

NETZSCH

Proven Excellence.

ECO-
LINE



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

Analyzing & Testing



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

The NETZSCH STA Eco-Line

70% LESS ENERGY AND COST.
NO EXTERNAL TEMPERATURE CONTROL NEEDED.



To obtain exact Thermogravimetric results with low drift behavior, most manufacturers have to resort to thermostatic control using a water cycle. Having to run the thermostat continuously requires a lot of energy and produces waste heat, which subsequently needs to be regulated by air conditioning.

NETZSCH has succeeded in removing the external thermostat. The weighing chamber's temperature is now regulated electronically, while maintaining excellent temperature stability. By removing the thermostat, the energy consumption of an STA 449 **F1 Jupiter**® will decrease by 70% for an average user.*

* When using the instrument 3 times a day on 250 days a year

Further advantages of the STA Eco-line are:

- 30% less waste heat
- Saves space
- Less maintenance
- Best performance



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 10⁻⁴ 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



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 ¹	Cooling system
Silver	-120°C to 675°C	liquid nitrogen ²
Copper	-150°C to 500°C	liquid nitrogen ²
Steel	-150°C to 1000°C	liquid nitrogen ²
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

¹ Corresponds to maximum sample temperature range

² 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.

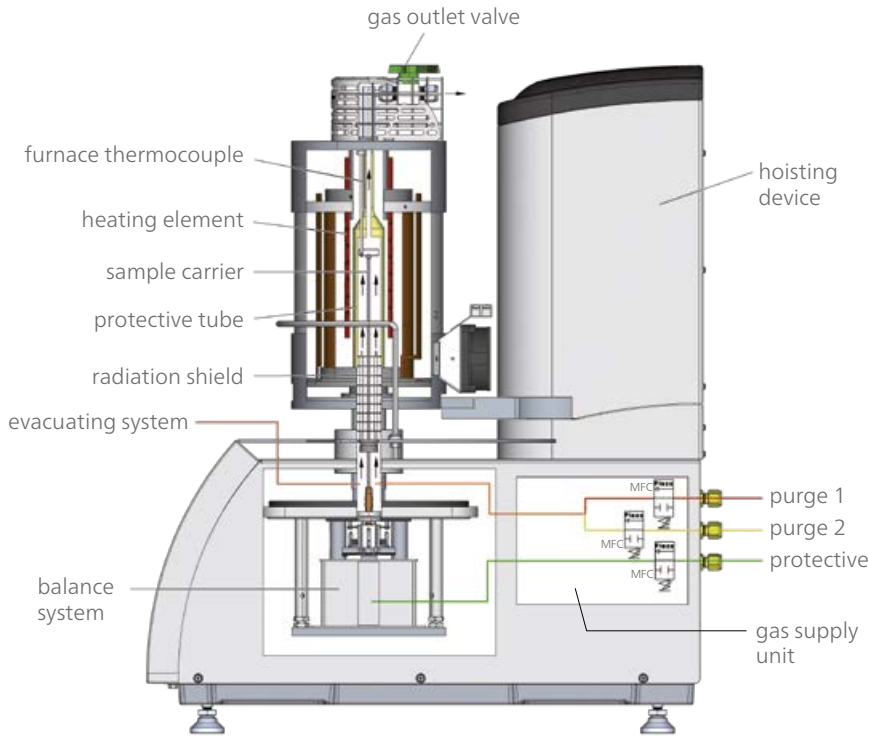
High and Highest Temperature Range

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



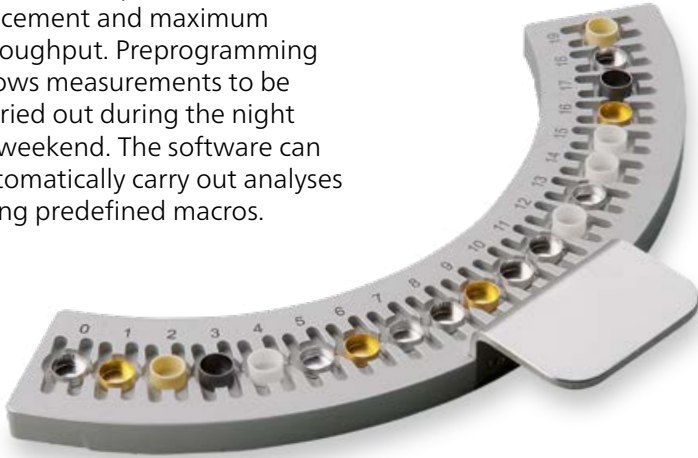
For evolved gas analysis, the system can be coupled to QMS and FT-IR individually or to a combination of QMS and FT-IR – even if equipped with an automatic sample changer – and GC-MS or a combination of FT-IR and GC-MS.

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



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*

Proteus[®] software is produced by an ISO-certified company and includes everything you need to evaluate the resulting data, and even perform complicated analyses.

STA 449 *F1 Jupiter*[®] with *Proteus*[®] 8.0

OUR POWERFUL ANALYTICAL SOFTWARE

AutoEvaluation – The World's Only Truly Self-Acting Evaluation

The unique *AutoEvaluation* function detects and evaluates thermal effects – i.e., peaks, glass transitions or mass changes – without user intervention. Intelligent algorithms are capable of handling DSC and TGA curves fully automatically. This generates completely objective test results.

Not only is this tool helpful for beginners, but experienced users can also use the results as a “second opinion”. The operator has full control at all times. Values can be recalculated or further manual evaluations added.

Identify – One Click Results

Identify marks a real turning point in the field of thermal analysis. This software package allows for the identification and classification of materials via database comparison with just one click.

In the case of DSC and TGA, the curve comparison is effect-based, which ensures fast and efficient processing. The result is a similarity hit list.

Besides one-on-one comparisons with individual curves or literature data, it is also possible to check whether a particular curve belongs to a certain class.

The database (more than 2000 entries, 1200 of which are already included in *Identify*) is open for adding users' own libraries and classes; it can be easily expanded with experiments and knowledge of their own.



SIMULATIVE OPTIMIZATION OF DEBINDING

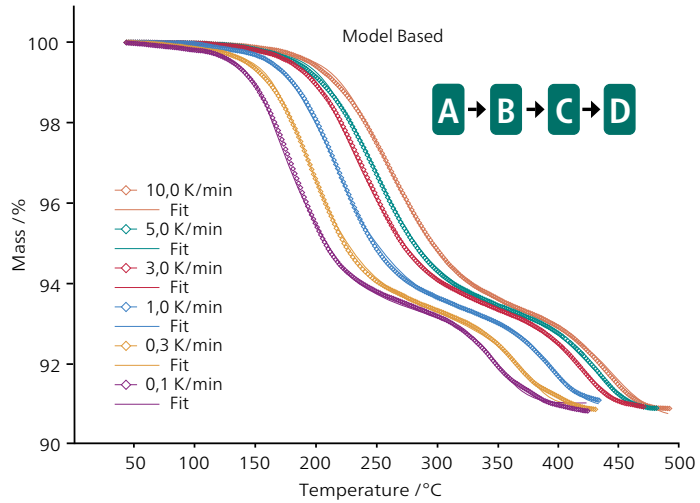


Fig. 1: Experimental TGA data (symbols) are in good agreement with the results of the simulation (solid lines) according to a 3-step kinetic model for heating rates at 0.1, 0.3, 1, 5 and 10 K/min.

Optimization of the Burn-Out Process for a Polymer Binder

In sinter metallurgy, a polymeric binder is added to a metal powder to improve adhesion. During the sintering process, the binder is carefully removed to prevent micro cracks caused by the release of gases. Slow heating results in time loss during production, while fast heating results in quality loss due to intensive gas development during polymer decomposition.

With these conditions in mind, the objective here is to find the optimum temperature program for a tunnel kiln. The production process is simulated by six TGA measurements (Fig. 1) conducted at different heating rates and a kinetic model based on them. In this example, a mass loss rate of 0.05%/min yields optimal material quality. Under laboratory conditions, the temperature program in Fig. 2 achieves this. Fig. 3 shows the optimum temperature curve for the different zones in the tunnel kiln.

We also offer "Kinetics as a Service". For more information, please refer to www.kinetics.netzsch.com

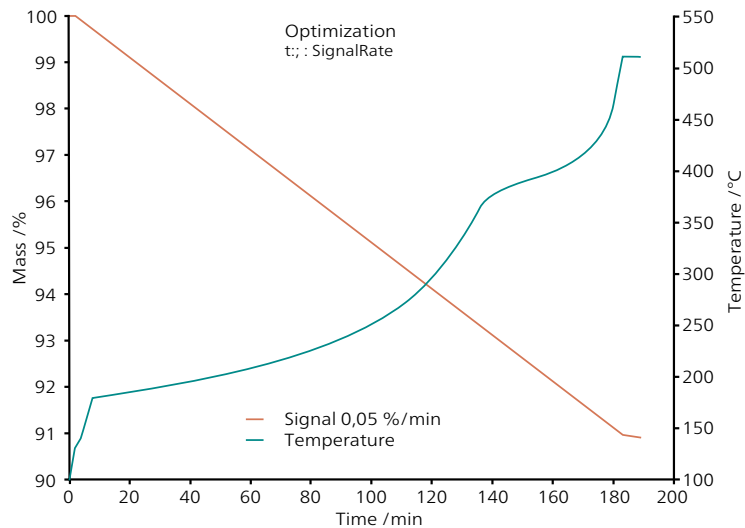


Fig. 2: Optimized temperature program for the burn-out of the polymer binder under laboratory conditions

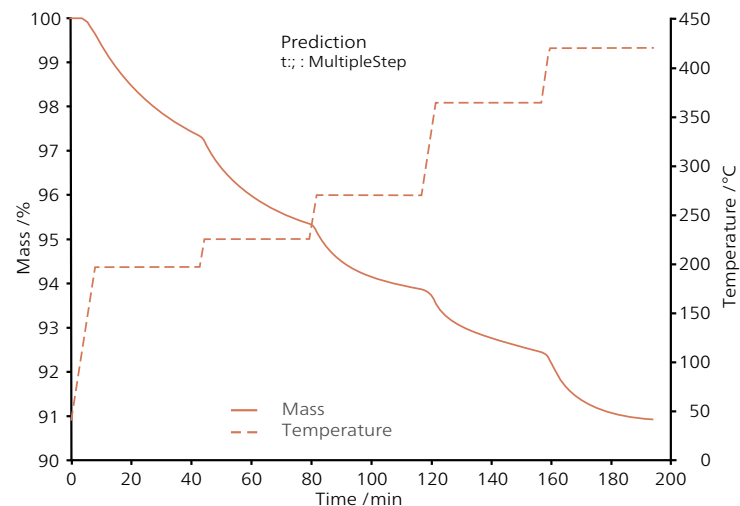


Fig. 3: Optimized zone temperatures for the burn-out of the polymer binder in the tunnel kiln during the production process

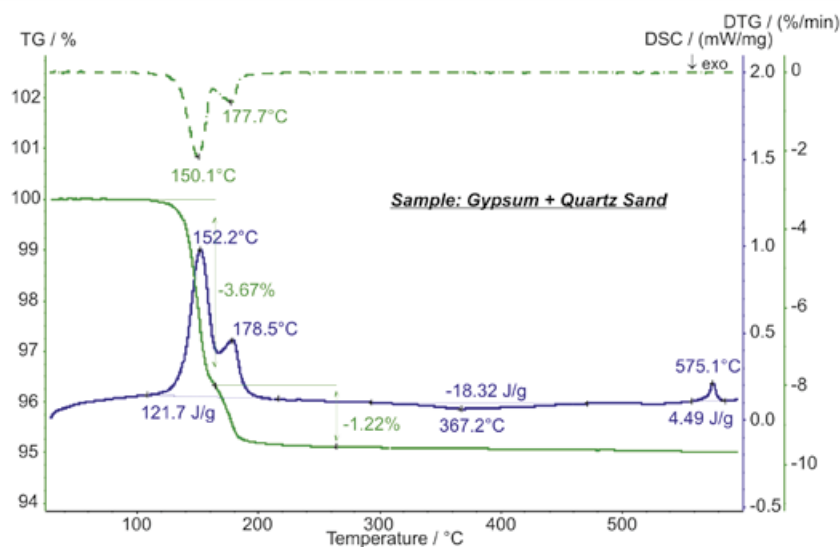
APPLICATION EXAMPLES

Meaningful Material Characterization

LOW-TEMPERATURE



Building Materials: Gypsum and Quartz Sand

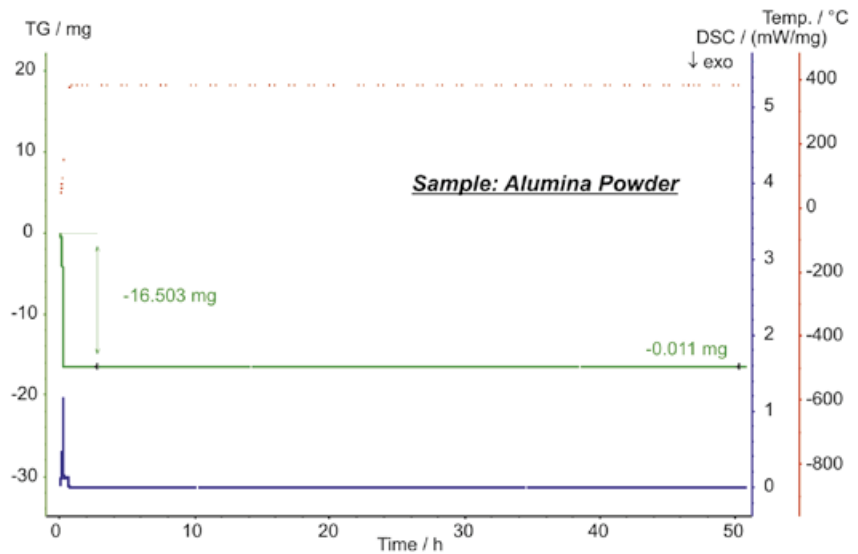


The measurement was carried out at a heating rate of 10 K/min under an air atmosphere in the SiC furnace. The sample mass amounted to 34.30 mg.

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 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 SiO_2).

Excellent Long-Term Stability

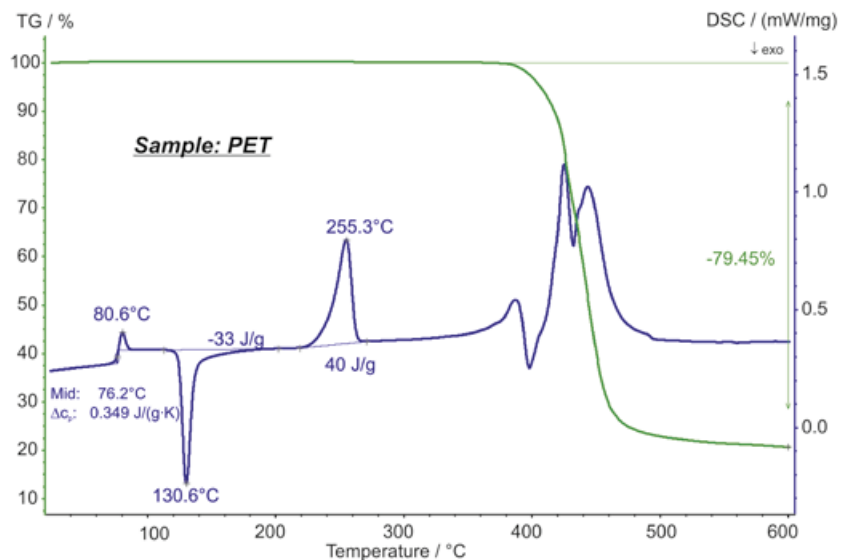
While heating an alumina powder sample (initial mass of 120.0 mg) to 400°C, a mass loss of 16.50 mg occurred, caused by the evaporation of humidity. This was accompanied by an endothermic DSC peak. During the 50-hour isothermal phase, the mass changed by only 11 µg, demonstrating the excellent long-term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

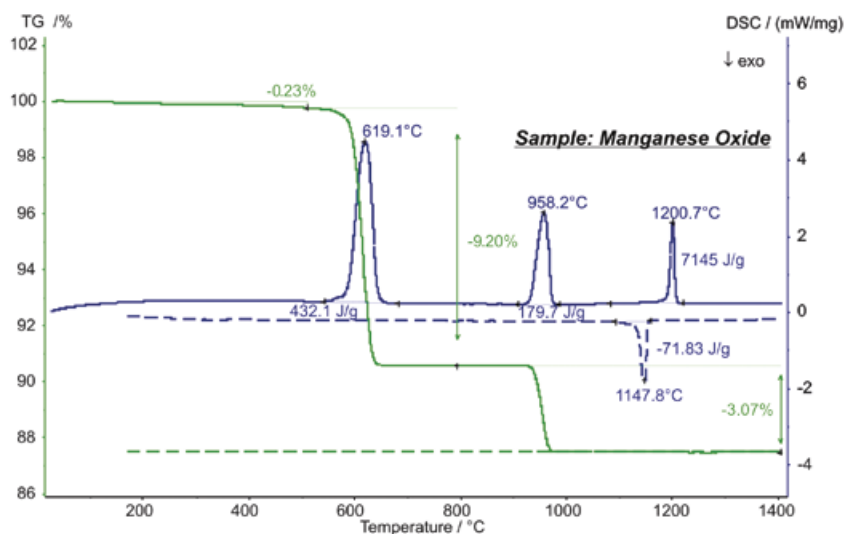
APPLICATION EXAMPLES

Meaningful Material Characterization

HIGH-TEMPERATURE

Reduction of Manganese Dioxide

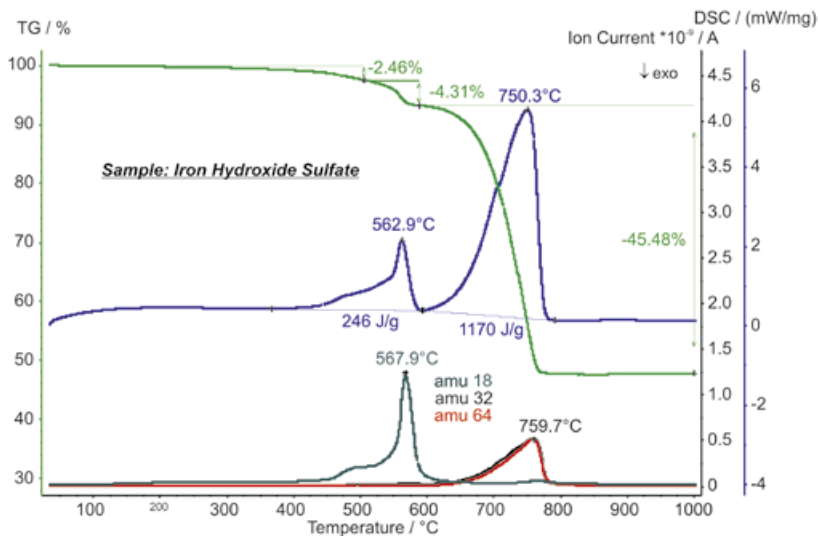
Manganese dioxide (MnO_2) is often used in chemistry as an oxidizer but is also used, for example, as a cathode material in batteries. This STA measurement shows mass loss steps at approx. 600°C and 950°C which are due to the reduction of MnO_2 into Mn_2O_3 and finally into Mn_3O_4 . The values of 9.20% 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).



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.



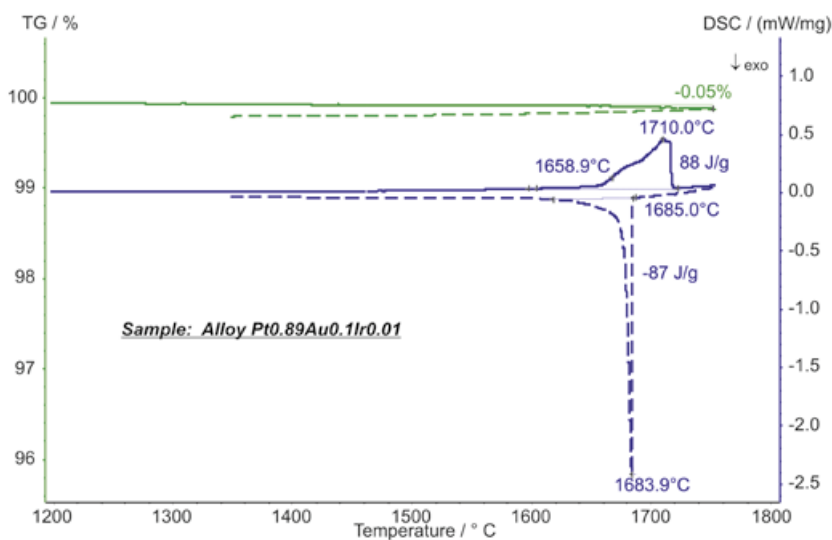
Decomposition of Iron Hydroxide Sulfate



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 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

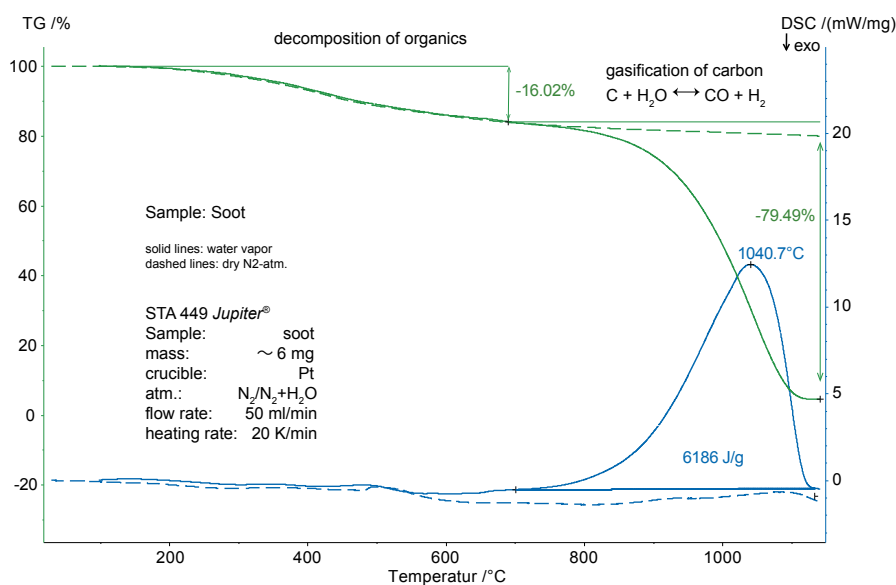


This dental alloy (202.13 mg) was measured in the graphite furnace in an argon atmosphere and at a heating rate of 10 K/min.

$\text{Pt}_{0.89}\text{Au}_{0.10}\text{Ir}_{0.01}$ 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"> ▪ 0.001 to 50 K/min (furnace-dependent) ▪ High-speed furnace: up to 1000 K/min
Sensors	TGA, TGA-DTA, TGA-DSC, TGA-DSC (c _p), special sensors for hanging samples. Sensors can be changed out in a matter of seconds.
Vacuum-tight	10 ⁻⁴ mbar ¹
Evacuation system	<ul style="list-style-type: none"> ▪ <i>AutoVac</i> for software-controlled automatic evacuation ▪ Pump systems for one or two furnaces
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"> ▪ TGA: up to 5 ml ▪ DSC: 0.19 ml ▪ DTA: 0.9 ml
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"> ▪ Glove box version ▪ Corrosion-resistant version

¹ Actual achievable vacuum depends on the selected evacuation system

Technical Specifications



The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 3,700 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

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

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

Proven Excellence.■

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

NETZSCH®

www.netzsch.com