Simultaneous Thermal Analysis





STA 449 F3

Leading Thermal Analysis.

STA 449 F3 Jupiter® – Introduction to the Method

For over 45 years, NETZSCH Analyzing & Testing has been a leading manufacturer of highperformance thermal analysis systems - flexible, sophisticated and technically outstanding. Our customers' wishes and requirements are our guidelines. This, combined with experience and innovation, allows us to consistently set new benchmarks and standards in the field of thermal analysis instrumentation. Our success is a result of the creativity and enthusiasm of our engineers and scientists and our close cooperation with you as our customer.



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



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 new STA 449 F3 Jupiter®. It meets nearly all 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.

Simultaneous Thermal Analysis

Simultaneous Thermal Analyzer STA 449 F3 Jupiter®

The NETZSCH STA 449 F3 Jupiter® is a robust, flexible, easy-to-operate instrument for determining caloric effects (transformation temperatures and enthalpies) and mass changes at the same time. The top-loading Simultaneous Thermal Analyzer can easily be adjusted to nearly all possible applications by selecting the optimum furnace, installing the ideal sensor and using the proper accessories. It combines a high-performance Heat-Flux DSC with a microgramresolution thermobalance, thereby offering an unmatched sample load and measurement range.

The STA 449 **F3** Jupiter[®] comprises a robust system for high-quality TG

and DSC measurements. The thermobalance can measure samples up to 35 g (the measurement range is 35 grams, as well). The resolution of this low-drift balance system is 1 µg. The system can operate in a temperature range between -150°C and 2000°C using various interchangeable sensors and furnaces. Different possible pump systems as well as the optional mass flow control device allow measurements under well-defined atmospheres. A double furnace hoist and automatic sample changer (ASC) are also optionally available and improve sample throughput to increase the efficiency of operation for this highperformance thermal analyzer.

The various TG-DSC sensors offer true DSC performance over an unmatched temperature range (-150 ... 1750°C). TG and TG-DTA sensors can be used for the highest temperatures of up to 2000°C.

The robust system setup, userfriendly software and flexible design along with the wide range of different options make the system an ideal tool for quality control and research for material characterization in your laboratory.

For evolved gas analysis, the system can be coupled to a QMS or FTIR system – or to both at the same time – even if equipped with an automatic sample changer.



STA 449 F3 Jupiter® – Groundbreaking Technology

Top-loading - the standard for balance systems

The STA 449 **F3** Jupiter® is a toploading system using a balance design that has been standard for a long time for other types of scales – in laboratories and even in the kitchen at home or in a supermarket, most balances have been top-loading for decades. The reasons are simple. These systems combine ideal performance with easy handling. Why should your thermobalance be any different?

Stability, low drift and high sample loads

The balance system of the STA 449 **F3** Jupiter® offers high sample loads (up to 35 grams) and measurement range (35 grams) as well as high resolution (1 μ g) and low drift (in the microgram range over hours). Another outstanding feature of the balance section of the STA is its high accuracy.

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Vacuum tight design - defined atmosphere conditions

The STA 449 **F3** Jupiter[®] is vacuum tight by design. Several pump systems can be connected to the STA which allow evacuation down to 10⁻² mbar and back-filling with well-defined atmospheres. The OTS[™] accessory can be used to reduce the oxygen partial pressure at the sample.

Various gas flow controllers

The gas flow is generally controlled by frits which are installed in the 3 gas flow channels (2 purge gases, 1 protective gas). Optionally available is a metal housed mass flow control system (MFC) for purge and protective gases offering optimum control of the atmosphere around the sample. Well defined gas flow conditions are crucial for an accurate interpretation of the measured effects, e.g. to differentiate between oxidation and decomposition reactions.

Various furnace systems

The STA 449 **F3** Jupiter® can be equipped with a wide range of different furnaces accommodating different temperature and application ranges. A double furnace hoist allows the simultaneous installation of two different furnaces for improved sample throughput or lowand 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.

Furnaces:

Furnace type

Steel furnace Platinum furnace Silicon carbide furnace Rhodium furnace Graphite furnace Water vapor furnace -150 ... 1000°C RT ... 1500°C RT ... 1550°C RT ... 1650°C RT ... 2000°C RT ... 1300°C

Temperature range

Cooling system

liquid nitrogen forced air forced air forced air tap water forced air

Various sensors

The STA 449 F3 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 results. 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 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.

BeFlat® and DSC correction

Optionally available software features such as *BeFlat*[®] and DSC correction allow a fully automatic baseline correction as well as correction for system time constants. All routines are fully software based. This can be optimized for your specific measurement conditions. Furthermore, the raw data signal can be accessed at any time. 먭 I S

Sensors:

Sensor thermocouple

ΤG

Type E Type K Type S Type B Type W Type S protected -150 ... 700°C -150 ... 800°C RT ... 1650°C 150 ... 1750°C RT ... 2000°C RT ... 1600°C

Temperature range

Sensor types

DSC-c_n

TG, TG-DTA, TG-DSC (c_p) TG, TG-DTA, TG-DSC (c_p) TG, TG-DTA, TG-DSC (c_p) TG, TG-DTA, TG-DSC TG, TG-DTA TG, TG-DTA

Atmospheres

DTA

inert, oxid., vac. inert, oxid., red., vac. inert, oxid., red., vac. inert, oxid., red., vac. inert, red., vac. inert, oxid., red., vac., corr.

STA 449 F3 Jupiter® – Proteus® Software

The STA 449 **F3** Jupiter[®] runs under a 32-bit Windows[®] software package which includes everything you need to carry out a measurement and evaluate the resulting data. Through the combination of easy-to-understand menus and automated routines, a tool has been created that is extremely user friendly and, at the same time, allows sophisticated analysis.

General Software Features:

- Windows[®] software: for Windows[®] XP and Vista[®] (Enterprise, Business) operating systems
- Multi-tasking: simultaneous measurement and evaluation
- Multi-moduling: operation of different instruments with one computer
- Combined analysis: comparison and/or evaluation of STA, DSC, TGA, DIL, TMA and DMA measurements in one plot
- Labeling: input and free placement of text elements
- Calculation of 1st and 2nd derivative
- Selectable scaling
- Graphic and data export
- Selectable colors and line types
- Storage and restoration of analyses
- Macro recorder (optional)
- Context-sensitive help system
- Temperature calibration
- Compatible with advanced software packages
 (Boak Separation Thermokin
- (Peak Separation, *Thermokinetics*)
- Software produced by ISO-certified company





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 (optional)
- BeFlat[®] for automatic baseline correction (optional)
- DSC correction (optional): evaluation of exo- and endothermal effects under consideration of system time constants and thermal resistance values

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)



Advanced Software (options)

- Peak Separation Software: allows accurate separation and evaluation of overlapping transitions
- NETZSCH *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
- Purity determination

STA 449 F3 Jupiter® – Applications

Characterization of porcelain raw material

This STA-MS measurement on porcelain raw material shows three mass-loss steps. Below approx. 250°C, the evaporation of humidity occurred. At temperatures between 250°C and 450°C, the burn-up of organic binder was observed, during which 156 J/g of energy was released. The dehydration of kaolin occurred above 450°C and required 262 J/g. The mass spectrometer signals for mass numbers 18 and 44 reflect the corresponding release of H₂O and CO₂. The exothermic DSC peak at 1006°C with an enthalpy of -56 J/g is due to a solid-solid transition.







Building material: glass wool

Glass wool is often used for the insulation of houses and heating pipes. The measurement shows three mass-loss steps below approx. 600°C, which are due to the evaporation of humidity and the burn-up of organic binder. The latter can be seen from the strongly exothermic DSC signal in this temperature range. The step in the DSC signal at 728°C is due to the glass transition (increase in the specific heat of 0.41 J/[$g\cdot$ K]). The exothermic DSC peak at 950°C with an enthalpy of -287 J/g is due to crystallization; the endothermic effects between approx. 1050°C and 1250°C with an entire enthalpy of 549 J/g are due to melting. The slight mass changes above 700°C are most probably due to oxidation and evaporation of impurities.

Burn-up of linoleum

The building material linoleum was invented in 1863 and is most often used as floor covering. It is very robust and has an insulating effect even at small thicknesses. This STA measurement in air reflects the natural contents of linoleum: after the evaporation of humidity below 150°C, the stepwise, strongly exothermic burn-up of linseed oil, natural resins, cork flour, wood flour and the substrate jute followed between approx. 200°C and 500°C. The entire heat released during the oxidation was 14.5 kJ/g. Between 600°C and 750°C, the endothermic decomposition of the filler CaCO₃ (chalk) Is observed. Above 750°C, the mass remains constant.







Characterization of explosives

The highly explosive material hexogen (also called RDX, T4, etc.) starts to sublime already at about 150°C, as can be seen from the TG curve. The endothermic DSC peak at an onset temperature of 206°C with an enthalpy of 123 J/g is due to melting of the sample. Already between 200°C and 250°C, the strongly exothermic decomposition occurred, releasing 1.38 kJ/g of energy. This experiment was carried out in a synthetic air atmosphere at a heating rate of 5 K/min using an initial sample mass of only 2.32 mg.

STA 449 F3 Jupiter® – Applications

Phase transitions of γ-TiAl

The refractory alloy γ-TiAl distinguishes itself through high temperature and corrosion resistance with a low specific weight. It is used, for instance, in turbo chargers, gas turbines, and motors as well as in aircraft and space applications. The DSC signal shows an endothermic effect (1323°C peak temperature) beginning at an extrapolated onset temperature of 1195°C; this is due to the structural $\alpha_2 \rightarrow \alpha$ transformation. At 1476°C (DSC peak temperature), the $\alpha \rightarrow \beta$ transformation occurred. The endothermic DSC peak at 1528°C is due to melting of the sample (onset at approx. 1490°C, liquidus temperature at about 1560°C). No significant mass changes were detected during the experiment.







Analysis of composite materials

Carbon fiber-reinforced polymers (CFRP) are very popular composite materials which consist of a polymer matrix and embedded carbon fibers. CFRPs are very light, and yet they exhibit high stability and rigidity at the same time. They are thus very suitable for automotive, aircraft and space applications. This STA measurement shows an endothermic DSC peak with an enthalpy of 25 J/g at 329°C which is due to melting of the polymer matrix. Between approx. 480°C and 620°C, the pyrolytic decomposition of the polymer occurred. At 650°C, the gas atmosphere was switched from N_2 to O_2 , resulting in the strongly exothermic decomposition of the carbon fiber content (24.7%). The residual mass of 0.0% at the end of the experiment indicates that no further inorganic fillers or glass fibers were in the sample.



STA 449 **F3** Jupiter[®] – Servicing Our Customers' Special Needs

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 **F3** *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 *F3 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.



Global Customer Support & Service Network

State-of-the-art technology combined with optimal customer support are NETZSCH trademarks. Our training department provides a complete range of programs tailored to the needs of our customers in research, education and industry. A wide range of different seminars, users' meetings and individual training programs are available to assist you in achieving optimum performance and benefit from your thermal analysis system.



NETZSCH is the fastest growing company in the field of thermal analysis and thermophysical properties testing in the world. This can be attributed not only to our superior technology and guality, but also to our unmatched pre- and after-sales service network. NETZSCH-certified staff at 45 service centers across the world provide fast and reliable customer support including qualified installation, calibration services, and maintenance contracts. In addition, our applications laboratories offer contract testing and support to address the most specific of thermal analyses.



The new STA 449 **F3** Jupiter[®] is the ideal tool for day-to-day work in your laboratory. The system is generally employed for:

- Product development
- Quality assurance
- Failure analysis

The STA 449 **F3** Jupiter® is part of the NETZSCH high-temperature series of instruments. Together with the DIL 402 PC/C (dilatometer), the TMA 202/402 (thermomechanical analyzer), the DMA 242 C (dynamic mechanical analyzer), the DSC 404 *Pegasus*® (differential scanning calorimeter), the DEA 230/231 series (dielectric analyzers) for cure monitoring, and other DSC and TGA systems, a full-scale thermal analysis of your materials and parts can be carried out. The key features of the STA 449 **F3** Jupiter[®] are:

- Maximum flexibility
- Top quality
- Easy to use
- Wide range of accessories
- Low cost of ownership

NETZSCH offers a full range of lowand high-temperature thermal analysis instruments for temperatures between -260 and 2800°C, including all conventional thermal analysis systems.

For thermophysical properties testing (measurement of the thermal diffusivity/conductivity), NETZSCH offers a broad range of heat flow meters (HFM), guarded hot-plate systems (GHP), flash devices (LFA) and other thermal conductivity testers (TCT systems).

Technical specifications subject to change



Leading Thermal Analysis.



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