<u>LINSEIS</u>

pushing boundaries

HDSC L62

High Temperature

Differential

High Tempera Differential Scanning Calorimeter





Since 1957 LINSEIS Corporation has been delivering outstanding service, know-how and leading innovative products in the field of thermal analysis and thermophysical properties.

Customer satisfaction, innovation, flexibility, and high quality are what LINSEIS represents. Thanks to these fundamentals, our company enjoys an exceptional reputation among the leading scientific and industrial organizations. LINSEIS has been offering highly innovative benchmark products for many years.

The LINSEIS business unit of thermal analysis is involved in the complete range of thermoanalytical equipment for R&D as well as quality control. We support applications in sectors such as polymers, chemical industry, inorganic building materials, and environmental analytics. In addition, thermophysical properties of solids, liquids, and melts can be analyzed.

Rooted in a strong family tradition, LINSEIS is proudly steered into its third generation, maintaining its core values and commitment to excellence, which have been passed down through the family leadership. This generational continuity strengthens our dedication to innovation and quality, embodying the essence of a true family-run business.

LINSEIS provides technological leadership. We develop and manufacture thermoanalytic and thermophysical testing equipment to the highest standards and precision. Due to our innovative drive and precision, we are a leading manufacturer of thermal analysis equipment.

The development of thermoanalytical testing machines requires significant research and a high degree of precision. LINSEIS Corp. invests in this research to the benefit of our customers.

CLAUS LINSEIS



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

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 to constantly develop new technologies to enable continued discovery in Science.



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High Temperature Differential Scanning Calorimeter

Differential Scanning Calorimetry (DSC) is one of the central methods of thermal analysis: Indispensable when it comes to precisely investigating endothermic and exothermic transitions. It provides detailed insights into the thermal behavior of materials – as a function of temperature and time with high significance.

The **LINSEIS HDSC L62** (High Temperature DSC) is designed to deliver highest calorimetric sensitivity, short time constants and a condensation free sample chamber. These features guarantee superior resolution and baseline stability over the entire instrument lifetime. This provides an indispensable tool for material development and quality control. The modular concept of the HDSC and DTA systems allows the use of different furnaces with a temperature range from -150 °C up to 1750 °C. The vacuum tight design enables quantitative enthalpy and Cp (Specific Heat) determination under the cleanest atmospheres and under vacuum of 10-5 mbar. The systems can be upgraded with an optional sample robot and coupled to a MS or FTIR.

Measuring System

User friendly exchangeable measuring systems such as a DTA Sensor and two different DSC Sensors are available. Each DSC Sensor is available as type E, K, S, B for the HDSC L62.

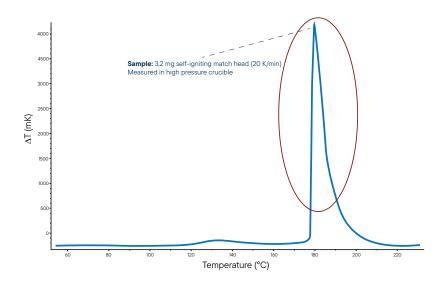
This allows the perfect choice for any application, temperature or atmosphere.



-150 °C up to 1750 °C

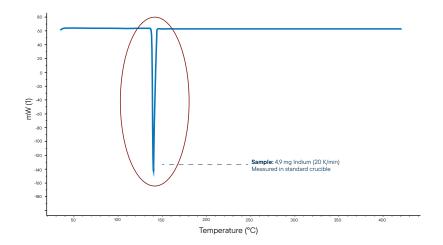






Exothermic Events

- Oxidation
- Cure Reactions
- Crystallization
- Polymorphic Transitions

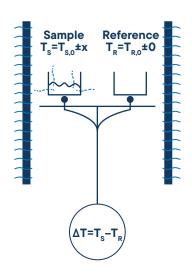


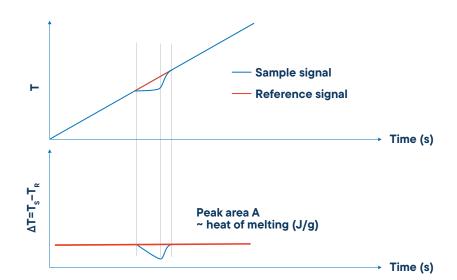
Endothermic Events

- Melting
- Glass transitions
- · Enthalpic Recovery
- Polymorphic Transitions
- Evoration/Volatilization
- Decomposition

DSC-True Heat Flow measurement

Quantitative DSC-signal





Differential Scanning Calorimetry (DSC)

A technique in where the difference in energy input into a substance and a reference material is measured as a function of temperature, while the substance and reference material are subjected to a controlled temperature program.

Differential Signal

The differential signal is displayed as a baseline. Effects, for example the melting of a metal, can be observed as a peak. The area of the peak gives the amount of enthalpy and the direction of the peak indicates the way of heat flux – endothermic or exothermic.

Temperature vs. Time

During an effect like a reaction, decomposition or phase transition, a temperature difference between the sample and the reference crucible can be measured by means of a thermocouples.

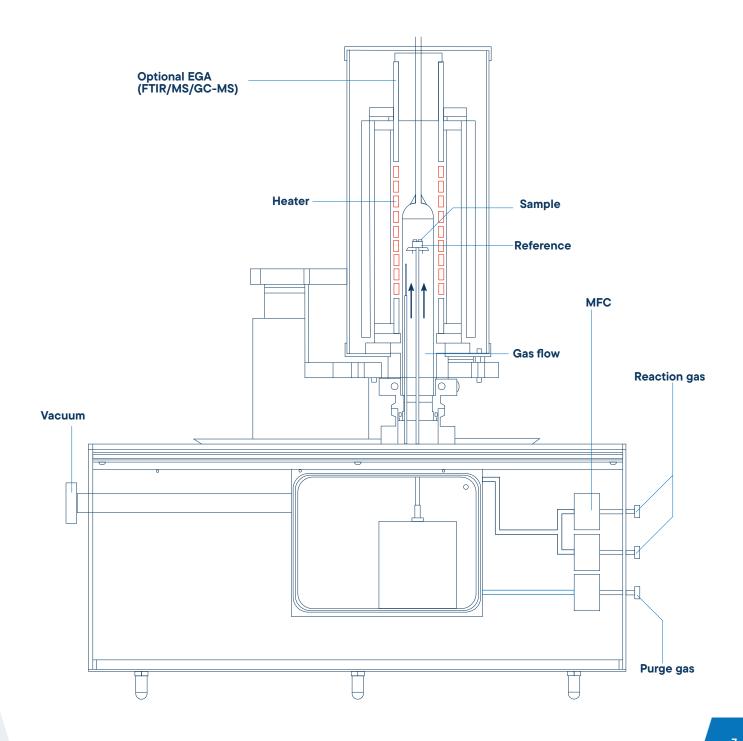
Measurable Properties

- Glass point
- Crystallinity
- · Thermal stability

- Oxidation stability
- Purity
- · Solidus relationship

- Liquidus relationship
- Product identification

Illustration of the HDSC L62



Unique features

Automatic Evacuation

The devices feature a built-in automatic evacuation capability, ensuring efficient processes and smooth operation.

Vacuum and controlled atmosphere

- Supports high vacuum, inert, reducing, oxidizing or humidified atmospheres
- Optional pressurization up to 5 bar overpressure
- Analysis of certain corrosive conditions with precautions
- Adaptability for residual gas analysis with optional heated capillary

Evolved gas analysis

Optional gas analysis with MS, FTIR or GCMS is possible, providing valuable additional information. The system can be configured with standalone and integrated MFC for gas dosing. Additionally, customer-specific options such as a heated inlet can also be integrated into the device.

Gas safety system

The gas safety system is designed to accommodate various gases such as hydrogen or carbon dioxide, ensuring secure operation and user safety.

Controlled humidity and water vapor

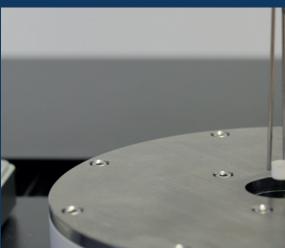
The device can regulate the humidity and water vapor during the measurement.

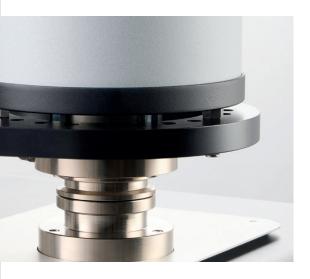
Sample robot

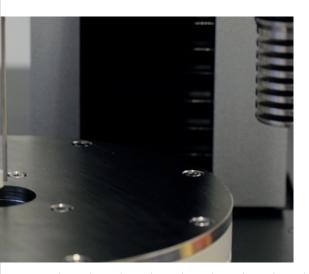
Our **HDSC L62** can be equiped with a proven sample robot for unattended sample measurements for highest throughput.











Wide temperature range -150 °C to 1750 °C

The **LINSEIS HDSC L62** instruments can be equipped with up to two furnaces at the same time. A broad variety of different furnaces are available to enable measurements in the widest temperature range on the market.

Automatic calibration

We offer an automatic calibration function in the software and hardware. With this function, our HDSC automatically calculates a calibration factor, which is also displayed.

Integrated LINSEIS platform

The integrated LINSEIS software offers a comprehensive solution, combining both hardware and software for maximum process security and precision. By providing a unified platform, it ensures seamless integration of components and devices from external partners, resulting in a highly robust system.

Customization

Close collaboration with the customers to tailor unique solutions, leveraging LINSEIS expertise to meet their specific needs.

Service

Our international presence across every continent enables us to deliver the best and fastest service possible.

Accessories starter kit

The starter kit guarantees fast and uncomplicated usage, serving as a complete system for instant application.







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Equipment for gas control and safety (H₂, CO, CO₂, etc...)



All LINSEIS instruments can be prepared for the use in hydrogen atmosphere with just minor adjustments. The most important thing is a safety system that can ensure that there is no leakage and no explosive atmosphere generated outside of the instrument. Therefore, the Linseis safety system uses hydrogen sensors that are coupled to an automatic gas control panel. If there is a leakage or unwanted hydrogen release, the instrument is automatically flooded with inert gas and the hydrogen valves are closed. This ensures a minimum risk level during operation. Besides that, the system contains an optional burn off unit where the gas outlet is connected to, to ensure that also the used gas of the measurement chamber is not just released into the environment. The system can also be operated with several combinations of inert gases and even water vapor besides hydrogen.

In summary, the Linseis safety system comes with the following benefits:

- Automatic evacuation function
- Gas flow control for multiple gases including water vapor and hydrogen
- Emergency shutdown function
- Gas detector system (H₂, CO, CO₂, etc...
- optional burn off unit
- Continuous monitoring to ensure safe operating conditions



Linseis equipment for operation under water vapor and controlled relative humidity



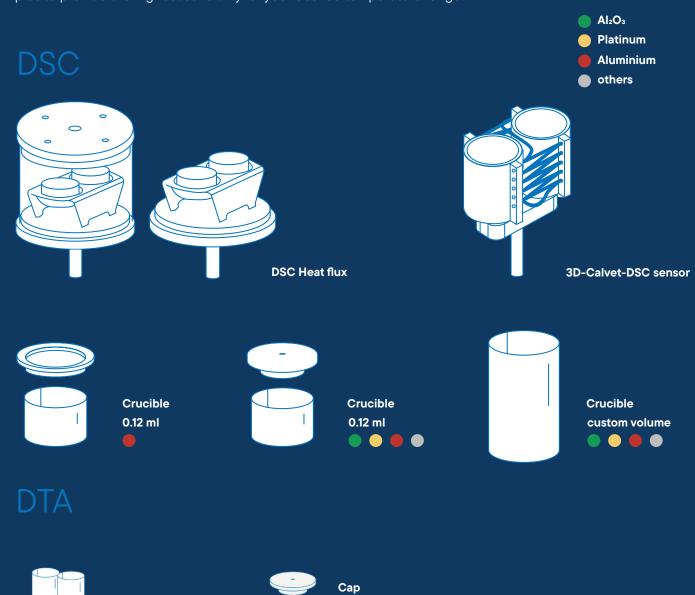
For many applications in thermal analysis, the atmosphere plays an important role as it may affect the sample behavior or activate reactions. Humidity influence on building materials, storage time of pharmaceuticals and foods or influence on mechanical properties of polymers are just some of the most common examples. Of course, the Linseis instruments are suitable for such experiments, however there is one fact that is often causing confusing and must be considered carefully: The difference between water vapor and relative humidty.

Relative Humidity Generators are most commonly used for experiments around room temperature, while water vapor applications are most often at higher temperatures. When water is heated to its boiling point or higher than that, the water changes its aggregate form from liquid to gaseous. It is then existing as water vapor (steam). If this steam is introduced into any kind of reaction chamber or instrument, it is called water vapor application. In contrast, every gas can transport and contain a certain amount of water at a given temperature. This is called humidity. Considering air as an example, there is always an amount of water contained in the air, even below the boiling point of water, which is defined as grade of humidity or relative humidity.



Sensors & Crucibles

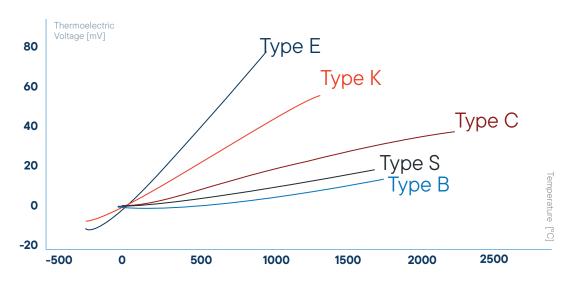
LINSEIS HDSC L62 can be equipped with an unmatched amount of different user exchangeable DSC sensors and crucibles. Each sensor is available with different thermocouples to provide the highest sensitivty for your desired temperature range.



0.3 ml or custom crucible volume

Crucible

DTA-sensor



All sensors available with the thermocouples illustrated. LINSEIS sensor combinations cover the broadest temperature range in the market (-180 $^{\circ}$ C up to 2400 $^{\circ}$ C).

Thermocouple types:

Type E (Chromel) Constantan: -50 °C up to 900 °C

Type K (Chromel) Alumel: -180 °C up to

1100 °C **Type S** (Platinum-Rho-

dium/10 %)
Platinum: 0 °C up to

1600 °C **Type B** (Platinum-Rhodium/ 30 %) Platinum-Rhodium (6 %): 25 °C up to 1800 °C

Type C (Tungsten/5 %) Rhenium: Tungsten (26 %)

Rhenium: 20 °C up to 2300 °C

| Sensor Type | (+) Leg | (-) Leg | Generated thermo voltage* change in μV (Reference junction at 0 °C) | | Approx. working temperature | Notes | |
|-------------|--------------------------------|------------------------------|--|--------|-----------------------------|----------------------|---|
| | | | 100 °C | 500 °C | 1000 °C | | |
| E | NiCr | CuNi | 68 | 81 | - | -200 °C up to 800 °C | Highest thermo voltage* per °C |
| К | Ni-Cr Cromel | Ni-Al Alumel | 42 | 43 | 39 | 0 °C up to 1100 °C | |
| s | Pt10Rh Platin10% Rhodium | Platinum | 8 | 9 | 11 | 0 °C up to 1550 °C | |
| В | Pt30Rh Platin30% Rhodium | Pt6Rh Platin6% Rhodium | 1 | 5 | 9 | 0 °C up to 1750 °C | Most suited for oxi- dizing atmosphere |

*Electro motive Force by seebeck coefficient

Software

The software greatly enhances your workflow as the intuitive data handling only requires minimum parameter input. LiEAP offers a valuable guidance for the user when evaluating standard processes such as melting and crystallization points.

The optional thermal library product identification tool, provides a database permitting an automatic identification tool for your tested materials such as polymers.

Data acquisition

Shared database:

One software for many devices

- Compatible with the latest Windows® operating systems
- Online updates
- Automatic gas control for multiple gases and hardware types (Optional)
- Unlimited heating, cooling and dwell time segments
- Multi-language versions such as English, German, French, Chinese, Japanese, etc. (user selectable)
- Optional password protection and user access levels
- Simultaneous data acquisition and evaluation

Data evaluation

Includes:

Signal correction and smoothing, derivate/ integral, arithmetic operations for curves, peak evaluation, glass point evaluation, onsetpoint determination, multiple curve overlay, annotation and drawing tools, copy to clipboard function, multiple export features for graphic and data export, reference based correction.

- Undo and redo function for all steps
- Complete evaluation history
- Export to various data formats
- Extendable via Python plugins





The LINSEIS Thermal Library software package comes as an option for the well-known, user friendly LINSEIS LiEAP (Linseis Evaluation and Acquisition Platform) software that is integrated in almost all our instruments.

The Thermal Library allows the sample material identification by comparing the measurement curve with a data base providing thousands of references and standard materials within only 1-2 seconds.

Multi-Instrument

LINSEIS instruments such as DSC, STA, TGA & LFA can be controlled with the same powerful LiEAP software platform.

Report Generator

Convenient template selection to generate customized measurement reports.

Database

Multi-User

State of the art database design enables easy data handling.

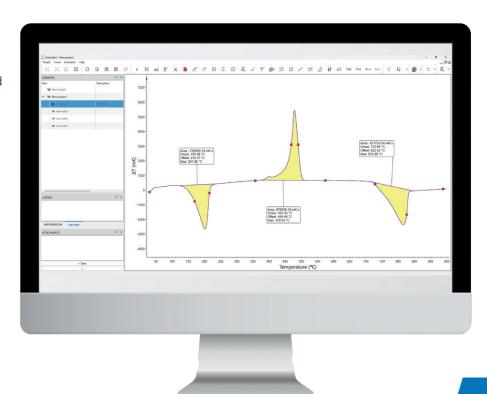
A optional Log file is also available.

The administrator can generate different user levels

providing different rights to operate the instrument.

Kinetic software

Kinetic analysis of DSC, DTA, TGA, EGA (TG-MS, TG-FTIR) data for the study of the thermal behavior of raw materials and products.



Technical Specifications

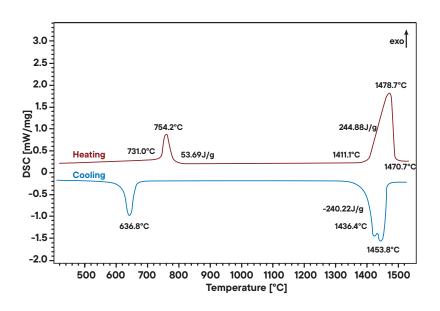
| HDSC L62 | | | |
|--------------------------|---|--|--|
| Temperature range | -150 °C up to 1750 °C | | |
| Vacuum | 10 ⁻⁵ mbar (depends on vacuum pump) | | |
| Pressure | up to 5 bar (optional) | | |
| Heating rate | 0.01 up to 100 °C/min (depends on furnace) | | |
| Temperature precision | 0.01 °C | | |
| Sample robot | 42 (optional) | | |
| DSC | | | |
| DSC-Sensors | E/ K/ S/ B/ C | | |
| DSC-Resolution | 0.3/ 0.4/ 1/ 1.2 μW | | |
| Calorimetric sensitivity | approx. 4/ 6/ 17.6/ 22.5 μW | | |
| DTA | | | |
| DTA-Resolution | 0.05 nV | | |
| Sensitivity | 1.5 μV/mW | | |
| DTA-Measuring ranges | 250 / 2500 μV | | |





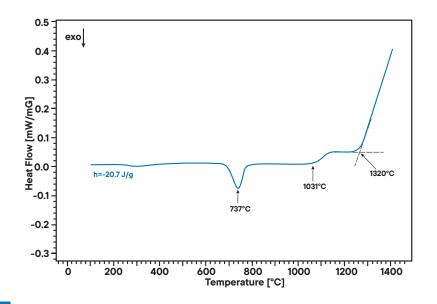
Applications

Steel (Low-Alloyed Steel)



The specific heat flow rate of a low-alloyed steel sample was measured by HDSC. At 734 °C a change in crystal structure from body-centered to face-centered cubic and a transition in magnetic properties from ferromagnetic to paramagnetic were observed. The melting point was detected at 1411 °C and the liquidus temperature at 1473 °C. All peaks are reversible and appear in the cooling segment as well. The phase transition back to the ferromagnetic state occurs at 637 °C and the crystallization range extends from 1454 °C to 1436 °C.

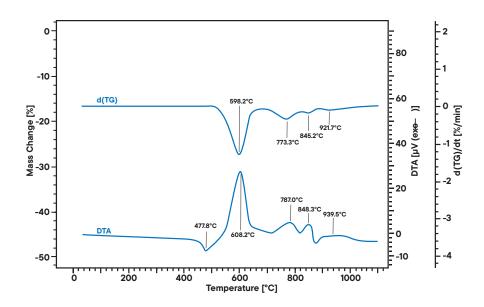
DSC DTA powder measurements of ferrites



The components used for production of magnetic ferrites are ZnO, Fe₂O₂ and Cr₂O₃. The Chrome oxide is added for modification of magnetic and electric properties. At 735°C the powder forms a mixed ferrite with a spinal structure (exothermal reaction: -20.6 J/g). Above 1034°C and 1321°C the heat flow changes into the endothermic direction due to melting of different phases. The LINSEIS HDSC L62 with type S measuring sensor provides a very stable baseline with an extremely low noise level up to 1600°C. This high sensitivity is essential to perform exact reaction enthalpy measurements and evaluations.

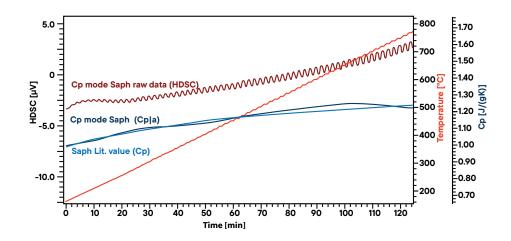


DSC Analysis of Talcum



Talcum $(Mg_3(OH)_2[Si_2O_5]_2)$ is a mineral that consists of hydrated magnesium silicate. It is used for the production of steatite bodies used as isolators with high resistance and a low dielectric loss factor. Its impurities (chlorite, carbonates) can be determined and detected using DSC. The measurement shows the dehydroxylation of chlorite that appears at 608°C and 848°C as endothermal DSC signals. At 768°C the removal of CO2 can be observed when the contained carbonates decompose into their oxides, releasing the CO2. Finally the dehydroxylation of talc can be seen at 937°C as an endothermal peak as well.

Modulated Cp determination



For highest possible accuracy of Cp, the LINSEIS HDSC allows the usage of modulated heating rate temperature profiles. This method causes a continuous change in heat flow of the sample and the system can monitor the heat uptake much better than with a linear heating profile. The deviation from the literature value is much smaller than with linear DSC runs. The modulated heat flow signal (dark red) leads to a significant better Cp resolution (dark blue) that is only slightly different from the literature (bright blue) over the full temperature range. The bright red curve shows the modulated heating profile.



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