



THERMAL TRENDS

6

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Dilatometry is a technique which measures the dimensional change of a substance as a function of temperature while the substance is subjected to a controlled temperature program.

Many international norms such as DIN 51045, ASTM E 831, ASTM E 228 and ASTM D 3386 describe this technique and the exact procedures in detail.

LINSEIS Dilatometers L75H (horizontal), and L75V (vertical) provide a powerful tool for the determination of the thermal expansion and expansion coefficient (CTE).

Further application examples are the evaluation of sintering processes of ceramics, metals and powder metals, the dimensional changes during chemical reactions (Oxidation) and phase changes of solid materials.

As a unique feature LINSEIS offers its range of Dilatometers either in horizontal or vertical mode of operation to provide the perfect solution for every application and budget.



Picture: Dilatometer L75 PT1600

Dilatometers are typically used in:

- Glass industry
- Ceramics industry
- Sintering of high tech ceramics
- Aerospace industry
- Metal/powder industry
- New material research
- Automotive industry
- Polymer industry

Dilatometers are frequently used for R&D and Quality Control of solids, liquids, powders and pastes to determine their:

- Linear thermal expansion (ΔL)
- Sinter-temperatures and sinter-steps
- Determination of glass transition (T_g)
- Phase changes
- Optimization of burning processes
- Determination of thermal expansion coefficient (CTE)
- Volume changes
- Rate controlled sintering (RCS)

The entire range of LINSEIS Dilatometers enables the perfect choice for any application.

LINSEIS L75 series are available in horizontal as well as vertical (Zero – Friction) mode of operation.

It offers a broad temperature range, many different sample holders, operation in vacuum or a controlled oxidizing and reducing atmosphere, while maintaining the highest accuracy and ease of use.

Measurement system:

All measuring systems are manufactured to the highest standards and equipped with an LVDT sensor which provides the maximum precision, repeatability, and accuracy.

Our Dilatometers have significantly benefited from the extensive research undertaken from the patented LINSEIS LASER Dilatometer.

Automatic pressure control:

The contact pressure can be continuously varied between 10 and 999mN depending on the application.

This feature continuously adjusts the contact pressure throughout expansion and/or shrinkage of the sample.

Vacuum atmosphere:

The vacuum tight construction (up to $10E-5$ mbar) of the L75 series permits measurements in the purest gas atmospheres.

This feature is essential in preventing unwanted effects due to sample oxidization.

Integrated DTA signal:

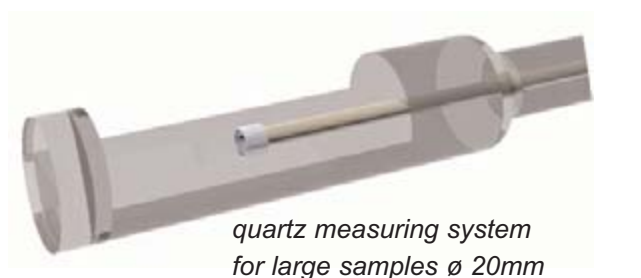
All LINSEIS L75 series dilatometers are optionally equipped with the DTA evaluation feature.

This provides the user with valuable additional endo- and exothermic sample information.

Special Dilatometers & Accessories:

LINSEIS manufactures a broad range of exceptional Dilatometers and accessories. Systems can be specifically designed to meet a broad range of unique applications.

Please call or visit our Webpage for more information.



*quartz measuring system
for large samples \varnothing 20mm*



*Al_2O_3 measuring system
standart*



*Al_2O_3 measuring system
contact free*



*quartz measuring system
 \varnothing 7/12 mm*



Adapter for powders and pastes

Software:

All LINSEIS thermo analytical instruments are PC controlled.

The individual software modules exclusively run under Microsoft® Windows® operating systems.

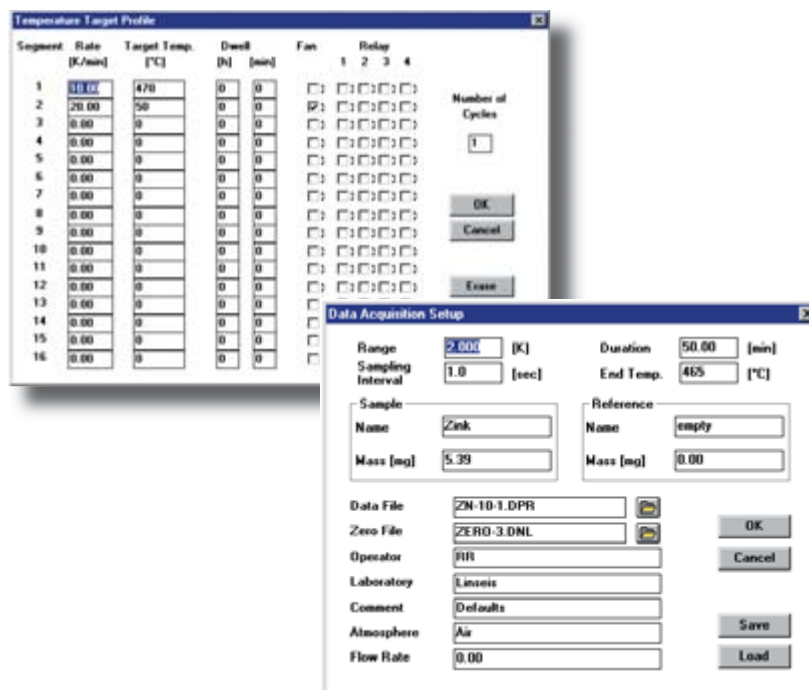
The complete software consists of 3 modules: temperature control, data acquisition and data evaluation.

The 32 bit software incorporates all essential features for measurement preparation, execution, and evaluation of a Dilatometer run.

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 measurement
- 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
- 1st and 2nd derivation
- Programmable gas control
- Statistical evaluation package
- Automatic axis re-scaling



DIL Features:

- Rate Controlled Sintering (RCS) software
- Interchangeable Thermocouples for various atmospheres
- Sinter process evaluation
- Glass transition and softening point evaluation
- Softening point determination and system shut down
- Linear thermal expansion evaluation
- Several system correction features
- Automatic zero point adjustment
- Auto-scheduler for up to 16 uninterrupted runs

Options:

- Automatic gas control
- Closed loop water cooler
- Water bath thermostat
- Gas humidity generator
- Two stage Rotary pump (10E-3 mbar)
- Turbo molecular pump (10E-5 mbar)

Horizontal - Dilatometer Furnaces:

Temperature	Type	Element	Atmosphere	TC-Type
-150 – 500°C	L75/264	Thermo coax	inert, oxid., red., vac.	Type K
RT – 1000°C	L75/220	Kanthal	inert, oxid., red., vac.	Type K
RT – 1400°C	L75/230	Kanthal	inert, oxid., red., vac.	Type S
RT – 1600°C	L75/240	SiC	inert, oxid., red., vac.	Type S
RT – 2000°C	L75/260	Graphite	inert, red., vac.	Type C

Vertical - Dilatometer Furnaces:

Temperature	Type	Element	Atmosphere	TC-Type
-150 – 500°C	L75/264	Thermo coax	inert, oxid., red., vac.	Type K
RT – 1000°C	L75/220	Kanthal	inert, oxid., red., vac.	Type K
RT – 1400°C	L75/230	Kanthal	inert, oxid., red., vac.	Type S
RT – 1600°C	L75/240	SiC	inert, oxid., red., vac.	Type S
RT – 1750°C	L75/250	Pyrox/MoSi2	inert, oxid., red., vac.	Type B
RT – 2000°C	L75/260	Graphite	inert, red., vac.	Type C
RT – 2400°C	L75/270	Graphite	N2/Vac.	Pyrometer

Model	L 75 Horizontal	L 75 Vertical
Temperature Range	L75H X LT (-150 – 700°C) L75H X 1000°C L75H X 1400°C L75H X 1600°C L75H X 2000°C -	L75V X LT (-150 – 700°C) L75V X 1000°C L75V X 1400°C L75V X 1600°C L75V X 1750°C L75V X 2000°C L75V X 2400°C

X = S = single Dilatometer
X = D = double Dilatometer

Technical Data:

• Temperature range	-150°C up to +2400°C
• Sample length	up to 50 mm
• Sample Ø	7 or 12 mm
• Measuring range	100 µm up to 5000 µm
• Resolution	0,125 nm/digit
• Gas	possible, oxid., red.
• Vacuum	10E-5 mbar
• Automatic pressure	0 - 1000 mN
• Calibrations standards	Al2O3, Sapphire, etc.

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 Platinum STA (simultaneous thermal analysis) can be used to determine simultaneous changes of mass (TG) and caloric reactions (DSC) of a sample in the temperature range $-150 \dots 1000^{\circ}\text{C}$.

The unique characteristics of this product are high precision, high resolution and long term drift stability.

The STA Platinum was especially developed to meet the high demands of the high temperature as well as low temperature applications.

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 is an indispensable tool for every thermo analytical user.



Picture: STA PT1000

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

TG:

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

**DSC:**

- 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



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.

TG – Features

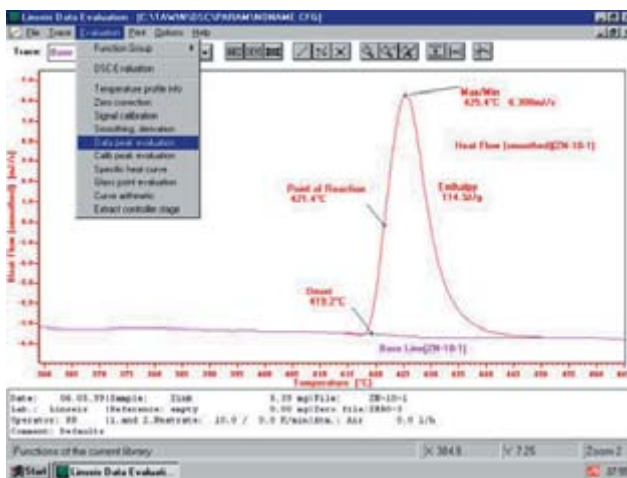
- Mass change as % and mg
- Rate Controlled Mass Loss
- Evaluation of mass loss
- Residue mass evaluation

DSC – 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 measuring procedures

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 measurement
- 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



Measurement system

All measurement systems are easily exchangeable to ensure user-friendly and quick system handling.

Vacuum and controlled atmosphere

The balance design provides 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.

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 L81 – Platinum STA:

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.

Specifications

Platinum 1000

Platinum 1600

Temperature features

Temperature range	RT – 1000°C	-150 ... 1750°C
Temperature accuracy	+/- 0.25°C	+/- 0.3°C
Temperature reproducibility	+/- 0.2°C	+/- 0.3°C
Heating rate	100K/min	50K/min
Cooling rate 1000 – 50°C	< 15 min	< 35 min

Balance

Sample weight (max.)	10g	25g
Resolution	0.5 µg	0.5 µg
RMS noise	< 1µg	< 1µg

Accessories

DSC

DSC resolution	0.3, 0.4, 1µW	0.3, 0.4, 1µW
DSC RMS noise	4, 6, 17.5µW	4, 6, 17.5µW
DSC sensor type	E, K, S	E, K, S, B

DTA

DTA Sensitivity	0.05µV	0.05µV
Coupling	MS/FTIR	MS/FTIR

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 Platinum STA (simultaneous thermal analysis) can be used to determine simultaneous changes of mass (TG) and caloric reactions (DSC) of a sample in the temperature range $-150 \dots 1750^{\circ}\text{C}$.

The unique characteristics of this product are high precision, high resolution and long term drift stability. The STA Platinum was especially developed to meet the high 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 is an indispensable tool for every thermo analytical user.



Picture: STA PT1600

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

TG:

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

DSC:

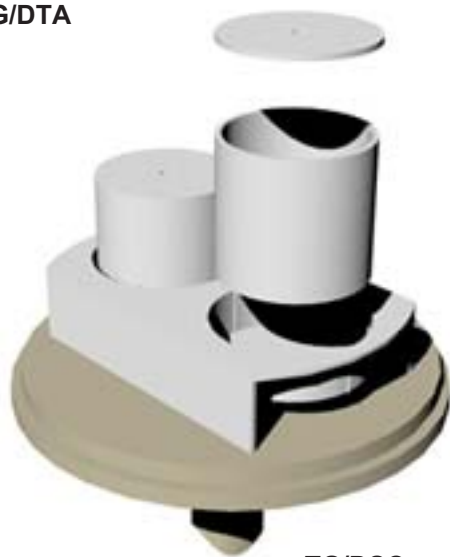
- Enthalpy, melting energy
- Specific heat
- Glass point
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TG/DTA



TG



TG/DSC



TG/DSC/Cp

Measurement system

The Linseis STA 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 ($10E-5$ mbar), 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 L81 Platinum can be equipped with four easily exchangeable furnaces. This allows measurements in the temperature range $-150 \dots 1750^{\circ}\text{C}$.

Stable and reproducible baselines are achieved due to the specially designed heating elements and system setup.

Sample holders

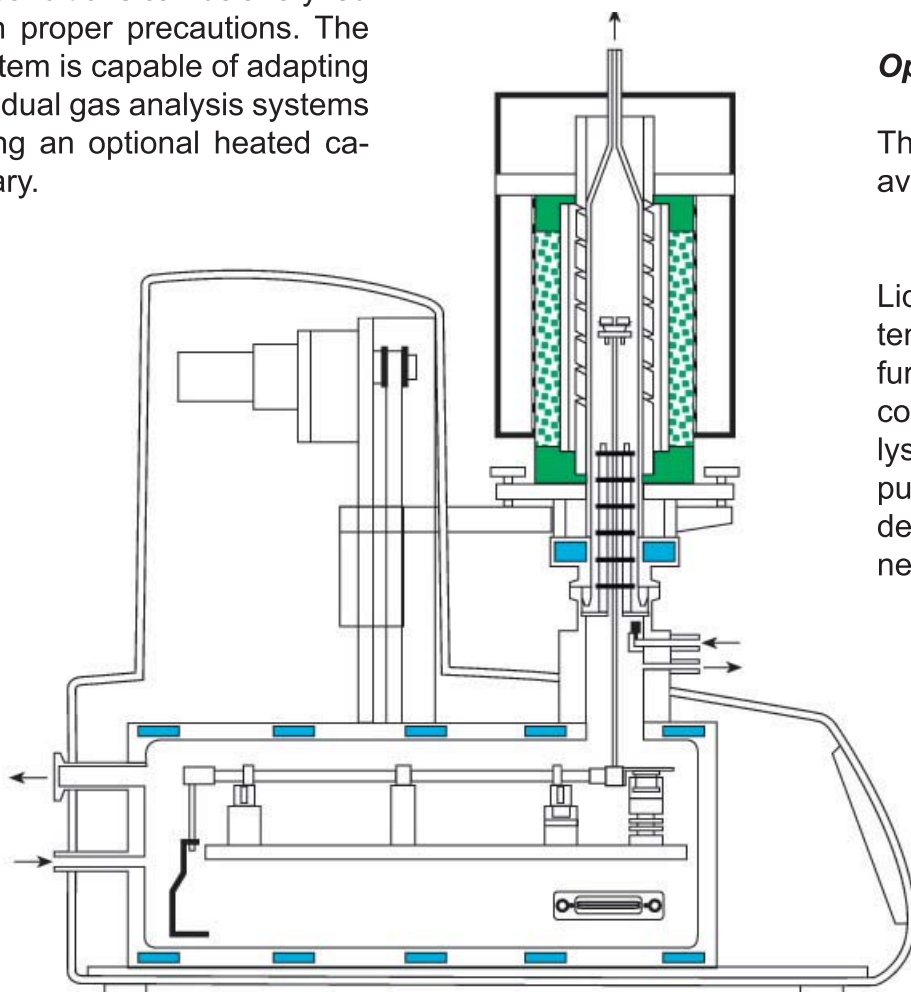
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Large amounts of overall sample can be measured to distinguish reactions from lesser components. This combination enables a very high resolution while assuring the best possible sample reactions.

Options

The following options are available for the Platinum STA:

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.



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.

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Features -Software

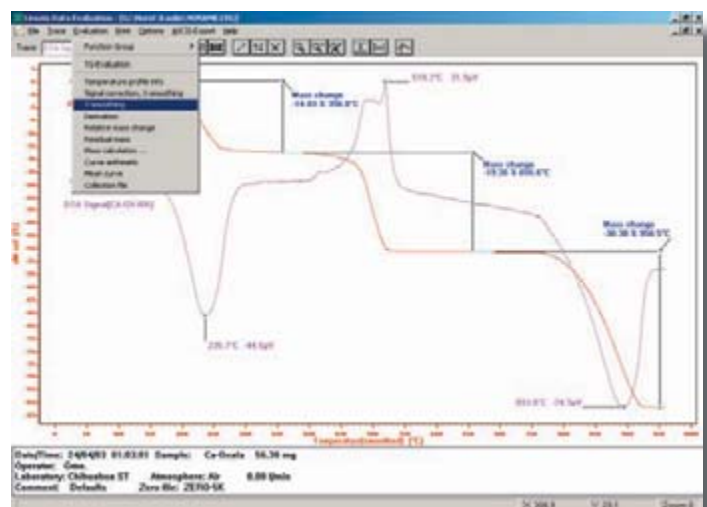
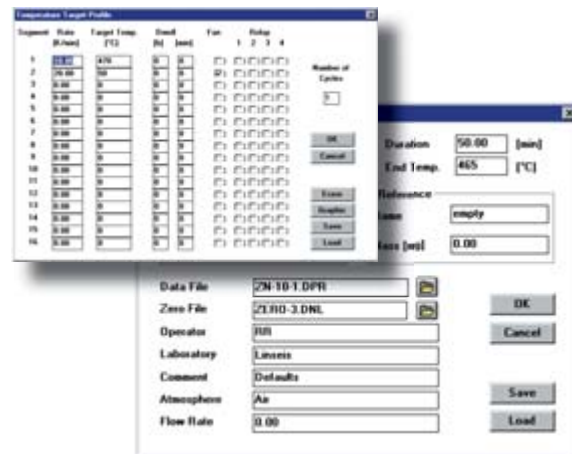
- Program capable of text editing
- Data security in case of power failure
- Thermocouple break protection
- Repetition measurements with minimum parameter input
- Evaluation of current measurement
- Curve comparison up to 32 curves
- Storage and export of evaluations
- Export and import of data ASCII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

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

DSC – 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 measuring procedures



Furnace program

TemperatureType	Element	Atmosphere	TC-Type
-150 – 700°C L81/264	LN2	inert, oxid., red., vac.	K
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 L81/260	MoSi2	inert, oxid., red., vac.	B

Specifications**Platinum 1000****Platinum 1600****Temperature features**

Temperature range	RT – 1000°C	-150 ... 1750°C
Temperature accuracy	+/- 0.25°C	+/- 0.3°C
Temperature reproducibility	+/- 0.2°C	+/- 0.3°C
Heating rate	100K/min	50K/min
Cooling rate 1000 – 50°C	< 15 min	< 35 min

Balance

Sample weight (max.)	10g	25g
Resolution	0.5 µg	0.5 µg
RMS noise	< 1µg	< 1µg

Accessories**DSC**

DSC resolution	0.3, 0.4, 1µW	0.3, 0.4, 1µW
DSC RMS noise	4, 6, 17.5µW	4, 6, 17.5µW
DSC sensor type	E, K, S	E, K, S, B

DTA

DTA Sensitivity	0.05µV	0.05µV
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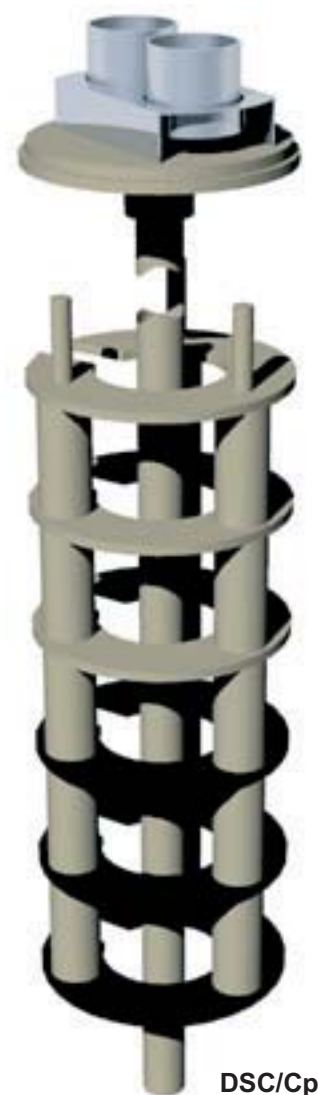
Coupling	MS/FTIR	MS/FTIR
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Differential Scanning Calorimetry, DSC, is well-known as a fast and easy-to-operate thermo analytical measuring technique according to industrial standards such as ISO, ASTM, DIN, ... for research & development, quality management, failure analysis, and process optimization.

Heat Flux DSC

A technique in which the temperature of the sample unit, formed by a sample and reference material, is varied in a specified program and the temperature difference between the sample and the reference material is measured as a function of temperature.

Many industrial standards provide a guideline of how to calibrate the equipment for specific material-, product- and characteristic-referred applications, result evaluation and interpretation.



Picture: DSC PT1600

The DSC measurement principle is used typically for:

- Research & Development
- Quality control
- Quality assurance
- Process optimization
- Error analysis

The main user groups of Differential Scanning Calorimetry (DSC) are:

- Powder Metallurgy
- High tech materials
- Ceramics
- And many more ...

The following characteristics can be determined by DSC measurements:

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

LINSEIS DSC PT1600 (Cp)

Features

- Temperature range -170°C up to +1750°C
- Different easy exchangeable furnaces
- Different easy exchangeable sensors
- Low temperature model with LN2 cooling or with Intercooler

The **DSC PT1600** was developed to specifically for the high – as well as low temperature range (-170 up to 1750°C).

For this broad temperature range a number of different exchangeable furnaces is available. Furthermore emphasis was placed on a stable baseline and high reproducibility.

Due to its unique features the DSC PT1600 is an indispensable tool for quality control and R&D.

The Instrument can be equipped with a number of different exchangeable furnaces, different measuring systems and numerous different crucibles. Measurements under vacuum, inert, reduced and oxidized atmospheres are possible.

The vacuum tight construction (10E-5 mbar) allows quantitative measurements under cleanest gas atmospheres.

Measuring system

User-friendly exchangeable measuring systems such as DTA, as well as different DSC sensors (Type E, K, S, B) are available for the DSC PT1600. This allows the perfect choice for any application or atmosphere.

Options

- LN2 cooling system
- Turbo molecular pump (10E-5 mbar)
- Two stage rotary pump (10E-3 mbar)
- Different protection tubes
- Coupling with MS/FTIR

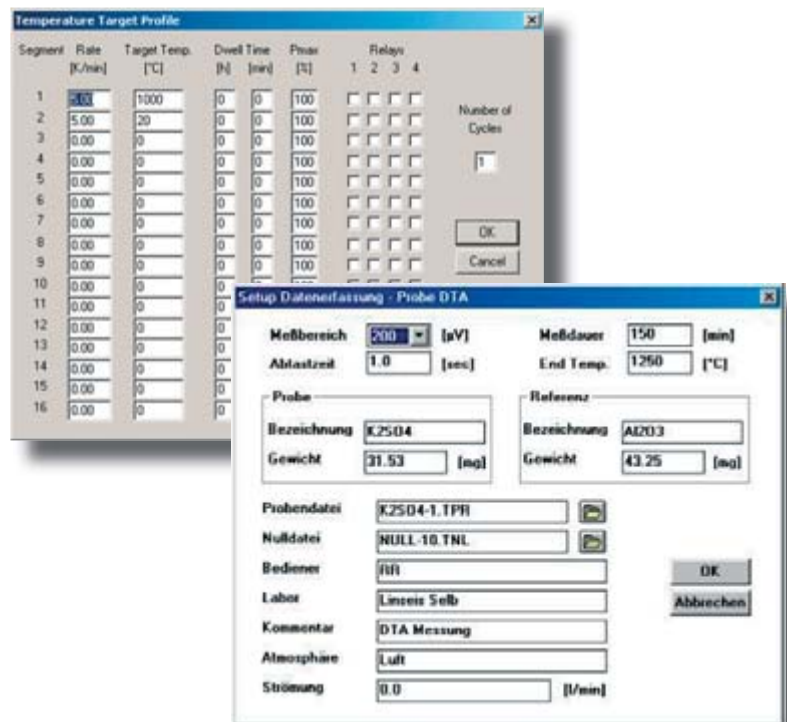
Software

All thermo analytical devices of LINSEIS 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 Linseis 32 – bit software encounters all essential features for measurement preparation, execution and evaluation with a DSC run, just like with other thermo analytical experiments.

Due to our specialists and application experts LINSEIS was able to develop this easy understandable and highly practical software.



Features

- Program capable of text editing
- Repetition measurements with minimum parameter input
- Evaluation of current measurement
- Curve comparison up to 32 curves
- Curve subtraction
- Multi-methods analysis (DSC TG, TMA, DIL, etc.)
- Zoom function
- 1. and 2. Derivative
- Complex peak evaluation
- Multipoint calibration for sample temperature
- Multipoint calibration for change of enthalpy
- Cp calibration for heat flow
- Storage and export of evaluations
- Export and import of data ASCII
- Data export to MS Excel
- Signal-steered measuring procedures

LINSEIS accessories

The versatility of the LINSEIS DSC is supported by the large selection of crucibles. Select for your application and samples the ideal crucible material, the best form and kind of sealing.

Crucibles made of metal; precious metal, graphite and oxide ceramics are available in different dimensions.

Aluminum crucibles can be sealed gas-tight in a handy locking press, so that samples can be protected from the influence of the Environment atmosphere and that gas splitting off from the samples is suppressed.

Specifications LINSEIS DSC systems

Temperature range	-170 ...1750°C*
Heating/Cooling rates	0,1 up to 50°C/min
Temperature accuracy	+/-0,5°C (substance calibration)
Time constant	7 s
Resolution	0,3, 0,4, 1µW**
RMS Noise	4, 6, 17.5µW**
Data acquisition rate	0,1 s up to 3600 s / data point
Atmospheres	N2, Argon, O2 etc., reducing and oxidizing
Measuring range	+/-250 ...+/-5000mW

*Different Furnaces

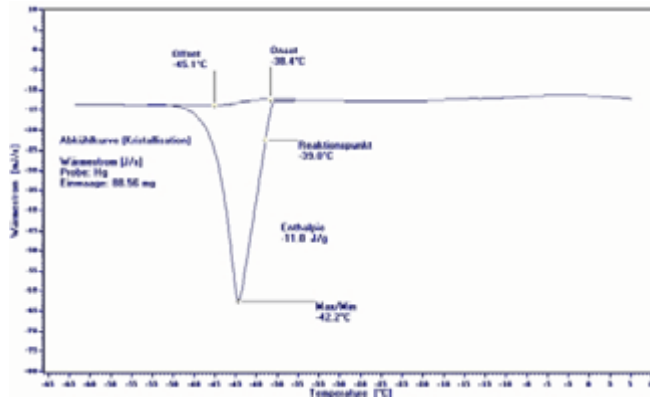
**Different Sensors

Calibrations material included

Calibration: recommended 6 month interval

Application examples for the DSC

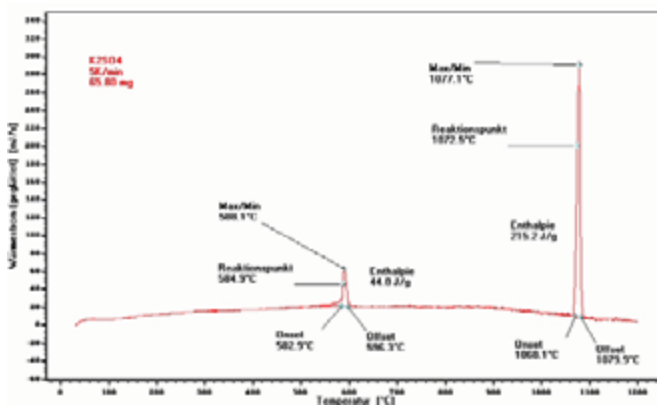
Picture 1, measurement of silversulfat



Silversulfat (AG2SO4)

Silversulfat (AG₂SO₄) changes its crystalline structure from a orthorhombic configuration to a hexagonal system at 424°C. The energy that is needed for this crystalline restructuring can be very good determined by means of the HDSC method (high temperature differential scanning calorimetry).

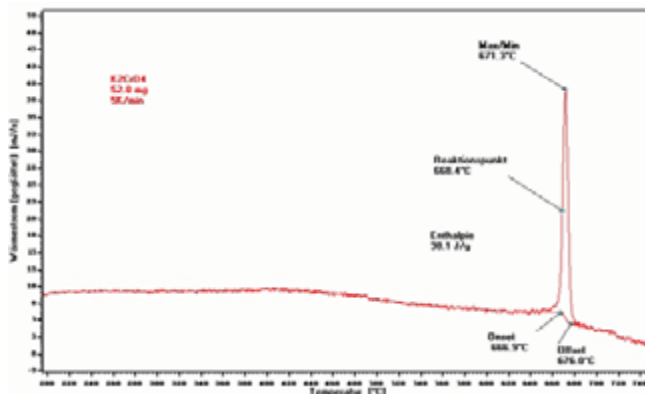
Picture 2, measurement of potassium sulfat



Temperature of potassium sulfat (K2SO4)

Potassium sulfat (K₂SO₄) is changing its crystal structure from a orthorhombic to a hexagonal system at 582°C. The energy that is needed for this restructuring can be determined with the HDSC method quantitatively (high temperature differential scanning calorimetry).

Picture 3, measurement of caliumchromat



Caliumchromat (K₂CrO₄)

The crystalline structure of K₂CrO₄ changes from a orthorhombic to a hexagonal structure at 665°C. This endothermal phase change can be evaluated by means of HDSC, relative to temperature, as well as relative to the energy, that is needed for the phase change.

TMA / DTMA the Method

Thermo mechanical analysis (TMA) easily and rapidly measures sample displacement (growth, shrinkage, movement, etc.) as a function of temperature, time, and applied force.

Traditionally, TMA is used to characterize linear expansion, glass transitions, and softening points of materials by applying a constant force to a specimen while varying temperature.

For expansion measurements, a probe rests on a sample on a stage with minimal downward pressure. Other constant force experiments include measurement of penetration, bending, tension, shrinkage, swelling, and creep (sample motion measured as a function of time under an applied load).

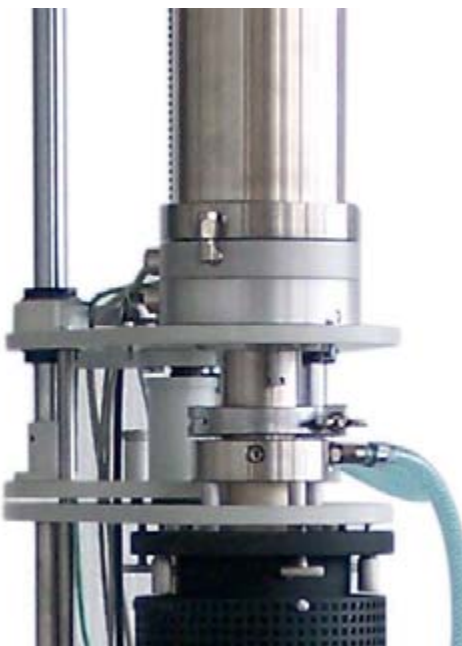


Picture: TMA PT100

In Dynamic TMA (DTMA), a known sinusoidal stress and linear temperature ramp are applied to the sample, and the resulting sinusoidal strain is measured.

Typical Applications

- Tension studies of the stress/strain properties of films and fibers
- Determination of softening behavior
- Glass transition temperatures and secondary transitions
- Phase change determination
- Determination of mechanical behavior under applied force
- Determination of expansion coefficient (dilatometry)
- Sintering behavior
- Volumetric expansion
- E modulus
- Slipping and friction resistance



The Concept

Sample Chamber

The easily accessible chamber is located in the center of the furnace. Both temperature and atmosphere can be controlled. In addition an optional digital mass flow controller is available for purge gas regulation.

Furnace

The TMA PT100 comes with a robust and reliable furnace. Its customized design enables rapid and cool down times and excellent heating rate control over the entire temperature range.

The Sensor

Every dimensional change of the sample is transmitted via the pushrod to the highly precise inductive transducer (LVDT sensor). Its precise and reliable response over the entire temperature range guarantees highest reproducibility of the TMA results.

Sample Holders

A broad range of sample holders is available for the TMA PT100. Hence the best method for testing can be selected for every application. Furthermore LINSEIS can certainly provide aid for special customer requirements.

Measurement systems



Automatic pressure control

The contact pressure can be 10mN and 1N depending on the application.

This feature continuously adjusts the contact pressure throughout expansion and/or shrinkage of the sample.

Integrated DTA signal

All LINSEIS TMA models are optionally equipped with the DTA evaluation feature.

This provides the user with valuable additional endo- and exothermic sample information.

Software

All LINSEIS thermo analytical instruments are PC controlled. The individual software modules exclusively run under Microsoft® Windows® operating systems.

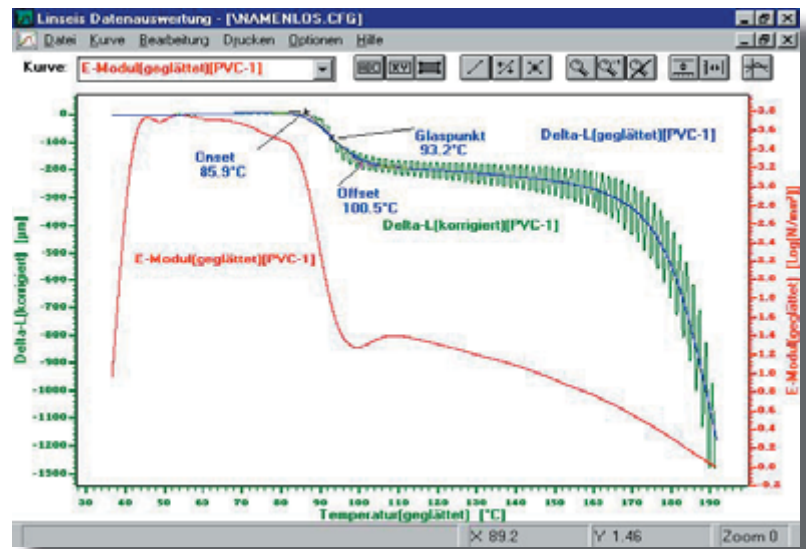
The complete software consists of 3 modules: temperature control, data acquisition and data evaluation.

The 32 bit software incorporates all essential features for measurement preparation, execution, and evaluation of a TMA/DTMA run.

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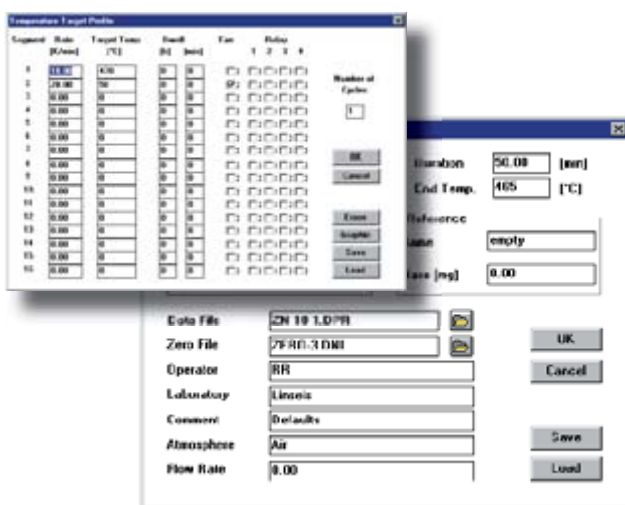
TMA / DTMA Features

- Rate Controlled Sintering (RCS) software
- E modulus
- Sinter process evaluation
- Glass transition and softening point evaluation
- Softening point determination and system shut down
- Linear thermal expansion evaluation
- Several system correction features
- Automatic zero point adjustment
- Auto-scheduler for up to 16 uninterrupted runs



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 measurement
- 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
- 1st and 2nd derivation
- Programmable gas control
- Statistical evaluation package
- Automatic axis re-scaling



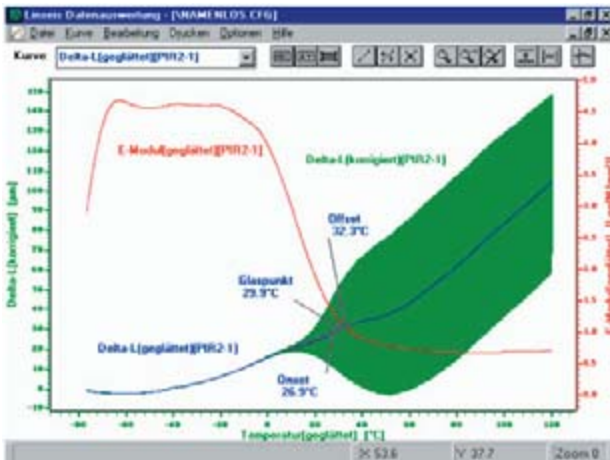
Elastomer

You can see an elastomer that was specifically developed for use at temperatures above 0 °C.

The glass point is at 29,9 °C.

If the temperature is further increased an additional dilatation of the material in the elastic range is visible.

The plastic range of the material is hereby not yet reached.

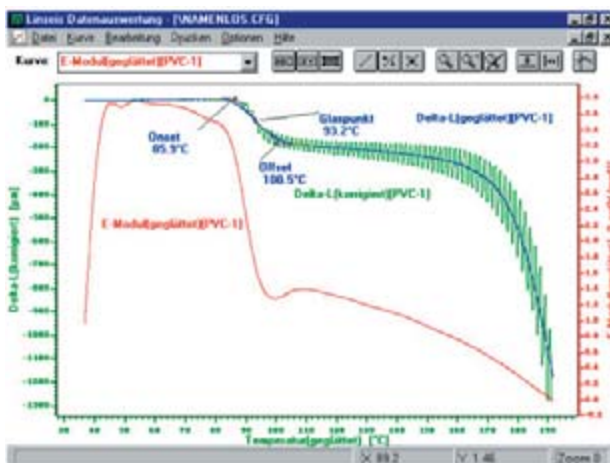


PVC sample

You can see the test run of a PVC sample starting from room temperature.

The glass point is at 93,2 °C.

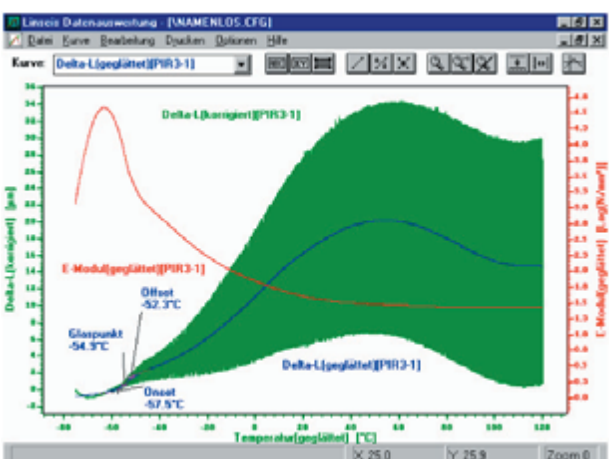
The elastic range starts from approx. 150°C. At higher temperatures the change into the plastic range is very good visible.



Silicon kautschuk

The evaluation of a silicon kautschuk is shown which was especially developed for use at low temperatures. The glass point is at -54,9 °C.

Furthermore the E-modules is shown over the complete temperature range and the mean value of the expansion. The change of length is up to about 50 °C an expansion (elastic range), and there after a change into the plastic range is visible.



	TMA-PT10	TMA-PT100	TMA-PT1600
Temperature Range	-30 up to +70°C	-150 up to 1000°C	RT up to 1600°C
Max. Sample size	50mm	30mm	50mm
Measurement precision	+/- 0,1%	+/- 0,1%	+/- 0,1%
Resolution	0,2nm	0,2nm	0,2nm
RMS Noise	3,5nm	3,5nm	3,5nm
Dynamic baseline drift	<1um	<1um	<1um
Force range	0,001 to 1 N	0,001 to 1N	0,001 to 1N
Force resolution	0,001 N	0,001 N	0,001 N
Frequency		0,01 to 1 Hz	Optional
Mass Flow Control	Optional	Optional	Optional
Atmosphere	Air	Inert, oxid. red.,vac.	Inert, oxid. red.,vac.

Operation Modes

Standard	Included	Included	Included
Stress/Strain		Included	Optional
Creep		Included	Optional
Stress Relaxation		Included	Optional
Dynamic TMA		Included	Optional

Optional

Dta feature	Optional	Optional	Optional
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ADVANCED TOOLS FOR THERMAL AGEING AND SAFETY ANALYSIS

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INTRODUCTION

Generally, all energetic materials evolve heat during decomposition. Processing, design, quality control, and operational applications all require an understanding of thermal hazards and an ability to predict safety limits and decomposition process in extended temperature ranges [1,2].

Several methods have been presented for predictions of the reaction progress of exothermic reactions under heat accumulation conditions [3]. However, because decomposition reactions usually have a multi-step nature, the accurate determination of the kinetic characteristics strongly influences the ability to correctly describe the progress of the reaction [4,5]. The use of simplified and conservative kinetic models for the assessment of runaway reactions leads to economic drawbacks, since they result in unnecessary large safety margins.

Applying the results obtained by :

- DTA (Differential Thermal Analysis)
- DSC (Differential Scanning Calorimetry)
- TG (Thermogravimetry)
- EGA (Evolved Gas Analysis MS or FTIR)

advanced numerical techniques such as AKTS-Thermokinetics and AKTS-Thermal Safety Software enable prediction of the reaction progress of materials in broad temperature range. In fact, numerical simulations are used to replace experiments in situations, which are not directly accessible to the experiment for timing or safety reasons.

ANALYSIS PROCESS

As a general rule, solid state reactions demonstrate profound multi-step characteristics as presented in figure 1. The assumption that the decomposition of an energetic material will obey a simple rate law is not often true. Using for example DSC data, the analysis process requires determination of the kinetic characteristics of the reaction.

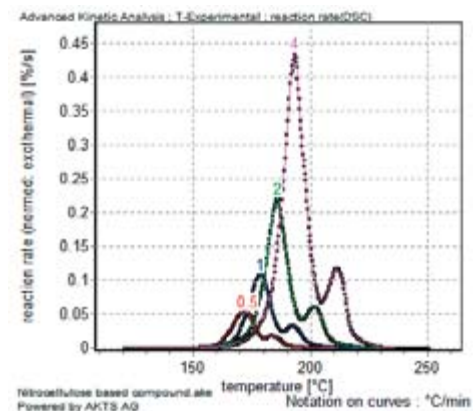


Figure 1: Analysis process. Normalized DSC-signals of a nitrocellulose based compound as a function of the temperature for a reaction involving at least two exothermic events. Experimental data are represented as symbols, solid lines represent the calculated signals. The values of the heating rate in °C/min are marked on the curves.

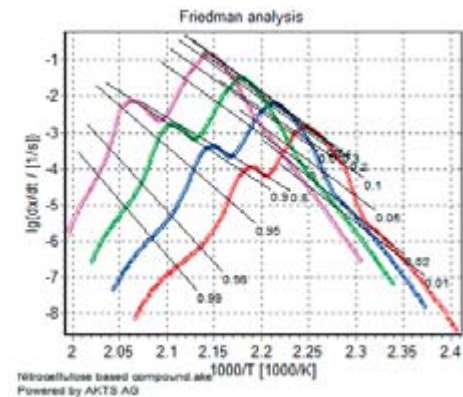


Figure 2: Friedman analysis of the examined material.

Friedman analysis, based on the Arrhenius equation, applies the logarithm of the conversion rate dx/dt as a function of the reciprocal temperature at different degrees of the conversion.

$$\ln \frac{dx}{dt} = \ln(A) - \frac{E}{RT_{i,j}} + \ln(f(x_i))$$

with i : index of conversion, j : index of heating rate, $f(x)$ the function dependent on the decomposition mechanism. As $f(x)$ is constant at each conversion degree x_i , the method is so-called 'isoconversional'.

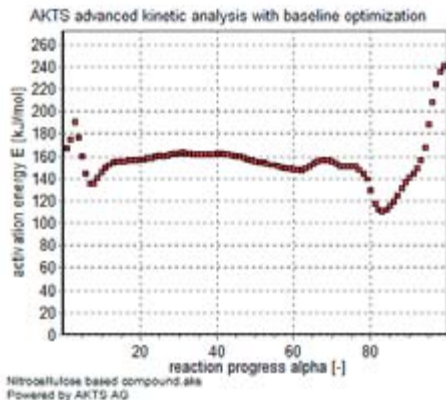


Figure 3: Activation energy as a function of the reaction progress for decomposition of the high energetic material (DSC closed crucibles).

The accurate determination of the kinetic parameters and optimization of the baseline which enable the correct fit of the experimental data is a prerequisite for prediction of the reaction progress under any new temperature profile (see figure 4).

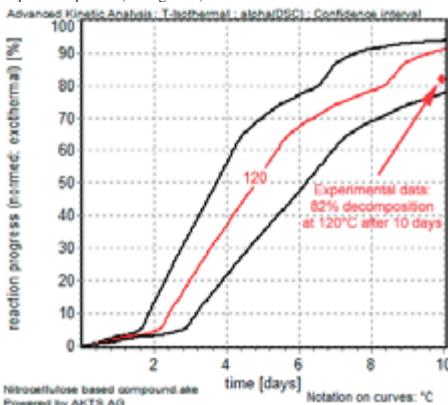


Figure 4: Prediction of the reaction extent (DSC, normalized signals) and confidence interval of a nitrocellulose based substance as a function of time under isothermal conditions ($T = 120^\circ\text{C}$). These values indicate that there is a 95% probability that the reaction progress after 10 days exposure at 120°C is greater than 77 and lower than 93%. These values are in good agreement with a subsequent measurement under isothermal conditions (82% decomposition at 120°C after 10 days).

TMR_{ad} SAFETY MARGINS & HEAT ACCUMULATION CONDITIONS

The calculated kinetic parameters can be subsequently employed to predict the reaction progress of the investigated samples under any given temperature mode. For chemical process safety adiabatic conditions are used for the prediction of the Time to Maximum Rate under adiabatic conditions (TMR_{ad}).

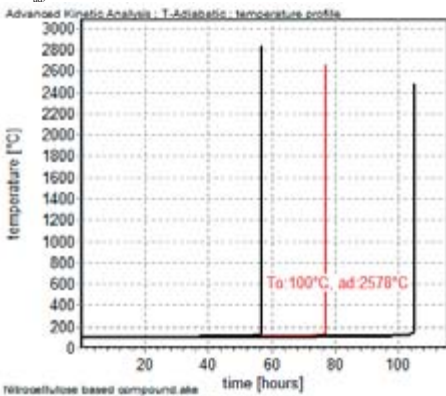


Figure 5: Adiabatic runaway curves showing the confidence interval for the prediction ($T_{\text{beg}}=100^\circ\text{C}$ and $\Delta T_{\text{ad}}=\Delta H/c_p=2578\pm 173^\circ\text{C}$).

The second field of application for numerical simulation techniques in process safety is the solution of partial differential equations as they are encountered in heat conduction problems. These problems arise when heat accumulation situations are to be analyzed [6].

$$\frac{dT}{dt} = \frac{\lambda}{c_p \rho} \left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} \right) \quad (\text{cartesian}) \quad \frac{dT}{dt} = \frac{\lambda}{c_p \rho} \left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} \right) \quad (\text{cylindrical})$$

Applications of Finite Element Methods (FEM) and accurate kinetic description enable the determination of the effect of scale, geometry, heat transfer (isolation), thermal conductivity and ambient temperature on the heat accumulation conditions. In fact, the assumption that it is safe to handle an energetic material at any temperature below the first appearance of an exothermic signal on the DSC curve can be often false. The highest safe temperature for handling any energetic material depends on several factors such as its size, shape, and previous thermal history. Due to insufficient thermal convection and limited thermal conductivity, a progressive temperature increase in the sample can easily take place, resulting in a thermal explosion. Safe operating conditions with tailored safety margins can be defined using numerical simulation. Presented examples illustrate a slow cook-off experiment and the calculations performed on pressed PBX.

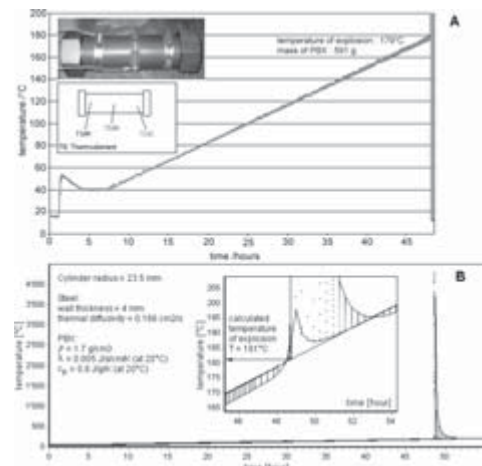


Figure 6: Slow cook-off experiments of pressed PBX based on 94% RDX (A) and simulation (B). As presented in the inset, the predicted temperature of explosion was 181°C . It is in good agreement with the slow cook-off experiments (179°C).

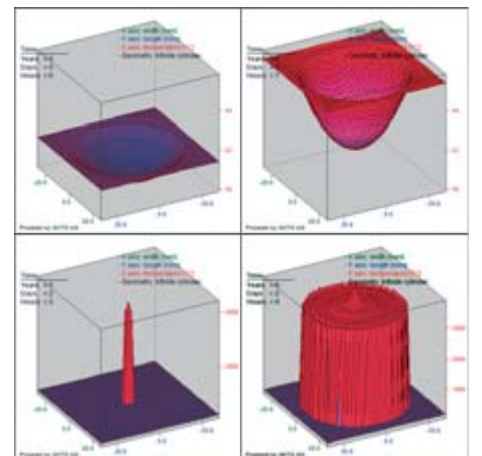


Figure 7: 3D calculations representing the different stages of the slow cook-off performed on pressed PBX.

CONCLUSIONS

Employing effective and efficient mathematical modeling, advanced kinetic analysis enables the calculations of the progress of decomposition reactions under temperature conditions different from those at which the original examinations were carried out. Applications of Finite Element Methods (FEM) and accurate kinetic description enable determination of the effect of scale, geometry, heat transfer, thermal conductivity and ambient temperature on the heat accumulation conditions.

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