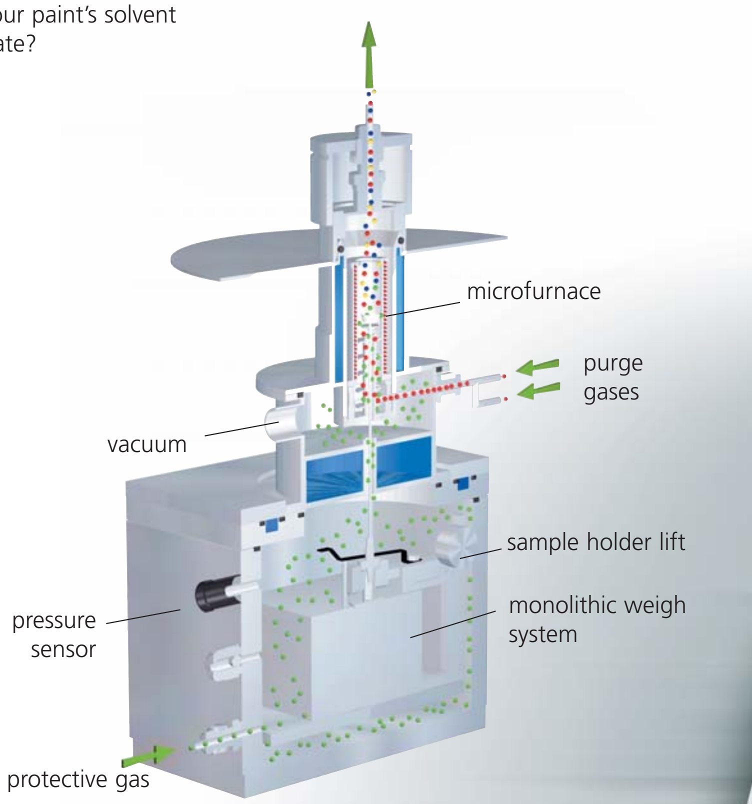
E

TG 209 *F1* Iris®

Thermo-Microbalance TG 209 *F1 Iris*®

- What is your rubber mixture's composition?
- What is the glass fiber content of your plastic?
- How high is the mass loss during polycondensation?
- How much plasticizer does your soft PVC contain?
- How high is the ash content?
- How does your metal's mass increase during oxidation?
- At what temperature does your technical ceramic's binder burn out?
- At what temperature does your pharmaceutical sample release water and solvents?
- Which polymer type is more temperature-resistant?
- What is your plastic's maximum working temperature?
- At what temperature does your paint's solvent evaporate?



These or similar questions can be answered with the help of thermogravimetry (TG) or thermogravimetric analysis (TGA) in research & development, quality assurance, failure analysis and process optimization.

Changes in mass as a function of temperature or time in a defined and controlled environment (atmosphere, flow rate, sample crucible, etc.) can be measured by means of TGA. Along with Differential Scanning Calorimetry (DSC), TGA is also a well-established method in polymer engineering and in the chemical and pharmaceutical industries. Additionally, ceramic materials, minerals and metals, as well as food, adhesives and paints can be analyzed thermogravimetrically.

In academic and industrial research, a thermobalance yielding accurate and reproducible data on the materials' properties is required. For industrial day-to-day routine, it is important to have a versatile and robust thermobalance - especially one with user-friendly automatic functions.

- one fits all

The well proven thermo-microbalance TG 209 *F1 Iris*® offers these state-of-the-art features:

- easily accessible, vertical, top-loading system
- wide temperature range: (10)20 ... 1000°C
- flexible heating and cooling rates: 0.001 ... 100 K/min
- cooling time: only 10 min (1000 ... 100°C) by means of water cooling (cooling thermostat) for high sample throughput
- broad measuring range: 2000 mg
- tare range: 2 g
- resolution: 0.1 µg
- internal mass calibration
- vacuum-tight design
- AutoVac®-ready
- integrated mass flow controllers for accurate gas flow of the two purge gases and the protective gas; controllable, recordable and evaluable via the software
- automatic gas change, programmable via the software
- c-*DTA*® for recording of transformation energetics by means of a calculated DTA curve and for reliable temperature calibration (optional)
- Super-Res® for rate-controlled mass change (optional)
- automatic sample changer (ASC) for 64 different crucibles, also available for the DSC 204 *F1 Phoenix*® (upgradable)
- different sample crucibles, e.g. Pt, Al_2O_3, Al
- coupling to FTIR and/or MS for gas analysis (optional), even for simultaneous operation with the ASC
- sophisticated *Proteus*® software (32-bit) for measurement and evaluation with MS® Windows®
- compatible with all other NETZSCH thermoanalytical instruments
- *Peak Separation* software for optimized DTG peak separation (optional)
- *Thermokinetics* software for the kinetic analysis of decomposition processes (optional)
- conforms to appropriate standards (e.g. DIN 51006, ASTM E 1131, ASTM E 1868, ISO 7111, ISO 11358)

TG 209 *F1 Iris*®
new platform
vacuum-tight design
versatile applications

Proteus® Software - user-friendly and versatile

Our extended 32-bit *Proteus*® software based on MS® Windows® is an extremely user-friendly tool allowing complicated analyses. This is confirmed by our users time and again. See for yourself how the comfortable flexibility of the user guidance and context-sensitive help system can simplify your work.

The *Proteus*® software gives you leeway for your individual evaluation style. Data security and absolute quality are prerequisites for GLP-, GMP- and 21 CFR, part 11 conform use.

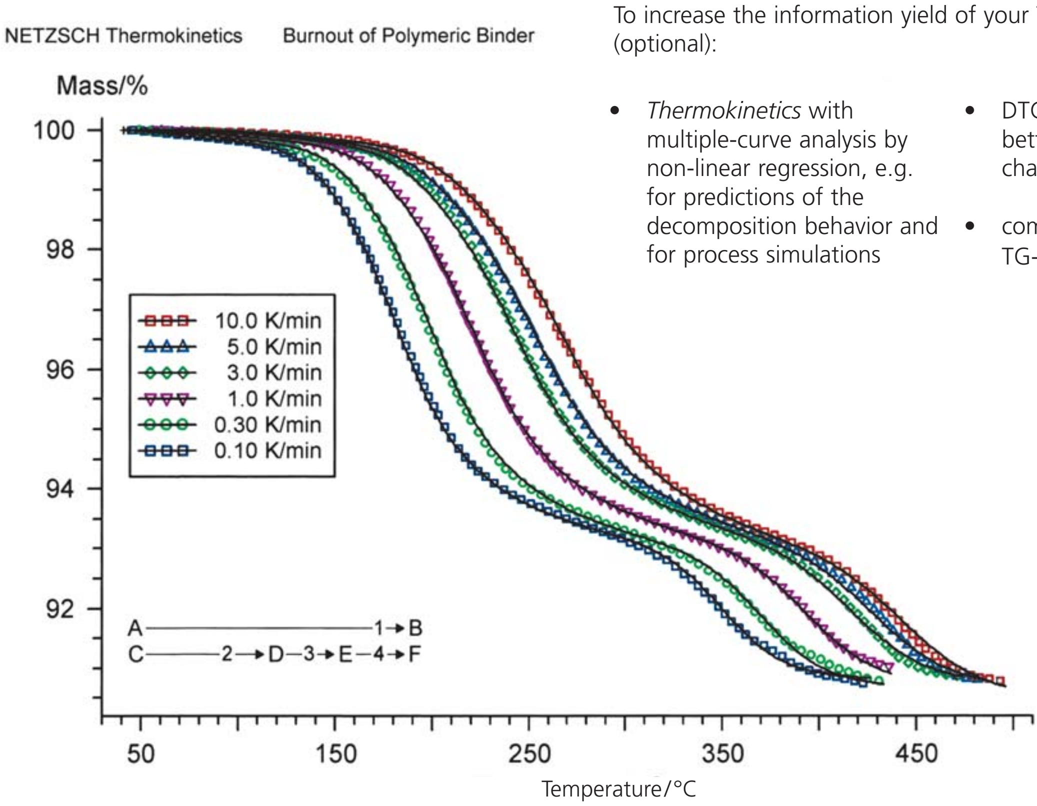
The multi-point temperature calibration, executed either by means of c-*DTA*® or by means of conventional Curie points, is permanently associated with both the raw data and the measurement values for buoyancy correction (baseline with empty crucibles). Evaluation routines conform to standardized methods and can therefore be easily validated.

Proteus®

flexible, intelligent, complete

- multi-tasking: simultaneous measurement and evaluation
- multi-moduling: operation of up to 4 different instruments with one computer
- multi-method analysis: comparison and/or evaluation of TGA, DSC, DIL, TMA and DMA measurements in one plot
- editable experimental program
- repetitive measurements with minimum parameter input
- programming while the measurement is running
- snapshot: on-line evaluation of the running measurement
- curve comparison of up to 32 curves
- curve subtraction
- picture-in-picture presentation (PIP and FLIP)
- selectable scaling
- selectable colors, line types and thicknesses
- zoom function
- 1st and 2nd derivative
- automatic evaluation of characteristic temperature and mass-change steps
- calculation of residual mass
- storage and export of evaluations
- export and import of data (ASCII)
- direct data export to MS® EXCEL
- multi-point calibration for the sample temperature
- context-related help system
- automatic dispatch of status messages or complete measurement results by e-mail
- fully automatic macro-evaluation (optional)
- c-*DTA*® (optional)
- Super-Res® (optional)

Advanced Software



To increase the information yield of your TGA measurements (optional):

- *Thermokinetics* with multiple-curve analysis by non-linear regression, e.g. for predictions of the decomposition behavior and for process simulations
- DTG peak separation for better separation of mass-change steps
- combined TG-FTIR and TG-MS software

TG 209 *F1 Iris*® – one fits all

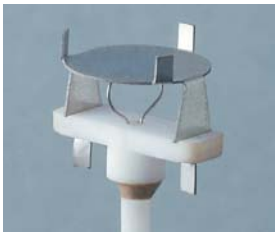
The TG 209 *F1 Iris*® represents the third generation of NETZSCH thermobalances specially designed for the temperature range up to 1000°C. Many of our users' requests and suggestions were integrated into the new concept; for example, the standard unit is now AutoVac®-ready. Also, by carrying out the measurement in a vacuum, low volatiles can be more clearly separated from a polymer (reduction of the boiling point).

Evacuation and re-filling, gas exchange, and adjustment of the purge and protective gas rates are activated via the measuring software. The signals generated for the flow rates in ml/min can be recorded by the software.

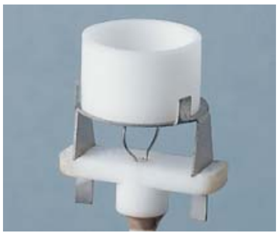
The sample is inserted into the robust sample carrier in a crucible of a material of your choice. The sample carrier is automatically raised out of the furnace chamber by pressing the button for the automatic lid-lifting device. This makes it easier and quicker to change the sample. By again pressing the button to close the lid, the sample carrier is returned into the furnace chamber and the measurement can be started.

The sample temperature is measured directly at the sample with a thermocouple of *Platinel*®, a PdPt/Au/AuPd alloy, and is used for the determination of the c-*DTA*® signal (calculated DTA curve). The c-*DTA*® is ideally suited for the temperature calibration of the system and for the analysis of energetic effects within the sample.

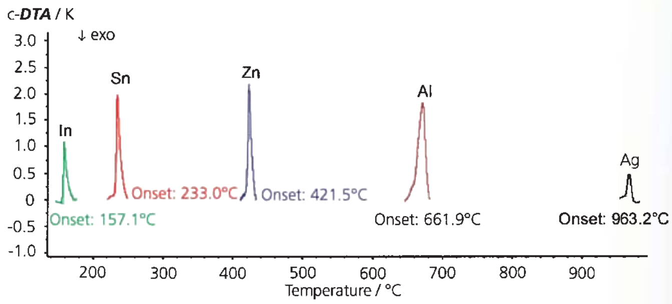
The sophisticated coupling technique for evolved gas analysis (TG-FTIR and/or TG-MS), with which your system can be upgraded, is presented on pages 8 and 9.



Sample carrier with *Platinel*® thermocouple



Sample carrier with Al_2O_3 sample crucible



Melting peaks (raw data) of reference metals for temperature calibration with c-*DTA*®

ASC - Automatic Sample Changer

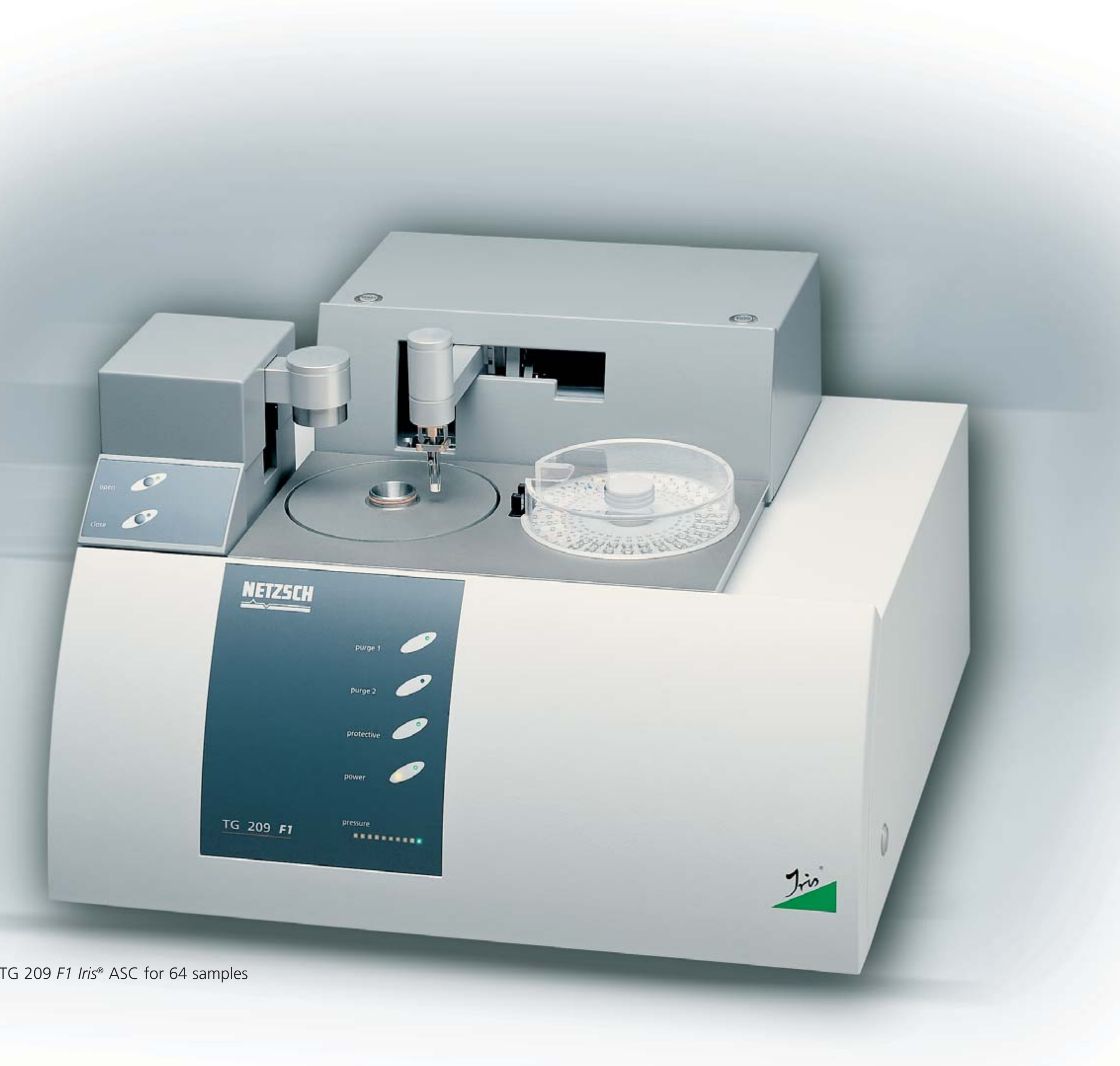
The automatic sample changer (ASC) for up to 64 samples changes different crucible types safely and reliably. The ASC can handle routine measurements for quality assurance as well as measurements for research and development.

This feature allows unattended operation around the clock, freeing the user to perform other tasks.

Of course, each sample can be assigned a different measurement and evaluation program. It is also possible to insert unplanned analyses into a preprogrammed series of measurements which is already in progress.

The ASC can also be employed for the DSC 204 *F1 Phoenix*® since it is based on the same platform concept.

For unstable samples or samples with volatile components, an automatic piercing device is available which opens the sealed crucible just prior to the start of the measurement.

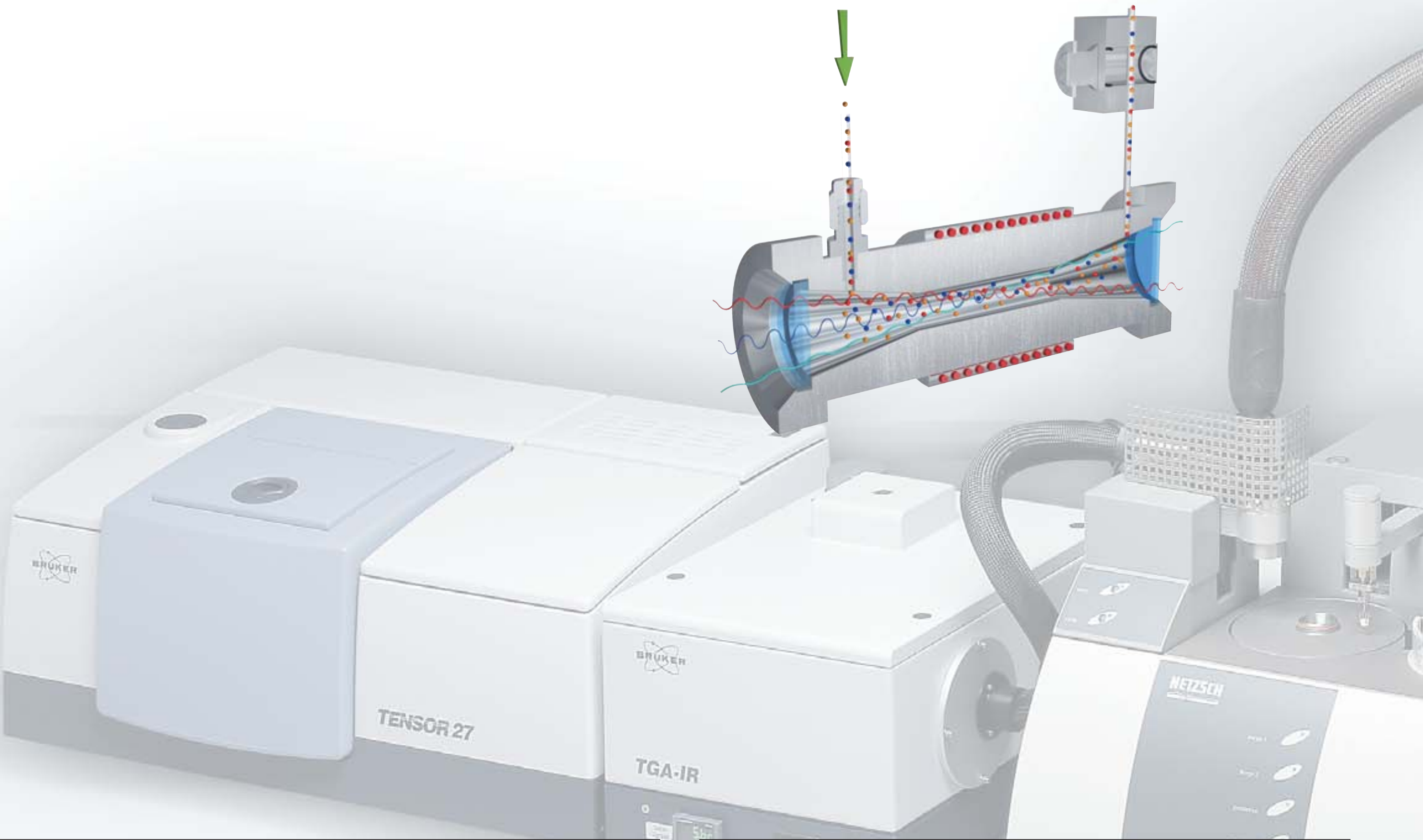


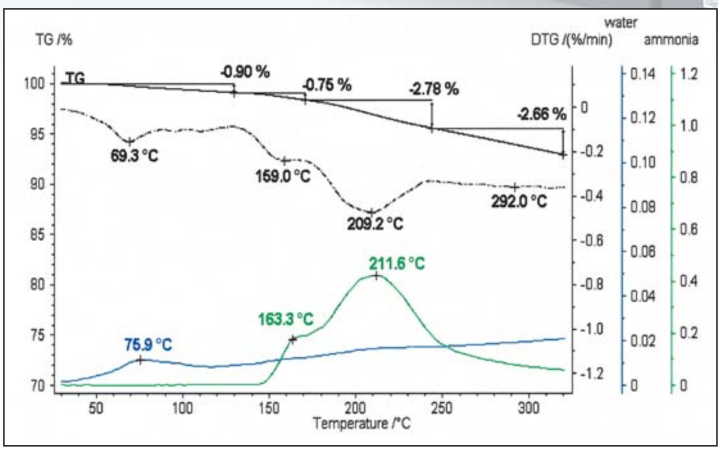
TG 209 *F1 Iris*® ASC for 64 samples

Coupling of the TG 209 *F1 Iris*® to an FTIR and MS

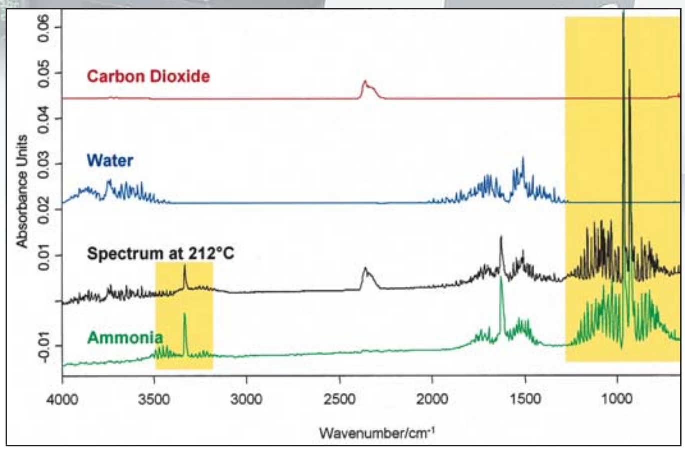
With thermogravimetry alone, you can obtain information about the properties, composition and stability of a sample subjected to controlled thermal treatment. More detailed conclusions concerning material changes associated with the mass changes of the sample can only be achieved by coupling the thermobalance to a gas analysis technique. This combination enables characterization of the unidentified sample by determining the decomposition products (fingerprints).

The two diagrams below portray the curing of a phenolic resin as analyzed by TG 209 *F1 Iris*® coupled to the FTIR Spectrometer via a heated transfer line. In addition to water, ammonia is also clearly detected during polycondensation of a phenolic resin at higher temperatures. This indicates amine as the hardener component.



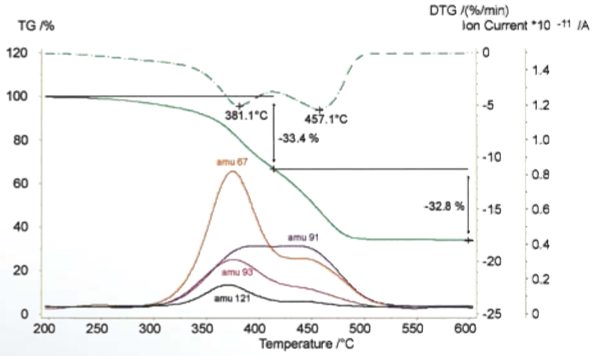


TGA and DTG curves as well as traces of water (blue curve) and ammonia (green curve) for the polycondensation of phenolic resin.



FTIR absorption spectrum at 212°C in comparison with the spectra from the database.

Complete Analysis System – also simultaneously ASC and MS/FTIR

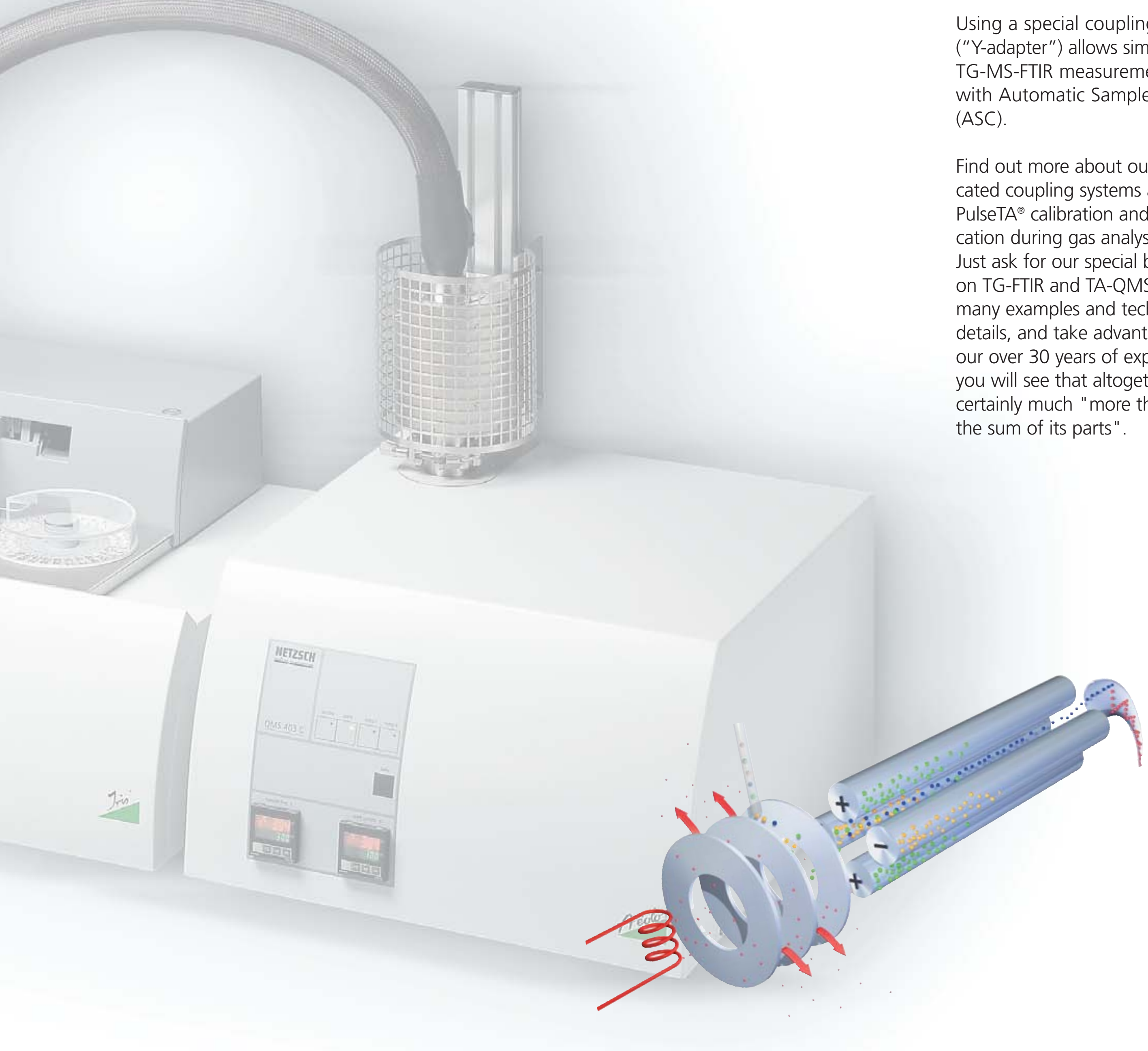


TGA, DTG and ion current curves for fragments with different atomic mass units (amu)

Material research and characterization at the highest level can be achieved by coupling the TG 209 *F1 Iris*® to our QMS 403 C *Aëolos*® quadrupole mass spectrometer. Gases evolved are introduced directly into the electron impact ion source of the MS through a quartz glass capillary heated to 300°C. Various hydrocarbon fragments can thereby be detected into the ppm range during the decomposition of a rubber compound (see plot).

Using a special coupling adapter ("Y-adapter") allows simultaneous TG-MS-FTIR measurements, even with Automatic Sample Changer (ASC).

Find out more about our sophisticated coupling systems and PulseTA® calibration and quantification during gas analysis.
Just ask for our special brochures on TG-FTIR and TA-QMS with many examples and technical details, and take advantage of our over 30 years of experience; you will see that altogether it is certainly much "more than just the sum of its parts".

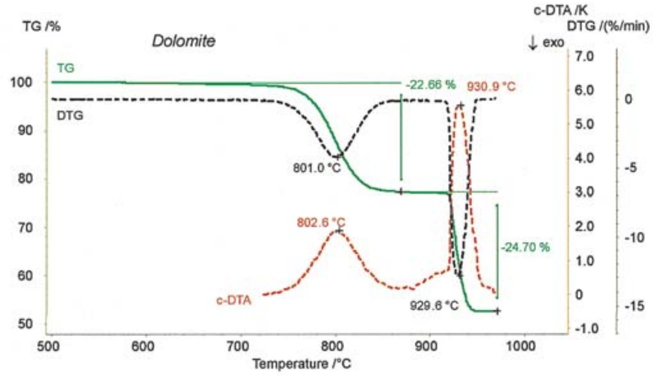


Bruker FTIR Tensor 27 – TG 209 *F1 Iris*® ASC – QMS 403 C *Aëolos*®

Applications

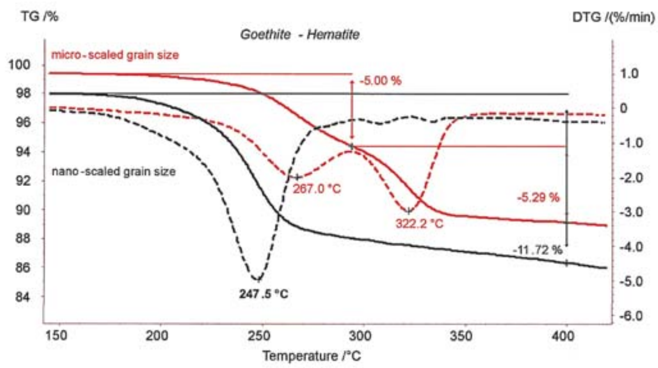
Thermal decomposition of dolomite in a CO_2 atmosphere

The mass loss steps during the thermal decomposition of dolomite [$CaMg(CO_3)_2$], overlap when the measurement is performed in a nitrogen atmosphere.
By using CO_2 as a purge gas, they can be clearly separated. The calculated DTA signal (c-*DTA*®) additionally yields the information that both effects are endothermal.



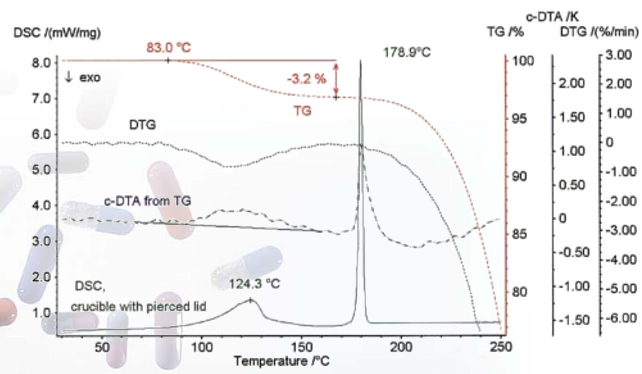
Inorganic Pigments

The mechanism of the thermal conversion of goethite into hematite ($2\alpha\text{-FeOOH} \Rightarrow \alpha\text{-Fe}_2O_3+H_2O$) is determined by the size of the microcrystalline goethite needles. With decreasing particle size, a decline in the complexity of the conversion can be observed.

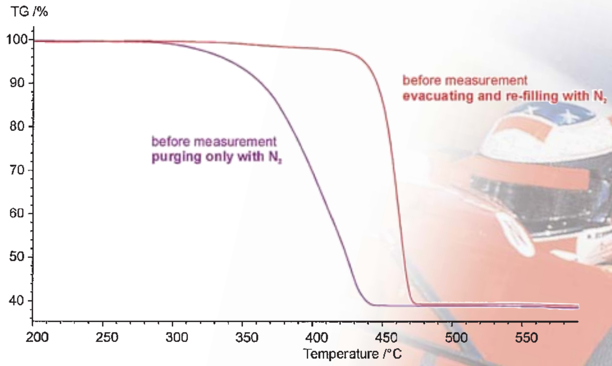


Dehydration of estradiol hemihydrate

The dehydration of the estrogen substitute begins at 83°C and yields a mass loss of 3.2%, which is exactly equivalent to the stoichiometric water content. With the c-*DTA*® curve, not only can one evaluate the associated endothermal effect at 120°C, but the melting at 179°C as well. The separately conducted DSC measurement verifies the c-*DTA*® result.

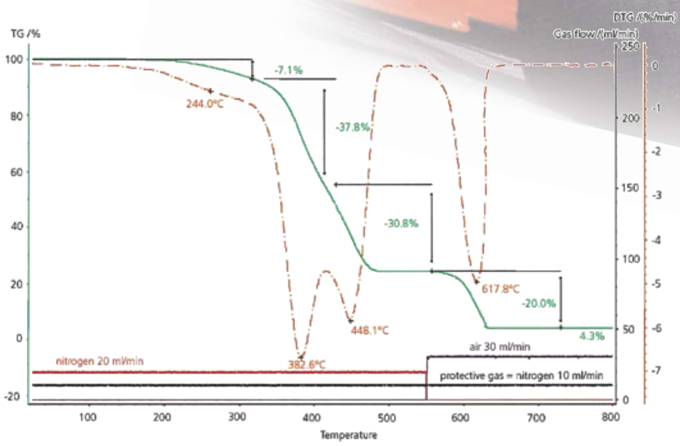


Applications



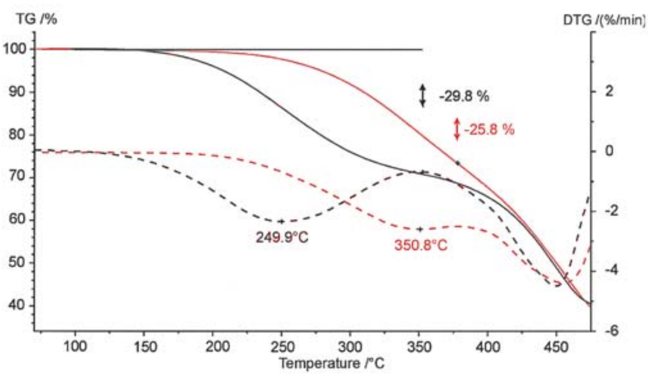
Decomposition of a talcum-filled polypropylene

In order to generate a defined inert gas atmosphere for pyrolysis, the thermobalance is evacuated before the measurement and re-filled with nitrogen gas. Extensive purging with inert gas before beginning the measurement is no substitute for evacuation; it cannot fully eliminate the possibility that residual oxygen may remain in the system. This causes an earlier onset of decomposition of oxidation-sensitive substances.



Components of a rubber compound for tires

Besides the plasticizer portion (ca. 7%), the first elastomer components during the decomposition of a rubber compound are derived at 383°C (38%) and the second at 448°C (31%). The carbon black portion is calculated at 20% and the ash content at 4%. The position of the peak temperatures of the derivated TGA curves (DTG) show that the compound is a carbon black-filled NR/SBR rubber blend.



Plasticizer content in a rubber compound

In a standard measurement (red curves), the evaporation of a low-molecular plasticizer is overlapped by the decomposition of the elastomer components. The plasticizer begins separating earlier due to the lower boiling point under vacuum conditions (blue curves). The plasticizer content can hereby be determined much more precisely.

TG 209 *F1 Iris*® - Technical Data

Weighing system
- Wide measuring range: 2000 mg
- Tare range: 2 g
- Resolution: 0.1 µg

Heating system
- Temperature range: (10) 20 ... 1000°C
- Heating and cooling rates: 0.001 ... 100 K/min
- Cooling time: approx. 10 min (1000 ... 100°C)

Sample atmosphere
- AutoVac®-ready
- Vacuum down to 10^{-2} mbar
- Static/dynamic: inert, reactive gases (non-toxic, non-flammable, non-explodable), forming gas (≤3% H_2)
- Gas control for two purge gases and a protective gas by means of integrated gas flow controllers and software; automatic gas change

Sample crucibles
- Alumina, platinum, aluminum, graphite, quartz glass, etc.
- Standard crucibles with an outer diameter of 6.8 mm and a volume of 0.085 ml
- Additional crucible materials and sizes up to 0.350 ml upon request

Proteus® software (32-bit) for MS® Windows®

Options
- Automatic sample changer (ASC) for up to 64 different crucible types (upgradable)
- Coupling to FTIR and/or MS
- c-*DTA*® (calculated DTA curve)
- Super-Res® (rate-controlled mass change)
- Macro recorder
- *Peak Separation* (DTG)
- *Thermokinetics* (decomposition kinetics)

Perfect Platform Concept for DSC and TGA

DSC 204 *F1 Phoenix*® with automatic sample changer (ASC)

same design,
same electronics,
same sample changer as
TG 209 *F1 Iris*®

NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · D-95100 Selb/Bavaria
Phone: +49 9287 881-0 · Fax: +49 9287 881144
e-mail: at@ngb.netzsch.com

www.netzsch-thermal-analysis.com

NGB · TG 209 F1 Iris · E · 1000 · 0406 · LH