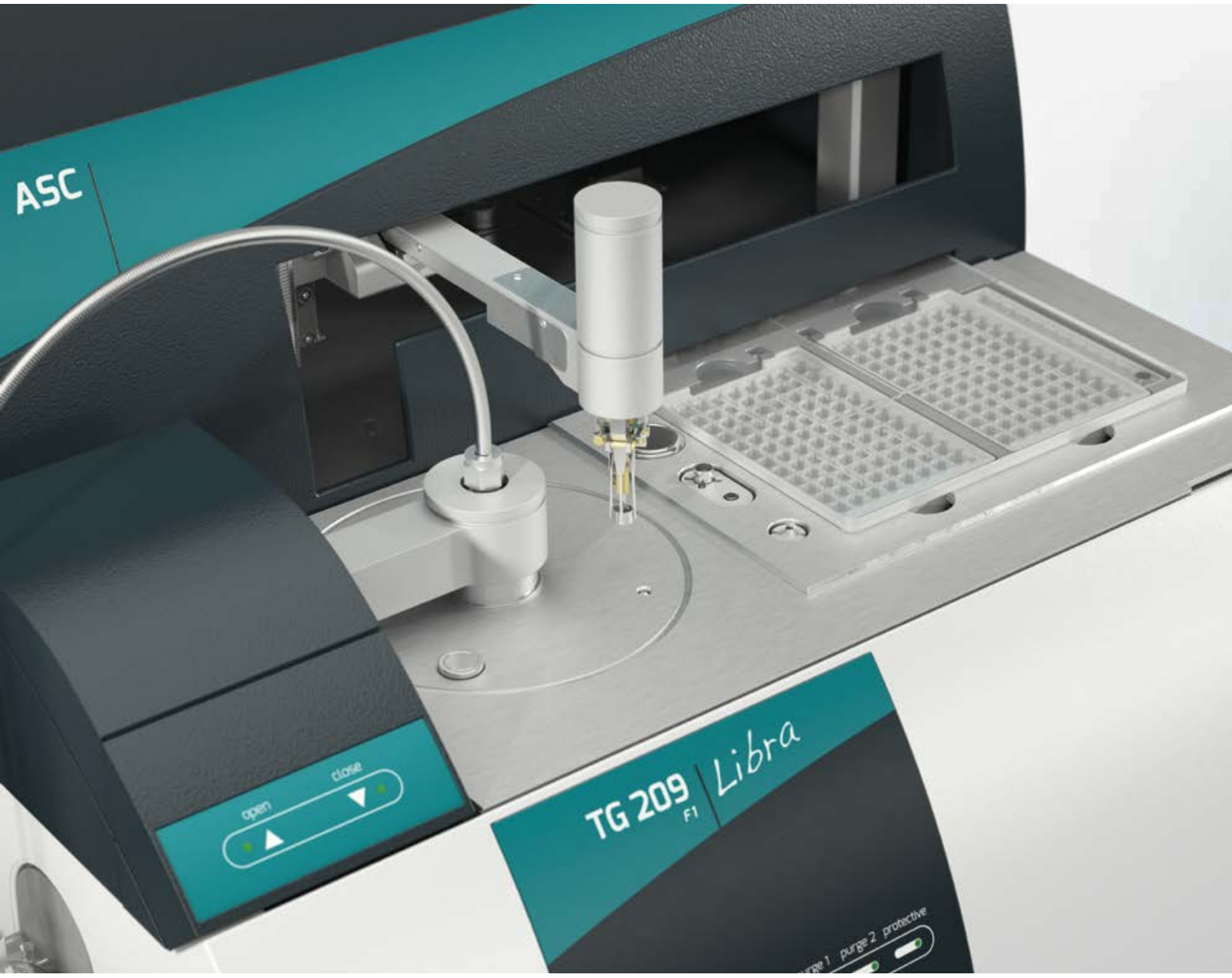
NETZSCH



TG 209 *F1* *Libra*®

Thermogravimetric Analysis – TGA
Method, Technique and Applications

Leading Thermal Analysis

TG 209 *F1* *Libra*®

Convincing Technology
Controlled by Intelligent Software

Made by NETZSCH

Vertical Design Combined with Top-Loading Ultra-Microbalance for Easy and Safe Handling

The design of the vacuum-tight TG 209 ***F1*** *Libra*® ensures free and safe access to the sample, easy crucible exchange (no hang-down wires or horizontal balance beam), and a constant and stable position for the sample carrier in the furnace. This results in homogeneous temperature distribution and high sample-to-sample reproducibility.

Robust Ceramic Furnace for Fast and Reliable Quality Control

The maximum temperature of the corrosion-resistant micro-furnace amounts to 1100°C (sample temperature). Its high heating rates of up to 200 K/min are suited for material identification by a fast QC check. A water-cooled jacket provides the micro-furnace with fast cooling and therefore allows for high sample throughput.

Large Sample Changer for 192 Samples – Routine Work at Its Best

The optional automatic sample changer (ASC) with many additional features takes on any routine measurement safely and reliably (more on pages 8 and 9).

Intelligent – *AutoEvaluation* and *Identify*

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

VERTICAL, TOP-LOADING DESIGN

PRECISE ULTRA-MICROBALANCE

VACUUM-TIGHT BY DESIGN

SMARTMODE

DETACHABLE SAMPLE CARRIER

AUTOEVALUATION

AUTO-CYCLE EVACUATION

AUTOMATIC SAMPLE CHANGER FOR UP TO 192 SAMPLES

CALCULATED DTA

INTERCHANGEABLE SAMPLE CARRIERS

OPTIMIZED COUPLING TO FT-IR, MS, GC-MS

IDENTIFY

ROBUST MICRO-FURNACE MADE OF ALUMINA

TG 209 ***F1*** *Libra*®

Trendsetting Technology

Micro-Furnace – A Dynamic System for Efficient Laboratory Work

The low-volume furnace of the NETZSCH TG 209 ***F1*** *Libra*® supports fast heating rates over the entire temperature range and fast ballistic cooling by chilling from 1100°C down to room temperature. The low purge gas flows ensure less dilution of the evolved gases (to be considered if coupling to evolved gas analysis is planned). The short gas path, low volume of the furnace and low dead volume above the sample help prevent condensation of the evolved gases in the furnace.

Safe and Easy Handling

When placing the crucible on the sample carrier, there is no effect to the microbalance since the carrier is lifted by an automatic hoisting device. Sample placement is therefore always trouble-free.

Precise Detection of 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! Simultaneous Monitoring of Caloric Effects

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

Advancements in Thermogravimetry – Taking Care of All Your Day-to-Day TGA Tasks

The Right Sample Carrier for Any Application

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. The sample carrier can be changed out in less than one minute.

Sample carrier made of Al_2O_3 for corrosive gases and sample carrier type P for c-*DTA*®

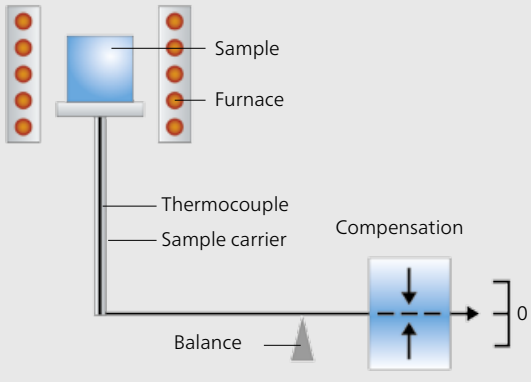
Interchangeable Sample Carriers[1]

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

Measuring Principle

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.



Vacuum-Tight by Design for Reproducible Measurement Conditions

Tests under Vacuum for Improved Separation of Decomposition Steps

Defined Gas Conditions for Reproducible Measurements

The vacuum-tight design allows a pure and defined inert gas atmosphere to be established for the pyrolysis of the sample. No superimposed oxidation by residual air can occur. The integrated gas supply unit with mass flow controllers (MFCs) accommodates up to two purge gases and one protective gas. They can be controlled, recorded and evaluated via the software.

AutoVac – Reproducible Results

The *AutoVac** feature allows for software-controlled automatic evacuation and gas filling, thus providing uniform measurement conditions.
When polymer mixtures or blends are measured at reduced pressure, boiling point depression can be realized for volatiles (e.g., solvents, plasticizers). This leads to better separation from the decomposition of the polymer component. After release of the solvent, it is possible to backfill with an inert gas followed by, for example, an oxidizing atmosphere to continue the measurement for the complete decomposition.

*optional

Crucible Types

Various crucible types made of different materials and in different volumes are available to fit your application needs. The table shows only a small selection of crucibles. Sample/crucible compatibility should always be taken into consideration (e.g., metallic crucibles may not be used for metal samples).

Selection of Crucibles[1]

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
Ideal 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
Ideal for c-*DTA*®, up to max. 600°C	Al (99.5%)	6.7 mm/2.7 mm	85 µl

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

TG 209 ***F1*** *Libra*®	
Design	Top-loading
Temperature range	(10°C) RT to 1100°C
Heating rate	0.001 K/min to 200 K/min
Cooling time	In nitrogen: ≈ 12 min from 1100°C to 100°C In helium[1]: ≈ 5 min from 1000°C to 100°C
Max. sample weight/ measuring range	2 g (including crucible)
TGA resolution	0.1 µg
Motorized sensor	Motorized sensor for easy and safe handling
Interchangeable sample carriers	For standard applications, high-volume samples and large masses; high sensitivity for functions such as c-*DTA*®; special coatings for high resistance to corrosive gases
Vacuum-tightness	10^{-2} mbar (1 Pa)
Gas atmospheres	Inert, oxidizing, reducing, measurements under vacuum (for tests such as rubber analysis)
Gas flow control	Three integrated mass flow controllers for purge and protective gases
AutoVac	Automatic evacuation and refilling of purge gas (optional)
Temperature calibration	c-*DTA*®, also for detection of endo- and exothermal effects; Curie standards
Crucibles	Pt, Al_2O_3, Au, SiO_2, Ag, ZrO_2, Al, etc.; more upon request.
Automatic sample changer (ASC)	Up to 192 samples (optional); various crucible types in one tray
Software	▪ Comprehensive evaluation routines including *SmartMode*, *ExpertMode*, *AutoCalibration* and TGA-*BeFlat*® ▪ *AutoEvaluation* and *Identify* ▪ *SuperRes*® (optional)
Coupling to evolved gas analysis (EGA)	Optional: FT-IR and/or MS or GC-MS, integrated FT-IR (*PERSEUS* TG)

[1] 21°C chiller temperature, 200 ml/min He (purge + protective gas); the maximum temperature of the TGA system depends on the He gas flow: at 200 ml/min, T_{max} is 1020°C.

Key Technical Data

AUTOMATIC SAMPLE

Thermogravimetry Was Never as Efficient as Today – Routine Work and Out-of-Turn Measurements Run Side by Side.

Unprecedented Flexibility

A novelty in thermogravimetry is the automatic sample changer (ASC); it comes with two disposable trays in microplate format for 96 samples each which can be quickly inserted and removed. This allows for measurement of up to 192 samples.

An additional fixed strip is reserved for up to 12 calibration materials or empty crucibles of different dimensions and materials for calibration and correction purposes. Of course, correction measurements with empty crucibles can also be taken on the trays.

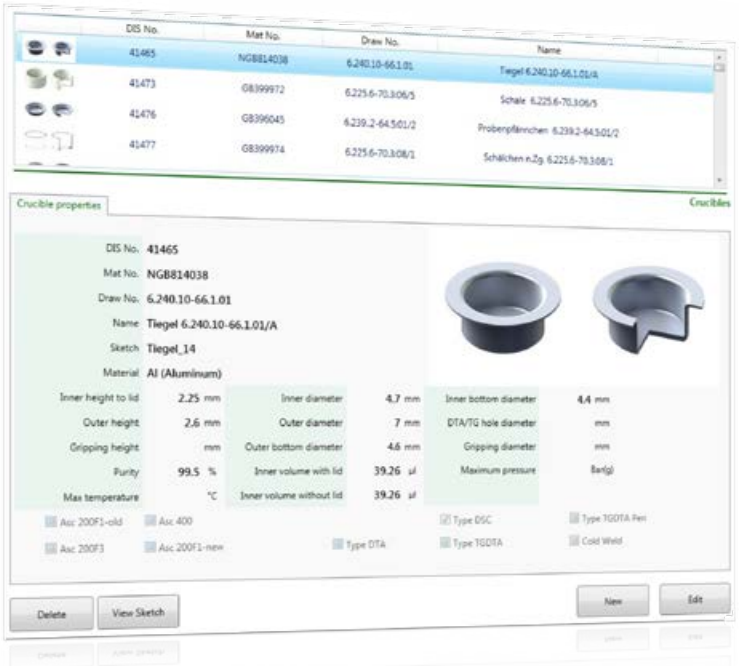
This large number of positions ensures total flexibility and more time left over for other important day-today tasks.

Crucible Recognition in Flight

Safe operation of the gripper is ensured by a laser beam; this beam is detected by a photo diode when the gripper passes over its centering position without any crucible in place. When a crucible is present, the laser beam just strikes the crucible bottom.

Smart Gripper Linked to the Crucible and Lid Database

The gripper with *SafeTouch* functionality is able to handle all kinds of different crucibles. When programming measurement parameters, the specifications for crucible and lid – including the appropriate gripping pressure for each crucible type – is taken from the crucible and lid database. With just a click on the desired crucible and lid, the system has all the necessary details.



CHANGER

Automatically Controlled Tray Cover Protects Samples from Dust, Humidity and Atmospheric Influences

Both trays are immediately covered after the 4-needle gripper has removed or repositioned a crucible. After closing the tray depots, the space can be purged by branching gas channels integrated into the cover. Directly after closing, the gas flow is increased and decreases if the cover remains closed. This entire process takes only a few seconds.

"Remove Lid" Function of the ASC Keeps Your Sample in the Crucible

In addition, a "remove lid" feature comes with the instrument. Placing a lid onto crucibles with unstable samples while they wait their turn to be inserted into the furnace minimizes the risk that they would evaporate or react with ambient humidity prior to the measurement. The gripper disposes of lids and non-reusable crucibles in the refuse bin, based on information stemming from the measurement program.

Sophisticated Down to the Last Detail

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

TG 209 ***F1*** *Libra*® with ASC for 192 samples and an additional 12 positions for calibration/correction samples/crucibles

PROTEUS®

The Backbone for Uncomplicated and

SmartMode – Run Professional Tests After Minutes of Introduction

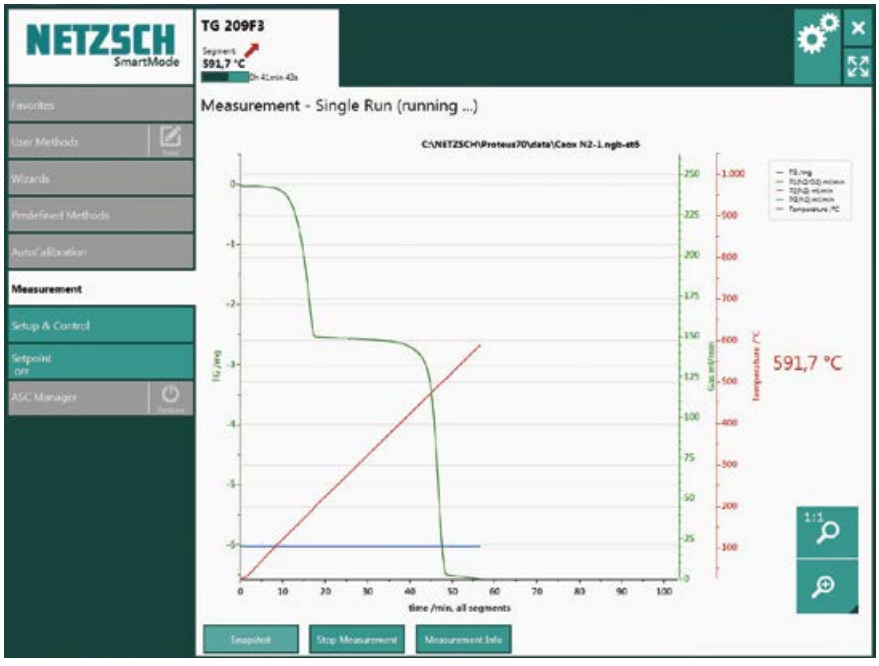
SmartMode boasts a clear structure, a consistent navigation design and easy-to-access buttons. Using wizards (measurement templates), it is possible to start a measurement with just a few inputs. Alternatively, customized methods or predefined methods can be selected to start an experiment. The predefined 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.

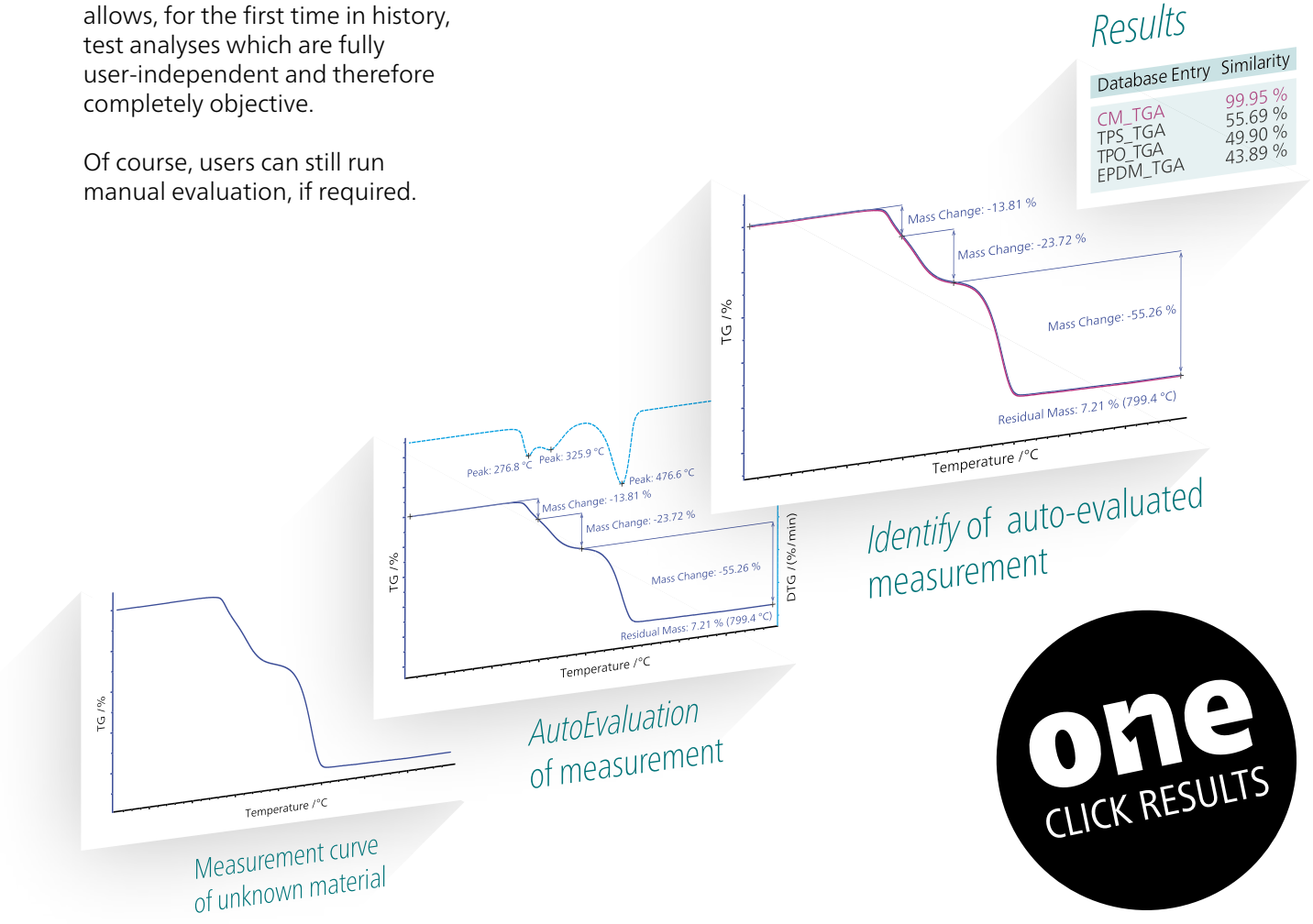
Of course, users can still run manual evaluation, if required.

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.

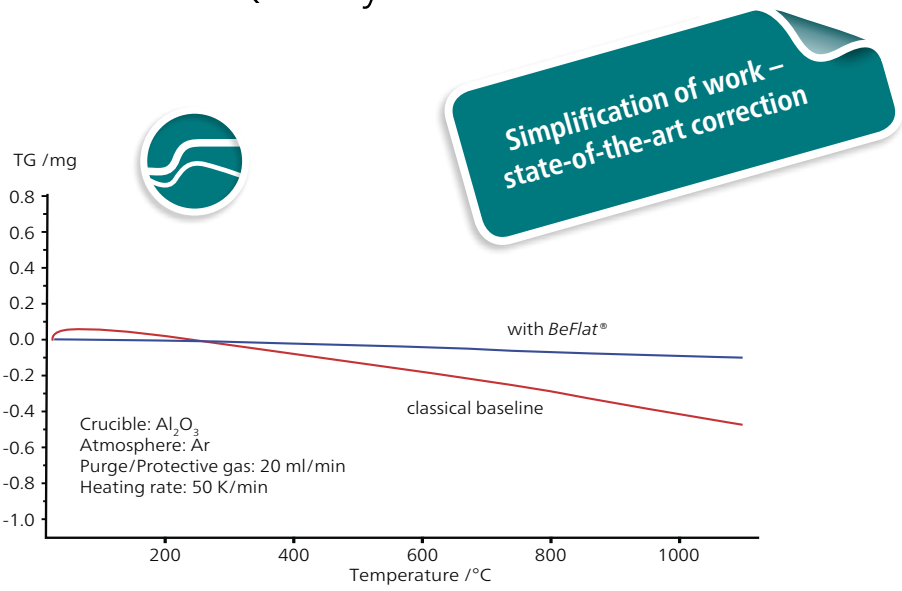


BeFlat®– Measurement Results Quickly Obtained

No More Baseline Run – Get to Your Results Faster

In order to ensure correct mass change values, a baseline run is typically carried out under identical test conditions such as heating rate, gas type, gas flow rate, crucible type and geometry, etc., and subtracted from the sample measurement. The baseline takes instrument and buoyancy influences into consideration.

In contrast, the TG 209 ***F1*** *Libra*® generally no longer requires a separate baseline run. This greatly simplifies routine test work, especially for quality control in industry.



Stable baseline (blue) thanks to automatic correction, TGA-*BeFlat*®, of external influences

Smart Software Tools

Extensions for Comprehensive Evaluation

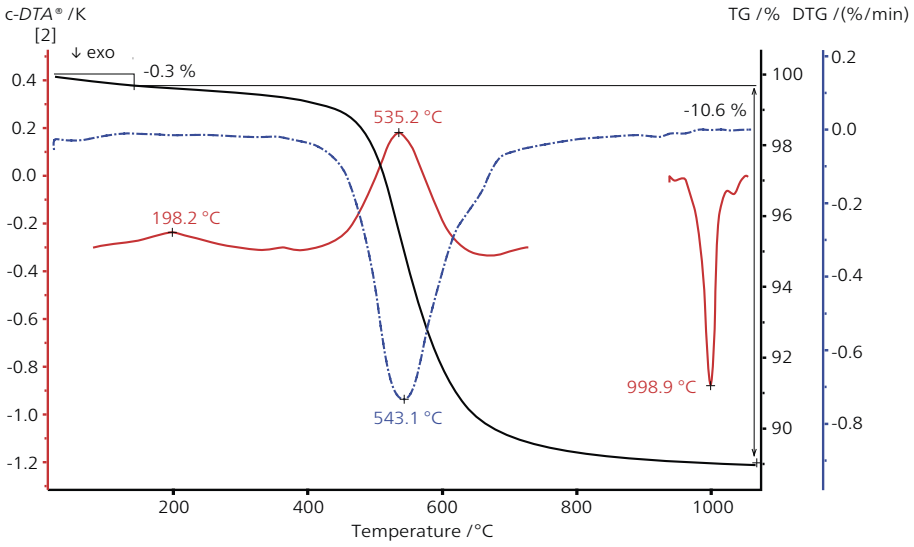
Thermokinetics – Process Optimization by Prediction

Thermokinetics creates kinetic models of decomposition and evaporation processes based on a series of measurements under different temperature conditions. With the unique NETZSCH *Thermokinetics* software, even multi-step processes can be precisely modelled. Kinetic parameters such as activation energy, pre-exponential factor and order of reaction can be determined. *Thermokinetics* can be used to predict the behavior of chemical systems under user-defined conditions for process optimization.

Peak Separation for Improved Determination of Overlapping Mass-Loss Steps

The separation of overlapped peaks in the DTG and DTA signal is achieved by using profiles from the Gaussian, Cauchy, pseudo-Voigt, Fraser-Suzuki, modified Laplace and Pearson peak types. With it, the analysis data is fitted as an additive superposition of peaks.

Calculated DTA for Endo- and Exothermic Effects by Using the Sample Temperature



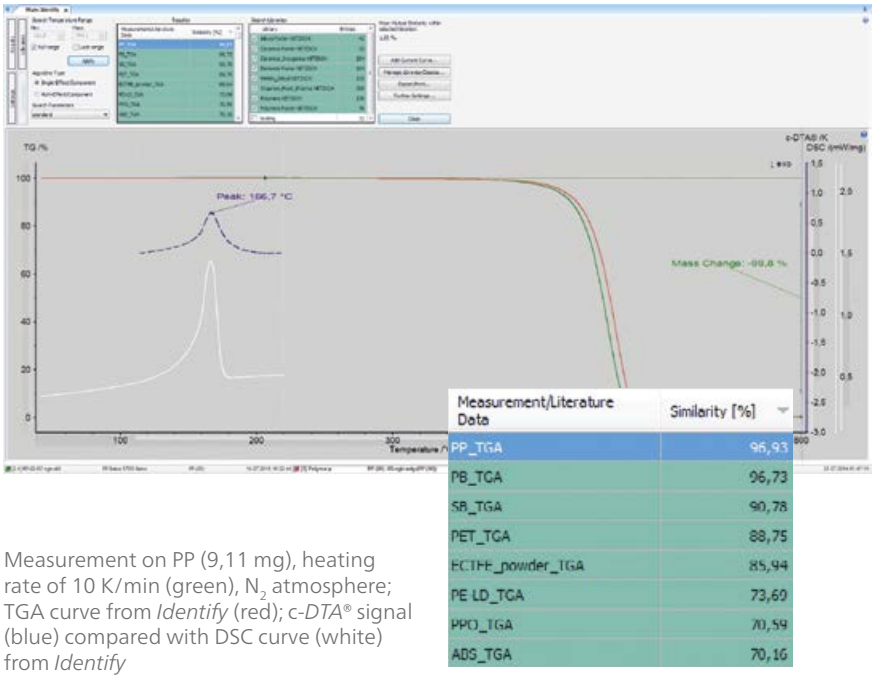
TGA measurement on a kaolinite sample (37.17 mg); joint presentation of the TGA curve (black), the DTG curve (blue, dashed line) and the c-*DTA*® curves (red)

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

In addition to the TGA and DTG curves, this plot depicts endothermic and exothermic effects determined by means of the calculated DTA signal, c-*DTA*®.

Kaolinite was heated to 1100°C in a nitrogen atmosphere at 10 K/min. The two endothermal c-*DTA*® peaks (198°C and 535°C) are directly related to the occurring mass-loss steps and are caused by dehydration and dehydroxylation: loss of adsorbed water from the surface (0.3%) and loss of water from inter layers (10.6%). The exothermal c-*DTA*® peak at 999°C represents the formation of mullite.

CLEVER USAGE IS HALF THE BATTLE!



Measurement on PP (9,11 mg), heating rate of 10 K/min (green), N_2 atmosphere; TGA curve from *Identify* (red); c-*DTA*® signal (blue) compared with DSC curve (white) from *Identify*

Combination of c-*DTA*® and *Identify*

This plot shows the decomposition of a thermoplastic material to 600°C. By using *Identify*, the TGA curve (green) can be assigned to PP (red curve) with a similarity of 96.3%. At 167°C, the c-*DTA*® signal (blue curve) exhibits an endothermic effect where no mass loss is detected. *Identify* also offers curve comparison of further curve types. By overlaying a DSC curve for PP (white), the c-*DTA*® peak can be clearly attributed to melting.

The TG 209 ***F1*** *Libra*® can be employed for the characterization of a great variety of materials including polymers, pharmaceuticals, textiles, foods, cosmetics, and other organic and inorganic materials. For researchers, the technique employed by this instrument is a fast and reliable tool. Its easy operation, quick analysis and standardized evaluation procedures make the TG 209 ***F1*** *Libra*® ideal for applications in quality assurance, failure analysis and research and development.

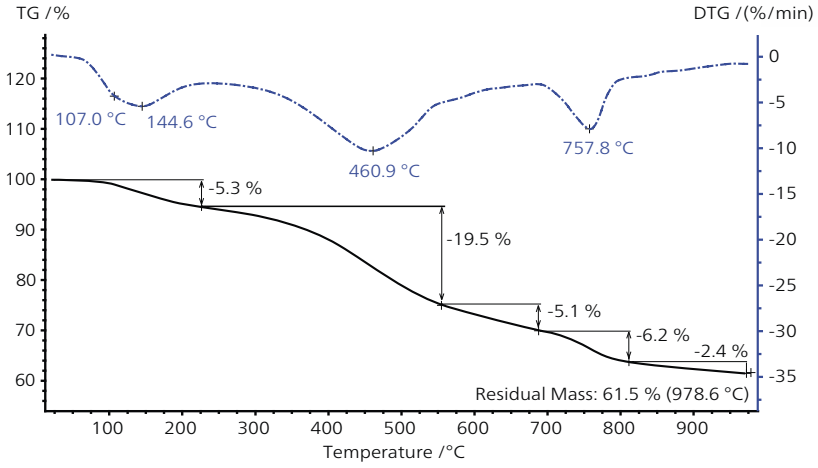
Various international standards describe the general principles of thermogravimetry for polymers (ISO 11358) or other specific applications, such as compositional analysis for rubber (ASTM D6370) and evaporation loss in lubricating oils (ASTM D6375).

APPLICATION FIELDS

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 ash content
- Determination of added carbon black
- Curie temperatures
- Reaction kinetics
- *Purity Determination*

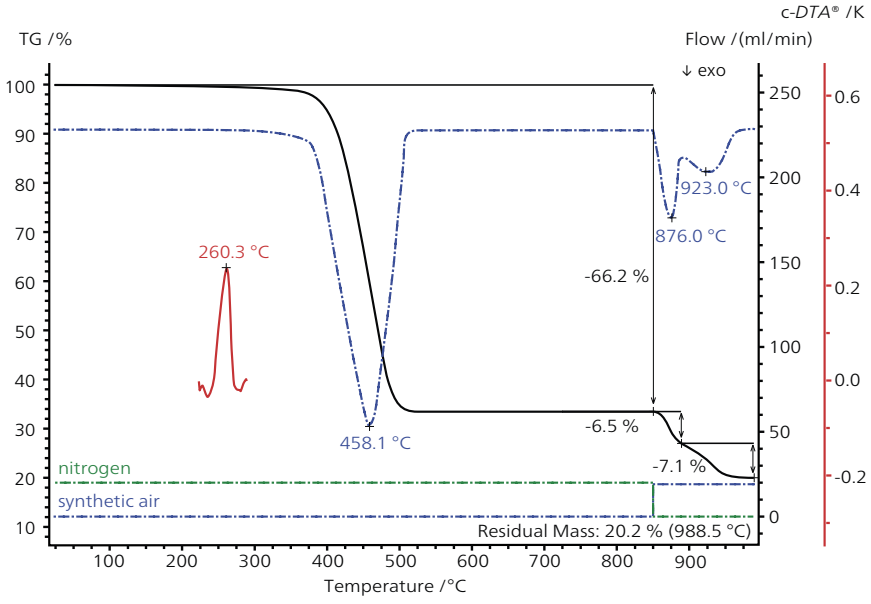
Tracing of Complex Processes – Coal Pyrolysis



TGA curve (black) and DTG curve (blue dashed) of a coal sample

Coal pyrolysis is a complex process involving a large number of chemical reactions. During heating, mainly volatiles (gases and tars) and solid carbon (coke) are produced. Here, 10 mg of coal was measured in a nitrogen atmosphere at a heating rate of 100 K/min. The first mass loss (below 210°C) can most probably be attributed to the release of moisture, while the other steps are mainly related to organic volatiles.

Determination of the Glass Fiber Content in PA66

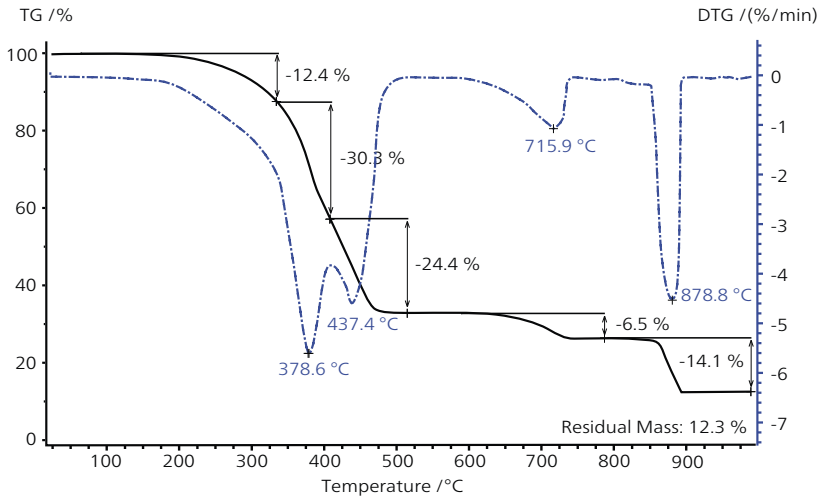


PA 66 measurement (15.2 mg) at a heating rate of 10 K/min; TGA curve (black), DTG curve (blue dashed), c-*DTA*® peak (red)

Polyamide 66 is a thermo-plastic polymer which is used for a wide variety of technical parts. The stiffness of the PA66 can be increased by suitable fillers such as glass fibers. TGA allows for determination of not only the decomposition of the polymer, but also the precise glass fiber content (residual mass 20.2% at 989°C). After switching from nitrogen to synthetic air, the TGA curve also shows the content of pyrolytic soot and added carbon black. The c-*DTA*® peak exhibits the melting peak of PA66 at 260°C (red curve).

Comprehensive Rubber Analysis Cannot Be Imagined without TGA

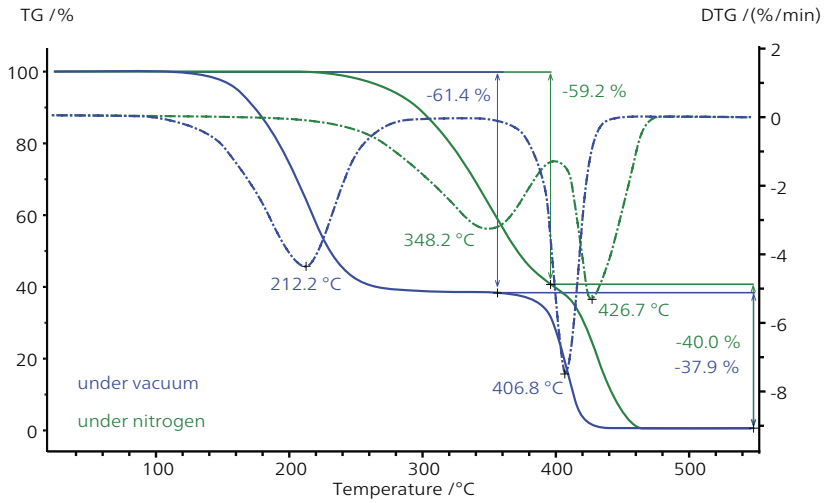
NR/SBR Rubber with Inorganic Filler



Rubber measurement (12.2 mg) at a heating rate of 10 K/min; TGA curve (black), DTG curve (blue dashed)

TGA analysis on rubber is a standard analytical method for the determination of plasticizer content and rubber components. Here, an example of an NR/SBR rubber mixture which releases a plasticizer content of 12.4% is shown. The two-step decomposition (NR and SBR) can be separated precisely. This compound also has chalk content as an inorganic filler. The DTG peak at 716°C and the corresponding mass-loss step of 6.5% can be attributed to the release of CO_2 resulting from the decomposition of chalk ($CaCO_3$). By switching to an oxidizing atmosphere at 850°C, burning of the carbon black was observed.

Vacuum Method for Improved Separation of Plasticizer and SEBS+PP



Comparison of standard test (green curves) and measurement under reduced pressure (blue curves) on SEBS+PP (10.4 mg); heating rate 10 K/min

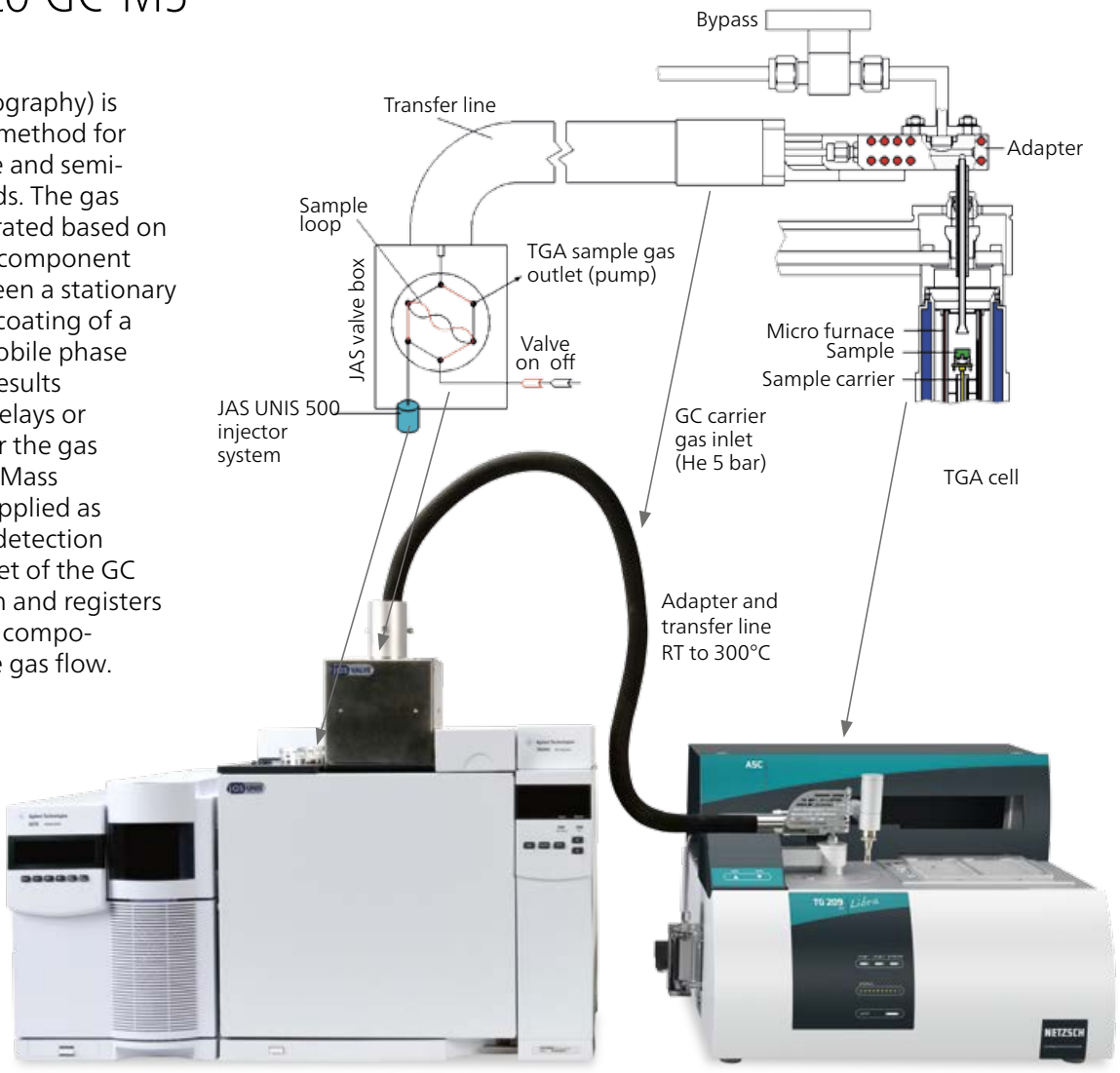
Thermoplastic elastomers are a class of copolymers or polymer mixtures with both thermoplastic and elastomeric properties. They can be used very easily in the manufacturing process, e.g., by injection molding. For investigating the plasticizer content, the advantage of conducting the TGA analysis under vacuum conditions is evident (blue curves). By reducing the boiling point of the plasticizer, two mass-loss steps were clearly separated.

By coupling the TG 209 ***F1*** *Libra*® to a gas analysis technique such as an FT-IR (Fourier Transform Infrared) spectrometer, MS (mass spectrometer), or GC-MS (gas chromatograph – mass spectrometer), information regarding the type of evolved gases as a function of time or temperature can be obtained, thus yielding a fingerprint of the analyzed material.

HYPHENATED TECHNIQUES

Coupling to GC-MS

GC (Gas Chromatography) is a high-resolution method for separating volatile and semi-volatile compounds. The gas mixtures are separated based on the differences in component distribution between a stationary phase (e.g., inner coating of a capillary) and a mobile phase (purge gas). This results in different time delays or retention times for the gas components. MS (Mass Spectrometry) is applied as a highly sensitive detection system at the outlet of the GC separation column and registers the separated gas components in the purge gas flow.



MSD
1.6 u to 1050 u

GC
35°C to 425°C

TG 209 ***F1*** *Libra*®
10°C to 1100°C, 0.1 µg

TG 209 ***F1*** *Libra*®

Always Ready for Coupling to Evolved Gas Analysis (EGA)

Coupling to FT-IR

"More than just the sum of its parts" is the slogan for our comprehensive coupling system incorporating an FT-IR (Fourier Transform Infrared) spectrometer manufactured by our collaborative partner, Bruker Optics.

The purge gas flow from the TGA carries the volatiles through a short heated transfer line to the vacuumtight gas cell of the FT-IR.

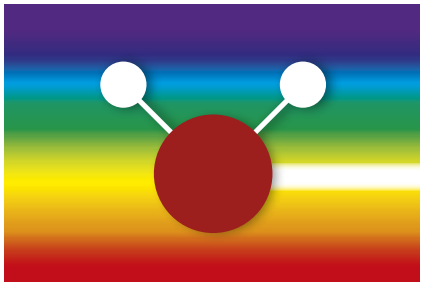
All evolved gases with a changing dipole moment are identified by their typical absorption spectrum, and complex gas mixtures can be spectroscopically separated.

PERSEUS TG 209 ***F1*** *Libra*®

With the *PERSEUS* TG 209 ***F1*** *Libra*®, a TG-FT-IR system was created that forms an excellent alliance with a compact Bruker Optics FT-IR spectrometer. The integrated design for the two systems sets new standards for state-of-the-art coupling techniques. The built-in heated gas cell is directly connected with the gas outlet of the TGA furnace. The low volume of the short transfer path guarantees fast transport and is advantageous for condensable gases.

Coupling to MS

High-level material research and characterization can be achieved by coupling the TG 209 ***F1*** *Libra*® to our QMS 403 *Aëolos*® quadrupole mass spectrometer. Any gases evolved are introduced directly into the electron impact ion source of the MS through a quartz glass capillary heated to 300°C.



Molecules with changing dipole moment can be identified by FT-IR

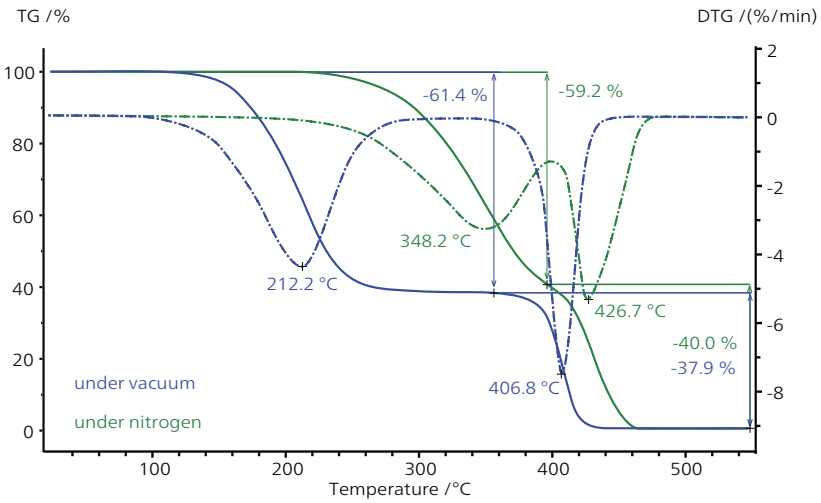
PERSEUS TG 209 ***F1*** *Libra*®

Simultaneous coupling of the TG 209 ***F1*** *Libra*® to FT-IR (Bruker TENSOR II) and MS (*Aëolos*®)

Simultaneous TGA-MS-FT-IR Coupling and *PulseTA*®

The unique heated coupling adapter allows simultaneous TGA-MS-FT-IR measurements, even when the automatic sample changer (ASC) is running. Only one operational software package on a single PC is needed for TGA-MS-FT-IR. Comprehensive evaluations can be displayed in one plot.

Calibration and quantification of the evolved gas components can be achieved by the *PulseTA*® technique. Take advantage of over 40 years of coupling experience and ask for our special coupling brochures.



Comprehensive evaluation of TGA, DTGA, FT-IR (Gram-Schmidt), and QMS (m/z) versus temperature

The NETZSCH Group is a mid-sized, family-owned German company engaging in the manufacture of machinery and instrumentation with worldwide production, sales, and service branches.

The three Business Units – Analyzing & Testing, Grinding & Dispersing and Pumps & Systems – provide tailored solutions for highest-level needs. Over 3,300 employees at 210 sales and production centers in 35 countries across the globe guarantee that expert service is never far from our customers.

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

Leading Thermal Analysis.

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