NETZSCH

Simultaneous Thermal Analyzer – STA 449 ***F5*** *Jupiter®*

Method, Technique and Applications

Leading Thermal Analysis

Easy Operation & Highest-Level Performance

EASY TO CHOOSE. EASY TO USE.

Simultaneous Thermal Analysis

Two Methods Going Well Together

Simultaneous Thermal Analysis (STA) generally refers to the simultaneous application of Thermogravimetry (TG, TGA) and Differential Thermal Analysis (DTA) or Differential Scanning Calorimetry (DSC) to the same sample in one instrument. In the STA, the test conditions are perfectly identical for the TGA and DTA/DSC signals (same atmosphere, gas flow rate, heating rate, thermal contact to the sample crucible and sensor, etc.). Sample throughput is also improved as more information is 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*
- *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

Top-loading – The Proven Design for Thermobalances

The STA 449 ***F5*** *Jupiter*® is a toploading system using a balance design that has been standard for years in laboratories. The reasons are simple: These systems combine ideal performance with easy handling.

Setting Benchmarks by Experience & Innovation

Easy Operation

The STA 449 ***F5*** *Jupiter*® is designed to guarantee easiest operation. Sample change can be performed safely via the motorized furnace hoist and top-loading principle of the balance system. The integrated software feature TGA-*BeFlat*® provides flat baselines and eliminates additional work for buoyancy correction. Not only the experienced users will appreciate this!

Unique Combination – True TGA-DSC and High-Volume TGA

Between ambient and 1600°C sample temperature, combined TGA and true DSC measurements can be performed with high precision and reproducibility.
TGA measurements are also possible even on large samples – crucibles up to 5 cm³ in volume are available.

Atmosphere – Perfectly Controlled by MFC and *AutoVac*

The three built-in mass flow controllers (MFC) for purge and protective gases provide an optimum control of the atmosphere around the sample.
The *AutoVac* feature allows for automatic evacuation and backfilling of the STA system. This function is designed to simplify the evacuation procedure especially when dealing with powders and other "critical-to-evacuate" samples. For totally softwarec-ontrolled *AutoVac*, a vane-type rotary pump system is included.

STA 449 ***F5*** *Jupiter®*

Trend-Setting Technology

Best Cost-Performance-Ratio

The system's built-in balance with high weighing and load range (both up to 35 g), high resolution of 0.1 µg and low drift behavior in the µg-range all combine with its sensitive DSC capabilities to allow it to handle any typical application task over a broad temperature range.

Fully Equipped

This vacuum-tight STA system comes with all hardware and software features which high-temperature applications demand in the fields of ceramics, metals, inorganics, building materials, etc.
No need to configure the instrument for your application. It comes exactly as you need it!

Accessories and Options Tailored to

Two True Measurement Techniques, Easy-to-Add Accessories and Ready for Gas Analysis

Two Methods – More Effective Together

The system is equipped with the TGA-DSC sensor. True DSC or simultaneous TGA-DSC measurements can also be performed simultaneously using the automatic sample changer (ASC).

In addition, a TGA sample carrier and a TGA-DTA sensor are available. These can be quite handy when critical or unknown samples need to be tested.

Variety of Crucibles

Crucibles made from different materials and in various dimensions are available. Standard crucibles are made of alumina or platinum. Many crucibles, e.g., gold, zirconia, etc., can be offered with solid or pierced lids. You can choose the right type for your application.

Selection of DSC crucibles

Large beaker crucibles for high-volume TGA tests

Your Application

Automatic Sample Changer Provides Peace of Mind

An automatic sample changer (ASC) for up to 20 samples is optionally available. It can be used for TGA or TGA-DSC measurements. The ASC guarantees optimal crucible placement and maximum throughput.Preprogramming allows measurements to be carried out during the night or weekend. By use of predefined methods, handling is even further facilitated.

Efficient and reliable – you don’t want to miss out on it.

All Set! – Ready for Coupling to Evolved Gas Analysis

For evolved gas analysis (EGA), the STA 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 ***F5*** *Jupiter*® with automatic sample changer coupled to FT-IR and QMS *Aëolos*®

Universal Software Includes

The STA 449 ***F5*** *Jupiter*® runs under the versatile *Proteus*® software on Windows® operating systems. There is no need for troublesome, customized software configurations. The interface is structured to ensure intuitive understanding of the menus; automated routines and a context sensitive help system are always available to permit effortless work flow and avoid timeconsuming delays. The *Proteus*® software is licensed with each instrument and can also be installed on other computer systems.

Complete

The universal *Proteus*® software includes everything you need to carry out a reliable measurement and evaluate the resulting data of TGA and DSC curves – or even perform complicated analyses.

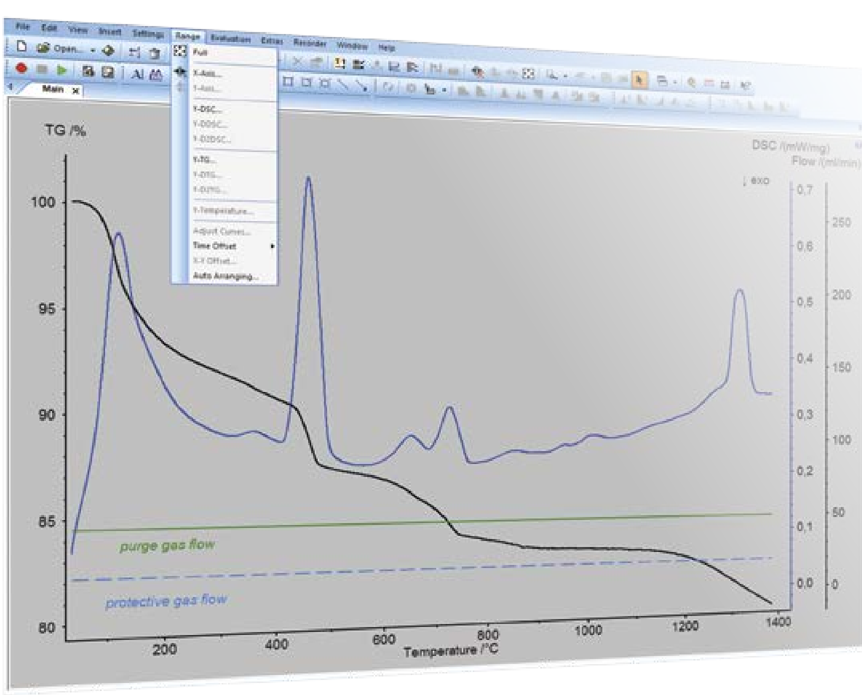
Intelligent

Proteus® is a multi-tasking system and offers simultaneous measurement and evaluation. In addition to the option to run multiple instruments on one computer, *Proteus*® also makes it possible to run combined analyses like STA, DSC, TGA, DIL, TMA and DMA together and has the capability to display all these measurements and evaluations in one plot. *Proteus*® is produced by an ISO-certified company.

Extended Insight

The Advanced software package makes your *Proteus*® even more powerful.

- *Peak Separation* for separation of peaks which are in close proximity. Improved quantitative determination of superimposed mass-change steps.
- *Thermokinetics* for accurate process prediction and optimization such as lifetime and composition behavior via multivariate nonlinear regression.



Everything You Need

BeFlat® – Our Intelligent Software Function Flat-Out Saves You Time!

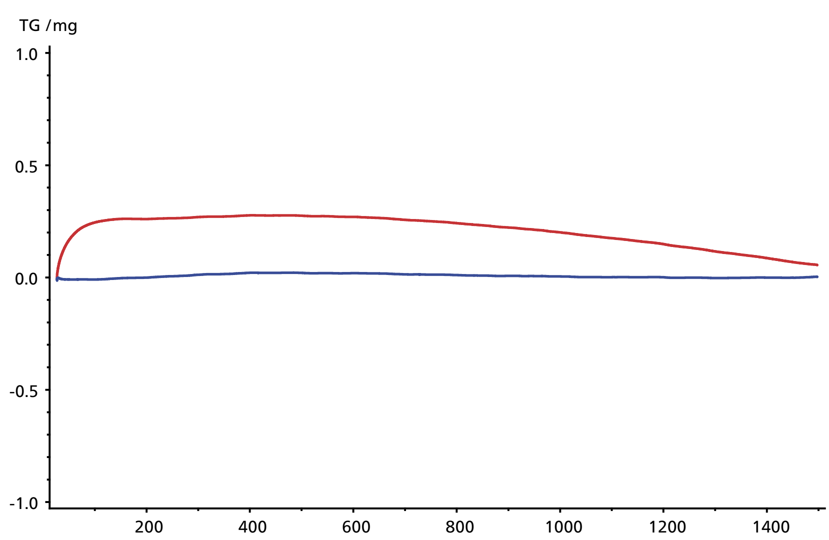
Thermogravimetric measurements require a correction of the buoyancy effect. It depends on variations in the measurement conditions – such as the crucible material and shape, type and rate of purge gas, and heating rate, which can affect the measurement results to varying degrees.

Typically, these influences are corrected by carrying out correction measurements under exactly the same measurement conditions for each respective measurement series.

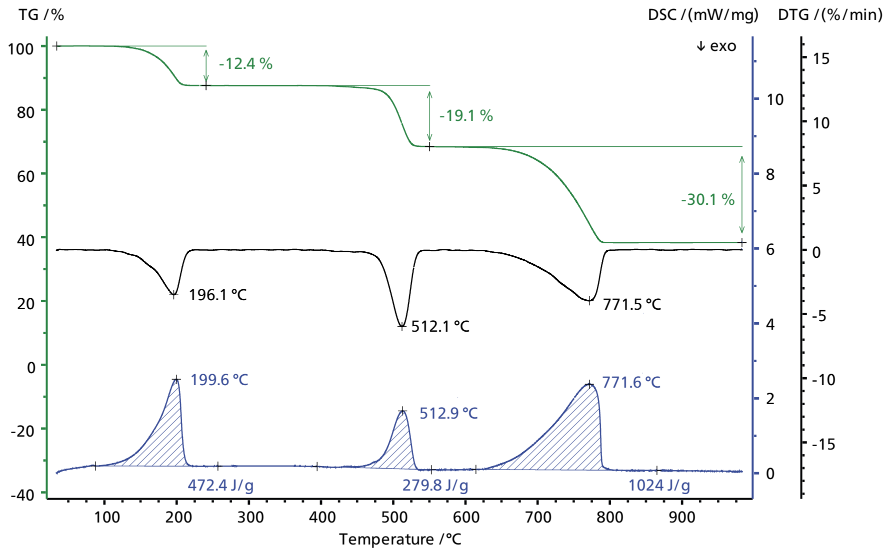
The novel software feature TGA-*BeFlat®* for STA systems keeps a record of the temperature dependence for the measuring influences, the heating rate, the different purge gases (such as argon, air and nitrogen) and the gas flow rates, and can therefore provide the appropriate correction for the selected measurement conditions without having to carry out a blank value determination in the form of correction measurements.

The advantage to using the TGA-*BeFlat®* correction is a huge savings in time afforded by eliminating additional correction measurements.

TGA-*BeFlat®* can be activated or deactivated at any time; the data set for the actual sample measurement (raw data) is always stored.



Influence of *BeFlat®*: The blue curve represents the TGA measurement with BeFlat® correction and the red curve without additional correction. Measurements were carried out on two empty crucibles under identical conditions.



STA measurement of calcium oxalate monohydrate (sample mass 12.79 mg) in Pt crucibles and at a heating rate of 10 K/min in nitrogen atmosphere (70 ml/min).

ACCURACY

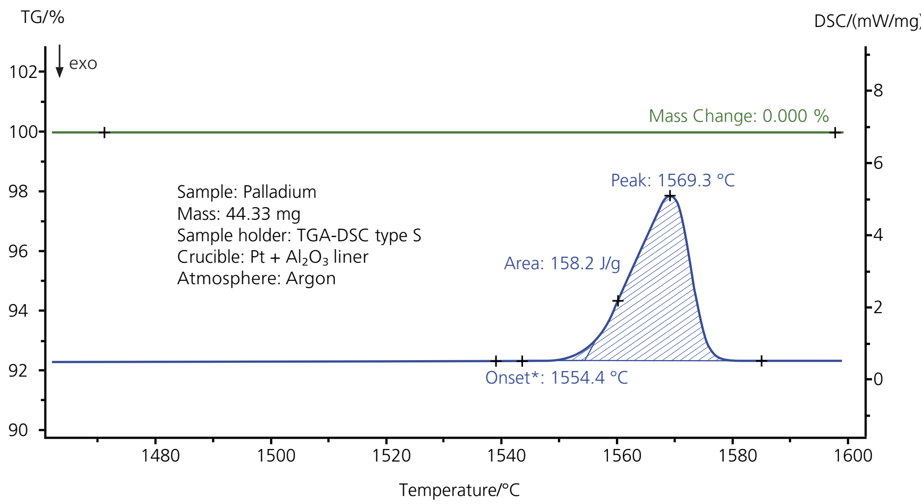
Accuracy of the TGA Signal

In thermal analysis, calcium oxalate monohydrate ($CaC_2O_4 \cdot H_2O$) is used to determine the accuracy of the TGA signal. The substance has a high stability and adsorbs little moisture from the laboratory environment. This makes it an ideal reference material for thermobalances. This plot shows the TGA and DSC curves of $CaC_2O_4 \cdot H_2O$ between room temperature and 1000°C. The 1st mass-loss step shows the release of water transforming the sample to calcium oxalate (CaC_2O_4). The 2nd mass-loss step is due to the release of CO which represents the transition from calcium oxalate to calcium carbonate ($CaCO_3$). Above 700°C, the carbonate decomposes by releasing CO_2; the residual mass consists of CaO. The detected mass losses correspond very well with literature data (<1%). This proves the high accuracy of this thermobalance.

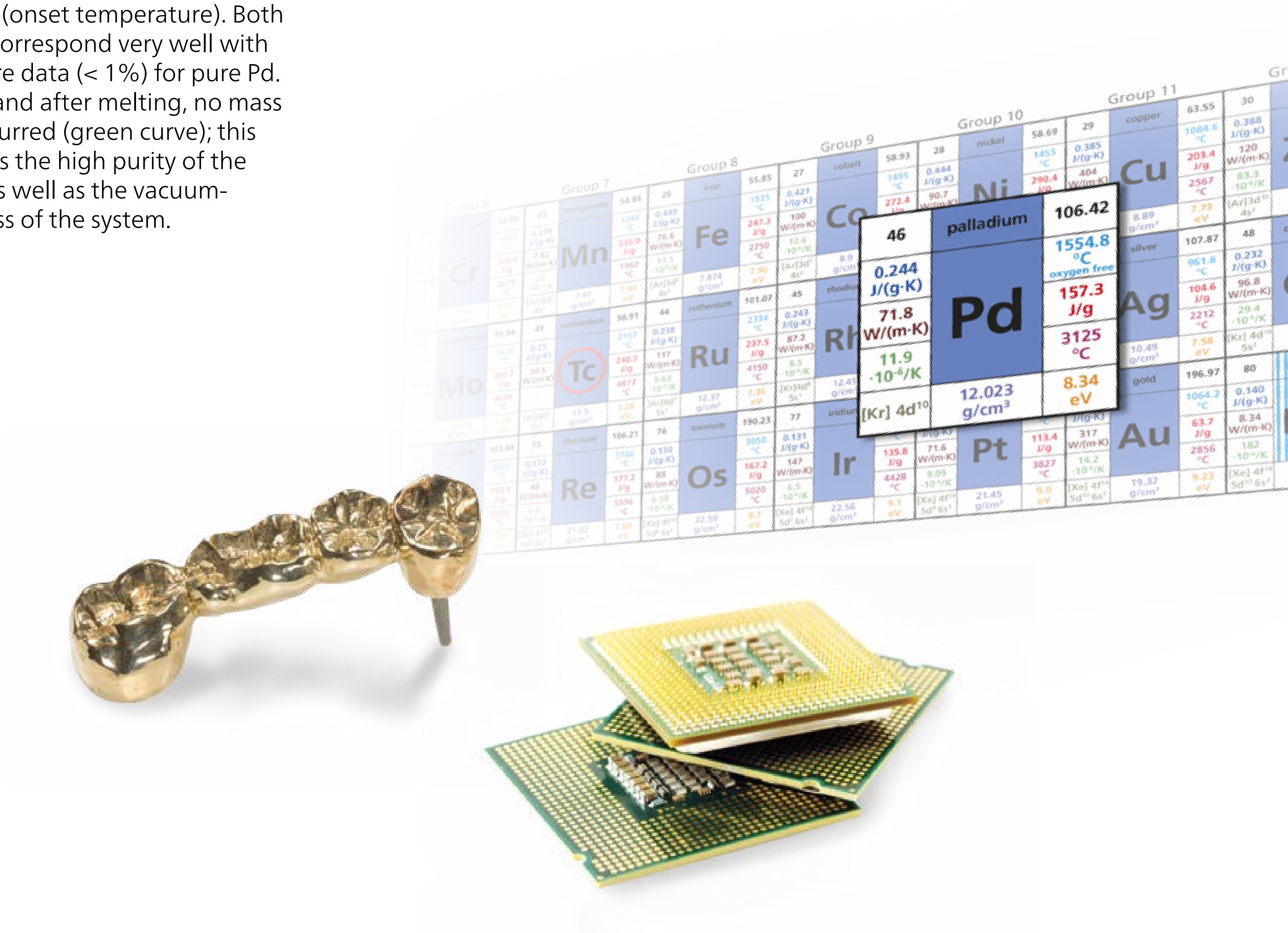
APPLICATIONS

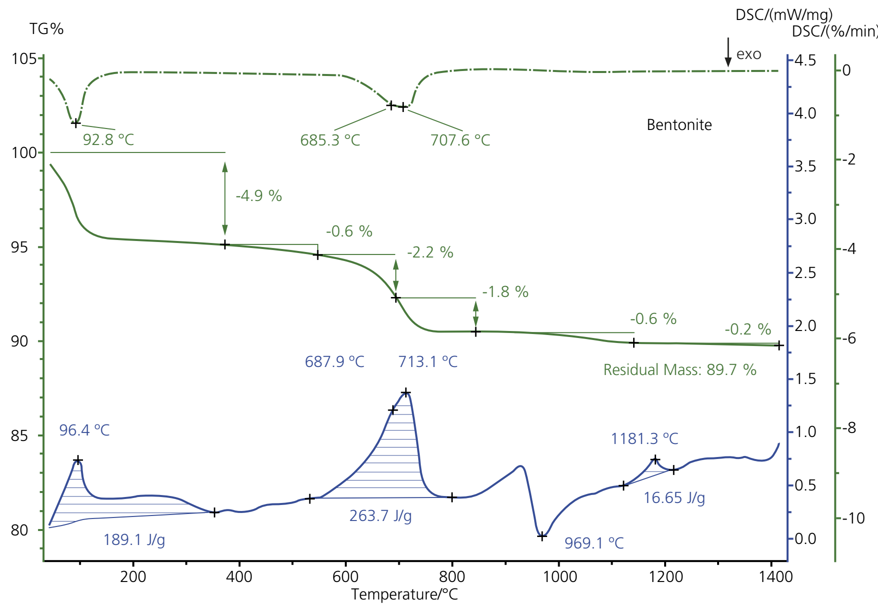
Melting Point of Palladium

The largest use of palladium (Pd) today is in catalytic converters. However, it is also used in, e.g., dentistry, aircraft spark plugs and surgical instruments and electrical contacts. Palladium shows no reaction with oxygen at normal temperature although when heated to 800°C in air will produce a layer of palladium(II) oxide (PdO). This plot exhibits the STA measurement on Pd up to a sample temperature of 1600°C. The DSC curve (blue) shows the melting with an enthalpy of 158 J/g (blue curve, DSC) at 1554°C (onset temperature). Both values correspond very well with literature data (< 1%) for pure Pd. Before and after melting, no mass loss occurred (green curve); this confirms the high purity of the metal as well as the vacuum-tightness of the system.



STA measurement on a Pd sample at a heating rate of 20 K/min

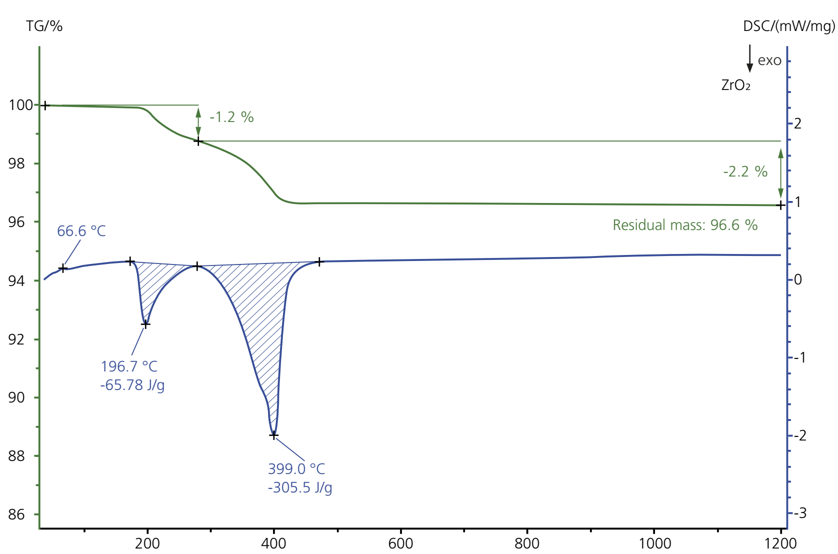




Complex thermal behavior of bentonite in Pt crucibles at a heating rate of 10 K/min in nitrogen atmosphere (70 ml/min)

Ceramic Mass

Bentonite is clay consisting mainly of montmorillonite and stands out due to its absorbent capabilities. This plot exhibits the TGA (green), DTGA (green dotted) and DSC (blue) curves. The 1st mass-loss step (DSC peak temperature 96°C) is due to a release of water followed by a small mass-loss step of 0.6%. This is most likely due to the release of SO_2 indicating a pyrite contamination. Above 600°C, water is released from the bentonite structure (DTGA at 685°C and 708°C). The exothermic DSC peak at 969°C represents the phase transition of this mineral. The endothermic peak at 1181°C is most likely due to a partial melting or a further SO_2 release.



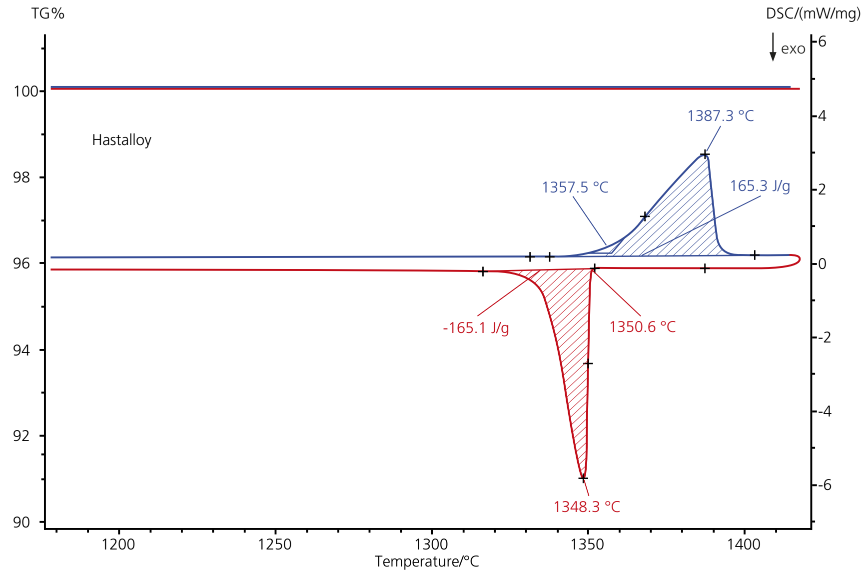
STA measurement on stabilized ZrO_2 (26.2 mg) in platinum crucibles

Binder-Burnout of Zirconia

Upon heating, zirconia undergoes disruptive phase changes. By adding small percentages of yttria, these phase changes are eliminated, and the resulting material has superior thermal, mechanical, and electrical properties. This measurement between room temperature and 1200°C exhibits two small losses up to 450°C (3.4% in total; green curve) which correspond very-well with the two exothermic peaks at 197°C and 399°C in the blue DSC curve. These effects (mass loss up to 500°C, exothermic peaks with high enthalpies) are due to the binder-burnout of this ceramic material. The small endothermic DSC peak at around 67°C is caused by the melting of the binder.

Corrosion-Resistant Metal Alloy

Hastelloy® is a nickel-chromium-molybdenum-tungsten alloy with outstanding high-temperature stability as evidenced by high ductility and corrosion resistance. It has excellent resistance to stress-corrosion cracking and to oxidizing atmospheres up to 1038°C. It is used in combustion gas desulfurization plants, chemical industry and incineration plants, etc.
The blue DSC curve depicts the melting of a hastelloy sample (alloy 22) at 1358°C (extrapolated onset) with an enthalpy of 165 J/g. During cooling, crystallization occurred at 1351°C (extrapolated endset) with nearly the same enthalpy change (red DSC curve). No mass loss or increase due to oxidation was observed.



Heating and cooling of Hastelloy® (39.02 mg) at the heating and cooling rate of 20 K/min in 70 ml/min Ar atmosphere; platinum crucibles with alumina liners were used.

STA 449 ***F5*** *Jupiter*®	
Design	Top-loading
Temperature range	RT… 1600°C (sample temperature)
Furnace	SiC furnace on motorized hoist for safe, simplified operation
Heating rate	0.001 to 50 K/min
Sensors	▪ TGA-DSC (standard in system version I) ▪ TGA-DSC_{ASC} (standard for system version II with automatic sample changer) ▪ TGA (optional for up to large sample sizes) ▪ TGA-DTA (optional) All sensors are easily interchangeable within seconds
Vacuum-tight	10^{-2} mbar
AutoVac	Integrated for software-controlled automatic evacuation
Evacuation system	Yes
Atmospheres	Inert, oxidizing, static, dynamic, vacuum
Automatic sample changer (ASC)	20 crucible positions (standard for system version II)
Gas flow control	3 mass flow controllers integrated for 1 protective and 2 purge gases
Temperature resolution	0.001 K
Balance resolution	0.1 µg (over the entire weighing range)
BeFlat®	Integrated for flat baselines → considers buoyancy correction due to influences by crucible, atmosphere, heating rate, etc.
Balance drift	< 5 µg/hour
Maximum sample load	35000 mg (incl. crucible), corresponds to TGA measuring range
Sample volume	Up to 5 cm^3 (for TGA crucibles)
DSC enthalpy accuracy	± 2% (for most materials)
Evolved gas analysis	QMS, GC-MS and/or FT-IR couplings (options)
Dimensions	600 x 700 x 650 (900) mm
Weight	83 kg (excl. computer)

Key Technical Data

Software Features

STA 449 ***F5*** *Jupiter*®	
Operating systems	Windows 7 and 8.1 32/64 bit, Professional, Windows 7 32/64 bit, Enterprise®, Windows 7 32/64 bit, Ultimate®
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^{st} and 2^{nd} derivative including peak temperatures ▪ Storage and restoration of analyses ▪ Context-sensitive help system ▪ Software produced by iso-certified company
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 ▪ 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 ▪ Automatic baseline correction TGA-*BeFlat*® for automatic correction of measuring influences ▪ 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)

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

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

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

Leading Thermal Analysis

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