Haas

Your knowledge our technology

SIMULTANEOUS THERMAL ANALYSIS

STA HP 3

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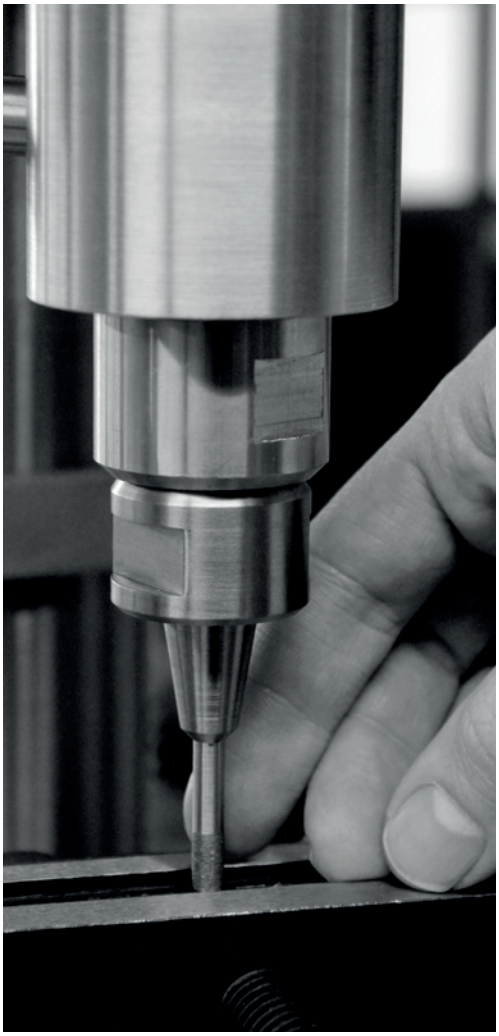
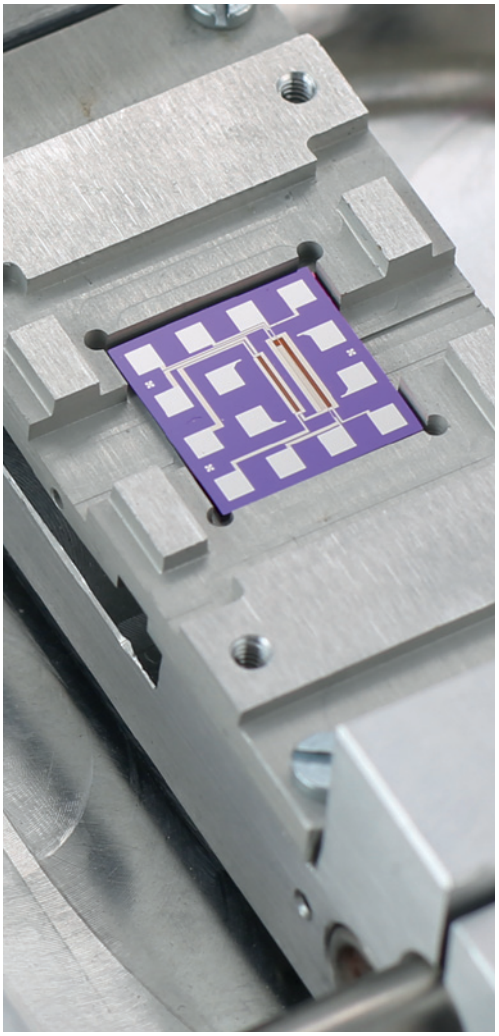




German engineering

The strive for the best due diligence and accountability is part of our DNA. Our history is affected by German engineering and strict quality control.

Innovation

We want to deliver the latest and best technology for our customers. LINSEIS continues to innovate and enhance our existing thermal analyzers. Our goal is constantly develop new technologies to enable continued discovery in Science.

HIGH PRESSURE STA

Linseis introduces the all new STA HP3 table top high pressure thermo-gravimetric, simultaneous thermal analysis instrument. The STA HP3 is the result of 25 years of experience thermal analysis under high pressure.

Worlds only top loading combined TG-DSC Thermogravimetry-differential scanning calorimetry. For experiments up to 1200°C and up to 150 bar in reactive or inert atmospheres.

The superior high speed micro-furnace with a maximum temperature of 1200°C, top loading microbalance, true TG-DSC Thermogravimetry-DifferentialScanning Calorimetry design permit exiting new opportunities. The table top design, optional vapor generator and different gas dosing systems provides utmost flexibility.

Various Sensors

Easy user exchangeable TGA or TG-DSC plug and play sensors. The right choice for any given experiment gives you the flexibility to analyze TGA only experiments with volumes up to 1 ml or combined TGA-DSC analysis with up to 0,12 or 0.3 ml volume.

TG-DSC Thermogravimetry-Differential Scanning Calorimetry arrangement permits the combined analysis of weight change and caloric events like endothermal or exothermal reactions or phase transitions in one run and under the same temperature, gas and pressure conditions.

High accurate sample temperature measurement. The thermocouple is in direct contact with the sample. The configuration of the TGA/STA HP 3 eliminates temperature measurement errors due to sample – thermocouple distance (unlike levitating MSB-Magnetic Suspension Setup).

High speed micro heater permits rapid heating and cooling (up to 300°C/min controlled heating rate and up to 150°C/min cooling rate).

Very small furnace volume allows rapid gas changes. In addition the low volume lowers the cost of ownership (gas consumption/energy requirement) drastically.

Flexible gas dosing and security design

Our gas dosing panels can be designed to your needs. Number of gases can be selected (1,2,3, 4, …) optional vapor generator, automatic evacuation, gas burn off and safety system are available for gases like hydrogen and hydrocarbons.

Linseis STA HP 3 total setup including water vapor generator and automated MFC gas control panel

High precision mass flow controllers (MFC) and intelligent gas dosing and pressure system of a Linseis STA HP 2 guarantee optimum performance under various conditions in the full pressure and temperature range

Linseis TG measurement system for high pressure range. The unique table-top pressure system can be equipped with TG or TG-DSC sensor heads

GAS EQUIPMENT

Customized gas control

The LINSEIS TGA/STA HP 3 series can be equipped with any number of mass flow controllers (MFCs), depending on the customer's needs, to control, mix and handle a wide range of gases. This allows full control of atmospheres from 10^{-4}mbar up to 150 bar in a temperature range from room temperature up to 1200°C.

Additionally, there is the possibility to add condensate traps, water vapor generators and also heated transfer lines for dosing of steam and other condensing gases. All gas control panels fulfill high German quality and safety standards and are user friendly designed to guarantee best possible performance.

SOFTWARE

The all new Platinum Software greatly enhances your workflow as the intuitive data handling only requires minimum parameter input.

AutoEval offers a valuable guidance for the user when evaluating standard processes such as glass transitions or melting points. Thermal library product identification tool, provides a database with 600 polymers permitting an auto-matic identification tool for your tested polymer. Instrument control and/or sur-veillance through mobile devices gives you control wherever you are.

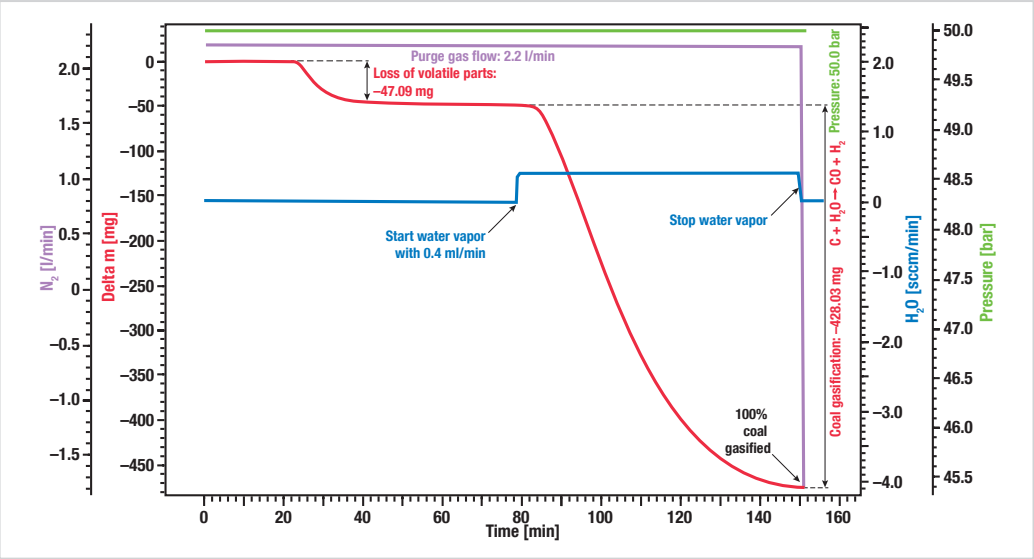
- Software packages are compatible with latest Windows operating system
- Set up menu entries
- All specific measuring parameters (User, Lab, Sample, Company, etc.)
- Optional password and user levels
- Undo and Redo function for all steps
- Infinite heating, cooling or dwell time segments
- Multiple language versions such as English, German, French, Spanish, Chinese, Japanese, Russian, etc. (user selectable)
- Evaluation software features a number of functions enabling a complete evaluation of all types of data
- Multiple smoothing models
- Complete evaluation history (all steps can be undone)
- Evaluation and data acquisition can be performed simultaneously
- Data can be corrected using zero and calibration correction
- Data evaluation includes: Peak separation software Signal correction and smoothing, first and second derivative, curve arithmetic, data peak evaluation, glass point evaluation, slope correction. Zoom / individual segment display, multiple curve overlay, annotation and drawing tools, copy to clip board function, multiple export features for graphic and data export, reference based correction

SPECIFICATIONS

	STA HP 3
Temperature range	RT up to 1200°C
Pressure range	up to 150 bar
Sample mass	Up to 5 g
Resolution	0.1 µg
Vacuum	10^{-4} mbar
TG-sensors	Type E/K/S/B/C
TG-DSC sensors	Type E/K/S/B/C
Electronics	Integrated or separation on electronics
Interface	USB or Ethernet
Vapor generator	Optional
Gas dosing	1,2 or 3 gases or more on request

APPLICATION

Coal gasification



A common known application for HDSC measurements is the investigation of the so called coal gasification or hydro gasification. This process, where carbon is heated in a water steam atmosphere is used in some catalytical processes, for example to remove carbon monoxide (CO) from exhaust fumes and especially to get valuable organic compounds out from resources like charcoal or biomass.

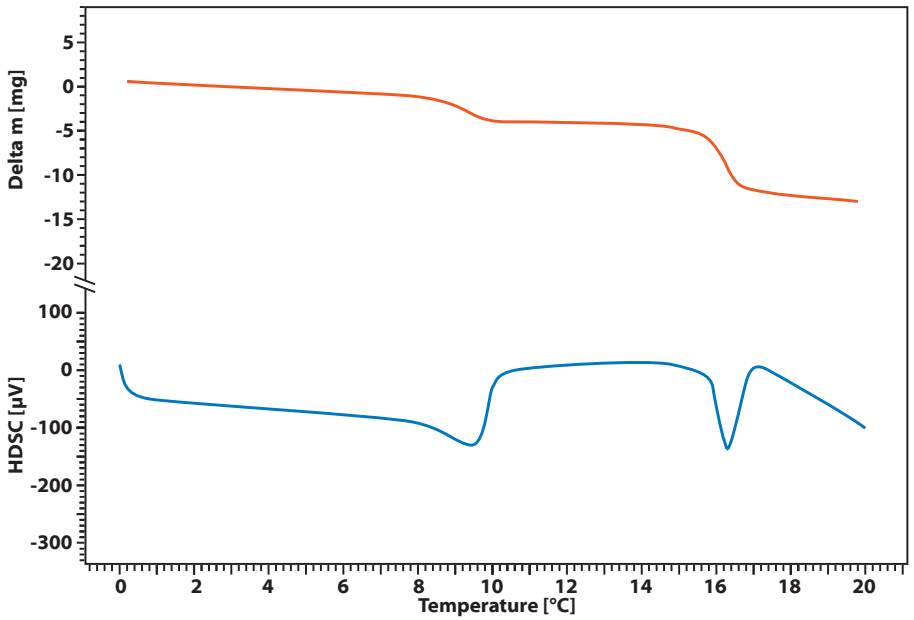
The whole process can be described like this:

Charcoal or the Carbon parts of Biomass react with water vapour to a mixture of carbon monoxide and hydrogen at higher temperatures ($C + H_2O \longrightarrow CO + H_2$).

This process can be done with or without additional oxygen. If an oxygen con-taining atmosphere is used, you will also get additional carbon monoxide ac-cording to ($C + O_2 \longrightarrow CO_2$ followed by $C + CO_2 \rightleftharpoons 2\,CO$) The third equation, no matter if you use oxygen or not, shows the reaction of carbon monoxide with water to get more hydrogen ($CO + H_2O \rightleftharpoons CO_2 + H_2$). So in the end you will get a mixture of carbon monoxide and hydrogen. Those two gases are involved in chemical balances and therefore sometimes it is also interesting, to know how much pressure you have in your system, be-cause the pressure determines on which side of the equation the balance will be. Finally, the purpose of the coal gasification is, that you can get methanol and methane out of the two created gases carbon monoxide and hydrogen ($CO + 2\,H_2 \rightleftharpoons CH_3OH$; $CH_3OH + H_2 \rightleftharpoons CH_4$) That means with this process you can get from any kind of carbon to the basic building block of almost every organic compound (drugs, polymers, oils, waxes, fatty acids, organic acids and so on).

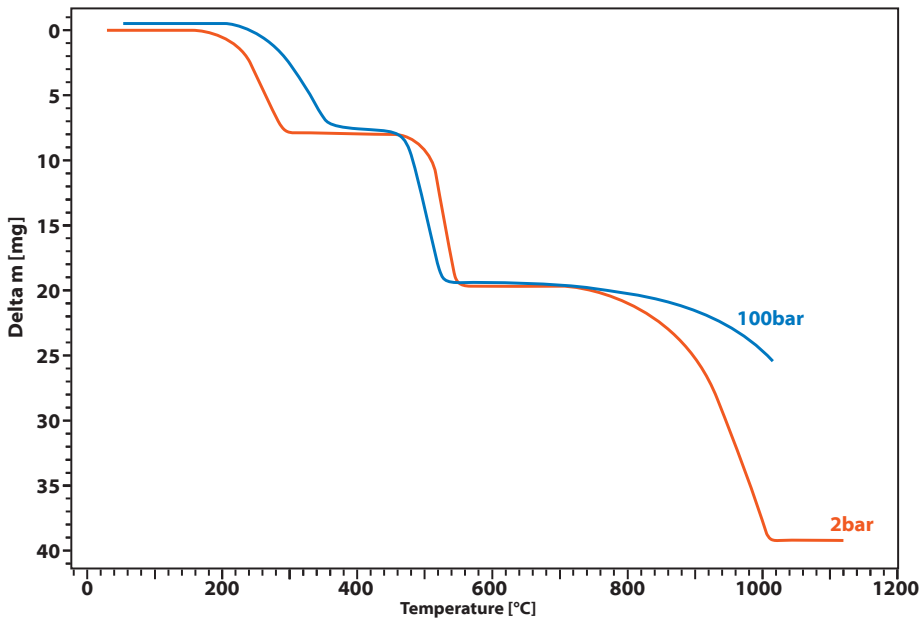
APPLICATIONS

Measurement of Calciumoxalate at 100bar constant pressure in STA HP3



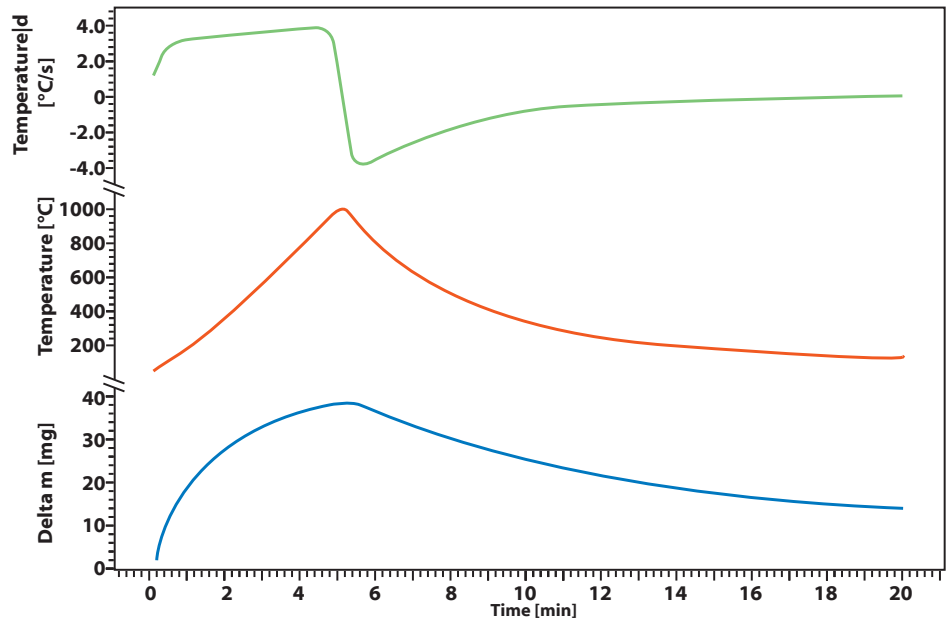
The STA HP3 can be operated also at high pressures such as 100bar. The scheme shows the DSC and TG signal of a calcium oxalate run in 100 bar static nitrogen atmosphere at a linear heating rate of 20K/min up to 600°C.
The red TG curve shows the first two mass loss steps that are well known for calcium oxalate. However the first effect is shifted in temperature due to the high ambient pressure. The first effect is the loss of water, where it is notable that the enthalpy peak ends clearly after the mass loss effect which means the water is released but still need more energy to evaporate due to the high ambient pressure. The second peak is the loss of carbon monoxide which happens at nearly the same temperature like at lower pressures. The reason is that this effect is a structural decomposition reaction that is happening independent from ambient pressure. Even if the CO is finally released it is not so much affected by the high pressure like the water in the first mass loss step as it is not in a chemical balance and part of an irreversible decomposition.

STA HP 3 pressure influence on the thermal decomposition of CalciumoxalateCalcium



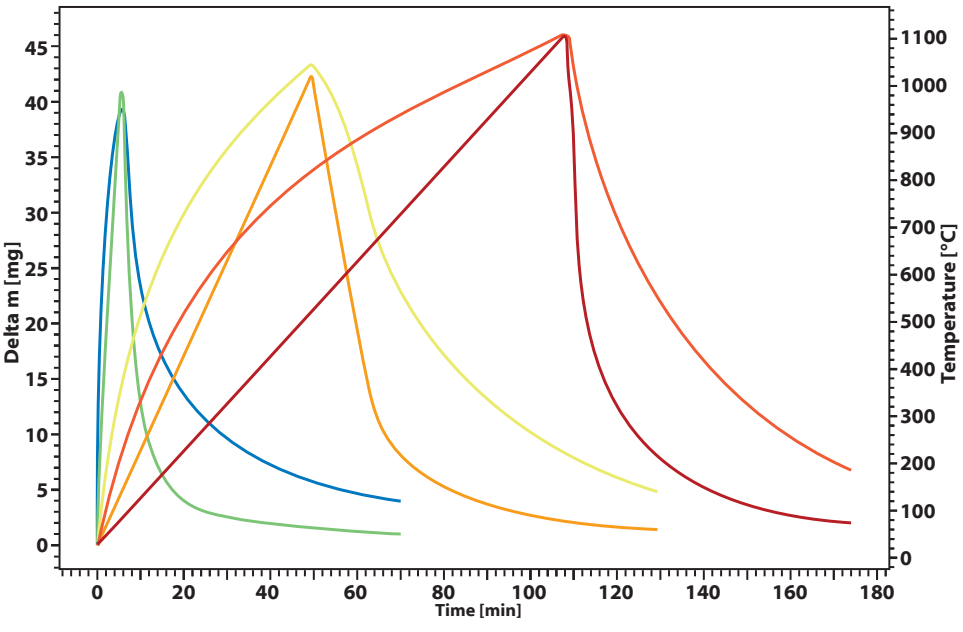
The substance calcium oxalate monohydrate is a common known reference material for thermo-balances and DSCs. It decomposes thermally in three definite mass loss steps that are caused by the release of water (a), the release of carbon monoxide (b) and the release of carbon dioxide (c).
Also in pressurized atmospheres, these effects can be seen, however they see a pressure depending shift on the temperature axis. The scheme shows the decomposition of calcium oxalate monohydrate at 2 bar and 100 bar nitrogen atmosphere, measured by Linseis STA HP3.
The first and last effect are clearly occurring later at the higher pressure (blue curve), while the second effect (b) is happening a little bit earlier. The reason is that the decomposition that releases the water and CO2 are reversible under higher pressure and therefore are delayed, while the decomposition step (b), the loss of CO is an irreversible decomposition reaction that is independent of ambient pressure.

Heating and cooling rates of STA HP3 at 100 bar constant pressure



The curves were measured with an empty crucible at 100bar constant ambient pressure using the STA HP3. The heating rates and cooling rates were determined using the derivative over time (red curve). The programmed heating rate was 200K/min which was reached after a short initial phase. The cooling was passive ballistic cooling, resulting in maximum cooling rates of up to 200 K/min at the beginning. Below 300°C, a constant cooling rate of 50K/min is still possible. As can be seen, the whole experiment (heating from room temperature to 1000°C and cooling down to 100°C) can be done in just 20 minutes, even at high pressures.

STA HP 3 comparison of fast heating rates



The unique furnace design of the STA HP 3 allows ultra-fast heating rates, even in pressurized experimental setups. The curves show the zero curves of an empty system at 10, 20 and 200 K/min at constant pressure of 10 bar nitrogen. As you can see even at fast heating speed, there is the same noise level and accuracy than with slower rates. Each curve was measured twice, to show the reproducibility which is also excellent as can be seen above.

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