

Simultaneous Thermal Analysis Method, Technique, Applications



Method

Simultaneous Thermal Analysis generally refers to the simultaneous application of Thermogravimetry (TG) and Differential Scanning Calorimetry (DSC) to one and the same sample in one instrument. The advantages are obvious: The test conditions are perfectly identical for the TG and DSC signals (same atmosphere, gas flow rate, vapor pressure on the sample, heating rate, thermal contact to the sample crucible and sensor, radiation effect, etc.). Furthermore, it improves sample throughput as more information is

gathered from each test run. Since the early years of its existence, NETZSCH has given high priority to the development and continuing optimization of its Simultaneous Thermal Analyzers yielding the STA 449 **F1 Jupiter**[®]. It meets respective instrument and application standards for TG and DSC systems including: ISO 11357, ISO 11358, ASTM E 967, ASTM E 968, ASTM E 793, ASTM D 3895, DIN 51004, DIN 51006, DIN 51007.



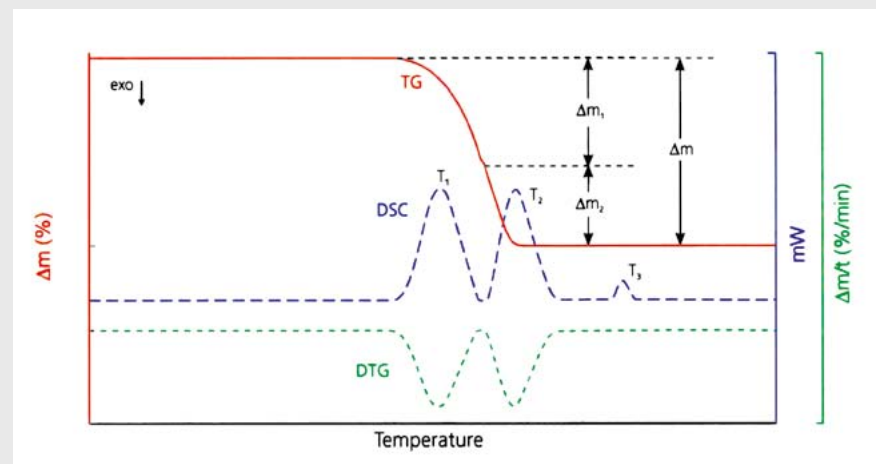
DSC analysis possibilities

- Melting/crystallization behavior
- Solid-solid transitions
- Polymorphism
- Degree of crystallinity
- Glass transitions
- Cross-linking reactions
- Oxidative stability
- Purity determination
- Specific heat
- Thermokinetics

TG analysis possibilities

- Mass changes
- Temperature stability
- Oxidation/reduction behavior
- Decomposition
- Corrosion studies
- Compositional analysis
- Thermokinetics

Measured signals in an STA



The STA 449 **F1 Jupiter**[®] allows determination of caloric effects (transformation temperatures and enthalpies) and mass changes at the same time with outstanding reliability, resolution and accuracy. The toploading Simultaneous Thermal Analyzer can easily be adjusted to almost any application by selecting the optimum furnace, installing the ideal sensor and using the proper accessories. It combines a high-performance Heat-Flux DSC with the world's first Thermo-Nanobalance, offering top-level DSC sensitivity and resolution with a high accuracy, long-time stable and top-resolution thermobalance. The STA 449 **F1 Jupiter**[®] comprises a balance system representing the benchmark for high-performance thermobalances and a DSC system capable of even specific heat measurements over an unmatched

temperature range. The system can operate from -150°C to 2400°C using various interchangeable sensors and furnaces. The optional double furnace hoist or automatic sample changer (ASC) system can improve sample throughput and the ASC allows operation during the night or over the weekend.

The STA 449 **F1 Jupiter**[®] offers highest TG resolution (0.025 µg, 25 ng) combined with a broad measurement range of 5 grams. The various DSC sensors offer true DSC performance over an unmatched temperature range (-150°C to 1750°C). Small phase transitions as well as specific heat can be analyzed with high accuracy.

The vacuum-tight design, along with high resolution, metal housed MFC systems, make the system an ideal tool for top-level TG and DSC research in academia and industry.

This fully equipped thermal analyzer can easily analyze small amounts of a new active pharmaceutical substance, minimal contaminations on a semiconductor waver, electronic component or medical implant, or deviations in the composition of an inorganic mixture.

For Evolved Gas Analysis (EGA), the system is well prepared for the coupling of mass spectrometers (MS), of GC-MS, and of FTIR. MS and FTIR can even work in parallel, when coupled simultaneously to the STA.



STA 449 **F1** Jupiter[®] – Trend-Setting Technology

Top-loading - the standard for balance systems

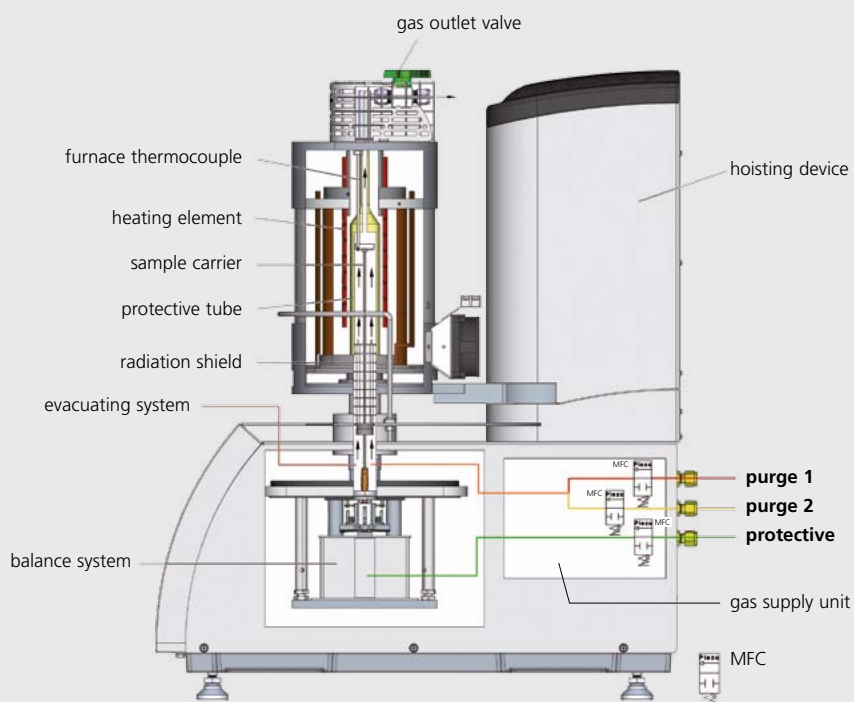
The STA 449 **F1** Jupiter[®] is a top-loading system using a balance design that has been standard for a long time in laboratories. The reasons are simple. These systems combine ideal performance with easy handling. Why should your thermobalance be any different?

The world's first Nanothermo-balance

The STA 449 **F1** Jupiter[®] comprises a balance system representing the benchmark for high-performance thermobalances. The system allows measurements on samples of up to 5 grams in weight and up to 5 ml in volume. Therefore, most complications resulting from sample inhomogeneity are simply eliminated by the large sample sizes that the STA 449 **F1** Jupiter[®] is capable of analyzing. You only have small sample masses available? No problem. STA 449 **F1** Jupiter[®] is the first commercial thermobalance with a digital resolution in the nano range (0.025 µg). This resolution spans the entire measurement range (5 grams). Further outstanding features of the balance section of this STA include lowest noise and a microgram stability over hours.

Vacuum-tight design – optimal atmosphere control

The STA 449 **F1** Jupiter[®] is vacuum-tight by design. Practically every component is designed to fulfill the requirements of high vacuum applications. Using a turbo molecular pump system, vacuum levels better than 10^{-4} mbar can be reached. The unique OTS[®] accessory can be used to reduce the oxygen concentration at the sample below 1 ppm. This, together with the built-in gas supply unit with three mass flow controllers (MFC) for purge and protective gases, offers optimum control of the atmosphere around the sample (e.g. pure inert conditions). This is crucial for an accurate interpretation of the measured effects, e.g. to differentiate between oxidation and decomposition reactions.



Further Information

www.sta449.info

Furnaces

Furnace type	Temperature range	Cooling system
Silver furnace	-120°C to 675°C	liquid nitrogen
Steel furnace	-150°C to 1000°C	liquid nitrogen
Platinum furnace	RT to 1500°C	forced air
Silicon carbide furnace	RT to 1550°C	forced air
Rhodium furnace	RT to 1650°C	forced air
Graphite furnace	RT to 2000°C	tap or chilled water
Water vapor furnace	RT to 1250°C	forced air
High-speed furnace	RT to 1250°C	forced air
Tungsten furnace	RT to 2400°C	tap or chilled water

Furnace and Furnace Hoists

Nine interchangeable furnaces are available to accommodate different application areas across the entire temperature range (-150°C to 2400°C). A double furnace hoist allows the simultaneous installation of two different furnaces for improved sample throughput or for low and high-temperature tests with the same instrument. The furnaces can easily be changed by the operator. Therefore, the system is adaptable to any future application range.

The high-speed furnace allows for high sample throughput for quality control as well as kinetic studies. Linear heating rates of up to 1000 K/min can be achieved at the sample with a final temperature of 1250°C.



Highest Precision – Maximum Flexibility

Various sensors

The STA 449 **F1 Jupiter**® can be equipped with different sensor types. TG sensors with plates or large crucibles (up to 5 ml) allow tests on large sample volumes and masses. TG-DTA sensors can be used for applications such as routine tests or measurements on aggressive sample substances. The TG-DSC and TG-DSC- c_p sensors are used for most tests and allow quantitative DSC testing simultaneous to the TG runs. The c_p versions additionally allow determination of the specific heat with high accuracy. For special applications such as tests under corrosive atmospheres, the protected

sensors can be employed.

The Fast-Fix connection of the sensors to the instrument allows sensors to be changed within seconds. The system can therefore easily be adapted among the various required applications.

Automatic sample changer

An automatic sample changer for up to 20 samples is optionally available. The sample changer guarantees optimum crucible placement and effective use of the instrument. Pre-programming allows measurements to be carried out during the night or over a weekend.

Innovative Software

Features such as *BeFlat*® and *Tau-R Mode*™ allow a fully automatic baseline correction as well as correction for system time constants. All routines are fully software based and can be optimized for your specific measurement conditions. Furthermore, the raw data signal can be accessed at any time. The STA 449 **F1 Jupiter**® is the first simultaneous thermal analyzer capable of doing modulated DSC measurements (The feature is not available in North America and in Japan).



Examples for STA 449 sample carrier and sensor types

Sensors

Sensor thermocouple	Temperature range	Sensor types	Atmospheres
Type E	-150°C to 700°C*	TG, TG-DTA, TG-DSC (c_p)	inert, oxid., red., vac.
Type K	-150°C to 800°C*	TG, TG-DTA, TG-DSC (c_p)	inert, oxid., red., vac.
Type S	RT to 1650°C	TG, TG-DTA, TG-DSC (c_p)	inert, oxid., red., vac.
Type P	-150°C to 1000°C	TG-DSC, TG-DSC (c_p)	inert, oxid., red., vac.
Type B	RT to 1750°C	TG, TG-DTA, TG-DSC	inert, oxid., red., vac.
Type W/RE	RT to 2400°C	TG, TG-DTA	inert, red., vac.
Type S protected	RT to 1650°C	TG, TG-DTA	inert, oxid., red., vac., corr.

* in oxid. atmosphere up to 500 °C

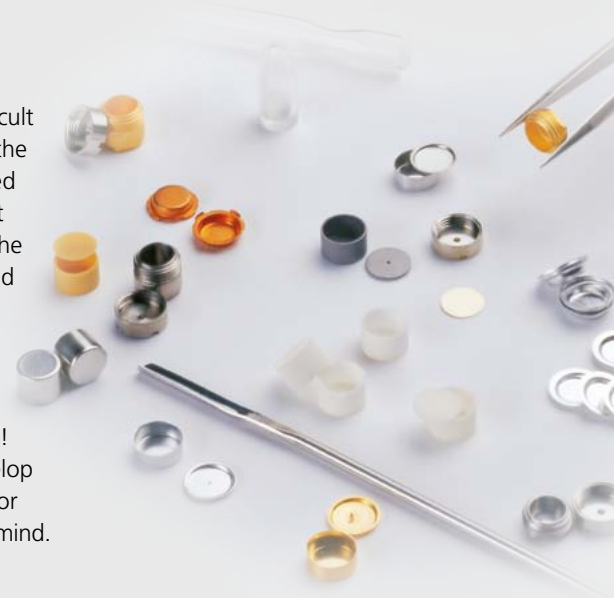
Accessories

A wide range of crucibles (aluminum, silver, gold, copper, platinum, alumina, zirconia, graphite, stainless steel, etc.) is available for nearly all possible applications and materials.

For working in critical atmospheres, a "CO version" of the STA 449 **F1 Jupiter**® can be supplied. This version is optimized for measurements under corrosive or reducing atmospheres. Gas flow control systems are prepared in a separate box and special sensors with protected thermocouple wires are

available. For measurements on difficult samples or radioactive substances, the STA 449 **F1 Jupiter**® can be prepared for installation in a glove box or hot cell. Electronics are removed from the measurement part and all cables and fittings are prepared for connection to an existing feedthrough.

If you have any other special application or test condition, ask us! Our engineers are prepared to develop special versions of instrumentation or software with your requirements in mind.



STA 449 **F1 Jupiter**® with Automatic Sample Changer (ASC)

Further Information

www.sta449.info

Proteus® Software for the STA 449 **F1 Jupiter**®

The STA 449 **F1 Jupiter**® runs under the versatile *Proteus*® software on a Windows® operating system. The *Proteus*® software includes everything you need to carry out a reliable measurement and evaluate the resulting data – or even carry out complicated analyses. The *Proteus*® software is licensed with the instrument and can also be installed on other computer systems. The data security and absolute quality that it provides are prerequisites for usage conforming to GLP and GMP.

General Software Characteristics:

- For Windows XP® and Windows® 7 operating systems
- Multi-tasking: Simultaneous measurement and evaluation
- Multi-moduling: Operation of several pieces of equipment via a single PC
- Combined analysis: Comparison and/or evaluation of STA, DSC, TGA, DIL, TMA and DMA measurements in a single presentation
- Labeling: Text elements can be entered and positioned freely
- Calculation of 1st and 2nd derivative
- Selectable scaling
- Selectable colors and line types
- Export of graphics and data
- Saving and recovery of the analysis status
- Temperature calibration
- Compatible with advanced software packages (*Peak Separation, Thermokinetics*)
- Macro-recorder (optional)
- Context-based Help system
- Software developed by ISO-certified enterprise of the NETZSCH Group

DSC Features

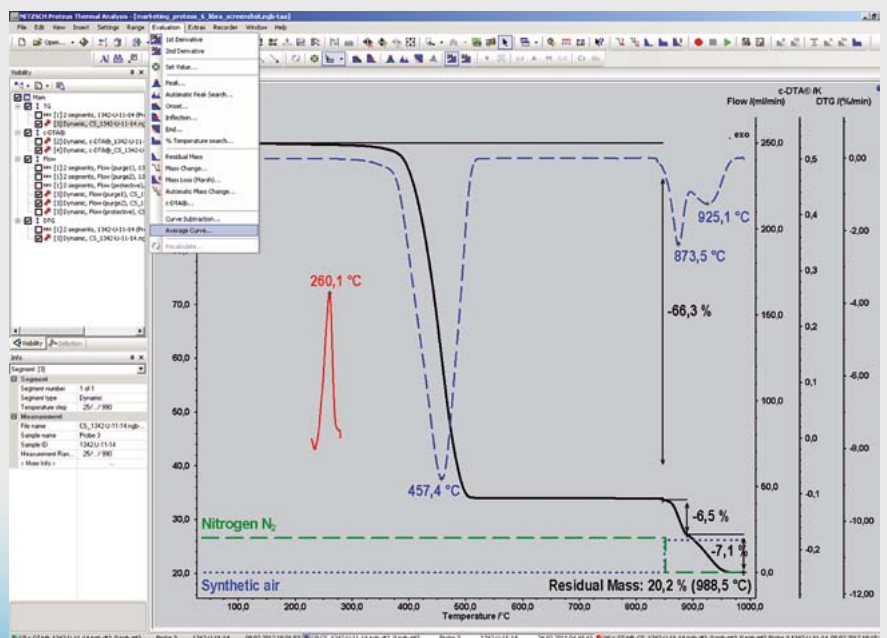
- Determination of onset, peak, inflection and end temperatures
- Automatic peak search
- Transformation enthalpies: analysis of peak areas (enthalpies) with selectable baseline and partial peak area analysis
- Comprehensive glass transition analysis
- Automatic baseline correction
- Degree of crystallinity
- O.I.T. (oxidative induction time) evaluation
- Specific heat determination
- *BeFlat*[®] for automatic baseline correction
- *Tau-R Mode*[™]: evaluation of exo- and endo-thermal effects under consideration of system time constants and thermal resistance values
- *Purity Determination* (optional)

Advanced Software (options)

- TM-DSC module software extension for temperature-modulated DSC tests (This feature is not available in North America and Japan)
- *Peak Separation Software*: allows accurate separation and evaluation of overlapping transitions
- *Thermokinetics*: allows advanced characterization of reactions and kinetic parameters on the basis of multiple-step kinetic analysis on up to 16 curves, also provides predictions of the process

TG Features

- Mass changes in % or mg
- Automatic evaluation of mass change steps
- Determination of the residual mass
- Extrapolated onset and endset
- Peak temperatures of the 1st and 2nd derivatives of the mass change curve
- Automatic baseline correction
- c-DTA[®] for the calculated DTA signal with evaluation of characteristic temperatures and peak area (optional for TG measurements)
- *Super-Res*[®] for rate-controlled mass change (optional)

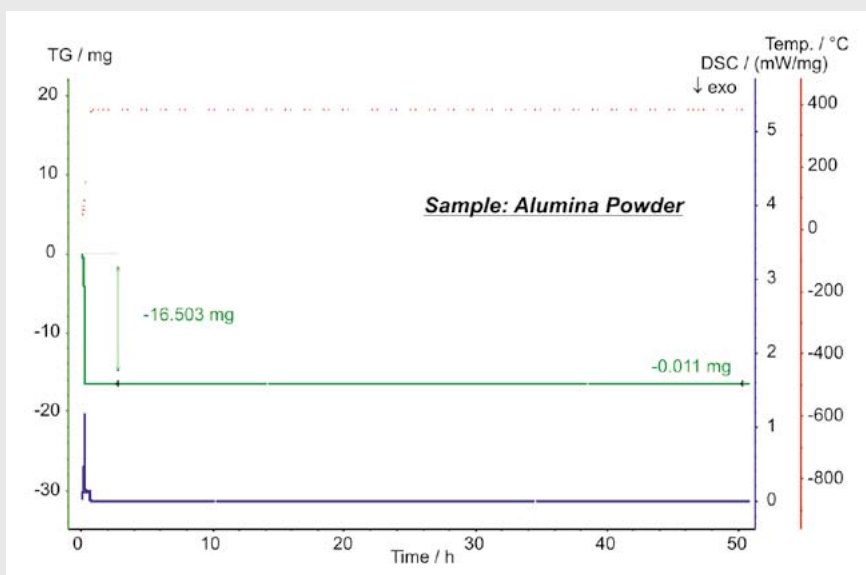


Meaningful Material Characterization in the Low-Temperature Fields

Application Examples

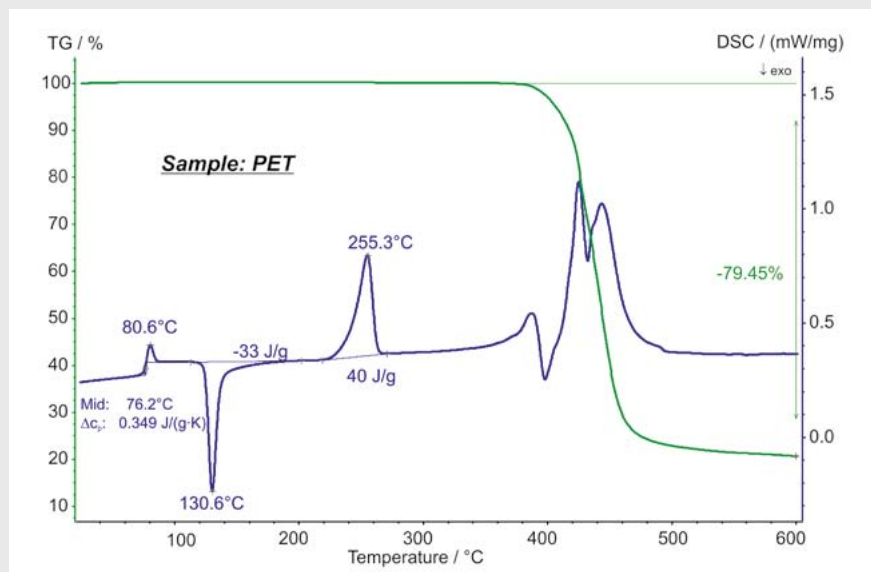
Excellent long-term stability

While heating an alumina powder sample (initial mass of 120.0 mg) to 400°C, a mass loss of 16.50 mg occurred caused by the evaporation of humidity. This was accompanied by an endothermic DSC peak. During the 50-hour isothermal phase, the mass changed by only 11 micrograms demonstrating the excellent long-term stability of the balance system.



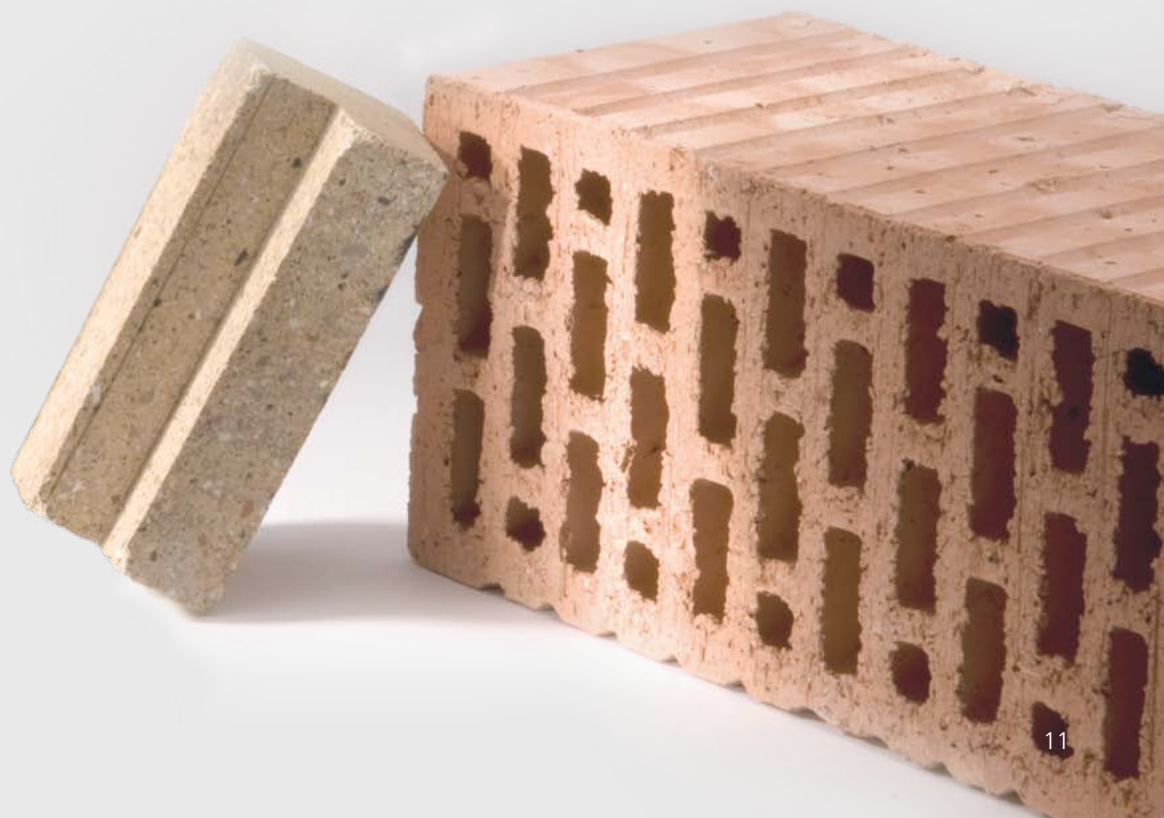
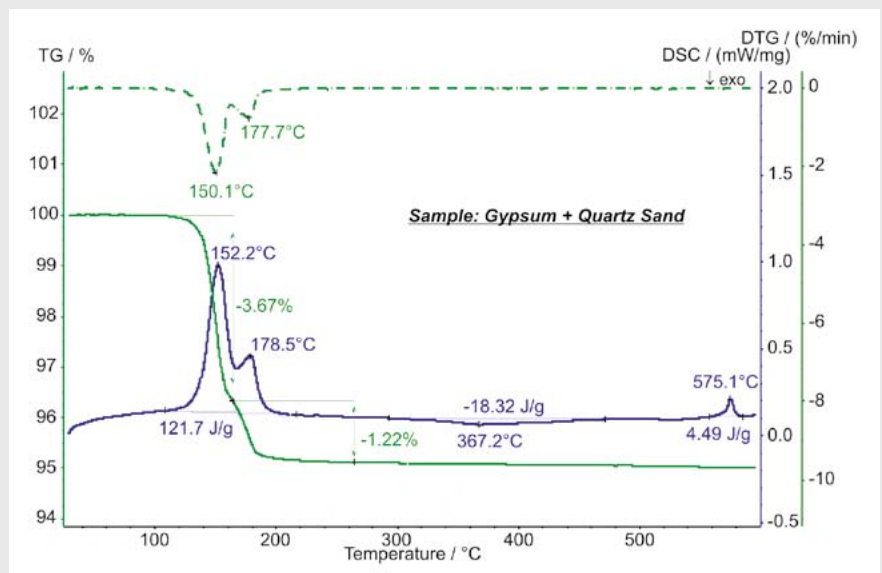
Plastics

Plastic bottles, textile fibers and films (for example packaging for food) are well known applications of the polymer PET (polyethylene terephthalate). The STA measurement under nitrogen exhibits a step in the DSC signal below 100°C which is due to the glass transition. A corresponding increase in specific heat of 0.35 J/(g·K) was detected. The endothermic DSC peak at 81°C is due to relaxation, the exothermic peak at 131°C is due to crystallization and the endothermic peak at 255°C is due to melting. At temperatures above 360°C, the pyrolytic decomposition of the sample occurred with an entire mass loss of 79.5%.



Building materials: gypsum and quartz sand

Gypsum and quartz sand are often used in building materials such as plaster and mortar. The gypsum part of the sample shows a two-step dehydration below 250°C from $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (dihydrate) into $\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$ (half-hydrate) and finally into CaSO_4 (anhydrite). This requires a total energy of 122 J/g. Quantitative analysis reveals that the gypsum was a pure dihydrate with a mass fraction of 23.4% in the sample. Between approx. 300°C and 450°C, the exothermic formation of $\beta\text{-CaSO}_4$ with a released energy of 18.3 J/g occurred. The endothermic effect at an extrapolated onset temperature of 573°C is due to the structural $\alpha \rightarrow \beta$ transition of quartz (crystalline SiO_2).

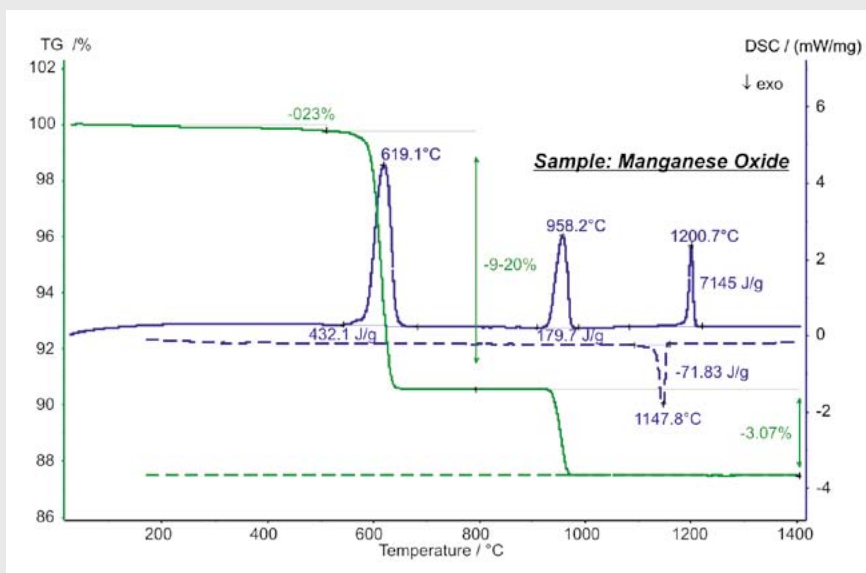


Meaningful Material Characterization in the High-Temperature Fields

Application Examples

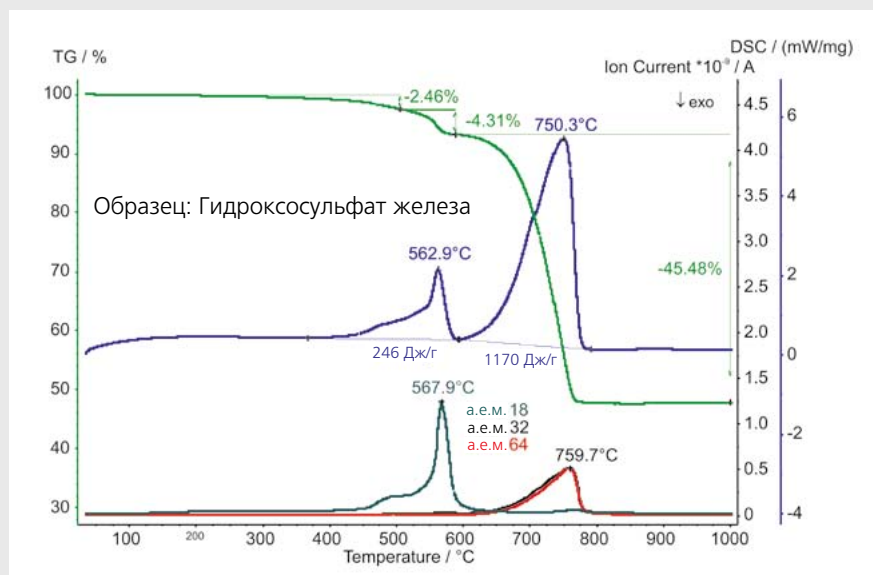
Reduction of manganese dioxide

Manganese dioxide (MnO_2) is often used in chemistry as an oxidizer but is also used, for example, as a cathode material in batteries. This STA measurement shows mass loss steps at approx. 600°C and 950°C which are due to the reduction of MnO_2 into Mn_2O_3 and finally into Mn_3O_4 . The values of 9.20% and 3.07% match the stoichiometrical values exactly, thus reflecting the high accuracy of the balance system. Endothermic DSC peaks with enthalpies of 432 J/g and 180 J/g were detected during the reduction steps. The endothermic DSC peak at 1201°C is due to a reversible structural transformation of Mn_3O_4 which was observed at the peak temperature of 1148°C upon cooling (dashed lines).



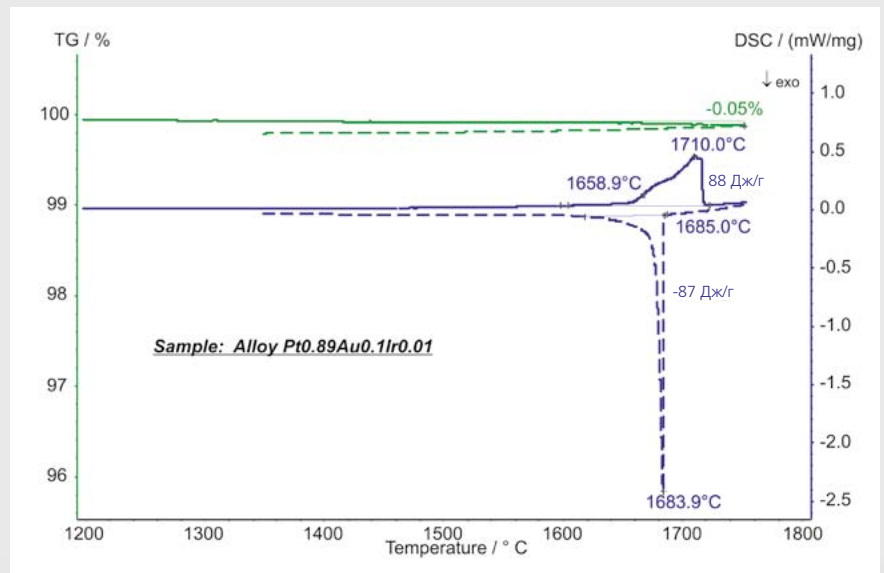
Decomposition of iron hydroxide sulfate

Iron hydroxide sulfate $\text{Fe}(\text{OH})\text{SO}_4$ is a possible base material for the synthesis of iron oxide particles. These can be used, for example, as a pigment or as a magnetic storage medium. So called ferrofluids contain superparamagnetic iron oxide nanoparticles which can serve as a contrast agent for MRI. Below 600°C, the STA-MS measurement exhibits a two-step release of H_2O with mass number 18, and between 600°C and 800°C, a release of SO_2 and O_2 with mass numbers 64 and 32, respectively. The final product is Fe_2O_3 (hematite).



Phase diagrams of alloys

$Pt_{0.89}Au_{0.10}Ir_{0.01}$ is a possible dental alloy generally used for inlays, crowns and bridges. Dental alloys must be shapeable but robust, corrosion-resistant and biocompatible. The measurement shows an endothermic DSC effect with an enthalpy of 88 J/g beginning at an extrapolated onset temperature of 1659°C upon heating (solid lines). This effect is due to melting. Upon cooling (dashed lines), an exothermic DSC peak (peak temperature 1684°C) with an enthalpy of -87 J/g occurred at 1685°C onset temperature due to recrystallization of the alloy. The mass loss of 0.05% observed at highest temperatures may be due to the start of evaporation.



Expertise in Service



Our Expertise – Service

All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, 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.

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

Summary of Our Services

- Installation and commissioning
- Hotline service
- Preventive maintenance
- Calibration service
- IQ /OQ/PQ
- On-site repairs with emergency service for NETZSCH components
- Moving/exchange service
- Technical information service
- Spare parts assistance

Our Expertise – Applications Laboratories

The NETZSCH Analyzing & Testing applications laboratories are proficient partners for nearly any Thermal Analysis issue. Our diverse measuring methods and over 30 different state-of-the-art test stations will provide ready solutions for all your thermal needs.

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 our expert team. This will enable you to precisely define new materials and components before actual deployment, minimize risks of failure, and gain decisive advantages over your competitors.

For production problems, we can work with you to analyze causal issues and work out solution concepts. The option of commercial testing is a relatively low investment which generally pays itself off in a few days or weeks and provides fast and reliable support for research on new materials or resolving any kinds of production issues.



The NETZSCH Group is an owner-managed, internationally operating technology company headquartered in Germany.

The three Business Units – Analyzing & Testing, Grinding & Dispersing and Pumps & Systems – provide tailored solutions for highest-level needs. Over 2,500 employees at 130 sales and production centers in 23 countries across the globe guarantee that expert service is never far from our customers.

When it comes to Thermal Analysis, Adiabatic Reaction Calorimetry 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.

NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42
95100 Selb
Germany
Tel: +49 9287 881-0
Fax: +49 9287 881-505
at@netsch.com

www.netsch.com