



## Simultaneous Thermal Analyzer – STA 449 **F1** Jupiter®

Method, Technique, Applications



# At Technology's Cutting Edge

Simultaneous Thermal Analysis generally refers to the simultaneous application of Thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) to one and the same sample in a single instrument. The advantages are obvious: The test conditions are perfectly identical for the TGA 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, sample throughput is improved as more information can be gathered from each test run.

## DSC Possibilities

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

## TGA Possibilities

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

| Standard* | Description   |
|-----------|---|
| ISO 11358 | Plastics – Thermogravimetry (TG) of Polymers  |
| ASTM E793 | Standard Test Method for Enthalpies of Fusion and Crystallization by Differential Scanning Calorimetry            |
| DIN 51004 | Thermal Analysis; Determination of Melting Temperatures of Crystalline Materials by Differential Thermal Analysis |
| DIN 51006 | Thermal Analysis (TA); Thermogravimetry (TG); Principles  |
| DIN 51007 | Thermal Analysis; Differential Thermal Analysis; Principles   |

\* Depending on instrument setup

STA 449 **F1** Jupiter®

# World-Leading Combination of Flexibility and Precision

– 50 YEARS –  
Leading Manufacturer of  
High-Performance  
Thermal Analysis Systems



The STA 449 **F1 Jupiter**® combines a high-performance *Heat-Flux* DSC with a nanogram-resolution thermobalance, thereby offering a high sample load and wide measurement range. This STA system can easily be adjusted to nearly any possible application by selecting the optimum furnace, installing the most appropriate sensor and using the proper accessories. It is a unique and ideal tool for material characterization in research and development.

Setting New Benchmarks through  
Experience & Innovation

### The World's First Thermo-Nanobalance

The STA 449 **F1 Jupiter**® has set a new 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. Such large possible sample sizes simply eliminate most potential complications resulting from sample inhomogeneity and impurities. Only have small sample masses available?

No problem. The STA 449 **F1 Jupiter**® is the first thermobalance on the market with a digital resolution in the nano range (0.025 µg) that spans the entire measurement range (5 grams). Additional outstanding features of the balance section include the lowest available noise levels and microgram stability over a period of hours.

### 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?

### Defined Atmosphere Conditions – Vacuum-Tight Design

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 of better than 10<sup>-4</sup> mbar can be reached.

The unique OTS® accessory can be used to reduce the oxygen concentration at the sample to below 1 ppm.





# Select the Appropriate

Ten interchangeable furnaces are available to accommodate different application areas across the entire temperature range (-150°C to 2000°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.

## Day-to-Day Work Done Safely

For standard STA measurements, the silicon carbide furnace (SiC) is the robust workhorse in your laboratory, operating from ambient temperature to 1600°C. For measurements under corrosive atmosphere, the SiC furnace can be equipped with a protected TGA-DTA sensor guaranteeing instrument-safe conditions.

## Measurements in the Lower-Temperature Range

The silver and steel furnaces allow for measurements in the subambient temperature range by using devices for controlled cooling. Whereas the silver furnace is ideally suited for the determination of the specific heat capacity, the steel furnace offers a broad temperature range from -150°C to 1000°C.

## Specific Heat Capacity at Higher Temperatures

The platinum and the rhodium furnaces in combination with dedicated DSC sensors are specifically suited for determination of the specific heat capacity in the higher temperature range.

## Your Results Achieved at the Highest Speed

The high-speed furnace allows for the simulation of realistic heating processes with linear heating rates up to 1000 K/min. Additionally, the high heating rates are useful when implementing kinetic studies.



STA 449 FI Jupiter

close

# Furnace

## *for Your Application!*

### Highest Temperatures

The tungsten heating element allows for measurements under helium atmosphere from room temperature to 2000°C and high-vacuum measurements from 400°C to 2000°C.

### Measurements in Humid Atmospheres

The water-vapor furnace covers the broad temperature range from room temperature to 1250°C. The furnace can be connected to a humidity generator, or to a vapor generator which produces steam by evaporating water. A molar concentration of up to 100% can be achieved.

The copper furnace can be used for conventional STA measurements including determination of the specific heat capacity up to 500°C. It is ideal for measurements under relative humidity between room temperature and 100°C. For this purpose, a humidity generator is available which offers a maximum dewpoint of 80°C corresponding to 47% molar concentration.

| Furnace type    | Temperature range <sup>1</sup> | Cooling system               |
|-----------------|--------------------------------|------------------------------|
| Silver          | -120°C to 675°C                | liquid nitrogen <sup>2</sup> |
| Copper          | -150°C to 500°C                | liquid nitrogen <sup>2</sup> |
| Steel           | -150°C to 1000°C               | liquid nitrogen <sup>2</sup> |
| Platinum        | RT to 1500°C                   | forced air                   |
| Silicon carbide | RT to 1600°C                   | forced air                   |
| Rhodium         | RT to 1650°C                   | forced air                   |
| Graphite        | RT to 2000°C                   | tap or chilled water         |
| Water-vapor     | RT to 1250°C                   | forced air                   |
| High-speed      | RT to 1250°C                   | forced air                   |

<sup>1</sup> Corresponds to maximum sample temperature range

<sup>2</sup> Alternative vortex cooling allows for start temperatures around 0°C.

*The Right Sensor for Your Demands*

# Various Sensors

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

The *Quick-Connect* system of connecting sensors to the instrument allows sensors to be changed in a matter of seconds. This allows the system to be easily adapted to any of the various potential applications.

| Sensor thermocouple | Temperature range | Sensor types                    | Atmospheres                     |
|---------------------|-------------------|---------------------------------|---------------------------------|
| Type E              | -150°C to 700°C*  | TGA-DTA, TGA-DSC ( $c_p$ )      | inert, red., oxid., vac.        |
| Type K              | -150°C to 800°C*  | TGA-DTA, TGA-DSC ( $c_p$ )      | inert, red., oxid., vac.        |
| Type S              | RT to 1650°C      | TGA, TGA-DTA, TGA-DSC ( $c_p$ ) | inert, red., oxid., vac.        |
| Type S protected    | RT to 1650°C      | TGA, TGA-DTA                    | inert, red., oxid., vac., corr. |
| Type P              | -150°C to 1000°C  | TGA, TGA-DSC, TGA-DSC ( $c_p$ ) | inert, red., oxid., vac.        |
| Type B              | RT to 1750°C      | TGA, TGA-DTA, TGA-DSC           | inert, red., oxid., vac.        |
| Type W              | RT to 2000°C      | TGA, TGA-DTA                    | inert, red., vac.               |

\* in oxid. atmosphere up to 500°C



### Standard Type S Sensors – Workhorse and Specialty

In the high-temperature range, type S sensors combine a broad temperature range from room temperature to 1650°C with high sensitivity. For measurements in the presence of corrosive gases, the TGA-DTA sensor with protected thermocouples provides safe conditions without adversely affecting the sensitivity.

### High Sensitivity in the Lower Temperature Range

The type P sensors are standard in the lower temperature range; they are ideally suited for the steel furnace. All sensors equipped with thermocouple E or K are characterized by the highest levels of sensitivity and resolution. They are particularly well suited for detecting small effects.

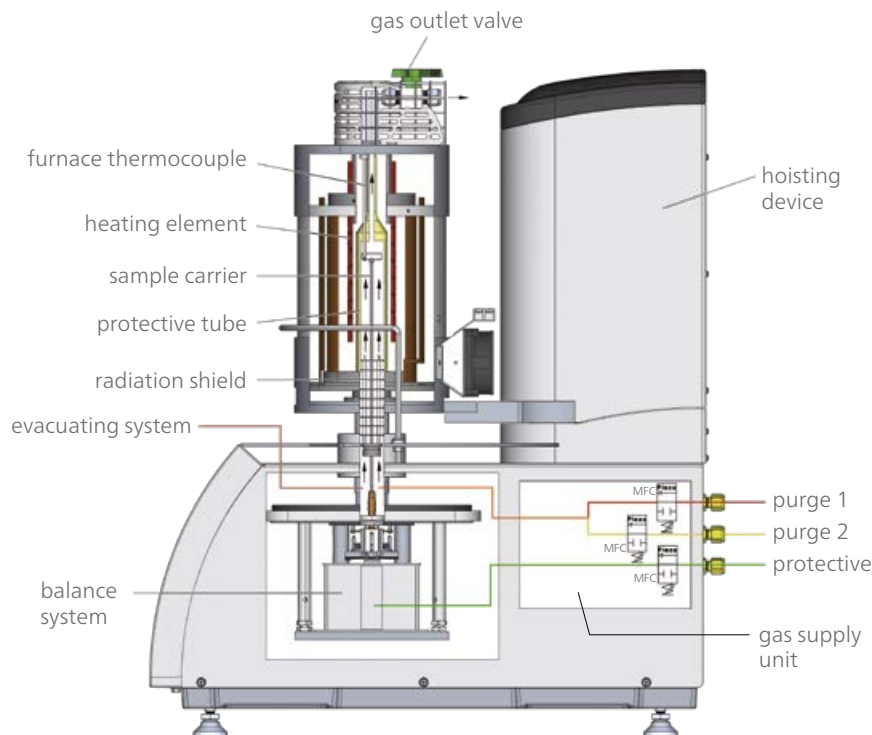
### High and Highest Temperature Range

True DSC measurements up to 1750°C can be monitored by using the type B sensor. At the highest temperatures up to 2000°C, the type W sensor for TGA and TGA-DTA can be used under inert, reducing, and vacuum conditions.

*HIGHEST PRECISION*

Maximum Flexibility





## Optimal Atmosphere Control

The built-in gas supply unit with three mass flow controllers (MFCs) for purge and protective gases offers optimum control of the atmosphere around the sample (e.g., pure inert conditions). This is crucial for accurate interpretation of the measured effects; e.g., to differentiate between oxidation and decomposition reactions.

## Coupling to Evolved Gas Analysis



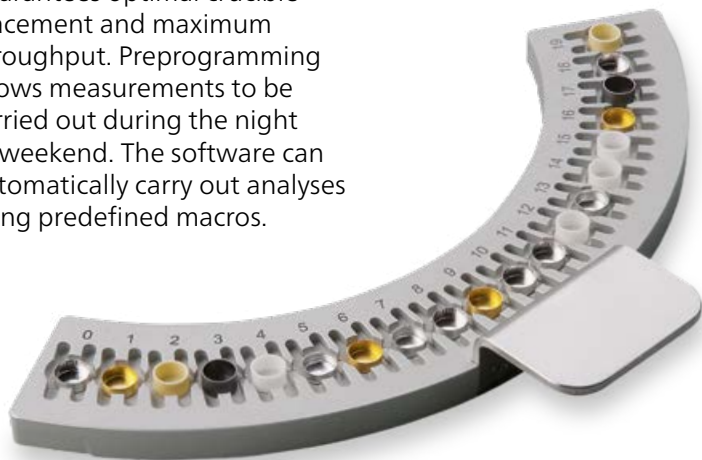
For evolved gas analysis, the system can be coupled to QMS and FT-IR individually or to a combination of QMS and FT-IR – even if equipped with an automatic sample changer – and GC-MS or a combination of FT-IR and GC-MS.

STA 449 **F1** Jupiter® with automatic sample changer coupled to QMS 403 Aëolos® Quadro



## Automatic Sample Changer

An automatic sample changer for up to 20 samples is optionally available. The sample changer guarantees optimal crucible placement and maximum throughput. Preprogramming allows measurements to be carried out during the night or weekend. The software can automatically carry out analyses using predefined macros.



## 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 "corrosive gas version" of the STA 449 **F1 Jupiter**® can be supplied. This version is optimized for measurements under corrosive atmospheres, such as reducing.

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.



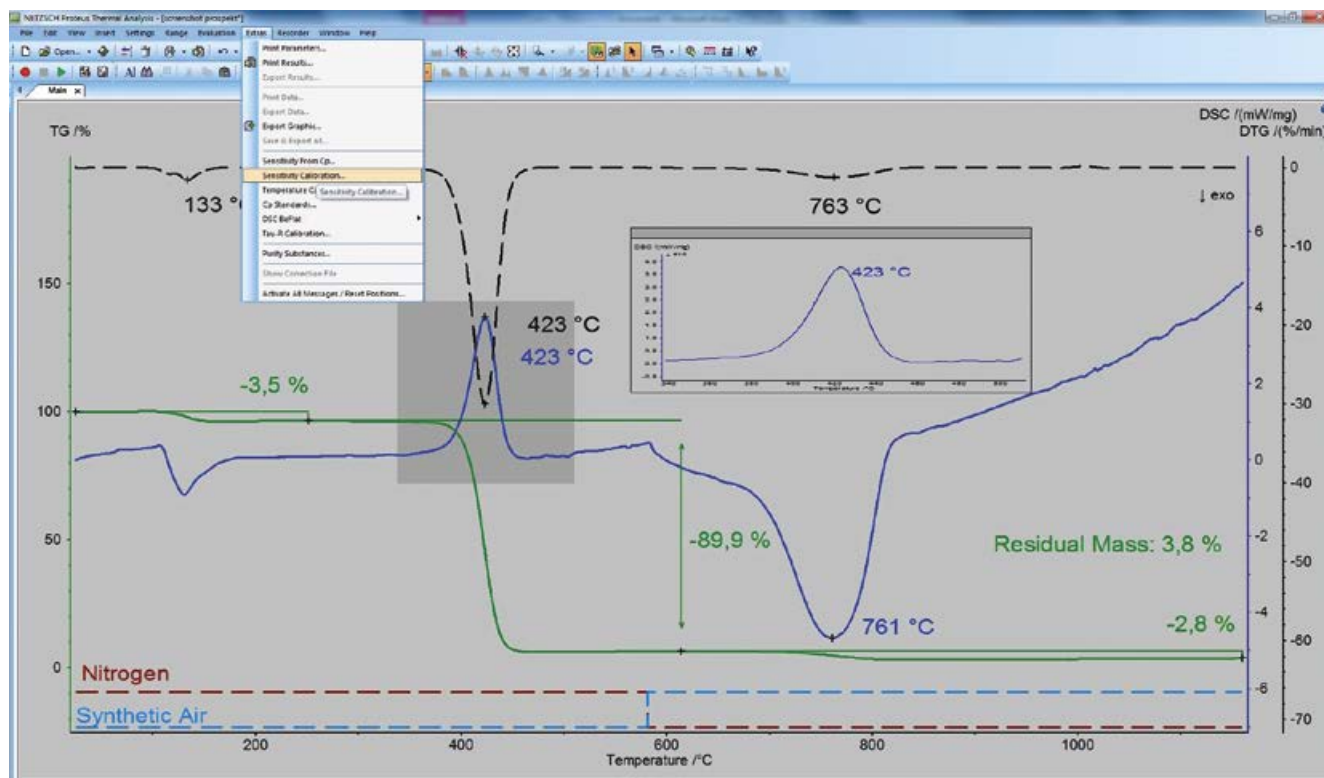
*Robust and  
Easy-to-Operate*



The STA 449 **F1 Jupiter**® runs under the versatile *Proteus*® software and 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. This software is produced by an ISO-certified company.

# Proteus® Software

for the STA 449 **F1 Jupiter**®



User interface during evaluation: TGA curve (green line) together with the DTG curve (black dotted line), DSC curve (blue line), two curves representing the gas flow of nitrogen (red dotted line) and synthetic air (blue dotted line); PIP presentation (picture-in-picture) for the DSC peak at 423°C.

## Advanced Software (options)

- *Peak Separation* allows for accurate separation and evaluation of overlapping transitions
- *NETZSCH Thermokinetics* offers 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
- Specific heat capacity determination
- *Purity Determination* via analysis of the DSC melting peak
- *Tau-R®* Mode enables evaluation of exo/endothermal effects under consideration of time constants and thermal resistance values
- *Thermal Simulations* allows for a description of the decomposition reaction process in order to enable accurate predictions

## Software Features

Operating systems    Windows® operating system

General  
software features

- Multi-tasking: simultaneous measurement and evaluation
- Multi-moduling: operation of different instruments from one computer
- Combined analysis: comparison and/or evaluation of STA, DSC, TGA, DIL, TMA and DMA measurements in one plot
- Selectable scaling
- Graphic and data export
- Calculation of 1<sup>st</sup> and 2<sup>nd</sup> derivative including peak temperatures
- Storage and restoration of analyses
- Context-sensitive help system

DSC-specific features

- Determination of onset, peak, inflection and end temperatures, incl. automatic peak search
- Analysis of exothermal and endothermal peak areas (enthalpies) with selectable baseline and partial peak area analysis
- DSC-integral curve
- Comprehensive glass transition analysis
- Degree of crystallinity
- OIT (Oxidative-Induction Time)

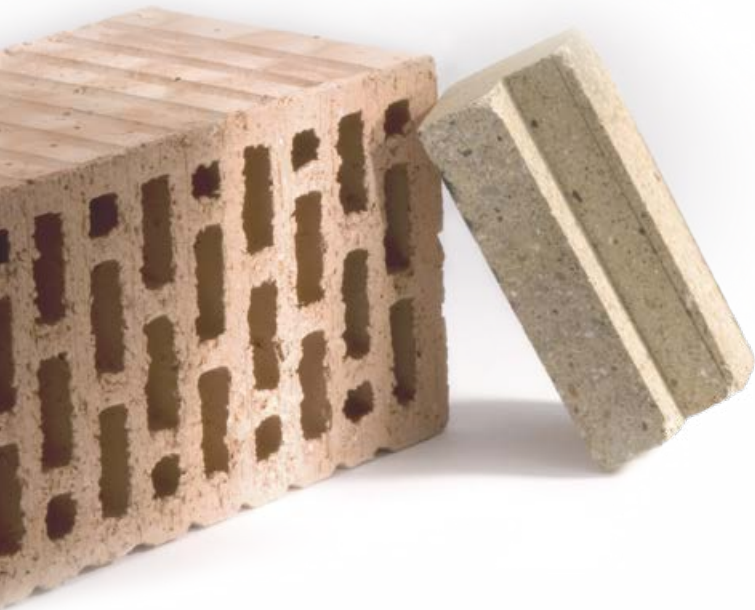
TGA-specific features

- Mass changes in % or mg
- Automatic evaluation of mass-change steps including determination of residual mass
- Extrapolated onset and endset
- c-DTA® for calculation of the DTA signal with evaluation of characteristic temperatures and peak area, optional for TGA measurements
- *Super-Res®* for rate-controlled mass change (optional)

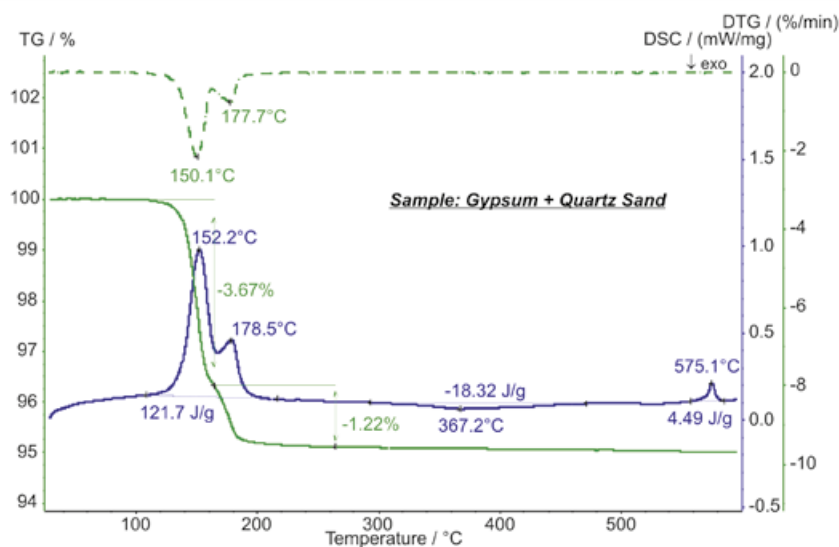
## APPLICATION EXAMPLES

### Meaningful Material Characterization

# LOW-TEMPERATURE



#### Building Materials: Gypsum and Quartz Sand

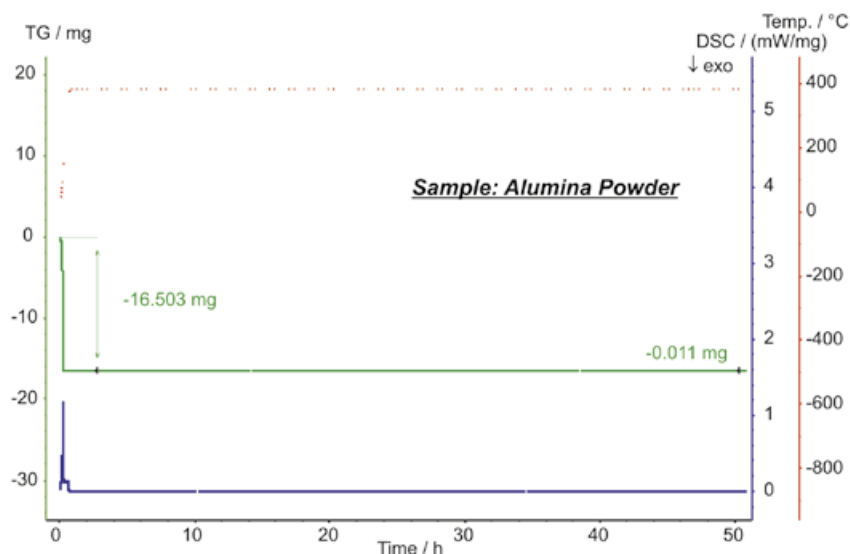


The measurement was carried out at a heating rate of 10 K/min under an air atmosphere in the SiC furnace. The sample mass amounted to 34.30 mg.

Gypsum and quartz sand are often used in building materials such as plaster and mortar. The gypsum part of the sample exhibits a two-step dehydration below 250°C from  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (dihydrate) into  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  (half-hydrate) and finally into  $\text{CaSO}_4$  (anhydrite). This requires a total energy of 122 J/g. Quantitative analysis by partial peak area determination 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 a peak temperature of 575°C is due to the structural  $\alpha \rightarrow \beta$  transition of quartz (crystalline  $\text{SiO}_2$ ).

## Excellent Long-Term Stability

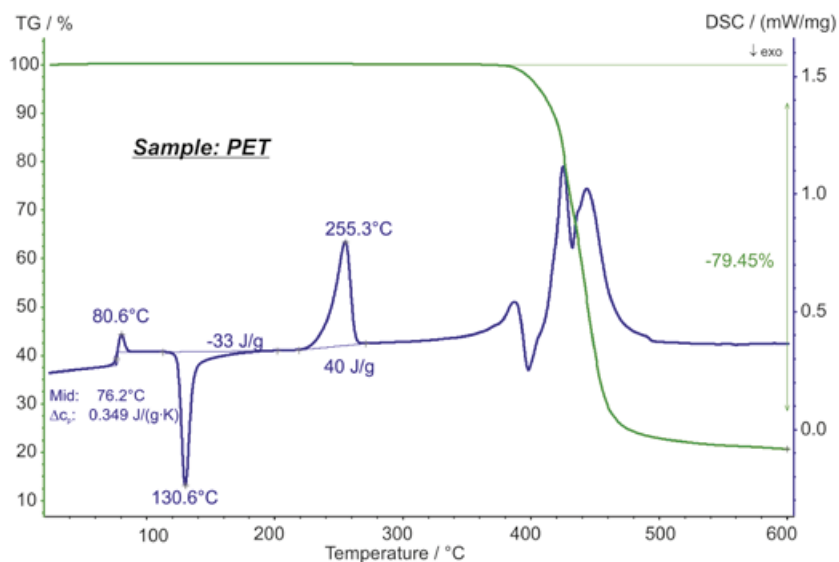
While heating an alumina powder sample (initial mass of 120.0 mg) to 400°C, a mass loss of 16.50 mg occurred, caused by the evaporation of humidity. This was accompanied by an endothermic DSC peak. During the 50-hour isothermal phase, the mass changed by only 11 µg, demonstrating the excellent long-term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



The long-term measurement on an alumina powder was performed at 400°C over 50 hours in the platinum furnace.

## Plastics: PET

Plastic bottles, textile fibers and films (for example, packaging for food) are well known applications of the polymer PET (polyethylene terephthalate). This 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 a total mass loss of 79.5%.



The SiC furnace was used for the investigation of the thermal behavior of a PET sample (10.13 mg) in a nitrogen atmosphere. The heating rate amounted to 10 K/min.

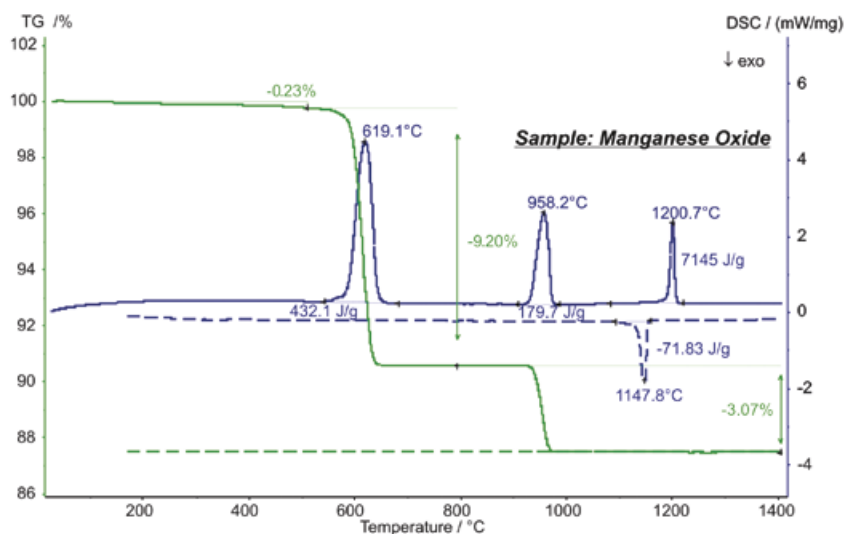


## APPLICATION EXAMPLES

# Meaningful Material Characterization HIGH-TEMPERATURE

### Reduction of Manganese Dioxide

Manganese dioxide ( $\text{MnO}_2$ ) 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  $\text{MnO}_2$  into  $\text{Mn}_2\text{O}_3$  and finally into  $\text{Mn}_3\text{O}_4$ . 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  $\text{Mn}_3\text{O}_4$  which was observed at the peak temperature of 1148°C upon cooling (dashed lines).

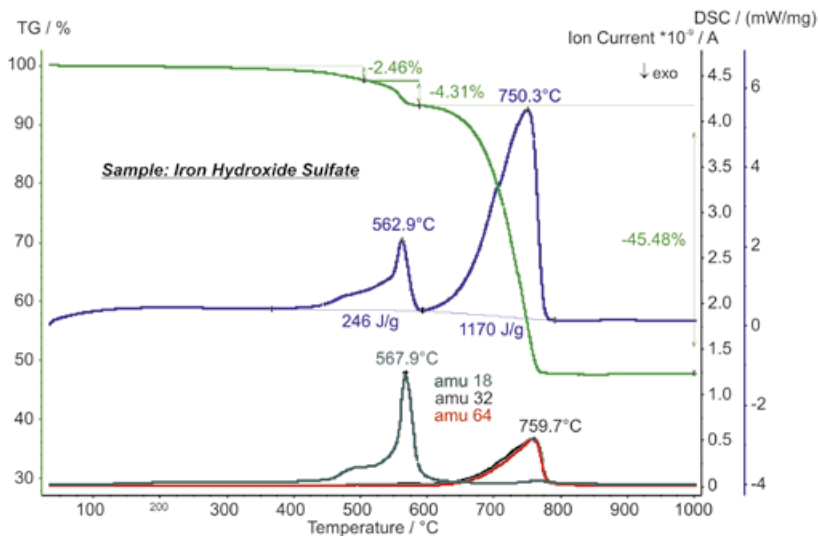


This STA measurement was carried out in the rhodium furnace under an air atmosphere. The oxide (32.14 mg) was heated at a rate of 20 K/min.





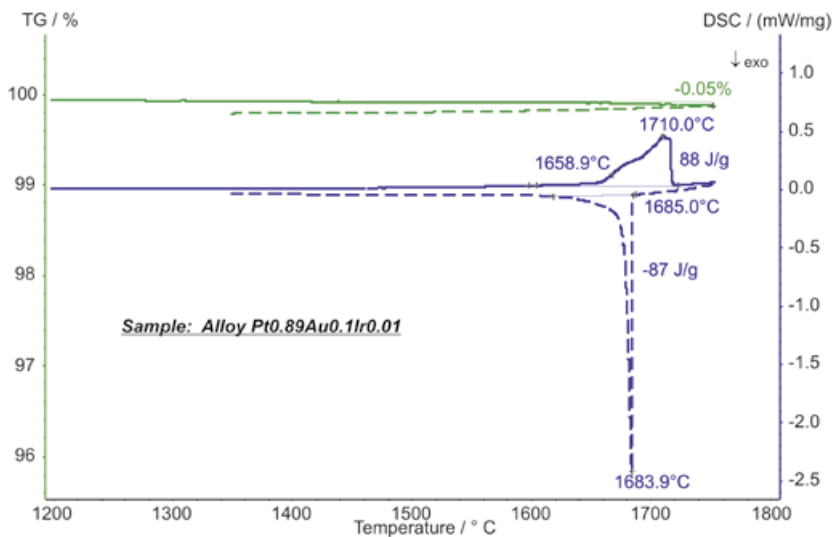
## Decomposition of Iron Hydroxide Sulfate



This sample (30.58 mg) was tested in the SiC furnace under a nitrogen atmosphere; the heating rate amounted to 20 K/min.

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. Substances known as ferrofluids contain superparamagnetic iron oxide nanoparticles which can serve as a contrast agent for MRI (magnet resonance imaging). Below 600°C, the STA-MS measurement exhibits a two-step release of  $\text{H}_2\text{O}$  with mass number 18, and between 600°C and 800°C, a release of  $\text{SO}_2$  and  $\text{O}_2$  with mass numbers 64 and 32, respectively. The final product is  $\text{Fe}_2\text{O}_3$  (hematite).

## Phase Diagrams of Alloys

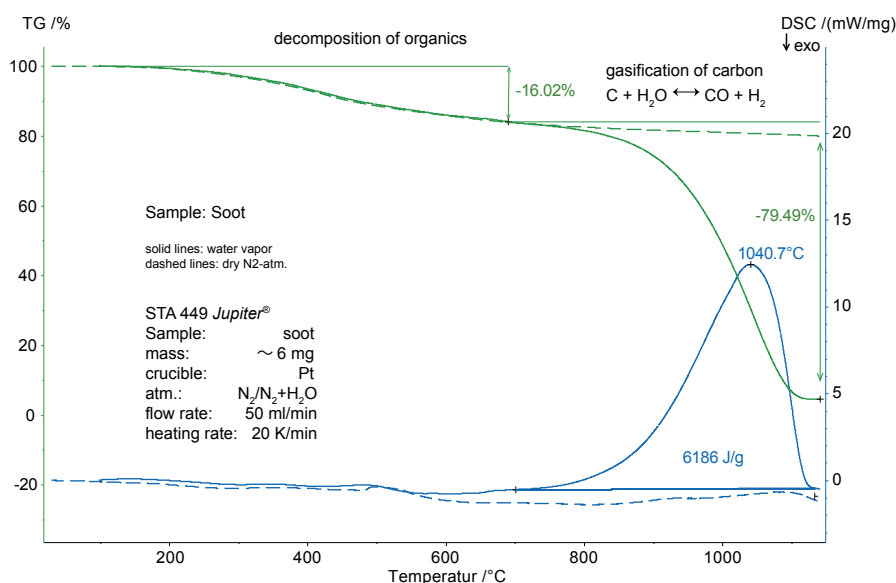


This dental alloy (202.13 mg) was measured in the graphite furnace in an argon atmosphere and at a heating rate of 10 K/min.

$\text{Pt}_{0.89}\text{Au}_{0.10}\text{Ir}_{0.01}$  is a possible dental alloy generally used for inlays, crowns and bridges. Dental alloys must be shapeable but robust, corrosion-resistant and biocompatible. This 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 the highest temperatures may be due to the start of evaporation.



## Gasification of Carbon under Water Vapor



For these tests, the water-vapor furnace was used for one measurement in a nitrogen atmosphere (dashed lines) and a second measurement in water vapor (100%; solid line). The sample masses amounted to approximately 6 mg; heating rates to 20 K/min.


The gasification process occurs as charcoal reacts with steam to produce carbon monoxide and hydrogen. This gas mixture is used for energy production or synthesis of basic chemicals (e.g., methanol).

Two soot samples were heated to 1150°C; one under a nitrogen atmosphere and the other under a humid atmosphere. Both measurements revealed a mass loss of 16% below ~700°C which is most probably due to the decomposition of organics. However, a mass loss of 79.5% was observed at higher temperatures when measured under water vapor. This mass loss is due to the gasification, which is the reaction of the carbon powder with water vapor into carbon monoxide and hydrogen; the process required 6.2 kJ/g of energy. This becomes evident via the endothermic DSC signal in the same temperature range.

## STA 449 **F1** Jupiter®

|                                 |  |
|---------------------------------|--|
| Design                          | Top-loading  |
| Temperature range               | -150°C to 2000°C   |
| Furnace                         | Variety of furnaces incl. high-speed, water-vapor, low to highest temperature, e.g., silver, platinum, etc.  |
| Motorized furnace hoist         | Double hoist for two furnaces or one furnace + automatic sample changer  |
| Heating rate                    | <ul style="list-style-type: none"> <li>0.001 to 50 K/min (furnace-dependent)</li> <li>High-speed furnace: up to 1000 K/min</li> </ul>                |
| Sensors                         | TGA, TGA-DTA, TGA-DSC, TGA-DSC (c <sub>p</sub> ), special sensors for hanging samples.<br>Sensors can be changed out in a matter of seconds.         |
| Vacuum-tight                    | 10 <sup>-4</sup> mbar  |
| Evacuation system               | <ul style="list-style-type: none"> <li>AutoVac for software-controlled automatic evacuation</li> <li>Pump systems for one or two furnaces</li> </ul> |
| Atmospheres                     | Inert, oxidizing, static, dynamic, vacuum  |
| Oxygen trap system (OTS®)       | Optional   |
| Automatic sample changer (ASC)  | 20 crucible positions (optional)   |
| Gas flow control                | 3 mass flow controllers  |
| Temperature resolution          | 0.001 K  |
| Balance resolution              | 0.025 µg   |
| Balance drift                   | < 2 µg/hour  |
| Maximum sample load             | 5000 mg (corresponds to weighing range)  |
| Sample volume (max.)            | <ul style="list-style-type: none"> <li>TGA: up to 5 ml</li> <li>DSC: 0.19 ml</li> <li>DTA: 0.9 ml</li> </ul>   |
| DSC enthalpy accuracy           | ± 2% (for most materials)  |
| Evolved gas analysis            | QMS, GC-MS and/or FT-IR couplings, <i>PulseTA</i> ® (options)  |
| Optional instrument specialties | <ul style="list-style-type: none"> <li>Glove box version</li> <li>Corrosion-resistant version</li> </ul>   |

# Technical Specifications



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 3,400 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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