NETZSCH

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STA 409

-160 … 2000°C

Simultaneous Thermal Analysis

Thermal Analysis (DIN 51005) is a series of techniques that study the effect of temperature on material properties. Differential Scanning Calorimetry, Differential Thermal Analysis and Thermogravimetry have proved themselves particularly suitable for research and quality control applications in industrial and university environments.

Thermogravimetry (TG)

TG measures any change in weight of a sample as a function of temperature or time, caused by the interaction of the sample with its surrounding atmosphere.

Differential Scanning Calorimetry (DSC)
Differential Thermal Analysis (DTA)

The DTA/DSC technique measures enthalpy changes during physical transitions and chemical reactions. Either the temperature difference (DTA) between the sample and a reference material or the heat flux difference (DSC) is determined.

Simultaneous Thermal Analysis (STA)

TG and DTA/DSC are applied simultaneously to the same sample. The results of both techniques can be directly compared, as reaction equilibria are not affected by differing sample atmospheres which can occur with single measurements. In addition, the thermal effects measured in this way are easier to interpret and enthalpy changes can be corrected for weight changes. Moreover, Simultaneous Thermal Analysis is able to eliminate the uncertainties arising with separate TG and DTA/DSC measurements, caused by the inhomogeneity and geometry of samples and inaccuracy in the temperature.

STA is superior to single measurements.

A sample can be characterized almost completely if the TG and DTA/DSC results can be assigned to it exactly. This is especially true of complex reactions. Continuous recording of the changes in weight makes an exact determination of the enthalpy changes possible.

STA 409

STA 409

The NETZSCH STA 409 Simultaneous Thermal Analyzer combines Simultaneous Thermogravimetry and Differential Scanning Calorimetry (DTA/DSC) over the temperature range -160 … 2000°C. The measuring part can be evacuated and both inert and reactive sample atmospheres can be used.
The application possibilities comprise the whole spectrum of TG and DTA/DSC analyses.
Typical parameters for the characterization of materials which can be determined with the STA 409 are:
– decomposition temperatures
– temperature stability
– oxidation stability
– determination of the different components
– reaction steps and kinetics
– characterization
– identification
– compatibility
– transition temperatures and transition enthalpies
– polymorphism
– temperatures for glass transition
– phase diagrams

When designing the STA 409, emphasis was placed on ease of handling, achievement of reproducible results, and application possibilities allowing universal use. The wide temperature range in which tests can be conducted, as well as the ability to perform simultaneous TG-DTA/DSC measurements in different atmospheres, allows broad applications of this technique in research and quality control, e.g.

– polymeric materials
– glass and ceramics
– technical ceramics and powder metallurgy
– petrochemistry
– metals and alloys
– building materials, cement and silicates
– soil science, clay and minerals
– fossile and nuclear fuels
– explosives
– environmental protection

Options available:
– double hoisting device for two furnaces
– turbomolecular vacuum pump
– device for c_p measurements
– water vapour or corrosive gas atmospheres
– high temperature furnace
– evolved gas analysis by mass spectrometry or FTIR
TA tests which are orientated towards practice and the simulation of a production process are possible with these options.

Software

Standard Software Features:

- Windows Software: fully compatible with other MS® Windows™ programs
- multitasking: simultaneous measurement and evaluation
- multi-moduling: operation of up to 4 different instruments with one computer
- combined analysis: comparison and/or evaluation of DSC, TG and DIL 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 at any evaluation step of the analysis
- snapshot for evaluation of a measurement in progress
- import of mass spectrometer and FTIR data as ASCII files
- temperature calibration
- context-sensitive help system

TG Features:

- mass changes in wt% or mg
- extrapolated onset
- peak temperatures in the 1st and 2nd derivative
- automatic evaluation of mass-loss steps
- calculation of pairs of values for mass loss/temperature
- calculation of residual mass

Options:

- Macro Recorder
- Peak Separation Software
- Thermokinetics Software
- Purity Software
- Multicomponent Analysis
- Thermal Safety Software
- Combined STA-QMS and STA-FTIR Software
- simple computation of the specific heat, C_p
- Rate Controlled Mass Loss: 3 different modes: start/stop, stepwise isothermal, dynamic

DSC Features:

- characteristic temperatures: determination of onset, peak, inflection and end temperatures
- automatic peak search
- transformation enthalpies:
  - determination of peak areas (enthalpies) with up to 5 selectable baseline types and partial peak area analysis
  - peak area determination under consideration of the mass loss
- glass transitions: fully-automated, comprehensive glass transition analysis
- complex peak analysis: determination of all characteristic parameters of a peak with a single keystroke
- automatic baseline correction

Technical Attributes

An electromagnetically compensated balance system is used to measure changes in weight. The top loading arrangement of the sample holder and easy upward movement of the furnace give good access to the crucibles for quick and simple sample loading. The standard STA 409 is vacuum tight and has all connections for vacuum and inert or reactive gases. The gas piping is made of stainless steel for operation under corrosive gases.

The junction between the furnace and the weighing system is held at a constant temperature by a water-cooled collar so that the influence of heat conduction is eliminated.

Temperature ranges

The furnace used will depend on the requirements of a particular application. The unique design of the STA 409 gives it the widest temperature range of any thermal analyzer on the market.

-160°C… 500°C (Kanthal)
-120°C… 675°C (sheathed heater)
25°C… 850°C (sheathed heater)
25°C…1350°C (Kanthal)
25°C…1500°C (Pt/Rh)
25°C…1600°C (SiC)
25°C…1650°C (Rh)
25°C…1700°C (Super-Kanthal 33)
25°C…2000°C (Graphite)

The STA 409 can be equipped with two furnaces using a double hoist system; a full temperature range of -160°C …1700°C can then be achieved with one measuring part.

A maximum temperature of 2000°C can be reached by using a graphite furnace.

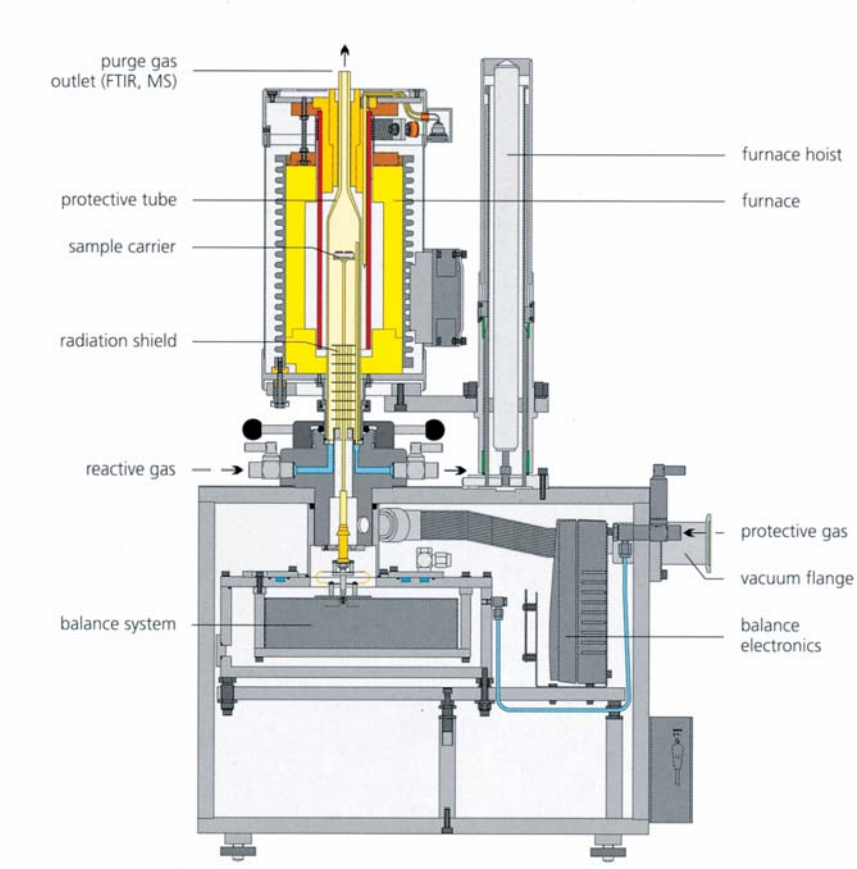
The cooling device is capable of achieving constant cooling and heating rates for measurements below ambient temperature.

For simultaneous measurements, the TG sample holder is removed and replaced by TG-DSC or TG-DTA systems. These plug-in TG-DSC/DTA sample holders can be supplied with different types of thermocouple to give the highest calorimetric sensitivity in a particular temperature range. This ensures the best possible sample results.

Simultaneous TG-DSC measurements up to 1650°C

The development of a new TG-DSC sample carrier system with high sensitivity and peak resolution allows simultaneous measurements in the temperature range -120°C…1650°C. A special high-temperature furnace with a very low temperature gradient in the sample area guarantees a stable DSC baseline up to the maximum operation temperature.

Scheme STA 409



Sample atmosphere

Fully automatic thermoanalytical tests can be carried out on samples under high vaccum (turbomolecular pump) in static or dynamic, inert or reactive gas atmospheres.

Other options for the STA 409 include: automatic gas change, protective tubes for vacuum and gas purge (inert, reactive). Corrosive gas atmospheres can be realized in a special version of the STA 409, for example SO_2. A special furnace permits measurements in a water vapour atmosphere up to 900°C.

Specific heat capacity c_p

A special high-temperature furnace with an almost non-existent temperature gradient in the sample chamber ensures a stable baseline up to the temperature limit. The specific heat capacity (c_p) can be determined up to a temperature of 1500°C with a TG-DSC sample carrier, a centering device and a supplementary software package. Three measurements are necessary: baseline, calibration and sample run.

Highest temperature model up to 2000°C

The STA is available with a 2000°C graphite furnace. The furnace is water-cooled. Measurements are conducted in an inert atmosphere, for example Ar. Water-cooling lowers the surface temperature of the furnace and allows more rapid cooling. A protective tube of alumina can be incorporated into the furnace to separate the sample area from the heating element. This allows measurements in oxidizing atmospheres up to 1680°C.

Mass spectrometer coupling

It is possible to determine evolved gases during decomposition by using simultaneous gas analysis. Two standard coupling systems are available for the model STA 409 :

– Capillary Coupling System
  MS 403 *Aëolos®*
  up to 2000°C,
  mass range: 1 - 300 amu

– Skimmer Coupling Systems
  403/5 up to 1500°C
  and 2000°C
  mass range: 1 - 300 amu
  1 - 512 amu
  1 - 1024 amu

Due to the combined software for Thermal Analysis (TA) and Mass Spectrometry (MS) only one computer system is necessary. TA and MS data are acquired in parallel, stored, and can be directly compared during the evaluation. Measurements can be made in SCAN-mode as well as in MID-mode (Multiple Ion Detection, up to 64 mass numbers m/e).

For details about coupling and software, please see our TA-MS/FTIR leaflet.

FTIR-Coupling

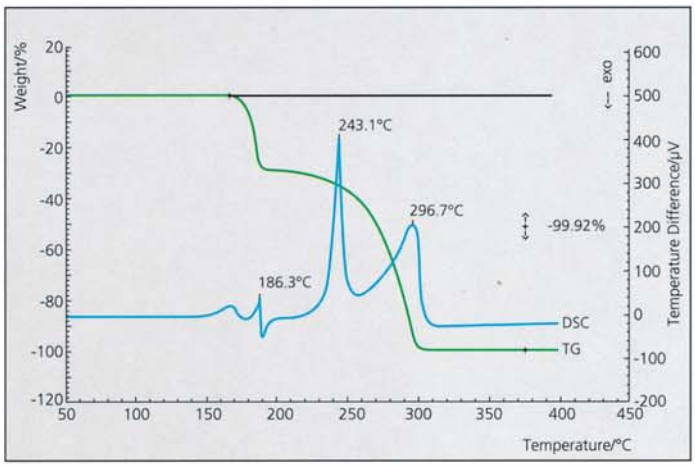
Infrared spectroscopy is a well-proven gas analysis method, especially in the field of polymers. Modern FTIR systems are coupled by a heated, flexible transfer line to the STA furnace with gas flow system (up to 1600°C).

The STA 409 is a user-friendly system that can be used in quality control, but at the same time it meets the high demands of research. The measuring part can be adapted to every single requirement in the different application areas.

Applications

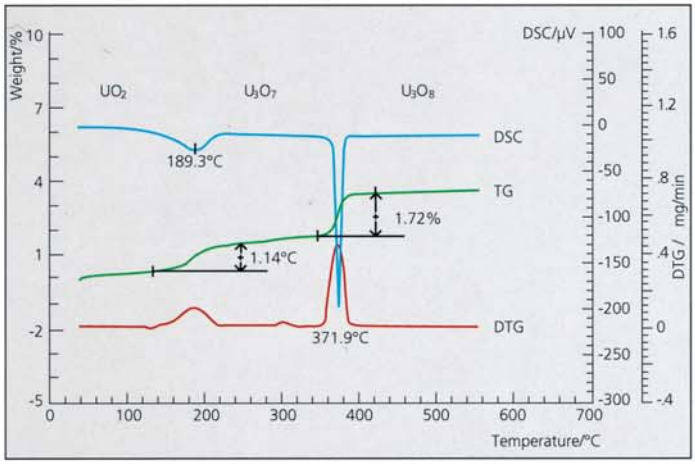
Quinolinic Acid

During the endothermal melting of quinolinic acid in dynamic N_2 atmosphere (100ml/min, quantity 30.6 mg, heating rate 5 K/min), the substance decomposes exothermally separating CO_2 and nicotinic acid.
The latter starts to sublime and shows a melting peak at 243°C. The complete sample mass has evaporated by approximately 300°C.



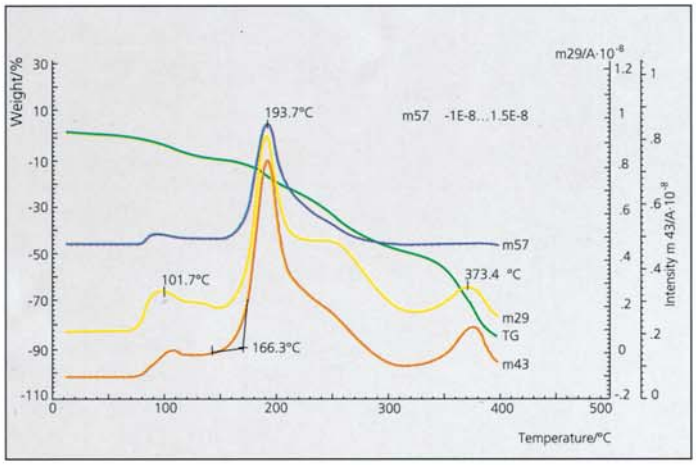
Uranium Dioxide

A 47.6 mg sample uranium dioxide was heated in air at a rate of 10 K/min. The TG signal shows a multiple step oxidation of the sample from UO_2 to U_3O_7 (2 steps) and then to U_3O_8. Simultaneously the DSC curve shows characteristic exothermal peaks at temperatures of 189°C and 372°C.



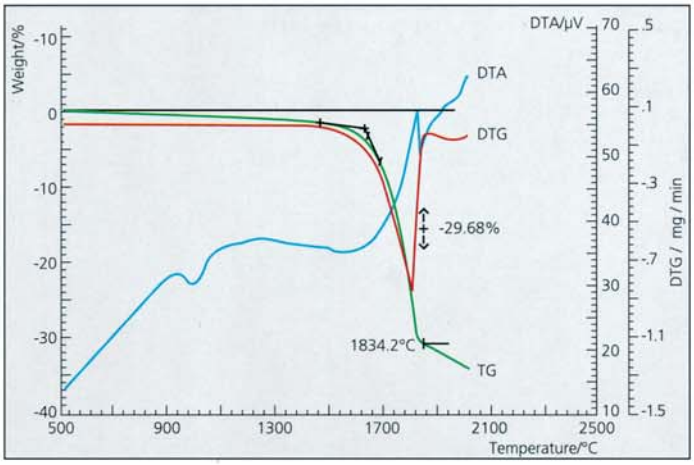
Dipping varnish

STA-MS measurement of a cathodic dipping varnish for the automobile industry. An isocyanate blocker has been qualitatively detected, fragments of which are set free at approx. 170°C so that the curing reaction can take place between the two lacquer components.



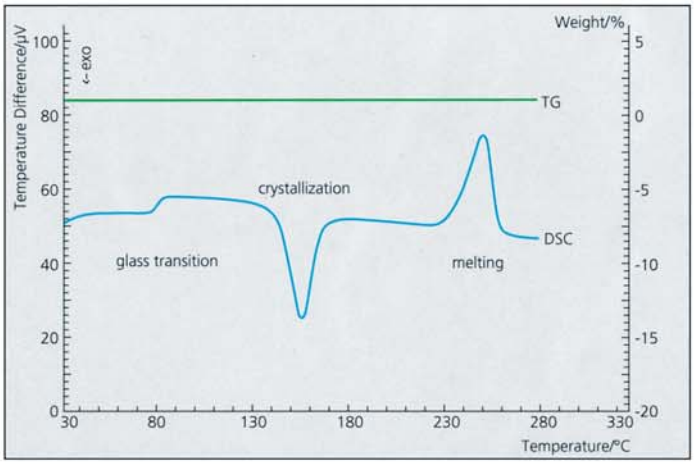
SiC – Sinter blend

STA measurement of a $SiC/Al_2O_3/Y_2O_3$ mixture in the STA 409/7.
Heating rate: 20 K/min;
Crucibles: Graphite;
Sample athmosphere:
He dynamic (100 ml/min);
DTA sample carrier: WRe.
The exothermal DTA peak at 1015°C can be traced back to the formation of the eutecticum of Al_2O_3-Y_2O_3. The two-step decomposition of the mixture starts at 1650°C (extrapolated onset). The maximum weight loss of the first TG step was determined at 1834°C. The TG steps are correlated with the endothermal DTA peaks (peak temperature of the first DTA peak is 1849°C).



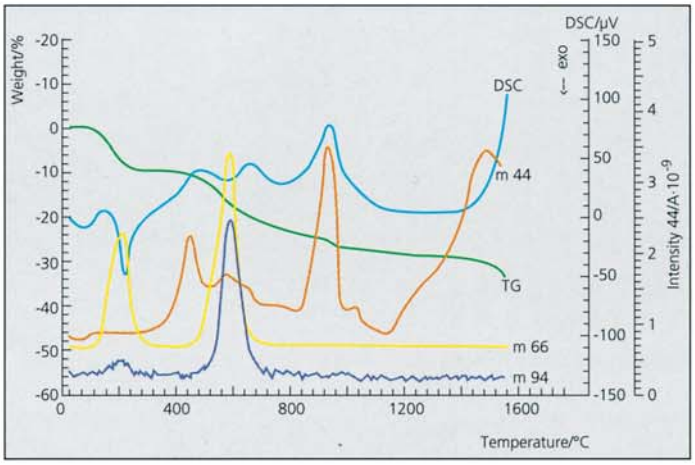
Polyethyleneterephthalate

The STA measurement of Polyethyleneterephthalate (PET) clearly shows the glass transition at 80°C, the crystallization peak at 158°C and the melting peak at 258°C. The measurement was conducted in a static He atmosphere at a heating rate of 10 K/min. In this temperature range, the weight clearly remains stable.



Phenolic Resin with Filler

Combined STA/mass spectrometry is a powerful tool to unravel complicated reactions which occur in polymeric material. This figure shows the TG-DSC curve corresponding to the release of CO_2 (m/z = 44), Phenol (m/z = 94) and the fragment with mass number 66 for a heating rate of 50 K/min (He atmosphere). The exothermal effect in the DSC curve at 200°C indicates a condensation reaction which is related to the curing of the material.



Technical Data

Temperature range
-160°C ... 2000°C
(different furnaces)

Thermogravimetry

Sensitivity:	5 µg
Sample weight:	max. 15 g
Measuring range:	15 g
Taring:	software-controlled

Calorimetry

Sensitivity:	depending on the sample carrier system used (e.g. DSC, type E – 18 µV/mW at the melting point of indium)
Measuring range:	5000 µV

Sample atmosphere

Static/dynamic:	oxidizing, reducing, inert, some corrosive gases (non-toxic, non-flammable, non-explosive), 0 ... 150 ml/min
Vacuum:	approx. 1 x 10^{-4} mbar

TA System Controller TASC 414/4
Microprocessor-controlled, for temperature control and data acquisition.
Programming and analogous output of temperature segments for 7 different temperature sensor types in the range of -200°C ... 2400°C, linear heating/cooling rates 0.1 ... 999.9 K/min, in steps of 0.01 K/min, isothermal segments of 0 ... 99 h 59 min, in steps of 1 min, high precision PID controller.
Resolution of analogue signals in ±2 Mio digits, max. 8 signals, sampling interval max. 160 values/s, integrated linearization of the signals of 7 different temperature sensor types, programming of the preamplifiers and optional control of external systems.

Computer system and software (MS® Windows™)
Standard PC e.g.
Pentium 500 MHz, ≥ 64 MB RAM, hard disk ≥ 6,4 GB etc.
1 IEEE interface, MS® Windows™.
Software for programming of the test run, data storage and evaluation. Software for kinetics, mass spectrometry or c_p determination (options).

Dimensions (w x h x d) and **Weights** (net)
measuring unit:
310 mm x 970 mm x 400 mm
approx. 50 kg
control cabinet TASC 414/3:
470 mm x 80 mm x 460 mm,
approx. 11 kg
Power supply for furnaces up to 1700°C:
370 mm x 200 mm x 470 mm,
approx. 30 kg, max. 3 KVA
for furnaces up to 2000°C:
545 mm x 1160 mm x 560 mm,
approx. 120 kg, max. 5 KVA
Connection: 2 x 230 V, 50 Hz
or 2 x 115V, 60 Hz

Technical data subject to change

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