inside the TG 209 ***F3*** *Tarsus*®

Micro-Furnace for Dynamics in Day-to-Day Lab Tasks

The low-volume furnace of the gas-tight NETZSCH TG 209 ***F3*** *Tarsus*® consists of an SiO_2-coated monolithic metallic heater with an integrated air cooler. Between room temperature and 1000°C, a heating rate of 200 K/min can be achieved for a high sample throughput. Safe handling is ensured by safety features. This includes purging of the balance chamber with protective gas.

Precise Detection of the Sample Temperature

The sample temperature is detected by a thermocouple in direct contact with the sample crucible. This ensures accurate reading of the sample temperature and makes it nearly independent from the atmosphere, gas flow or heating rate.

More Than Just a TGA! Monitoring Caloric Effects without Mass Loss

The calculated DTA signal, c-*DTA*®, is ideal for easy temperature calibration without the need of a magnet and disassembling of the instrument. It also yields information regarding endothermic and exothermic processes (e.g., melting without mass loss or evaporation with mass loss).

Auto-Cycle Evacuation – Fast Gas Exchange and Saving Purge Gas

The instrument's three gas connections allow for fast gas exchange when changing the atmosphere (e.g., from inert to oxidizing). In addition to two purge gases, an inert gas is introduced into the balance for protection purposes.
The time-controlled auto-cycle evacuation prior to measurement automatically evacuates and refills the system. It ensures less purging time before the measurement starts and therefore less gas consumption.

Automatic Sample Changer (ASC) – Makes Routine Work Easy

The ASC for up to 20 samples allows predefined methods to be included for each sample on the tray. Depending on the method, evaluation of the sample measurements can be carried out automatically. For quality control purposes, the temperature limits of earmarked effects can be set.

PROTEUS®

The Backbone for Uncomplicated and

SmartMode – Run Professional Tests After Minutes of Introduction

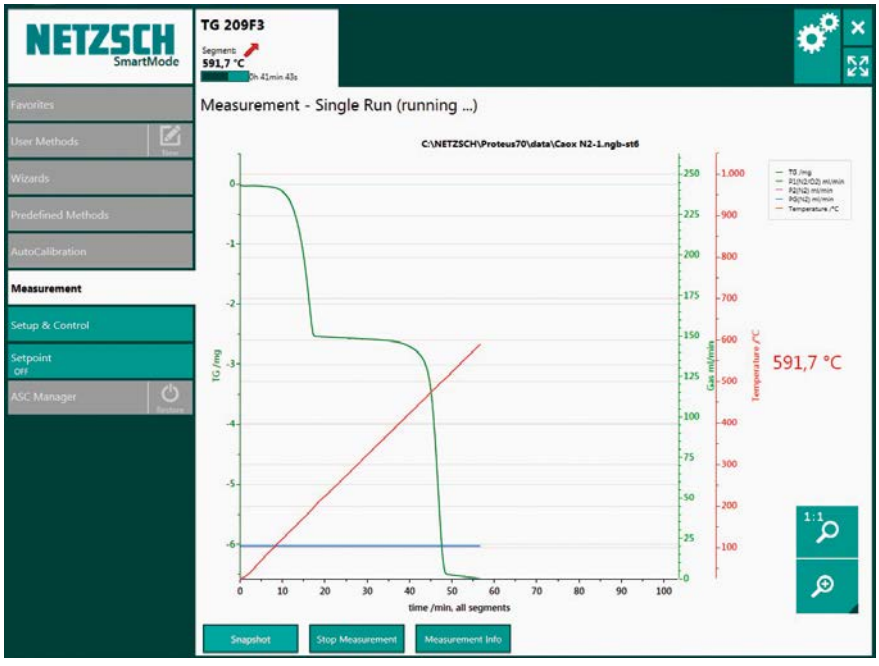
SmartMode boasts a clear structure, a consistent navigation design and easy-to-access buttons. Using wizards (measurement templates), it is possible to start a measurement with just a few inputs. Alternatively, customized methods or predefined methods can be selected to start an experiment. The predefined methods are based on different standards or measurements under reduced pressure. Even customers unfamiliar with the software immediately know how to proceed.

ExpertMode – Perefct for Beginners and Professionals

For those who wish to dive deeper into the software for enhanced option setting or for method definition, switching from *SmartMode* over to *ExpertMode* is the answer. Here, the user has access to the established *Proteus*® software functionality, including dozens of features and all adjustment settings.

AutoCalibration Allows for Full Concentration on the Measurement Tasks

Calibration procedures should be simple, fast and – ideally – done along the way. *AutoCalibration* provides automatic creation routines for all calibration curves – it automatically loads the current temperature calibrations (with consideration to the selected measurement conditions) and verifies their validity periods (watchdog function).



SmartMode – There's no need to be an expert in TGA to start a measurement!

Fast Test Start Through to Autonomous Evaluation

*AutoEvaluation** – Autonomous Evaluation

AutoEvaluation is a self-acting software package which evaluates the thermogravimetric effects, i.e., mass changes (decomposition reactions) without using stored evaluation macros.

For all those who haven't seen such measurements yet, *AutoEvaluation* will handle the curve independently – without any effort on the operator's part.

This ground-breaking technology allows, for the first time in history, test analyses which are fully user-independent and therefore completely objective.

Of course, users can still run manual evaluation, if required.

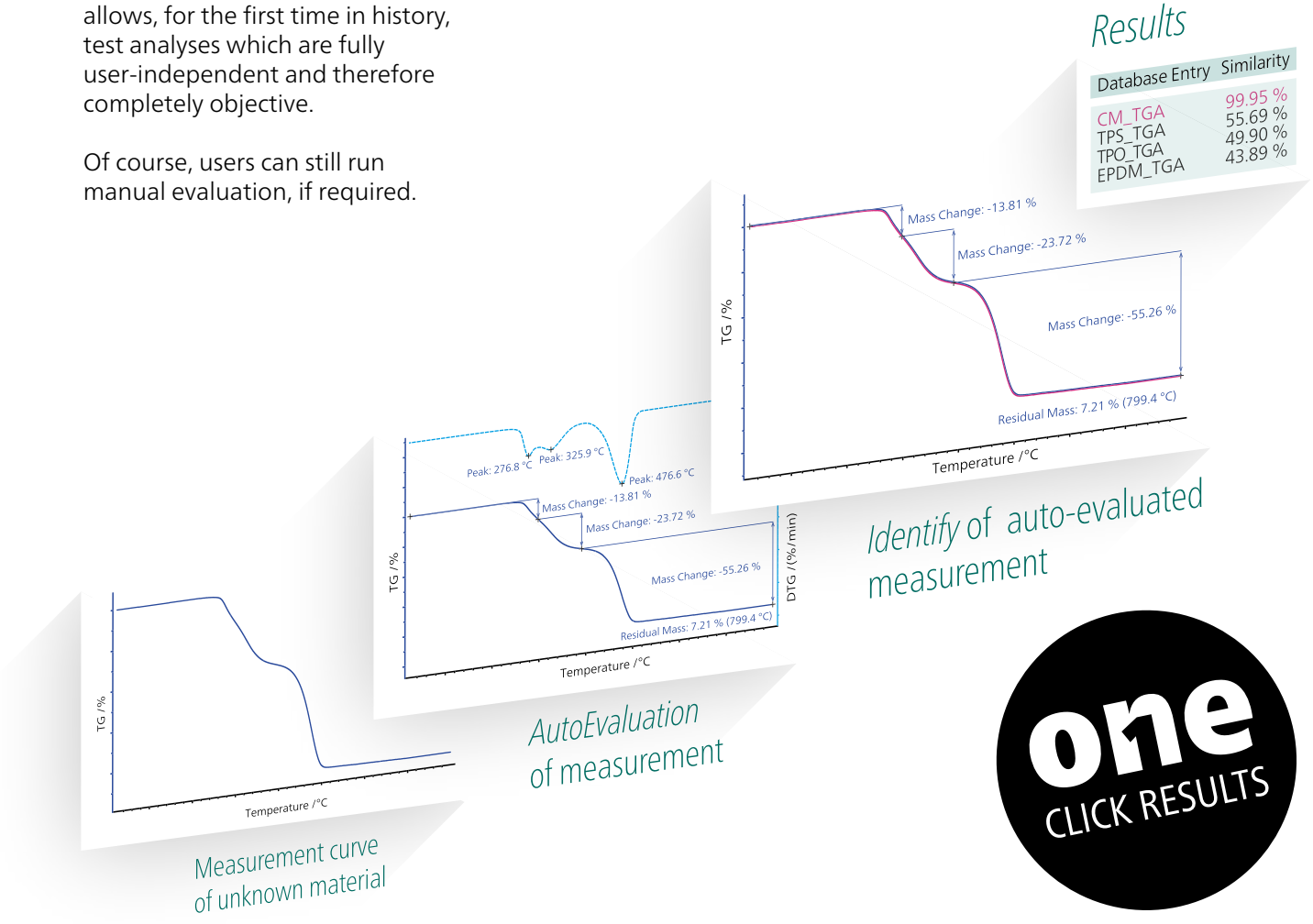
*Identify** – A Step Ahead with the Database

The *Identify* software searches for similar results stored in libraries, providing instantaneous interpretation of the measurement at hand.

With the *Identify* software package, it is possible to carry out one-on-one comparisons with individual curves or literature data from selected libraries, or to check whether a particular curve belongs to a certain class. These classes may contain sets of data for various types of the same material (e.g., several types of PE for polymers) but also curves, such as ones which are classified as PASS or FAIL in terms of quality control.

Both the libraries and classes are boundless and users can expand them with experiments and knowledge of their own.

*optional software extensions



APPLICATIONS

Application Fields

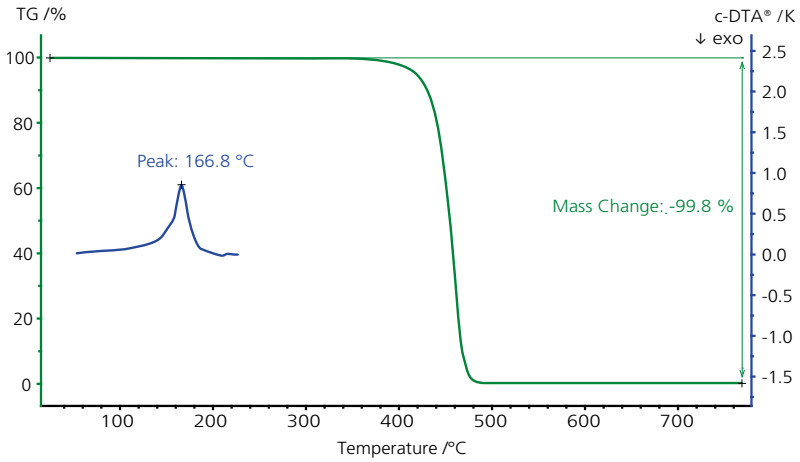
The TG 209 ***F3*** *Tarsus*® can be employed for the characterization of a great variety of materials and applications including polymers, pharmaceuticals, textiles, foods, cosmetics, and other organic and inorganic materials. For researchers in fields such as automotive, pharmaceuticals, textiles, etc., the technique employed by this instrument is a fast and reliable research tool. Its easy operation, fast analysis time and standardized evaluation procedures make the TG 209 ***F3*** *Tarsus*® ideal for application in quality assurance and failure analysis.

TGA Information

- Mass changes
- Identification
- Compositional analysis
- Decomposition
- Oxidation
- Thermal stability
- Reduction behavior
- Corrosion studies
- Determination of filler content
- Influence of aging
- Determination of plasticizer content and other additives
- Determination of moisture content
- Determination of added carbon black
- Determination of ash content
- Curie temperatures
- Reaction kinetics
- *Purity Determination*

More Information Through Caloric Effects – c-*DTA*®

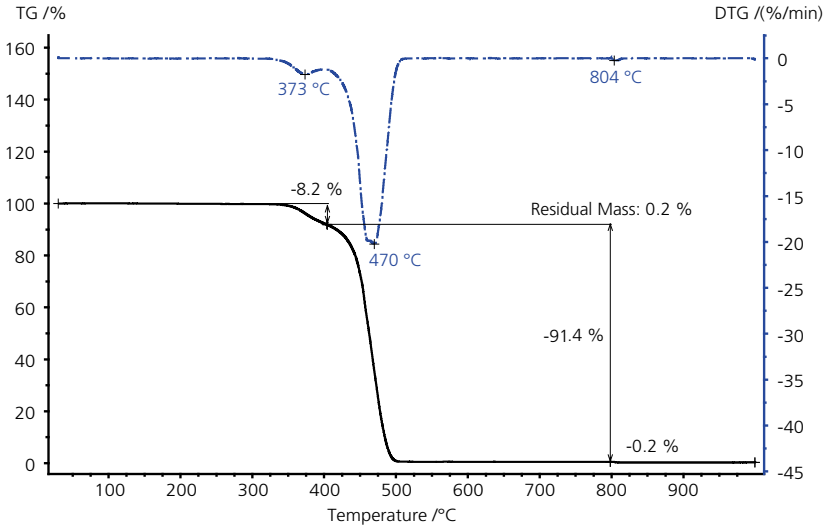
In addition to the TGA curve, this plot depicts an endothermic effect determined by means of the calculated DTA signal, c-*DTA*®, in the region where no mass loss is detected.
The TGA measurement shows the decomposition of PP in a nitrogen atmosphere (green curve). The c-*DTA*® peak at 167°C represents the melting of the polymer.



Polymer Identification Made Easy – Even for Complex Elastomer Materials

Characterization of Butadiene Rubber (BR)

The plot shows a two-step decomposition of BR at 373°C (DTG peak) and at 470°C (DTG peak) in a nitrogen atmosphere (N_2). The first step is due to unsaturated alkanes, the second to the cracking of the polymer back bone. In both decomposition steps, the sample nearly totally decomposes.
The small amount of remaining mass in the N_2 atmosphere most probably consists of pyrolytic carbon, which can be concluded from the fact that there is a mass loss of 0.2% after switching from nitrogen to synthetic air at 800°C.

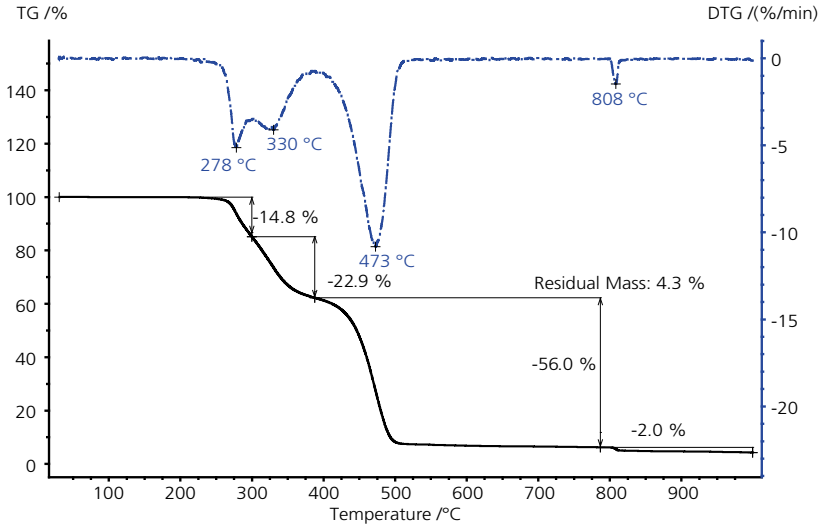


TGA measurement on BR at a heating rate of 10 K/min in nitrogen (40 ml/min), from 800°C to 1000°C, the measurement was continued in synthetic air; sample mass amounted to 9.90 mg; crucible made of alumina.

Complex Decomposition Behavior of CM Presented Simply by TGA

The measurement plot demonstrates the complex decomposition behavior of CM in a nitrogen atmosphere up to 800°C and then its mass loss behavior in synthetic air up to 1000°C.

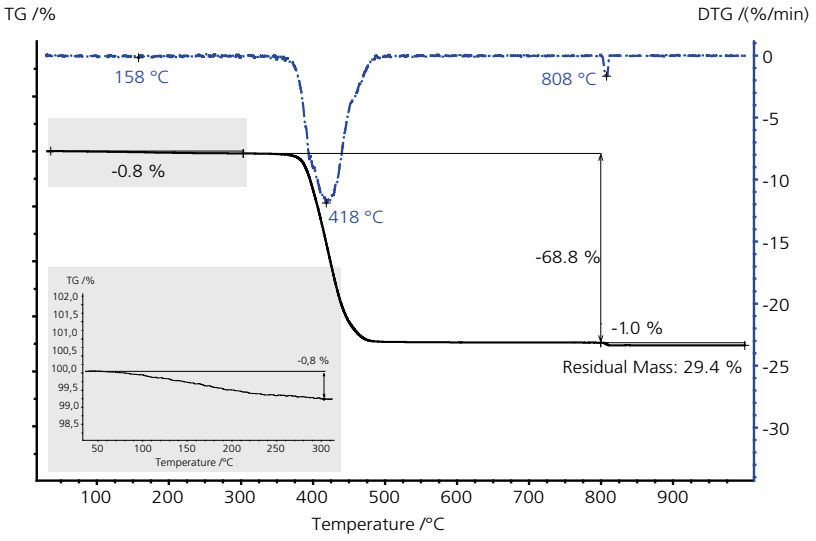
Typical for polymers containing chlorine is the release of HCl which can be seen in the two-step decomposition with DTG peaks at 278°C and 330°C. The main decomposition step at 473°C (DTG peak) shows the PE degradation with a mass loss of 56%. After switching to synthetic air, pyrolytic carbon burns to 2%. The remaining ash content amounts to 4.3%.



TGA measurement on CM at a heating rate of 10 K/min in nitrogen (40ml/min); from 800°C to 1000°C, the measurement was continued in synthetic air; sample mass amounted to 9.94 mg; crucible made of alumina.

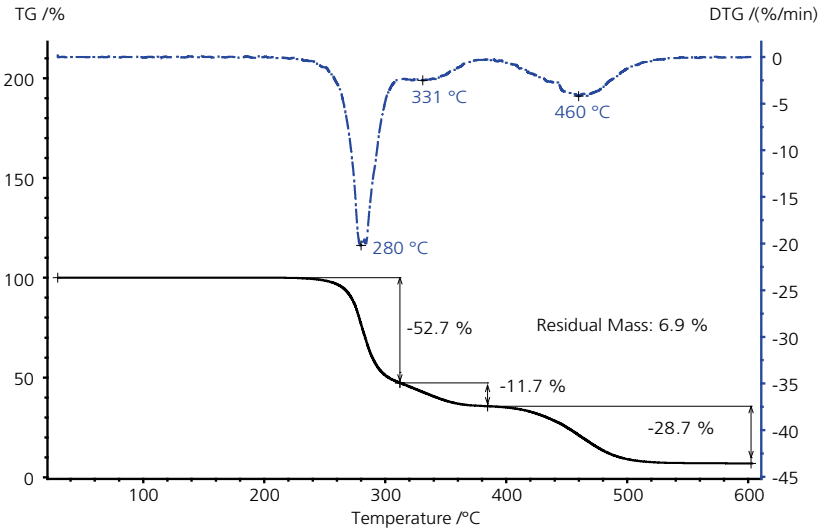
Determination of the Glass Fiber Content – Polyamide 66

This PA66/GF sample was heated to 800°C in an inert gas atmosphere at 20 K/min. Already between 70°C and 250°C, a considerable mass loss of 0.8% is determined (picture-in-picture presentation). Decomposition (68.8%) starts at approx. 360°C with a maximum decomposition rate at 418°C (DTG peak). After switching to an oxidizing atmosphere at 800°C, the pyrolytic carbon is burnt (1.0%). The remaining residue corresponds to the glass fiber amount (29.4%).



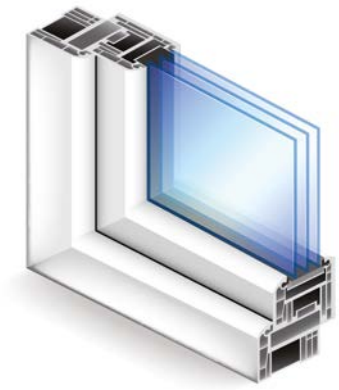
Picture-in-picture plot of the measurement on PA66/GF, heating rate of 20 K/min in a nitrogen atmosphere (40ml/min), from 800°C to 1000°C the measurement was continued in synthetic air; sample mass 10.47 mg, crucible made of alumina.

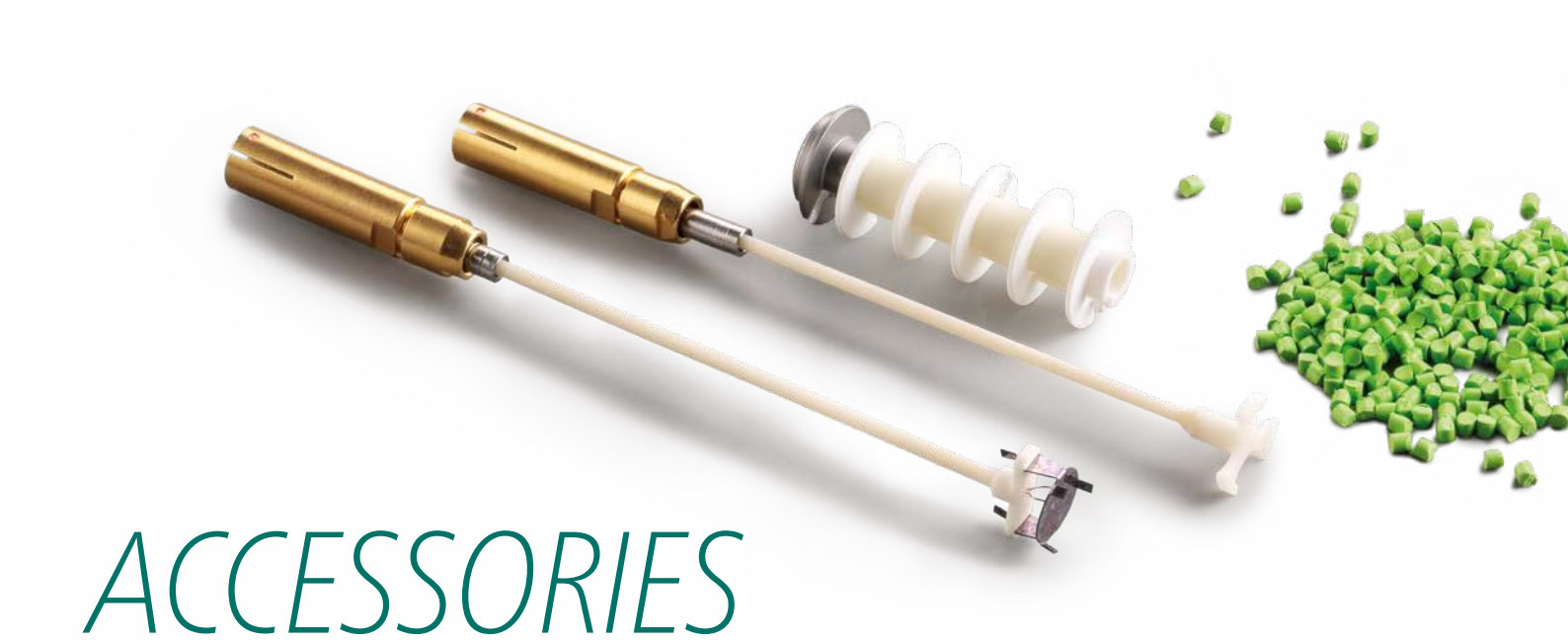
Thermal Stability of a Modified Rigid PVC-U Profile



Decomposition behavior of filled PVC-U in a nitrogen atmosphere (40ml/min) at a heating rate of 10 K/min up to 600°C; sample mass 10.08 mg, crucible made of alumina

The decomposition of a PVC-U sample can be followed by means of a TGA measurement in an N_2 atmosphere. This plot shows several mass-loss steps between room temperature and 600°C. The first two mass changes (DTG peaks at 280°C and 331°C) are due to the release of chlorinated components. The subsequent cracking of the hydrocarbon backbone can be observed at 460°C (DTG peak). The residue of 6.9% at 600°C can be attributed to pyrolytic carbon and an inorganic filler.





ACCESSORIES

For Each Application the Right Sample Carrier

The NETZSCH TG 209 ***F3*** *Tarsus*® offers multiple sample carriers – some with a special coating for high resistance to corrosive gases, others for large samples and yet others providing high sensitivity for combined c-*DTA*®. Thanks to the sample holder lift, all sample carriers can be detached from the balance system within a few seconds.

Interchangeable Sample Carriers[1]

Application	Material of Sample Carrier	Sensor Type	For Crucible Type
Standard TGA	Al_2O_3	Type P	Ø 7 mm to 9 mm, 85 µl to 350 µl
Ideal for c-*DTA*®	Platinel®	Type P (disk)	Ø 7 mm to 9 mm, 85 µl to 350 µl
For corrosive media	Al_2O_3	Type P, protected	Ø 7 mm to 9 mm, 85 µl to 350 µl

[1] For ASC: max. diameter of crucible is 8 mm

Crucible Types for Various Applications[2,3]

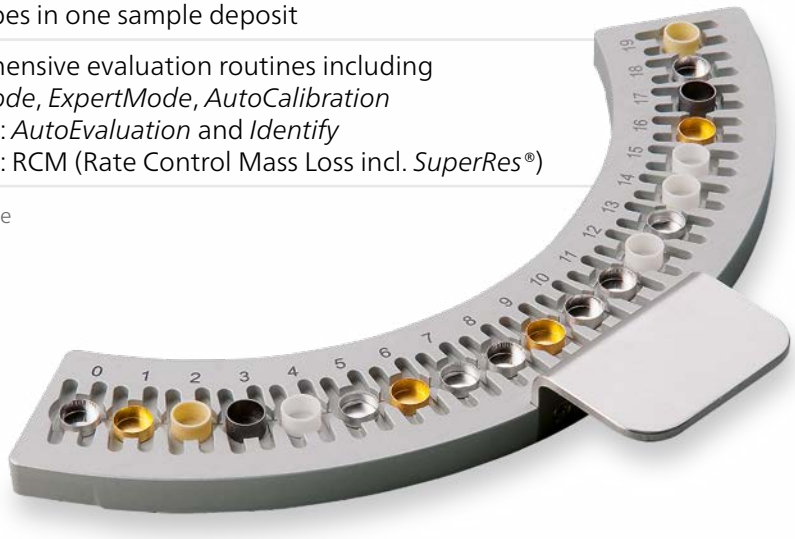
Application	Material	Diameter / Height	Volume
Standard TGA tests	Al_2O_3	6.8 mm/4 mm	85 µl
Standard TGA tests, high sample mass or volume	Al_2O_3	8.0 mm/8 mm; 9.0 mm/7 mm	300 µl; 350 µl
Especially for c-*DTA*®; high sample mass or volume	Pt/Rh (80/20)	6.8 mm/2.7 mm; 6.8 mm/6 mm	85 µl; 190 µl
Especially for c-*DTA*®, up to max. 600°C	Al (99.5%)	6.7 mm/2.7 mm	85 µl

[2] More crucibles available in other materials
[3] Please note crucible-sample compatibility

Technical Specifications

TG 209 ***F3*** *Tarsus*®	
Design	Top-loading
Temperature range	RT to 1000°C
Heating rate	0.001 K/min to 200 K/min
Cooling time	Approx. 25 min (free cooling in inert atmosphere); 12 min in He*
Max. sample weight/ measuring range	2 g
TGA resolution	0.1 µg
Motorized sensor lift	For easy and safe handling of sensor change
Interchangeable sensor types	▪ High volume samples / large masses ▪ High sensitivity (c-*DTA*®) ▪ Corrosion-resistant
Gas atmospheres	Inert, oxidizing, static and dynamic
Gas flow control	▪ Integrated frits ▪ Optional: mass flow controllers, free-standing gas control device
Time-controlled auto-cycle evacuation	Prior to measurement
Temperature calibration	c-*DTA*®, also for detection of endo- and exothermal effects; Curie standards
Mass calibration	Automated routine via integrated mass of 2 g ± 0.006 mg
Caloric effects	Endothermal and exothermal effects by c-*DTA*®
Crucibles	Pt, Al_2O_3, Au, SiO_2, Ag, ZrO_2, Al, etc. More upon request.
Automatic sample changer (ASC)	Up to 20 samples (optional)
Crucibles for use in ASC	Various types in one sample deposit
Software	▪ Comprehensive evaluation routines including *SmartMode*, *ExpertMode*, *AutoCalibration* ▪ Optional: *AutoEvaluation* and *Identify* ▪ Optional: RCM (Rate Control Mass Loss incl. *SuperRes*®)

* At 22°C ambient temperature, 23°C chiller temperature



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
- Exchange Service
- IQ/OQ Documents
- Calibration Service
- Spare Part Assistance
- Moving Service

TRAINING

- Basic Seminars
- NETZSCH Online Academy
- Comprehensive Instrument and Method Training

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 3,800 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

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

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

Proven Excellence.

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