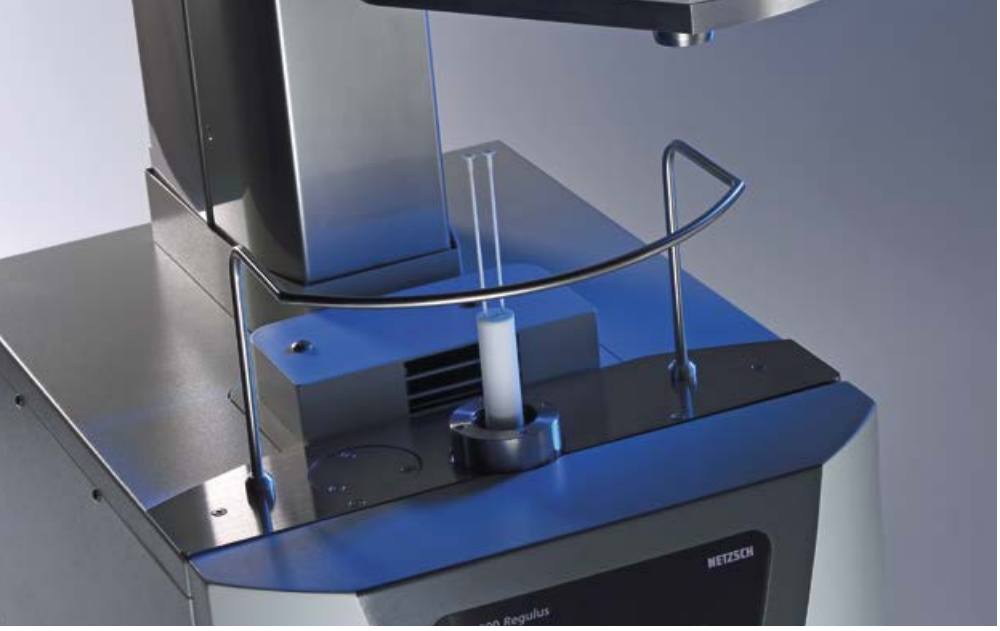
NETZSCH

STA 2500 *Regulus*®
Simultaneous Thermal Analysis

Technique, Instrument, Applications

Leading Thermal Analysis



Technique

Simultaneous thermal analysis refers to the simultaneous application of two or more thermoanalytical methods to the same sample in one instrument. This term, however, is in most cases used for simultaneous measurement of the mass changes and caloric effects on a sample under thermal treatment.

The benefits of such a system are obvious:

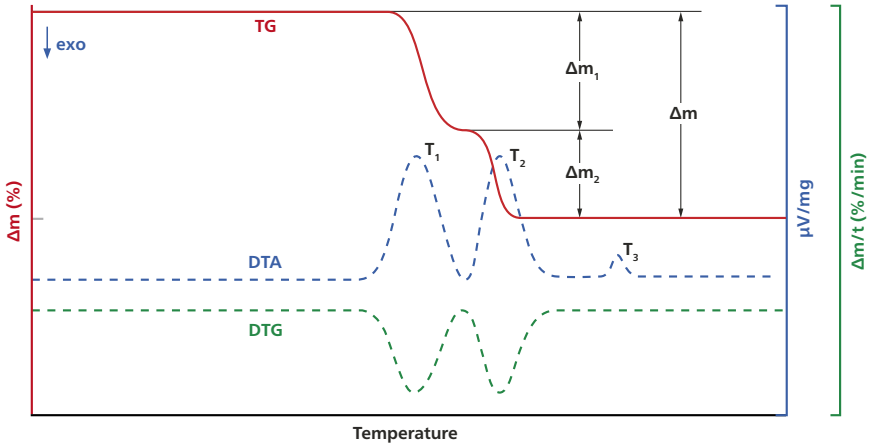
- Often, the material available for testing is expensive or difficult to produce. The transformation energetics and mass change are obtained on one sample in one run; the time necessary for the tests is cut in half.
- Influences of the measurement conditions and/or sample preparation can be eliminated. Comparability of characteristic temperatures of the mass changes and caloric effects is ensured.
- Problems resulting from differences in the sample composition for the two simultaneous measurements are removed for inhomogeneous materials.

Caloric Effects

- Phase transition temperatures
- Melting/crystallization behavior
- Phase diagrams
- Reactivity
- Modification changes

Thermogravimetric Effects

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



Measured signals in a TGA-DTA

Key Features at a Glance

Top-Loading and Unique Differential Balance Arrangement

The natural gas flow in the top-loading system guarantees automatic protection of the balance against condensation and contamination. The tailor-made differential balance system cancels out buoyancy and convection influences over a broad temperature range. This provides easy operation without the need for time-consuming baseline runs.

Various Atmospheres Available

Measurements can be performed in inert and oxidizing atmospheres but also under vacuum. The atmosphere can be dynamic or static. The built-in mass flow controllers (MFCs) are software-controlled and allow gas changes at any time during the test.

Ideal for Evolved Gas Analysis

The top-loading design of the STA allows for convenient coupling to gas analysis systems such as FT-IR (Fourier Transform Infrared) spectrometer, MS (mass spectrometer), or GC-MS (gas chromatograph – mass spectrometer). During thermal treatment, the gases evolved can be simultaneously analyzed.

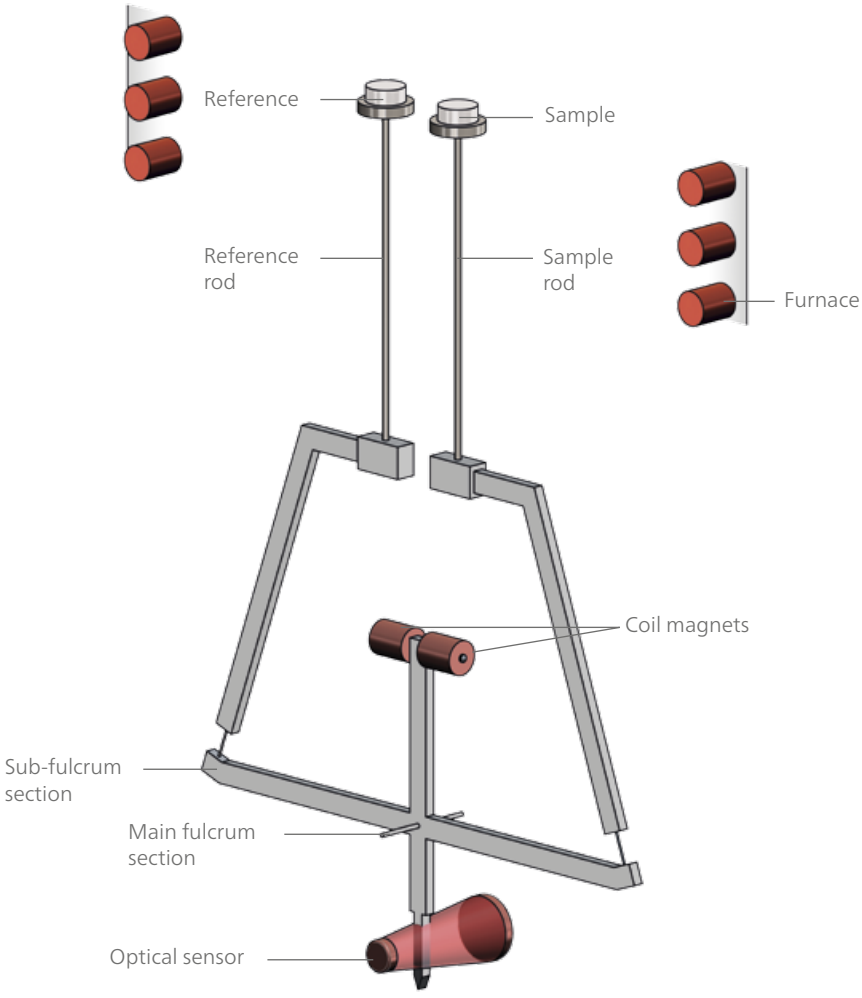
Excellent Cost Performance

The STA 2500 *Regulus*® features a highly reliable, fully equipped instrument package for simultaneous measurements up to 1600°C at an attractive price.

Standard Solution with Trendsetting Technology

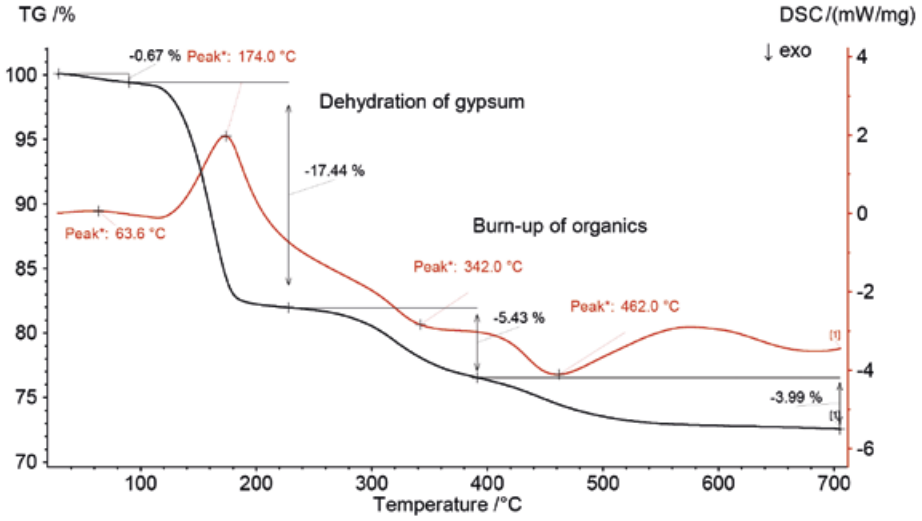
Advantages of the Unique Top-Loading Differential Balance Arrangement

The STA 2500 *Regulus*® is equipped with a tailor-made ultra-micro balance with a resolution of 0.03 µg. The self-compensating differential balance operates with two symmetric balance arms, connected to the reference and sample rod in the furnace. The compensation system (coil magnets) is controlled by a high-resolution optical sensor. This avoids rod movements resulting in a stable, high-resolution balance with a weighing range of ± 250 mg. The differential balance set-up compensates for buoyancy effects, even during changes in gas flow.



Schematic design of the top-loading differential balance of the STA 2500 *Regulus*®

STA 2500 *Regulus*®	
Design	Top-loading, vertical & differential balance
Furnace data	
Temperature range	RT to 1100°C/1600°C (2 easily exchangeable furnaces)
Heating rate	0.001 to 100 K/min (depending on the furnace)
Temperature precision (repeatability)	0.3 K
Motorized furnace hoist	Single hoist
Balance and Sensor Data	
Design	Differential balance system
Weighing range	± 250 mg, max. sample load 1 g
Thermogravimetric resolution	0.03 µg
Thermocouple	Type S
Atmosphere	
Vacuum-tight	10^{-4} mbar (10^{-2} Pa), depending on the evacuation system
Gas atmospheres	Inert, oxidizing, vacuum
Gas control	Built-in mass flow controllers

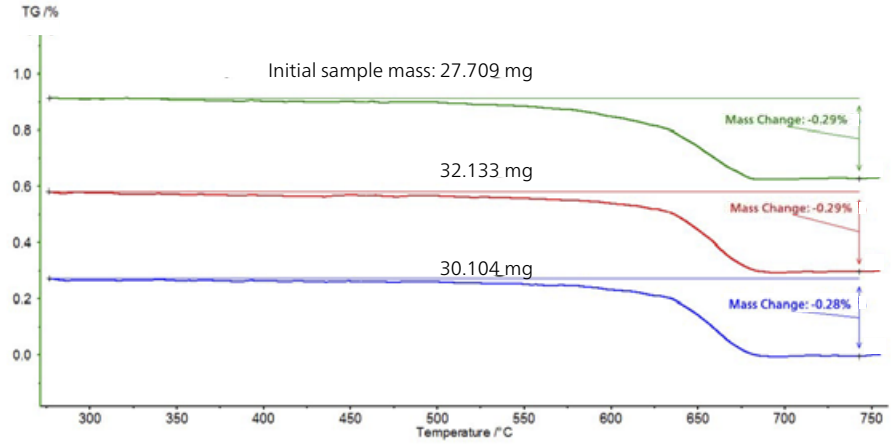


STA measurement on an aged gypsum board (8.32 mg) in air atmosphere (80 ml/min) at a heating rate of 40 K/min in open alumina crucibles. Here, the DTA signal is converted in mW/mg (DSC).

Gypsum Waste

Gypsum boards are specifically designed where high fire protection is required. Water is incorporated into its structure which in the presence of fire, vaporizes. It is fully recyclable and, as a consequence, it is one of the few construction materials for which closed loop recycling is possible. Here, gypsum waste powder was measured between RT and 700°C under air atmosphere for the simulation of both its behavior under fire exposure and the recycling process. Up to 250°C the endothermic dehydration of gypsum (red curve) can be observed with a mass loss of approximately 18% (black curve). This is followed by the exothermal burn-up of organics which might overlap the phase transition from Anhydrite III in II. Above 600°C, nearly constant mass is achieved.

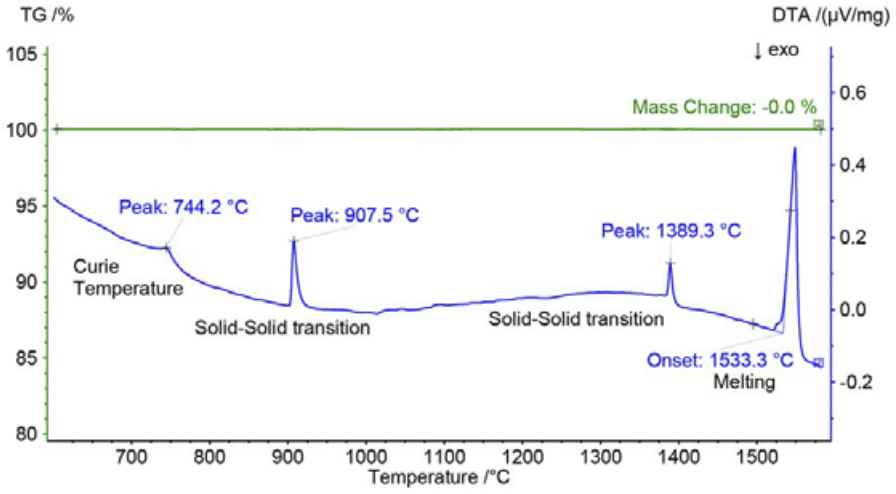
Excellent Reproducibility



Three rubber samples from the same batch after changing the atmosphere from nitrogen to air. Heating rate 20 K/min, Pt crucibles

The carbon black content of rubber materials is of essential importance and very often underlies a quality control process. In this example, three different rubber mixtures of the same batch were investigated specifically for their carbon black content. After monitoring the decomposition behavior under inert atmosphere, the atmosphere was switched to air and the mass change determined to 0.282 ±0.006% which proves the outstanding reliability and reproducibility of the STA 2500 *Regulus*®.

Phase Transitions of Iron



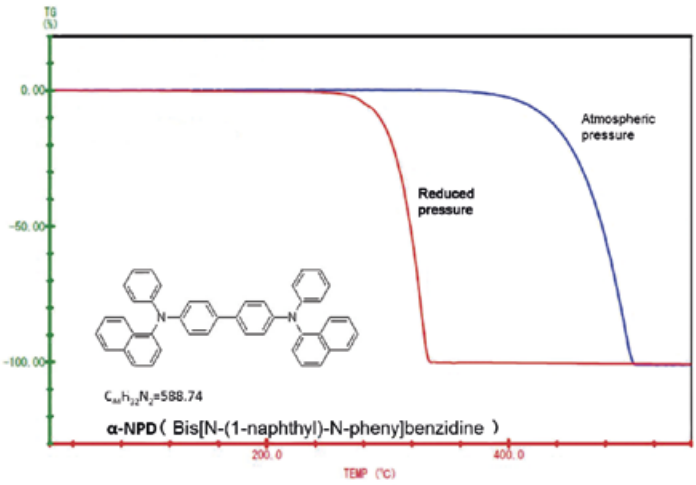
STA measurement of pure iron at a heating rate of 20 K/min. The phase transitions (blue curve) occurred without mass loss which indicates a high purity of iron and no oxidation due to the vacuum-tight design of the instrument.

The DTA sensitivity of the STA 2500 *Regulus*® allows for the detection of even weaker phase transitions. In addition, pure atmosphere can be achieved by using the *AutoVac* option for automatic evacuation and refilling of the system. Shown is the STA measurement of an iron sample between RT and 1600°C. The caloric effect at 744°C indicates a change in the magnetic properties of the material (blue curve). At peak temperatures of 908°C and 1389°C, two further changes in the crystal structure occurred. The endothermic peak at 1533°C (extrapolated onset) is due to the melting[1]. No mass loss could be detected in the TGA signal, indicating the vacuum-tightness of the system and high purity of the sample.

[1] Das Techniker Handbuch, Grundlagen und Anwendungen der Maschinenbau-Technik, 15. Auflage, Herausgeber Alfred Böge, Vieweg Verlag, 1999

Sublimation of an OEL Layer

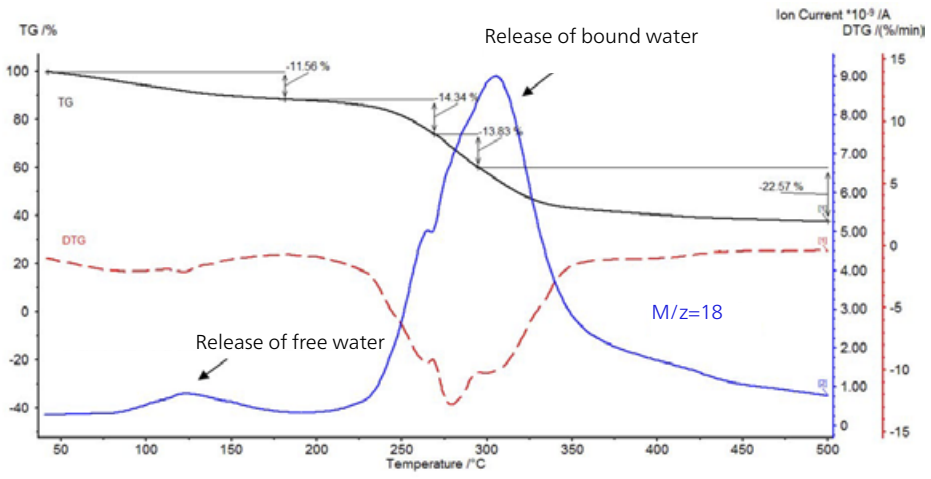
Multilayered organic structures can have unusual optical and physical properties which offer new possibilities for optoelectronic devices, including e.g., organic light emitting diodes (OLEDs). In organic multilayers, α-NPD is a hole transporting material. The vacuum-tight STA 2500 *Regulus*® allows for measurements under reduced pressure. Here α-NPD was measured under reduced pressure which resulted in a decrease of the sublimation temperature from 380°C at atmospheric pressure (blue curve) to 240°C at reduced pressure (red curve).



α-NPD under atmospheric and reduced pressure

Capsule in Tobacco Filter

Recently, the tobacco industry has introduced cigarettes with a new technology which makes cigarettes more attractive as it offers a capsule filled with a flavoring solution, embedded in the cigarette filter. The capsule can alter, for example, the taste of tobacco or keep it moist. This plot shows the TGA-MS measurement on a water-immersed capsule between room temperature and 500°C. The release of water associated with several mass-loss steps (TGA curve black and DTGA curve dotted red), is confirmed by the MS signal for mass number 18. A fast stabilization of the balance before the start of the measurement allows for the detection of the evaporation of free water prior to the release of the bound water.



TG-MS measurement on a capsule

STA 2500 *Regulus*®

WIDE VARIETY OF APPLICATIONS

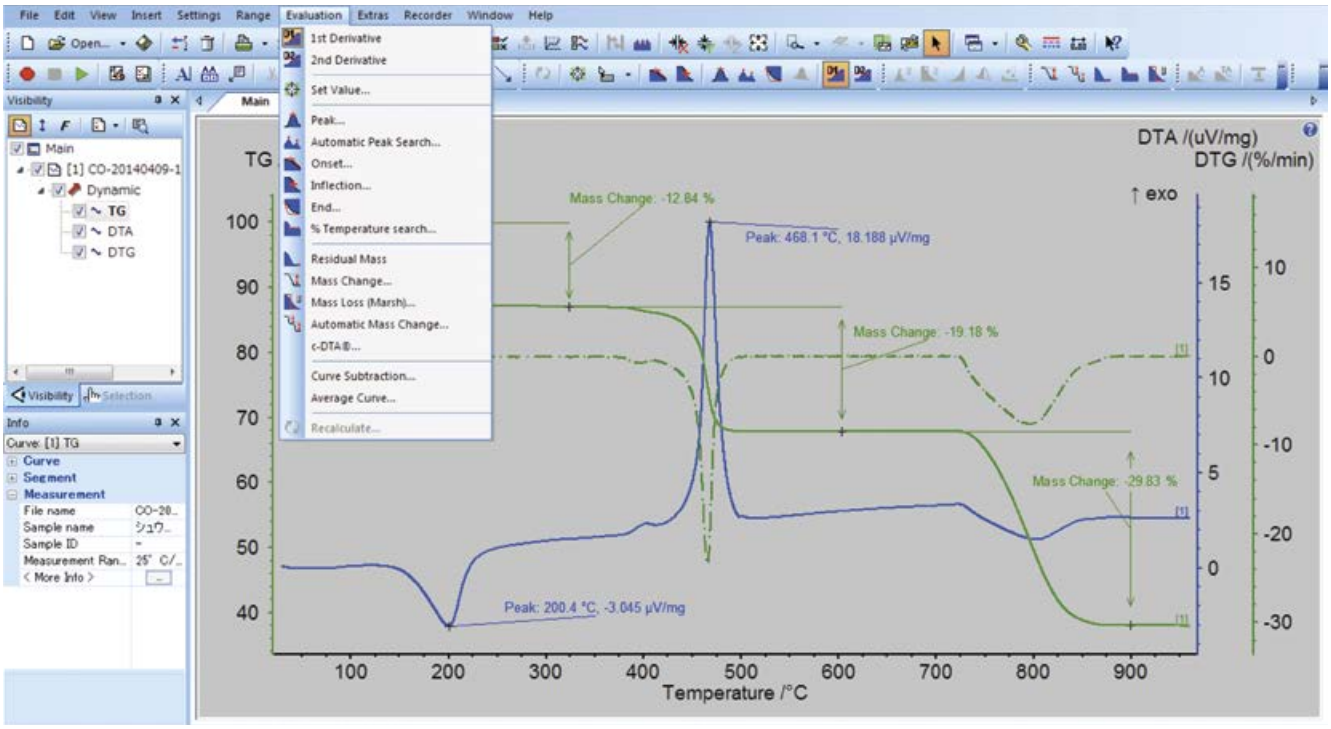
Proteus® Software

Highly Efficient and User-Friendly

The STA 2500 *Regulus*® runs on a Windows® operating system and includes everything you need to carry out a measurement and evaluate the resulting data. User-friendly menus combined with automated routines make *Proteus*® very easy to use while providing sophisticated analyses.

Key Features of the General Software

- Software produced by iso-certified company NETZSCH for Windows® 7/8.1 operating systems
- Simultaneous measurement and evaluation
- Operation of different instruments with one computer
- Combined analysis: Comparison and/or evaluation of STA, DSC, TGA, DIL, TMA and DMA measurements in a single presentation
- Input and free placement of text elements
- Calculation of 1st and 2nd derivative
- Selectable colors and line types
- Storage and restoration of analyses
- Context-sensitive help system
- Results by e-mail
- Automatic detection of instrument settings (e.g., furnace, sensor, etc.)
- Data export in Excel®-compatible CSV-format
- Calibration and correction routines for temperature, sensitivity, baseline
- Picture-in-picture presentation (PIP/FLIP)
- Snapshot for online evaluation of a measurement in progress



User interface of the *Proteus*® software during evaluation

STA-Specific Features

- TGA curves in absolute mass change (mg) or relative mass (%)
- Automatic evaluation of mass change steps and characteristic temperatures
- Extrapolated onset and endset
- Peak temperatures and values of the 1st and 2nd derivatives
- TGA stability check for early termination of the measurement
- Rate-controlled mass loss
- DTA curves in absolute (µV) or relative (µV/mg) units
- Determination of onset, peak, inflection and end temperatures
- Automatic peak search
- Exothermal presentation in accordance with DIN or ASTM (selectable)
- Calculation for conversion of DTA in DSC signal

Advanced Software (optional)

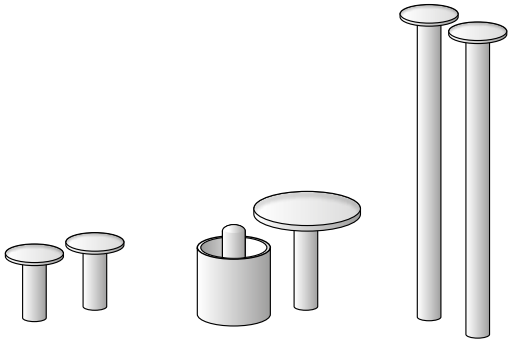
- *Peak Separation* for accurate separation and evaluation of overlapping transitions
- NETZSCH *Thermokinetics* for advanced characterization of reactions and kinetic parameters also provides predictions of the process

ACCESSORIES

Sample Pans, Crucibles and Plates

A variety of crucibles made of alumina, platinum, aluminum and quartz are available in different shapes and sizes. The STA 2500 *Regulus*® is delivered with slip-on plates which allow for nearly all kinds of sample pans (see table below). The large-volume crucible requires the large slip-on plate.

Slip-on plate



Standard | Large volume | Corrosion-protected

Slip-on plate types

Crucible Types for Various Applications

Material (Purity)	Diameter/Height	Volume	Maximum Temperature
Al (99.5)	Ø 5.2/2.6 mm	45 µl	600°C
Al (99.5)	Ø 5.2/5.1 mm	95 µl	600°C
Al_2O_3 (99.7)	Ø 5.2/2.6 mm	40 µl	1700°C
Al_2O_3 (99.7)	Ø 5.2/5.1 mm	80 µl	1700°C
Pt/Rh (90/10)	Ø 5.2/2.6 mm	45 µl	1600°C
Pt/Rh (90/10)	Ø 5.2/5.1 mm	95 µl	1600°C
Fused silica	Ø 5.0/2.6 mm	25 µl	1000°C
Fused silica	Ø 5.0/5.1 mm	55 µl	1000°C
Large volume Al_2O_3 (99.7)*	Ø 8.0/10.0 mm	400 µl	1700°C

* Available only with large slip-on plate

Coupling to Evolved Gas Analysis (EGA)

By coupling the STA 2500 *Regulus*® to a gas analysis system such as FT-IR (Fourier Transform Infrared) spectrometer, QMS (quadrupole mass spectrometer), or GC-MS (gas chromatograph – mass spectrometer), information on the type of gases evolved as a function of time or temperature can be obtained. This yields detailed information on the material to be tested and may provide fingerprint of the analyzed material.

- Connection to an FT-IR via transfer line
- Connection to a QMS via capillary
- Coupling to a GC-MS via transfer line

Coupling of STA 2500 *Regulus*® and GC-MS