Differential Scanning Calorimeter

Differential Thermal Analysis

BÄHR Thermo analyse

DSC 302 Differential Scanning Calorimeter

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The Differential Scanning Calorimeter (DSC) is used to measure the difference in heat flow between sample furnace and reference sample furnace as a function of temperature.

Setup and method of operation

The measuring cell is made of silver with a bifilar wound jacket heating conductor on the outside of the cylinder.

For low temperature ranges there is a cold exchanger located underneath the measuring cell, which can also be retrofitted.

The symmetric setup of the measuring cell ensures homogeneous heat flow between heating conductor and sample and to the inert probe, thus ensuring a stable baseline and an *excellent signal to noise ratio*.

Both the sensitive sensors for the measurement of heat flow are located in two spatially separated chambers.
The sensors are very dynamic and have a time constant of 2.5 seconds.
The spatial separation between both sensors also avoids crosstalk on the opposite side during strong reactions.

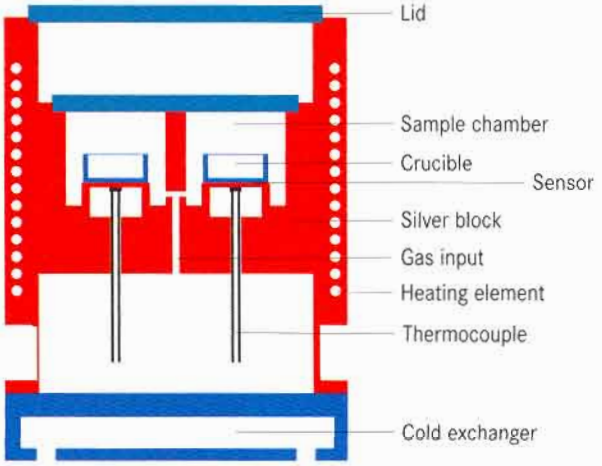
The DSC is also fitted as standard with two gas inputs, e.g. for air and nitrogen.
The valves are controlled by control tracks in the temperature programme. The gas is preheated before it reaches the measuring cell.

DSC302 features:

- heat flow principle
- separate sample chambers, therefore no signal crosstalk
- stable baseline due to homogeneous furnace area
- dynamic silver furnace
- integrated gas supply
- digital amplifier technology, therefore no signal drift
- own integrated processor system for independent operation
- network connections via LAN and WAN
- professional 32-bit WinTA9.0 software

Specifications:

Sensitivity:	1µW
Measuring range:	350mW
Signal noise level:	1µW
Enthalpy:	± 1%
Time constant:	2,5s
Temperature accuracy:	± 0,05°C
Baseline linearity:	± 50µW
Atmosphere:	air, purge gas
Temperature range:	RT - 700°C, -160°C - 700°C with LN_2 - 90°C - 400°C with kyrostat
Heating rate:	0,01 – 100K/min
Cooling rate:	max. 50K/min



Principle from the DSC

DSC 302 from the top

DSC 302 (RT – 700°C) measuring cell

Differential Scanning Calorimeter

DSC 302
DSC 302-HP

DSC 302-HP Differential Scanning Calorimeter

Detail

High-pressure DSC 302-HP

*Process and operating conditions can be simulated using the **high-pressure DSC302-HP.***
For this purpose, the measuring cell fitted to the standard DSC302 is located in a pressure vessel, which is designed for a pressure of 3MPa.

The external gas supply unit takes over pressure generation and control.
It has its own processor system. This allows it to create vacuums automatically, to regulate the gas pressure, and also to carry out appropriate cleaning of the measuring cell with inert gas during experiments under reduced atmospheres (e.g. hydrogen).

DSC 302 (-90°C – 400°C) measuring cell

DSC 302 (-90°C – 400°C) open measuring cell

DSC 302 with Dewar

Specifications:

Pressure:	max. 3MPa
Atmosphere:	Inert, oxidising, reducing
Temperature range:	RT – 400°C

Further technical specifications correspond to those of the DSC302.

Low-temperature fittings:

Using a cold exchanger which is fixed underneath the measuring cell, the DSC is, depending on the cooling medium fittings, cooled down to -160°C, -90°C and -50°C. *This cold exchanger can also be retrofitted to existing equipment.*

Working range -160°C to 700°C
***Cooling** takes place using liquid nitrogen (LN_2).*
The cold exchanger is supplied with LN_2 from a transport container (Dewar), which is subject to a continuous pressure of 0.5bar.
A proportional valve provides the cold exchanger with only the amount of LN_2 which it needs for creating the set temperature in the DSC.
The control variable is automatically calculated by the DSC processor. This method results in a considerable reduction in LN_2 consumption.

Working range -90°C to 400°C
***Cooling** is carried out by a two-stage kyrostat.* The kyrostat is connected with the cold exchanger by a feed and return pipe.
A circulation pump continuously transports the silicon oil, which has been cooled down to a standard temperature, through the cold exchanger.
The set temperature in the sample chamber is adjusted with the help of the DSC heating element.

Working range -50°C to 400°C
Here, a single-stage kyrostat is used.
Apart from this, the working principle is identical to that of the 90°C kyrostat.

Crucible • Reference material

DSC/ DTA Crucible

Crucible

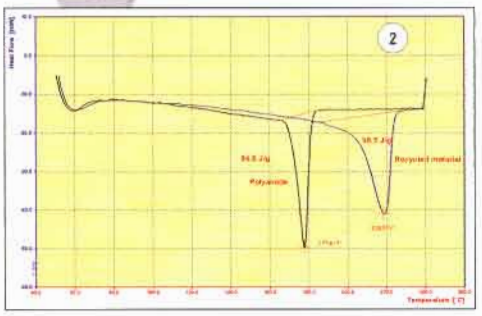
The crucible has considerable influence on the quality of the measured results.
The shape and heat capacity of the crucible are two of the factors influencing the calorimetric sensitivity and time constant.
The plane bottom side of the crucible enables optimum heat transfer between sample, crucible and sensor surface.

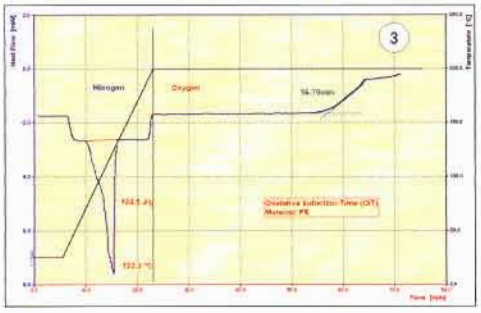
There is a range of crucibles available which vary both in material, such as Al, Au, Pt, Al_2O_3, W, fused silica and graphite, and also range in volume and diameters of 4, 6 and 9mm. **High-pressure crucibles** made of Inconel with replaceable bursting disks are designed for an internal pressure of 10MPa.

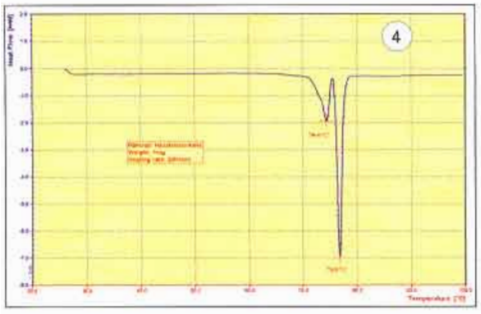
Crucibles made of aluminium *can be cold-welded and are designed for an internal pressure of 0.2MPa.*

A crucible press is available with replaceable pressing tools for simple sealing and for cold-welding.

DSC/ DTA Reference materials







1) Indium
2) Polyamide and recycled material
3) Oxidative Induction Time (OIT) from PE
4) Hexatricontane

Reference materials

Various metals, such as lead, gallium, indium, zinc, gold, and palladium are available for calibration of the DSC in addition to organic substances such as benzoic acid, caffeine, diphenyl ether, naphthalene, potassium nitrate, potassium perchlorate, silver sulphate, fused silica, potassium sulphate, potassium chromate, strontium carbonate and barium carbonate.
All reference materials are supplied with a certificate.

DSC Crucible press

DTA 703 with 1450°C Furnace

Differential Thermal Analysis DSC/DTA 703

Differential thermal analysis (DSC/DTA) is used to measure the temperature difference (ΔT) between the sample and an inert sample as a function of temperature.

Method of operation

Since the sensor is located free in the furnace area and therefore has no fixed connection to the heating element, **the energy from the heating element** is transferred to the sensor by radiation and convection.
In order to create a homogeneous temperature field around the sensor, the heating element is wound using different pitches.
The sensor surface contacting the crucible is completely plane.

If, for example, an exothermic or endothermic reaction occurs within the sample, a temperature difference is created between the inert sample and the sample.
This temperature difference is measured using thermocouples which are located underneath the sensor surface.
The temperature difference has a behaviour proportional to the reaction heat absorbed by the sample.

This enables quantitative determination of the specific heat of the transformation temperature and the enthalpy.
Calibration of temperature and enthalpy is carried out using reference materials.

DSC/DTA 703 features

- variable and pluggable sensors
- stable baseline due to homogeneous furnace area
- dynamic furnace
- digital amplifier technology, therefore no single drift
- an integrated processor system for independent operation
- network operation via LAN and WAN
- professional 32-bit WinTA9.0 software

Specifications:

Sensitivity:	5µW
Measuring range:	500mW
Signal noise level:	10µW
Enthalpy:	+/- 3%
Time constant:	7s (standard sensor)
Temperature accuracy:	+/- 0.1°C
Baseline linearity:	+/- 100µW
Atmosphere:	air, inert gas, reducing, vacuum 10^{-5}mbar
Temperature range:	-160°C - 700°C; RT - 1450°C 100°C-1650°C
Heating rate:	0.01 - 100K/min
Cooling rate:	max. 100K/min
Cooling-down.:	from 1500°C to 50°C in 10 min

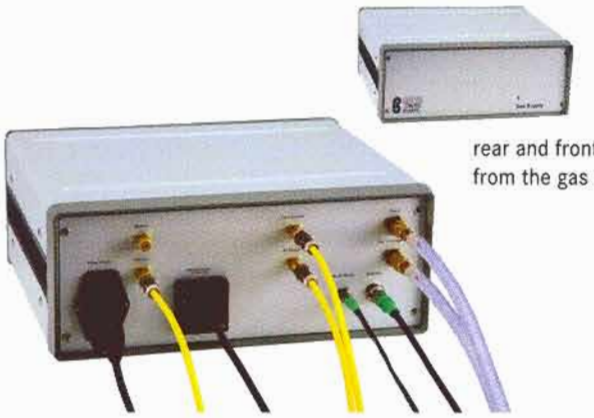
Furnace

Dynamic furnaces possess an excellent temperature profile and therefore guarantee a very good and stable baseline. This is achieved through the use of a heating element with differently wound pitches and a furnace shell with water cooling.

Heating-up speeds of max. 100K/min and a cooling-down time of 1450°C to 50°C in 10 min enable tests to be carried out very quickly.
As an alternative, temperature control can take place using the furnace thermocouple or using the sample thermocouple, whereby the latter guarantees extremely precise temperature control.

The following temperature ranges are available:
-160°C -700°C, RT - 1450°C and 100°C - 1650°C

DTA 703 Measuring head



rear and front side
from the gas supply

Gas supply

The DTA/DSC 703 is designed for operation under vacuum and dynamic or static gas atmospheres.

The gas supply unit regulates and controls the gas throughflow quantities and the vacuum unit.
You can switch between two gases as standard, and the gas throughflow quantities can be varied.

The control processor in the gas supply unit is connected to the DTA/DSC processor via a I²C-Bus.

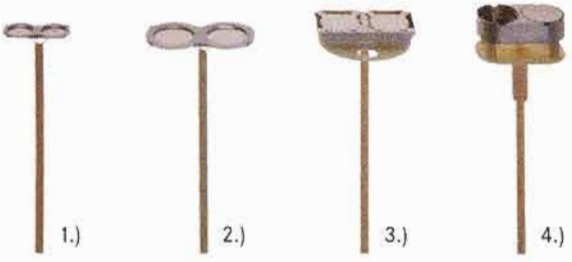
Gas throughflow regulation takes place using a mass-flow controller.

The effective gas throughflow quantity is a component of the dataset recorded during the measurement, which is subsequently made available for the evaluation.

DSC/DTA sensor

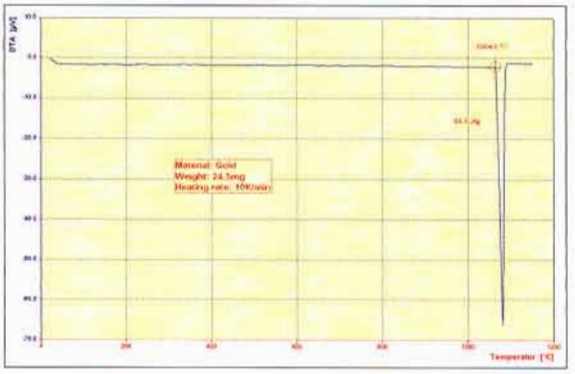
Various pluggable sensors enable adaptation to a wide diversity of measurement tasks taking the sensitivity, time constants and atmosphere into account.

The flat DSC/DTA sensor is made of Pt, Pt/Rh, Chromel or Platinel.
The thermocouples for temperature measurement are fixed to the bottom, and a positioning aid for the crucible is fixed to the top.

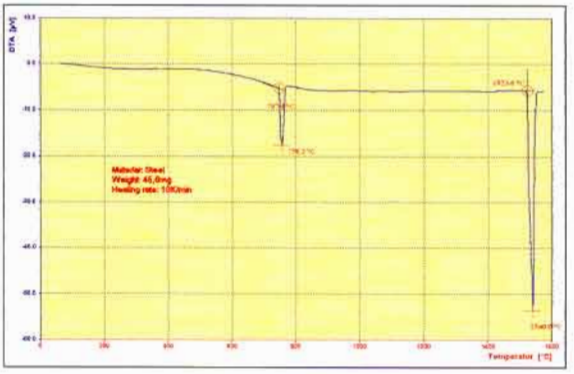


1) Standard sensor for 6mm crucible, time constant 7s
2) Large sensor for 9mm crucible
3) DSC sensor for 6 and 9mm crucible
4) cp sensor for 6mm crucible, metal block in Pt, gold or silver with integrated sensor

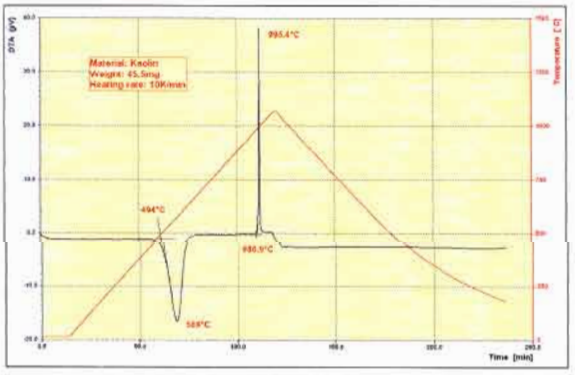
The sensors are usable in air, inert gas, vacuum and reducing atmospheres.
You should note that reducing atmospheres can shorten the sensor service life.
Sensors with Al_2O_3 coating can be supplied for working in aggressive media.



Gold



Steel



Kaolin

Application limits of DSC/DTA sensors:

Chromel	-120°C to 700°C
Platinel	-120°C to 1100°C
Pt	RT - 1500°C
Pt/Rh	100°C - 1700°C
W/Re	max. 2000°C