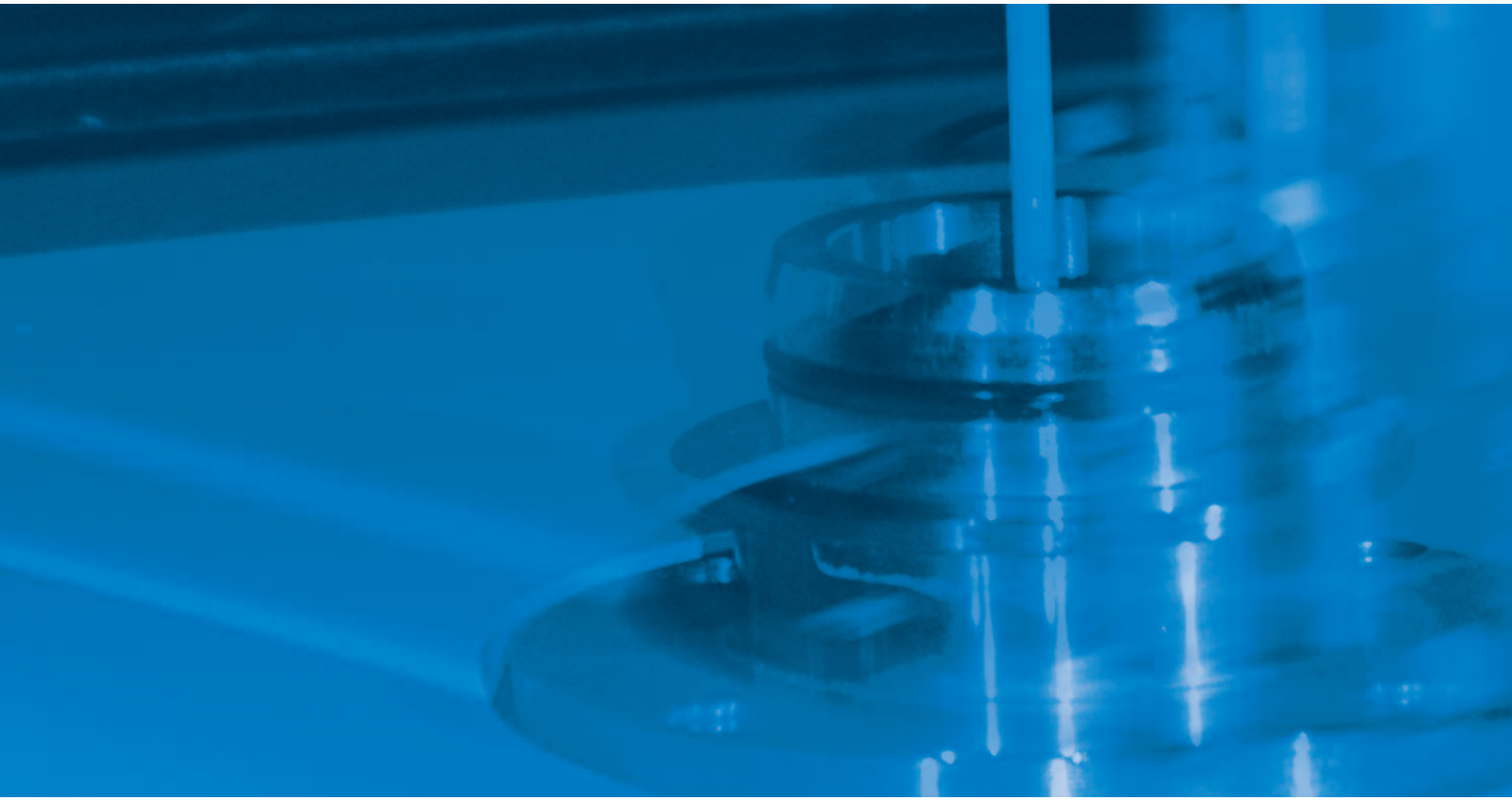
# LINSEIS

## STA–PLATINUM SERIES



Thermogravimetry is a technique in which the mass of the sample is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed.

This technique serves the determination of material compositions. It is common analysis method in the chemical and pharmaceutical industry. Thermogravimetric analysis (TGA) is performed on rubber, plastic, and ceramics as well as many other materials.

The LINSEIS STA Platinum Series (simultaneous thermal analysis) can be used to determine simultaneous changes of mass (TG) and caloric reactions (HDSC) of a sample in the temperature range –150 …1600°C. The unique characteristics of this product are high precision, high resolution and long term drift stability. The STA Platinum Series Platinum was especially developed to meet the challanging demands of the high temperature as well as low temperature applications. To cover this broad range several specifically designed furnace types are available. Furthermore MS (mass-spectrometer) and FTIR spectrometer couplings can be added to receive unique additional information. Due to its superior performance, user friendliness and modularity, the STA Platinum Series Platinum is an indispensable tool for every thermo analytical user.

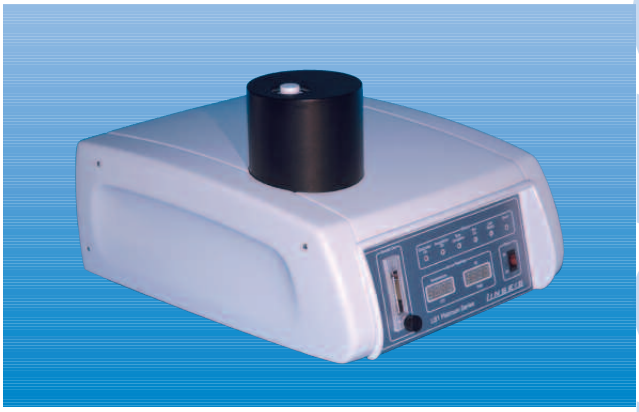
From the combination of TG and DTA or HDSC one receives a broad range of information, such as:

**TG:**

- Mass change
- Absolute sample temperature
- Temperature difference (sample / reference)

**HDSC:**

- Enthalpy, melting energy
- Specific heat
- Glass point
- Crystallinity
- Reaction enthalpy
- Thermal stability
- Oxidation stability
- Aging
- Purity
- Phase transformation
- Solidus / Liquidus - relationship
- Eutecticum
- Polymorphism
- Product identification



PRODUCTS

## Software

All LINSEIS thermo analytical instruments are PC controlled. The individual software modules exclusively run under Microsoft® Windows® operating systems. The complete software consists of 3 modules: temperature control, data acquisition and data evaluation. The 32 bit software incorporates all essential features for measurement preparation, execution, and evaluation of a Thermogravimetric measurement. Thanks to our specialists and application experts, LINSEIS was able to develop comprehensive easy to understand user friendly application software.

## Features -Software:

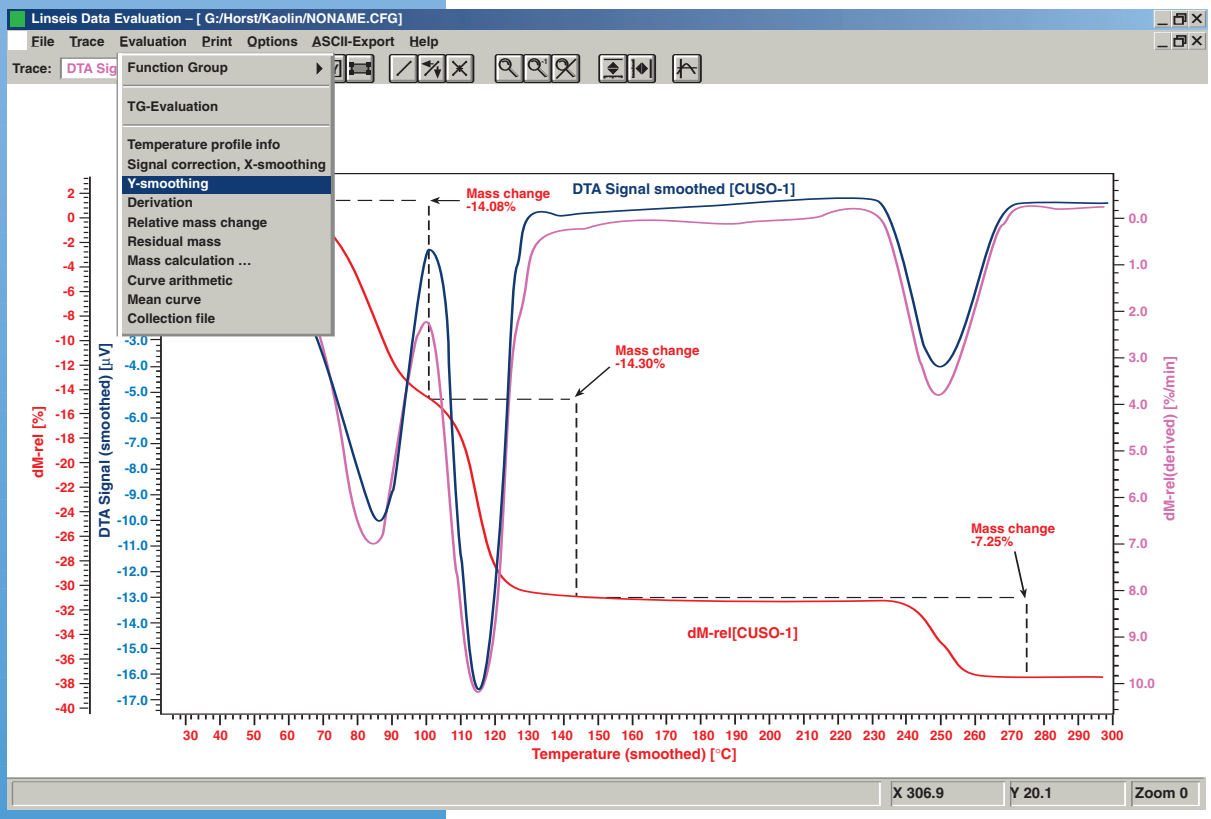
- Program capable of text editing
- Data security in case of power failure
- Thermocouple break protection
- Repetition measurements with minimum parameter input
- Evaluation of current measure ment
- Curve comparison up to 32 curves
- Storage and export of evaluations
- Export and import of data ASCII
- Data export to MS Excel
- Multi-methods analysis (DSC TG, TMA, DIL, etc.)
- Zoom function
- 1 and 2 derivation
- Programmable gas control
- Statistical evaluation package
- Free scaling

### TG – Features:

- Mass change as % and mg
- Rate Controlled Mass Loss
- Evaluation of mass loss
- Residue mass evaluation

### HDSC – Features:

- Glass transition temperature
- Curve subtraction
- Complex peak evaluation
- Multipoint calibration for sample temperature
- Multipoint calibration for change of enthalpy
- Cp calibration for heat flow
- Signal-steered measu ng proce dures




SOFTWARE

## Unique features:

**Measurement system:**

The Linseis L81 Thermo balances can be ordered as horizontal, vertical, or both mode of operation. All measurement systems are easily exchangeable to ensure user-friendly and quick system handling.

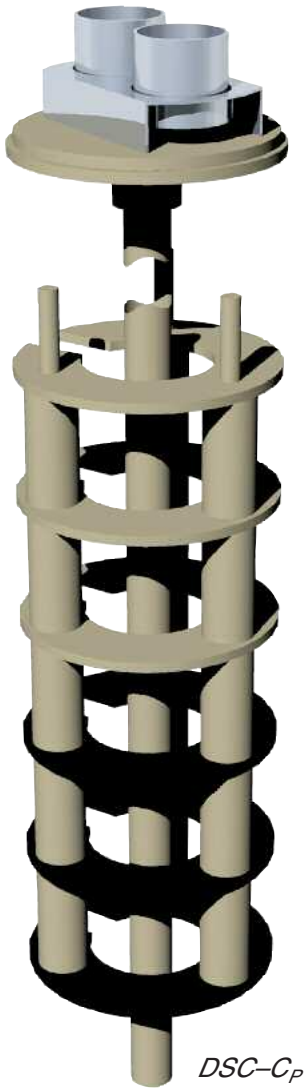
**Vacuum and controlled atmosphere**

The balance design provide for high vacuum (10E-5mbar), inert, reducing, flowing or humidified atmosphere. Corrosive conditions can be analyzed with proper precautions. The system is capable of adapting residual gas analysis systems using an optional heated capillary.

**Furnace program:**

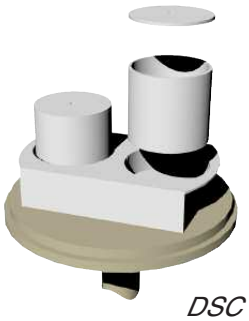
The exchangeable furnace program for horizontal operation allows measurements from –150 …1000°C. For vertical systems the temperature range goes from RT …2400°C. Stable and reproducible baselines are achieved due to the specially designed heating elements and system setup.

FEATURES



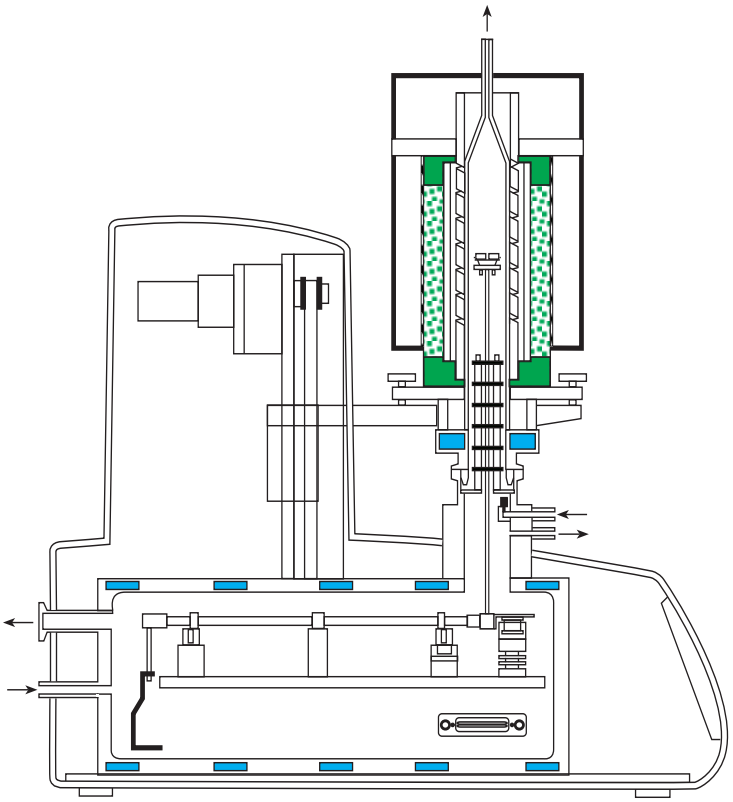
*DSC–$C_P$*

*TG*



*DSC*

*DTA*



*STA Platinum Series*

OPTIONS

**Sample holders:**

Several sample holders in different configurations are available. Because of the 25 gram capacity there is a clear advantage when measuring heterogeneous samples. Large amounts of overall sample can be measured to distinguish reactions from lesser components. This combination enables a very high resolution while assuring the best possible sample reactions.

## Options:

The following options are available for the STA Platinum Series:

Liquid nitrogen cooling system for low temperature furnace (KREG)

MS/FTIR coupling for evolved gas analysis (EGA)

Turbo-molecular pump: for measurements under highest vacuum and cleanest gas-atmospheres.

# FURNANCE PROGRAMM

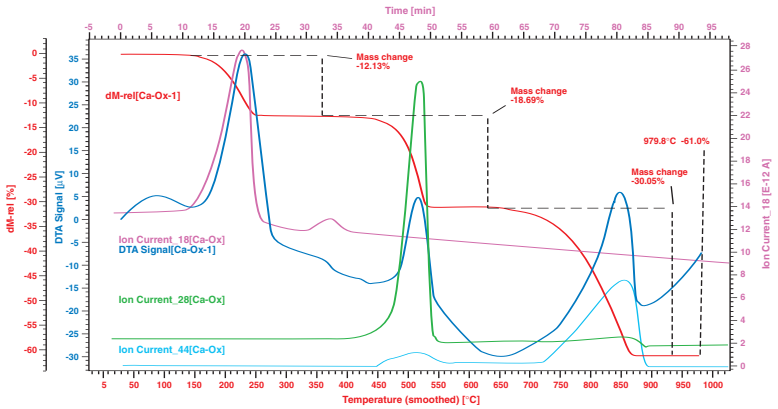
## Horizontal

| Temperature | Type | Element | Atmosphere | TC-Type |
|---|---|---|---|---|
| -150 – 500°C | L81/220 | LN2 | inert, oxid., red., vac. | K |
| RT – 1000°C | L81/264 | Kanthal | inert, oxid., red., vac. | K |

## Vertical

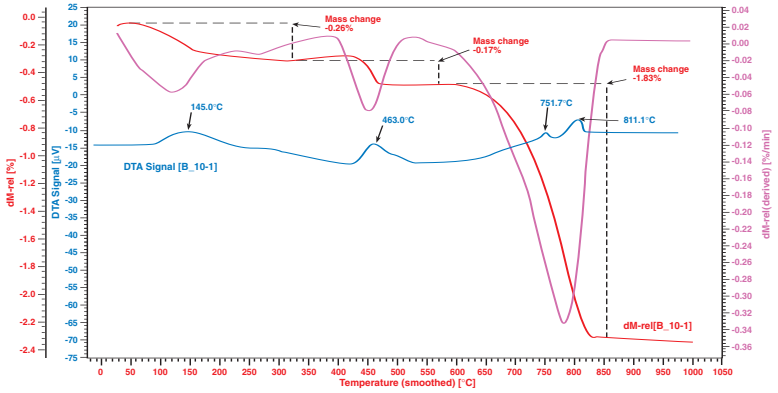
| Temperature | Type | Element | Atmosphere | TC-Type |
|---|---|---|---|---|
| RT – 1000°C | L81/220 | Kanthal | inert, oxid., red., vac. | K |
| RT – 1400°C | L81/230 | Kanthal | inert, oxid., red., vac. | S |
| RT – 1600°C | L81/240 | SiC | inert, oxid., red., vac. | S |
| RT – 1750°C | L81/250* | Pyrox | inert, oxid., red., vac. | B |
| RT – 2400°C | L81/270* | Tungsten | inert, red., vac. | C and/or Pyrometer |

(* only for L81 – I)



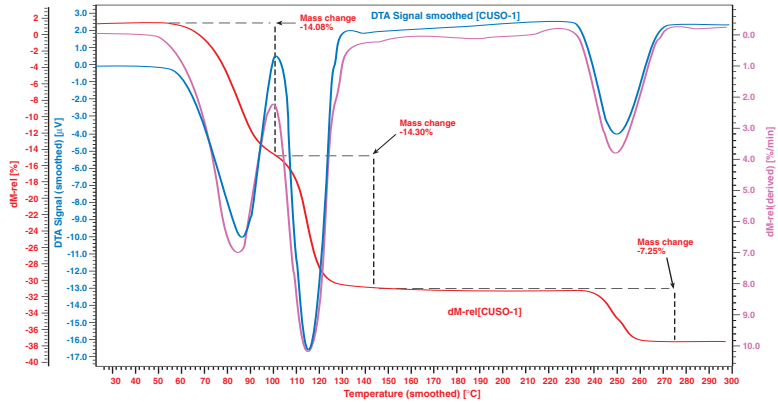

## 1. Decomposition of Calcium Oxalate monohydrate $CaC_2O_4$ under Argon Atmosphere

The evolved gases from the decomposition of Calcium Oxalat have been fed into the Mass Spectrometer with a heated capillary. The ion currents for mass numbers 18 (water), 28 (carbon monoxide) and 44 (carbon dioxide) have been imported into the graph.



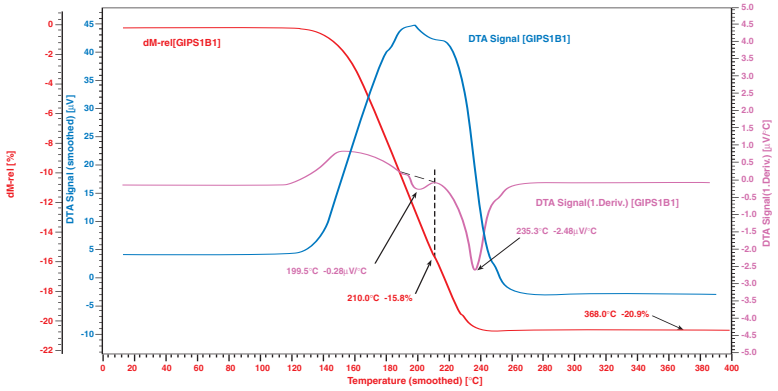

## 2. Cement

The main parts of cement are tri calcium silicate, di calcium silicate and tri calcium aluminates. After putting on the cement with water different hydrates slowly form. The absorbed water evaporates first, then hydrates of the calcium silicate decompose and at 570°C the hydroxides of calcium, magnesium and aluminum follow. Subsequently, calcium carbonate $CO_2$ splits off.



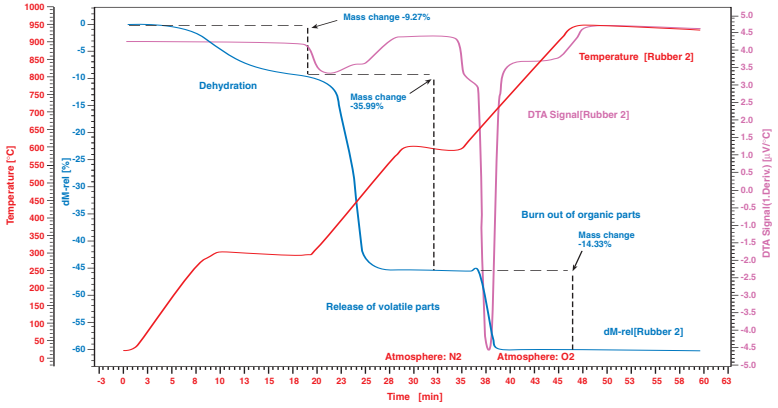

## 3. Vitriol of Copper

Inorganic salts first set their crystal water free. Vitriol of Copper heated with 10°C/min. results in a first TG stage, which corresponds to $4H_2O$. TG and DTA show that the curve is made up of two separate steps, one closely behind the other. At around 250°C the most strongly bound water evaporates.



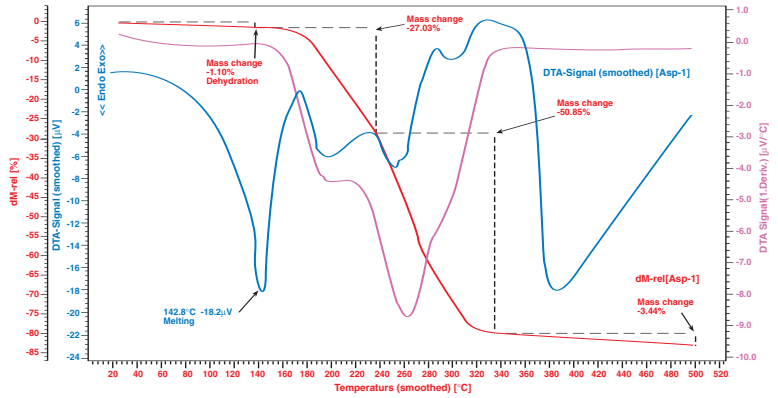

## 4. Dehydration of raw gypsum $CaSO_4 + 2\ H_2O$

The dehydrate is Raw gypsum. There are two molecules of water attached to a single $CaSO_4$ molecule. By heating up to app. 160°C the semi hydrate gypsum is built. 1,5 molecules of water are released; so two CaSO4 molecules are sharing a single water molecule. By heating to higher temperatures (400°C) the anhydrate is built. This is the so-called "dead burned gypsum", also known as alabaster. In this state no water at all is attached to the $CaSO_4$ molecules.




## 5. Decomposition of Rubber

In the first step of weight loss, the dehydration of the sample takes place. The amount of water was 9.27%. In the second reaction step, the volatile components are released by pyrolysis under $N_2$ atmosphere. The amount of these components is 35.99%. For the third reaction step, the atmosphere is changed to $O_2$ – all organic components are burned out. The loss in weight is 14.33%. The remaining rest of 40.41% are inorganic components like ashes, slake or fillers.




## 6. Aspirin

At the beginning of the heating process some adsorbed water is released, resulting in a weight loss of around 1%. At 140°C the melting point of the Aspirin is reached, resulting in an exothermic reaction, measured on the DTA trace. At 160°C the decomposition of the melted drug takes place in several stages. Since the decomposition products are volatile a weight loss of just about 80% occurs.

## 1.2 Model STA PT1600

Technical Data:
STA instrument (simultaneous thermal analyzer) complete with soft- and hardware, incl. calibration standards and accessories

| | |
|---|---|
| **Temperature range:** | RT up to 1600°C |
| **Vacuum:** | 10E-5mbar |
| **Heating rate:** | 0,1 up to 100°C/min (depends on furnace) |
| **Temperature resolution:** | ± 0,1°C |
| **Temperature accuracy:** | ± 0,3°C (substance calibration) |
| **Data evaluation rate:** | max. 10/s |
| **Base line drift:** | 5ug/K max. 10 ug/day |

### TG

| | |
|---|---|
| **Resolution:** | 0,5 µg, internal 0,05 ug |
| **RMS-noise:** | < 1 µg |
| **Max. sample weight:** | 25 g |
| **Measuring range:** | 25 / 2500 mg |

### DSC

| | |
|---|---|
| **DSC-sensors:** | E / K / S / B |
| **DSC resolution:** | 0,3 / 0,4 / 1 / 1,2 µW |
| **DSC-RMS-noise:** | 4 / 6 / 17,6 / 22,5 µW |

### DTA

| | |
|---|---|
| **DTA-resolution:** | 0,05 µV |
| **DTA-measuring ranges:** | 250 / 2500 µV |

## 1.3 System components

### Hardware

- STA basic unit consisting of:
- Desk, automatic furnace lift
- Measuring head for TG- or STA-application
- Vacuum tight protection tube Al2O3
- 1600°C SiC furnace
- USB connection to computer
- IBM compatible PC, State of the Art
- 17“ color monitor
- color inkjet printer

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