

THERMAL ANALYSIS & THERMOPHYSICAL PROPERTIES

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LINSEIS MESSGERÄTE GmbH



Claus Linseis Managing Director

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

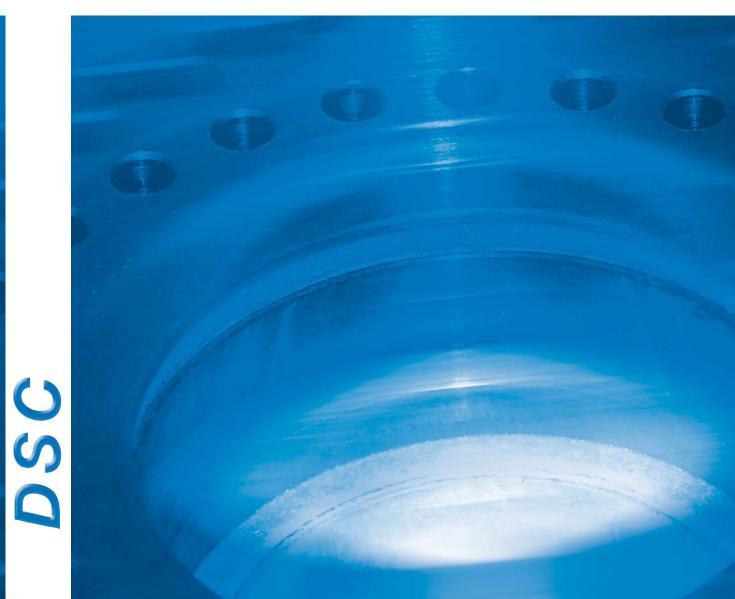
We are driven by innovation and customer satisfaction.

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

The LINSEIS business unit of thermal analysis is involved in the complete range of thermo analytical equipment for R&D and quality control in sectors such as polymers, chemical industry, inorganic building materials as well as environmental analytics. In addition, Thermophysical properties of solids, liquids and smelts can be analyzed. LINSEIS Corp.thrives for technological leadership. We develop and manufacture thermo analytic and Thermophysical testing equipment to the highest standard and precision. Do to our innovative drive and ultimate precision, we are a leading manufacturer of Thermal Analysis equipment.

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

Differential Scanning Calorimetry (DSC) is most popular thermal analysis technique it measures endothermic and exothermic transitions as a function of temperature - Endothermic = heat flows into a sample - Exothermic = heat flows out of the sample The instrument is used to characterize polymers, pharmaceuticals, foods/biologicals, organic chemicals and inorganics. Transitions measured include Tg, melting, crystallization, curing and cure kinetics, onset of oxidation and heat capacity.



Differential Scanning Calorimeter – DSC



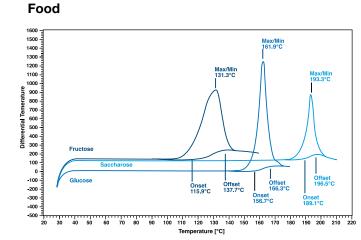
The DSC PT10 comprises the advantages of latest technology, highest resolution and a robust easy to use instrument design. The measurement principle of heat flux allows ultimate measurement precision. The sensor has a very high resolution and can be calibrated very accurately.

The optional software controlled gas box enables Oxidation Induction Time (OIT) measurements. The Kinetic software and many other features provide the perfect solution for any calorimetric experiment.



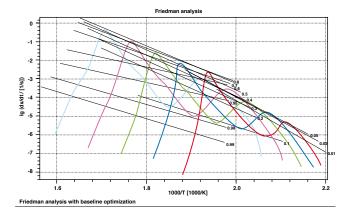
DSC PT 10

Temperature range: Heating and cooling rates: Resolution: Cooling: Gas control box: Atmosphere: -150 up to 700°C 0.01 up to 100°C/min 0.125 uW Air, LN2, intracooler up to 4 gases inert, oxid., red.



The three evaluated substances (Fructose,Glucose and Saccharose) show distinctive melting points. These melting points can be precisely determined by means of Differential Scanning Calorimetry (DSC). For this the analytical method is frequently used for the determination of unknown substances. Even mixtures with identical molecular weight such as Fructose and Glucose can thus be recognized.

Thermoplasts



PolyEthylenTherephtalat (PET) shows a significant endothermic glass point at about 77°C, which is quite special for partly crystalline thermoplasts. The relation between the exothermal cold crystallization at 131°C and the endothermic melting peak is a measure for the degree of crystallization of the material. In the case of (PET) the crystalline part is very small which results in a good transparency of the material.

Simultaneous TGA-DTA/DSC measures both heat flow and weight changes in a material as a function of temperature or time in a controlled atmosphere. Simultaneous measurement of these two material properties not only improves productivity but also simplifies interpretation of the results. The complimentary information obtained allows differentiation between endothermic and exothermic events which have no associated weight loss (e.g., melting and crystallization) and those which involve a weight loss (e.g., degradation).



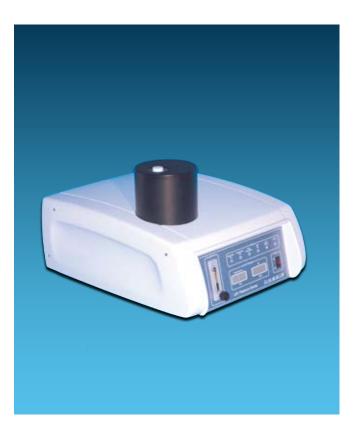
Simultaneous Thermal Analysis (STA) – TGA – DTA – DSC

STA PT1000

The LINSEIS STA PT1000 is a top loading Thermobalance, which offers a highly user-friendly design. Even at a sample weight of up to 10g the Tare is done electronically. The specially designed furnaces allow fast heating and cooling rates as well as a highly precise temperature control. Exchanging the different TGA, DTA or DSC measuring systems is only a question of minutes.

The STA PT 1000 and STA PT1000 HiRes combine both, the sensitivity of a Thermobalance and true Differential Scanning Calorimeter. Several different TG, TG-DTA and TG-DSC sample holders can be used to determine different reaction- and transition temperatures, enthalpies and specific heat. Thus the system can be perfectly adjusted for any type of application.

Because of the vacuum tight design of the instrument, static and dynamic atmospheres are possible. In addition a gas control box and a vacuum pump can be connected optionally.



STA PT 1000

	STA PT 1000	STA PT 100
Temperature range:	RT up to 1000°C	RT up to 10
Sample mass:	10g	5g
Resolution:	0.5µg	0.1µg
Measuring system:	E/K/S	E/K/S
Atmosphere:	inert, oxid., red., vac.	inert, oxid.,
Vacuum:	10E ⁻² mbar	10E ⁻² mbar
Pressure:		E/K/S
Sample carriers:	TG – DTA/DSC	
DSC measuring system:	E/K/S	

00 HiRes

000°C

, red., vac. rTG – DTA/DSC

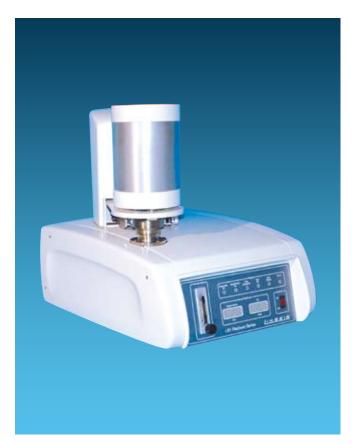


The STA PT1600 is the high end Simultaneous Thermobalance from LINSEIS. The system offers unparalleled TG and DSC resolution in combination with highest vacuum capabilities and TG drift stability. The system is modular due to five exchangeable furnaces, many different measuring systems and crucibles. The coupling ability and many other optional accessories guarantee the perfect setup for whatever application.

The STA PT 1600 and STA PT1600 HiRes combine both, the sensitivity of a Thermobalance and true Differential Scanning Calorimeter. Several different TG, TG-DTA and TG-DSC sample holders can be used to determine different reaction and transition temperatures, enthalpies and specific heat. Thus the system can be perfectly adjusted for any type of application.

Because of the vacuum tight design of the instrument, static and dynamic atmospheres are possible. In addition a gas control box and a vacuum pump can be connected optionally.

The evolving gases can be analyzed with our integrated QMS, FTIR or In-Situ EGA coupling options.



STA PT 1600

Temperature range

Sample mass Resolution: Measuring system: Atmosphere: Vacuum: Pressure: Sample carriers: DSC measuring system:

STA PT 1600

-150 up to 500°C RT up to 1400/1600/1750°C RT up to 2000/2400°C 25g 0.5μ g E/K/S/B Inert, oxid., red., vac. $10E^{-5}$ mbar optional 2/5 bar TG – DTA/DSC E/K/S/B

STA PT 1600 HiRes

-150°C up to 700°C RT up to 1500°C/1650°C

5g 0.1µg E/K/S/B Inert, oxid., red., vac. 10E⁻⁵ mbar

TG – DTA/DSC E/K/S/B





The LINSEIS L81 is designed as a top loading Thermobalance, providing excellent drift stability, a robust design and highest TG and DSC/DTA reproducibility. All measuring systems are easily user exchangeable to ensure quick and simple system handling. The broad selection of furnaces (optional turntable) ranging from 150°C up to 2400°C and the large sample mass of up to 25g/3ml are unique features of this system.

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.

Because of the vacuum tight design of the instrument, static and dynamic atmospheres are possible. An optional gas control box and a vacuum pump can be connected.



TG/STA L81-I

Temperature range

	RT up to 1750°C
	RT up to 2000°C
	RT up to 2400°C
Sample mass	25g
Resolution:	1µg
Measuring system:	E/K/S/B
Vacuum:	10E⁻⁵mbar
Automatic evacuation:	optional
Gas control (optional):	up to 4 gases
Sample holders:	TG / TG-DSC / TG-DTA

-150 up to 500°C RT up to 1600°C

Differential Scanning Calorimetry (DSC) is most popular thermal analysis technique it measures endothermic and exothermic transitions as a function of temperature - Endothermic = heat flows into a sample - Exothermic = heat flows out of the sample Linseis offers a unique line of high temperature DTA and DSC systems.



High Temperature DSC / DTA

This is the most common thermal analysis method due to its wide range of information provided. The LINSEIS high temperature DTA/DSC is designed to deliver highest calorimetric sensitivity, short time constants and a condensation free sample chamber. These features guarantee superior resolution, baseline stability over the entire instrument lifetime. Thus providing a indispensable tool for material development, R&D and quality control.

The modular concept of the DSC and DTA systems allows the use of different furnaces with a temperature range from -150 up to 2400°C, different measuring systems for DSC and DTA as well as many different crucibles. The vacuum tight design enables quantitative enthalpy and Cp (Specific Heat) determination under cleanest atmospheres as well as under vacuum 10E-5mbar. Additionally the systems can be coupled to a MS or FTIR.



DSC/DTA PT 1600

High Temperature DSC – DSC PT1600

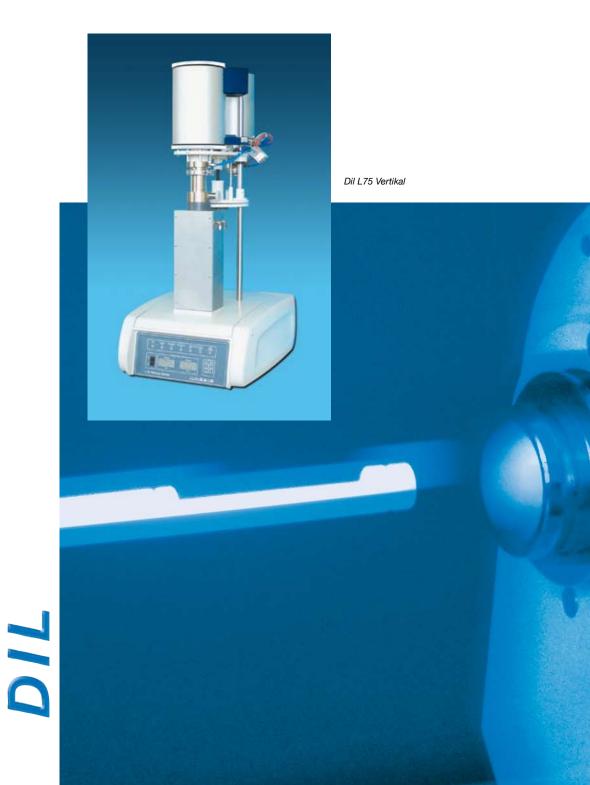
Temperature range:	-150 up to 500°C
	RT up to 1400°C
	RT up to 1500°C
	RT up to 1600°C
	RT up to 1650°C
	RT up to 1750°C
Sensors:	E/K/S/B
	DSC- Cp, DSC, DTA
Vacuum:	10⁻⁵ mbar
Atmospheres:	inert, oxid., red., vac.

High Temperature DTA – DTA PT 1600

Temperature range:	-150 up to 500°C
	RT up to 1400°C
	RT up to 1500°C
	RT up to 1600°C
	RT up to 1650°C
	RT up to 1750°C
	RT up to 2000°C
	RT up to 2400°C
Sensors:	different sensors
Vacuum:	10⁻⁵ mbar
Atmospheres:	inert, oxid., red., vac.



Dilatometry (DIL) is a technique in which a dimension of a substance under negligible load is measured as a function of temperature while the substance is subjected to a controlled temperature program in a specified atmosphere.



Dilatometry DIL



The LINSEIS Dilatometer series L76 PT combines user friendliness and high modularity to different applications with out¬standing performance in one system. The low cost dilatometer series is especially suitable for the ceramics and glass industry.





Temperature range: Resolution: Sample length: Sample diameter: Sample number: Atmosphere: Sample holders: RT up to 1000/1400/1600°C 1.25 nm/digit 25 / 50 mm 7 / 14 / 20 mm Single or differential measuring system inert, oxid. Fused Silica, Al₂O₄, Sappier

DIL L75PT Horizontal /Vertical operation mode

The high end pushrod dilatometer solves all measurement tasks when it comes to determining the thermal length change of solids, powders or pasts. The horizontal / vertical design as single or differential system provides the perfect solution for any type of expansion coefficient and material characteristics.

All models rely on a LVDT displacement sensor with almost indefinite resolution, furthermore the thermostacally controlled housing and perfect measuring design allow the highest precision and resolution measurements as well as long term drift stability.



DIL L75

Model

Temperature range:

Resolution: Sample length: Sample diameter: Sample number: Atmosphere: Sample holders:

L75 PT Horizontal

-150 up to 500°C RT up to 1000/1400/ 1600/2000°C 0.125 nm/digit 25/50 mm 7/14/20 mm Single or differential inert, oxid., red., vac. Fused Silica, Al₂O₃, Sapphire, Graphite

L75 PT Vertical

-150 up to 500°C RT up to 1000/1400/1600 1750/2000/2400/2800°C 0.125 nm/digit 25/50 mm 7/14 mm Single, differential or Quattro inert, oxid., red., vac. Fused Silica, Al_2O_3 , Sapphire, Graphite



The Laser Dilatometer is the next step in expansion measurements. The L75 Laser Dilatometer outperforms any conventional pushrod dilatometer by offering a 33 times higher resolution. The measurement principle is based on a Michelson interferometer thus eliminating all mechanical errors. This outstanding patented measurement principle can cope with latest high tech ultra low expansion (ULE) materials. LINSEIS has put much emphasis in the system design to secure a handling which is as easy as with our conventional type of dilatometers.

Sample preparation does not require any special preparation it is as simple as with any conventional dilatometer. Any kind of sample geometry can be measured as long as it fits into the maximum sample dimensions.

Applications

Highest precision expansion measurements of materials such as:

Carbon, Graphite, Composite materials, Glass, Alumina, Fused Silica, Substrates, semiconductor material, etc

Quality and entry control of materials with problematic expansions characteristics like, glass, bimetals, precision electronics components, etc.



DIL L75 Laser

Specifications

Method:

Sample length: Sample diameter: Resolution: Atmosphere: Vacuum: Sample preparation: Sample geometry:

Temperature range:

Laser Dilatometer "Michelson Prinzip" -180 up to 500°C RT up to 1000°C up to 20 mm up to 7 mm 0.3 nm inert, oxid., red., vac. 10E⁻⁵ mbar same as conventional dilatometer selectable



L78 RITA Quenching Dilatometer

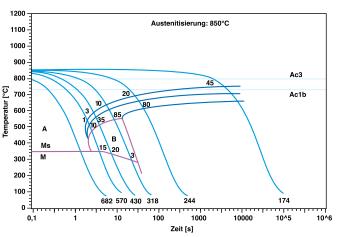
The Quenching Dilatometer L78 RITA is especially suitable for the determination of TTT, CHT and CCT diagrams. The special induction furnace enables heating and cooling speeds in excess of 400°C/s. The system complies with ASTM A1033.

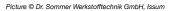
All critical parameters such as heat up and cool down speed, gas control and safety features are software controlled. The professional 32-bit software Linseis TA-WIN operates exclusively under the Microsoft© operation system. All routine (creation of CHT/CCT/TTT diagrams) and demanding applications are solved by the unique Software package which comes with the instrument.

Certainly export functions in ASCII-format as well as graphic output is possible.



L 78 RITA





L 78 RITA/Q Quenching Dilatometer

-100 up to 1600°C solid and hollow samples approx. 4 mm approx. 10 mm up to 200°C/s

L78 RITA/Q/D Deformation Dilatometer

-100 up to 1600°C solid samples approx. 5 mm approx. 10 mm up to 200°C/s 25 kN (0,01 - 125 mm)/s until 3 mm sample length

60 ms

Temperature range: Sample geometry: Sample Diameter: Sample Length: Heating / cooling rates: Deformation Force: Deformation Rate: Deformation Way: Minimum pause between two deformation steps:

Thermomechanical analysis (TMA) measures linear or volumetric changes in the dimensions of a sample as a function of time, temperature and force in a controlled atmosphere.



Thermomechanical Analysis – TMA



The Thermomechanical Analyzers TMA PT1000 and TMA PT1000 EM uniquely combine the flexibility of several measurement procedures under changing requirements. The instrument can measure expansion and deformation at highest precision.



TMA PT 1000

Model	TMA PT1000 EM	ΤΜΑ Ρ
Temperature range:	(-150-1000°C)	(-150-1
Cryo option:	available	availab
Force:	1/5.7N	1/5.7/2
Frequency:	1/5Hz	_
Resolution:	0.125nm	0.125n
Atmosphere:	inert, oxid	inert, o
	red., vac.	red., va
Vacuum:	10E⁻⁵mbar	10E-⁵m

TMA PT1000

(-150-1000°C) available 1/5.7/20N — 0.125nm inert, oxid red., vac. 10E⁻⁵mbar

🗾 ТМА РТ1600

The TMA PT1600 offers a broad temperature range (RT up to 1600°C) for all kinds of Thermomechanical investigations.

Model:	TMA PT1600
Temperature range:	-150 up to 500°C RT up to 1600°C
Force:	1N
Frequency:	1Hz
Resolution:	0.125nm
Atmosphere:	inert, oxid
	red., vac.
Vacuum:	10E⁵mbar



TMA PT 1600



When coupling a Thermal Analyzer with a Quadrupole Massspectrometer (QMS) or FTIR (Fourier Transformed Infrared Spectrometer) out gassing products can be determined and identified. The signal can then be time wise correlated with the signals received by the Thermal Analyzer.

With the optional Pulse – Analysis the outgasings can be quantified when using a QMS as well as a FTIR.



Gas Analysis/Couplings – EGA

🗾 Evolved Gas Analysis

The combination of a LINSEIS Thermal Analyzer with a FTIR (Fourier Transformed Infrared Spectrometer) is especially interesting in fields such as polymers, chemical and pharmaceutical industry. The coupling is more than the sum of the separate parts. Benefit from LINSEIS coupling knowledge and integrated hard- and software concept.

MS coupling	
Mass numbers:	100/200/300 amu
Detector:	Faraday and
	SEV (Channeltron)
Vacuum system:	Turbomolecular and
	Diaphragm pump
	(oil free)
Heating:	Adapter, heated capillary
	and QMS
Couplings:	DSC, TGA, STA, DIL by
	heated capillary
FTIR coupling	
Wave numbers: :	7500 cm ⁻¹ 370 cm ⁻¹
Resolution:	1 cm ⁻¹
Heating:	Transfer line and adap-
	ter: up to 230°C
Material transfer line:	PTFE (exchangeable)

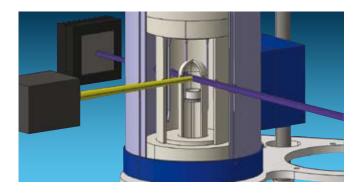


Main advantages

- Direct detection of gas compounds, not only mass numbers
- Real time in-situ measurement method
- No intrusion into the measurement system (as for extracting systems)
- No cooling of the analyzed gas
- No condensation of substances with high condensation temperatures
- No equilibrium shifts because of temperature changes
- No contamination of the sample gas in extracting lines
- Allows usage of principally all optical gas measurement systems (tested for FTIR, Raman, ELIF, among others)

Overview of proven Measuring Methods

• FTIR: Fourier Transform Infrared Spectroscopy Measurement of basic and trace gas components until



ppm range, for example H_2O , CO_2 , CO, H_2S , .. Polar Molecules are necessary

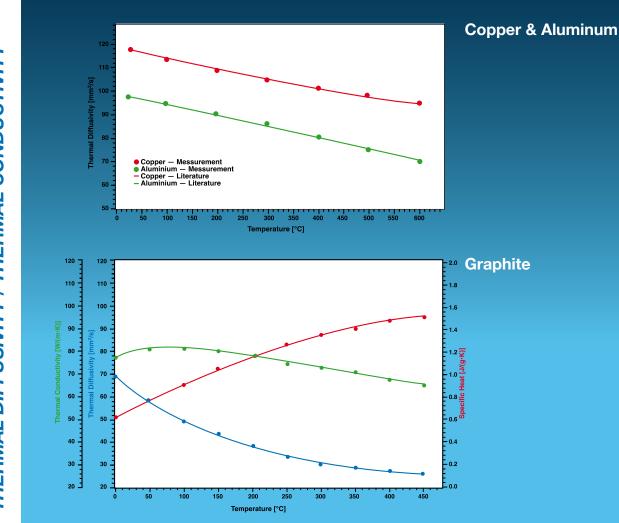
Raman-Spectroscopy

Measurement of basic gas components Also not polar molecules like $\rm H_{2}$ or $\rm N_{2}$ are measurable

• ELIF: Excimer Laser induced Fragmentation Fluorescence

UV-Laser-based Method for measuring of gaseous alkaline compounds (for example NaCl, NaOH, KCl, KOH) Also at 193 nm a entry through UV-Sapphire is possible

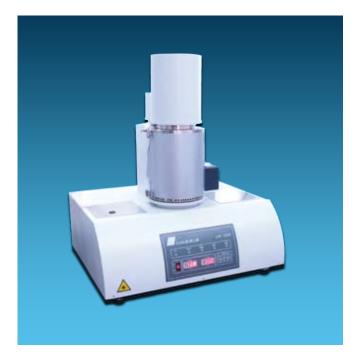
The sample is positioned horizontally on a sample robot, located in a furnace. The furnace is then held at a predetermined temperature. At this temperature the sample bottom is then irradiated with a programmed energy pulse (laser or xenon flash). This energy pulse results in a homogeneous temperature rise at the sample top. The resulting temperature rise of the sample top is measured by a high speed IR detector and thermal diffusivity values are computed from the temperature rise versus time data. The resulting measuring signal computes the thermal diffusivity, and in most cases the specific heat (Cp) data. If the density (r) is identified, the thermal conductivity can be calculated: $\lambda(T)=\alpha(T)\cdot Cp(T)\cdot p(T)$



APPLICATIONS THERMAL DIFFUSIVITY / THERMAL CONDUCTIVITY

Thermal Diffusivity / Thermal Conductivity – LFA/XFA





XFA 500/LFA 1000

LINSEIS offers a variety of instruments to measure the Thermal Diffusivity. The XFA 500 provides a cost effective solution for the temperature range RT up to 500°C. The highly modular design allows upgrade to the LFA 1000 the high end system whenever the measurement requires or the budget allows it. The LFA 1000 provides unbeaten sampling rates, up to 6 samples at the same measurement cycle, highest modularity, three different user exchangeable furnaces (-125 up to 1600°C) and two detectors as well as a high vacuum design (10E-5 mbar).

System Design

LINSEIS is offering an unparalleled modular system design for this Thermophysical properties Analyzer. It is possible to upgrade the temperature range (exchangeable furnaces/ measuring system) and the detector (InSb/MCT). This enables the user to start with a cost effective solution and upgrade the system whenever the budget allows or the measurement task requires it. Correspondence with International Standards.

The LINSEIS LFA and XFA operate in agreement with national and international standards such as ASTM E-1461, DIN 30905 and DIN EN 821.

Sample dimensions:

Max. Sample number:

Temperature range:

Vacuum: Atmosphere:

Measuring range:

Thermal Diffusivity: Thermal Conductivity: Pulse source: Pulse energy:

XFA 500

ø 10 mm, 0,1 to 6,0 mm thick ø 12.7 mm, 0,1 to 6,0 mm thick 10 x 10mm, 0,1 to 6,0 mm thick up to 6 samples, ø10,0 mm round up to 3 samples. ø25,4 mm round RT up to 500°C

10E⁻⁵ mbar inert, oxid. red., vak.

0,01 up to 1000 mm²/s 0,1 up to 2000 W/(m•K) Xenon Flash 10 J/Puls

LFA 1000

ø 10 mm, 0,1 to 6,0 mm thick ø 25,4 mm, 0,1 to 6,0 mm thick 10 x 10 mm, 0,1 to 6,0 mm thick up to 6 samples, ø10,0 mm round up to 3 samples. ø25,4 mm round -125 up to 500°C RT up to 1250°C/1600°C 10E⁻⁵ mbar inert, oxid. red., vak

0,01 up to 1000 mm²/s 0,1 up to 2000 W/(m•K) Nd: YAG Laser 25 J/Puls

Seebeck Effect & Electric Resistivity



The Thermal power, thermoelectric power, or Seebeck coefficient of a material measures the magnitude of an induced thermoelectric voltage in response to a temperature difference across that material. The thermal power has units of (V/K).

In recent years much interest has been shown in various methods of direct conversion of heat into electricity. Waste heat from hot engines and combustion systems could save billions of dollars if it could be captured and converted into electricity via thermoelectric devices. For this challenging application Linseis has developed a characteristic evaluating instrument for these materials and devices; the LSR -3 "LINSEIS - Seebeck & Electric Resistivity Unit".

Features

The LSR - 3 can simultaneously measure both Seebeck coefficient and electric resistance (Resistivity).

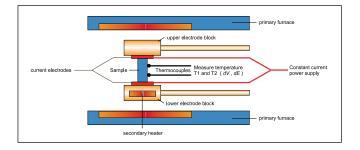
- Prism and cylindrical samples with a length between 6 to 23mm can be analyzed
- Wires and Foils can be analyzed with a unique measure ment adapter
- Three different exchangeable furnaces cover the temperature range from -100 up to 1500°C
- The design of the sample holder guarantees highest measurement reproducibility
- State of the art 32-Bit software enables automatic measurement procedures
- Measurement Data can be easily exported

Principles of Measurement

A sample of cylindrical or prism shape is vertically positioned between two electrodes. The lower electrode block contains a heater, while the entire measuring arrangement is located in a furnace. The furnace surrounding the measuring arrangement heats the sample to a specified temperature. At this temperature the secondary heater in the lower electrode block creates a set temperature gradient. Two contacting thermocouples then measure the temperature gradient T1 and T2. A unique thermocouple contact mechanism permits highest temperature accuracy measurements of the electromotive force dE at one wire of each of the two thermocouples.



LSR-3 Seebeck



The dc four-terminal method is used to measure the Electric Resistance. By applying a constant current (I) at both ends of the sample and measuring the change in voltage dV between one wires at each of the two thermocouple pares.

Temperature range :	-100°C up to 500°C and
	RT up to 800°C,
	RT up to 1100°C,
	RT up to 1500°C
Measurement method :	Seebeck coefficient:
	Static dc method
	Electric resistance: Four-terminal
	method
Specimen holder:	Sandwiched between two
	electrodes
Atmosphere:	inert, oxid., red., vac.
Sample size:	2 to 4 mm square or diameter \times 6
	to 23 mm long
Lead interval:	4, 6, 8 mm
Cooling water:	required



Software



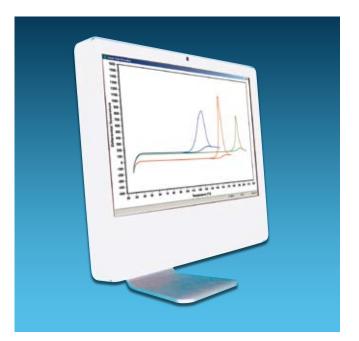
Features -Software:

- User-friendly
- Multi-methods analysis (DSC TG, TMA, DIL, etc.)
- Zoom function
- Online help menu
- Report generator
- Data export to MS Excel
- Export and import of data ASCII
- Program capable of text editing
- Data security in case of power failure
- Thermocouple break protection
- Repetition measurements with minimum parameter input
- Evaluation of current measurement
- Curve comparison up to 32 curves
- Storage and export of evaluations
- Programmable gas control
- Statistical evaluation package
- Smoothing of total or partial measurement
- Tangent intersection determination (automatic or manual)
- Free scaling

The information of a thermo analytical measurement can be increased when using the broad range of specialized Software.

Software Options

- Specific Heat determination (Cp)
- Rate Controlled Sintering (RCS)
- Calculated-DTA
- Quenching Dilatometer Software
- CHT / CCT / TTT Diagrams
- Thermo Kinetics Software





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Products: DIL, TG, STA, DSC, HDSC, DTA, TMA, MS/FTIR, In-Situ EGA, Laser Flash, Seebeck Effect Services: Service Lab, Calibration Service

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