



HP DSC 1

STAR® System

Innovative Technology

Versatile Modularity

Swiss Quality



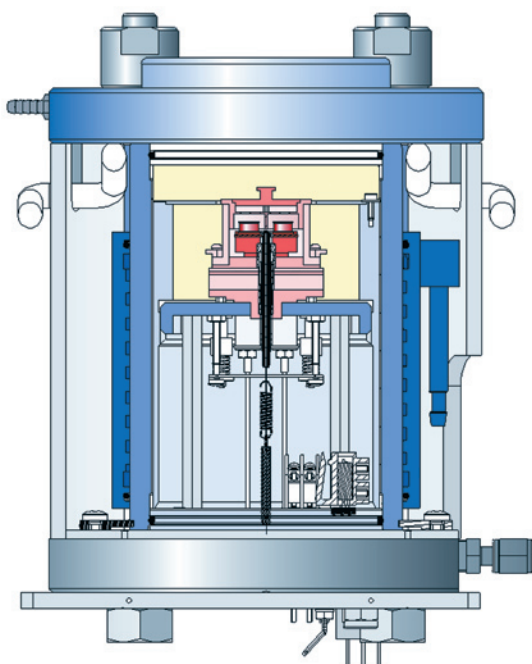
DSC Measurements under Pressure for Accelerated Materials Testing

METTLER TOLEDO

The Right Choice for High Pressure Applications

Increased pressure influences all physical changes and chemical reactions in which a change in volume occurs. For material testing, process development or quality control there is often no alternative to DSC measurements under pressure. Measurements performed under pressure expand the possibilities of thermal analysis.

- **Shorter analysis times** - higher pressure and temperature accelerate reactions
- **Measurements under process conditions** - simulation of practical reaction environments
- **Better interpretation** - effects can be separated by suppressing vaporization
- **Suppresses and delays vaporization** - separation of a reaction from overlapping vaporization
- **Higher gas concentrations** - reaction rate of heterogeneous reactions with gases is increased
- **Measurements under special atmospheres** - oxidative, oxygen-free conditions or measurements with toxic or combustible gases are possible
- **Possibility of working at different pressures** - more accurate measurement of adsorption and desorption behavior
- **Robust sensor with high resolution and outstanding sensitivity** - measurement of very weak effects as well as excellent temperature resolution of close-lying effects



In the HP DSC 1, the low-inertia, fast heating/cooling DSC 1 furnace is incorporated in a water-cooled pressure vessel. The furnace is specially insulated so that no temperature gradients occur. This guarantees a stable and reproducible baseline, even at higher pressures.

A double safety system limits the pressure to the permissible range (bursting disk and construction of the sealing system).

Three gas connections each with a control valve for:

- rapid filling (pressure build-up)
- purging the furnace chamber during the measurement (flow control)
- the gas outlet (pressure control).



The new high-pressure DSC cell is based on the successful Thermal Analysis Excellence DSC 1 technology and guarantees outstanding performance thanks to its FRS5 and HSS7 DSC sensors. The HP DSC 1 operates at overpressures from 0 to 10 MPa and from room temperature up to 700 °C. A pressure gage displays the actual pressure in the cell. An external pressure and flow controller is available as an option. This allows accurate pressure control in static and dynamically programmed atmospheres.



HP DSC-Chemiluminescence

The HP DSC 1 can easily be expanded to a high-pressure DSC-Chemiluminescence System. This enables you to simultaneously measure light emission and heat flow from a sample subjected to a precisely controlled gas pressure. Chemiluminescence measurements yield information on local oxidation rates and the influence of stabilizers.



HP DSC-Microscopy

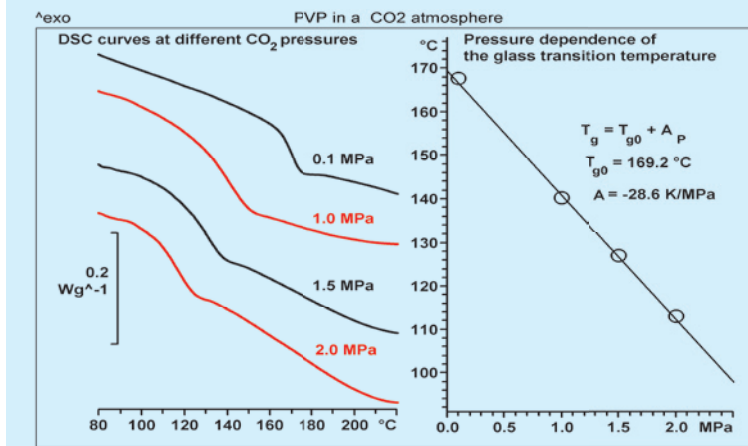
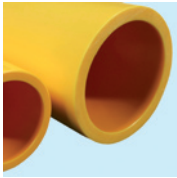
The HP DSC 1 can be used for high-pressure DSC-Microscopy applications. The sample is observed visually while it is heated or cooled in the DSC. Changes in the sample due to relaxation, melting or reaction processes are simultaneously recorded. This often facilitates the interpretation of DSC curves.

Characterization of Materials by High Pressure DSC

The high-pressure DSC allows you to measure samples under precisely defined atmospheres at pressures of up to 10 MPa as a function of temperature or time. Higher pressures and temperatures accelerate chemical reactions and shorten analysis times. Increased pressure suppresses vaporization and shifts the effect to higher temperatures. The high-pressure DSC is an excellent instrument for studying the influence of pressure and atmosphere on a sample or for separating an effect that is overlapped by vaporization.

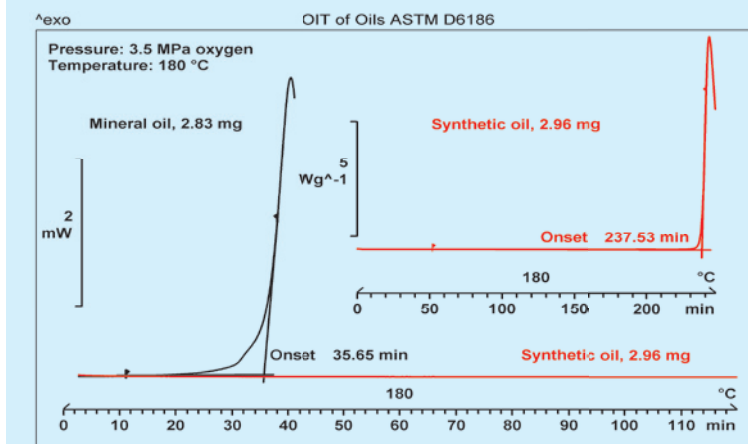
Industries and Application Areas

Chemical and pharmaceutical industries and universities	• Reactions with reactive gases such as O₂, H₂ and CO₂ (also combustible and toxic gases) • Safety investigations under pressure • Suppression of vaporization (through increase of the boiling point) • Separation of chemical reactions and vaporization processes that overlap at normal pressure • Investigation of reactions with volatile components • Catalytic reactions • Heterogeneous reactions • Adsorption and desorption • Measurement of the pressure dependence of the boiling point • Determination of the enthalpy of vaporization
Petrochemical	• Oxidation stability (e.g. testing of additives in lubricants) • Reactions with reactive gases
Plastics	• Curing reactions of polymers (e.g. polycondensation reactions) • Oxidation stability
Paints/lacquers/adhesives	• Cross-linking of adhesives • Suppression of vaporization (through increase of the boiling point)
Electronics	• Curing of resins (e.g. polycondensation reactions)
Foodstuffs	• Oxidation stability of fats and oils • Reactions with reactive gases



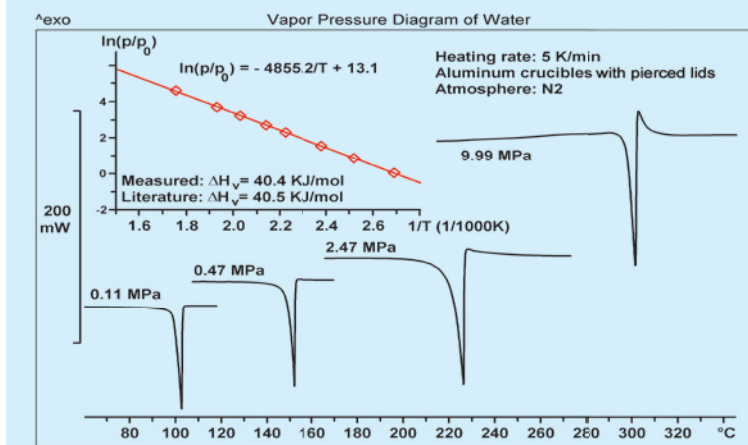
Influence of CO₂ pressure

The temperature range in which the glass transition occurs is often of great importance for the processing and application of polymers. Plasticizers are added to polymers to lower their glass transition temperature. Conventional plasticizers cannot always be used because they might cause adverse biological effects. In some cases, carbon dioxide (CO₂) acts as a plasticizer, e.g. with polyvinyl pyrrolidone (PVP). The DSC curves and the plot diagram show that increasing CO₂ pressure shifts the glass transition to lower temperature.



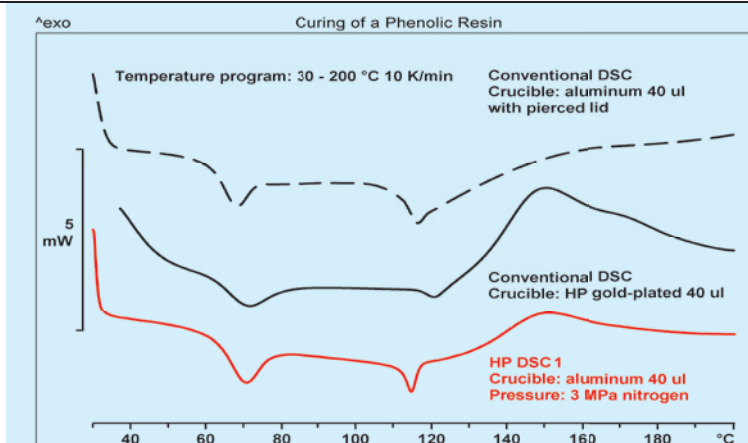
Oxidation stability ASTM D6186

The determination of the oxidation stability of oils is an important test in the petrochemical industry. The test is usually performed according to a standard, e.g. ASTM D6186. A sample of oil is held at 180 °C under increased oxygen pressure until oxidation begins. The onset of exothermic oxidation (intercept of the baseline with the inflectional tangent) is called the oxidation induction time, OIT. The curves show OIT measurements of two different motor oils. The synthetic oil takes much longer to oxidize, namely about 237 minutes, compared with just over 35 minutes for the mineral oil.



Vapor pressure diagram of water

A liquid boils when its vapor pressure equals that of its surroundings. The DSC curves show the boiling of water as a sharp endothermic peak at a temperature that depends on the total pressure. The exothermic effect immediately after the evaporation peak in the curves at higher pressures is due to the reaction between water vapor and the aluminum crucible. According to the modified Clausius-Clapeyron equation, the vapor pressure diagram can be plotted from the peak temperatures at different pressures. The enthalpy of vaporization is calculated from the slope of the measured vapor pressure curve and agrees well with the literature value.



Curing of phenolic resin

The DSC heating curve (top) of a phenolic resin exhibits a glass transition with relaxation, melting processes and a polycondensation reaction. Unfortunately, the heat flows from the exothermic reaction and the endothermic evaporation of water overlap. This makes it difficult to use the data for kinetic evaluations and hence for process optimization. The situation can be improved by suppressing evaporation. In a high-pressure capsule (middle), the effects are broadened due to the relatively large mass of the crucible. However, sharp peaks and a good separation are obtained when higher pressures are used (bottom).

Specifications HP DSC 1

Temperature data	
Range	22 ... 500 °C / 700 °C
Accuracy	±0.2 K
Reproducibility	±0.1 K
Heating rates	0.1 ... 50 K/min
Cooling system	double-walled jacket with water cooling (22 °C)

Pressure Data	
Range	0 ... 10 MPa (overpressure)
Gas flow	freely selectable, accurate control of purge gas flow rates and total pressure with external controller
Atmospheres	Measurements under different atmospheres: inert, oxidizing, reducing, reactive, e.g. nitrogen, oxygen, hydrogen, methyl bromide, ethylene, propylene, carbon dioxide, etc. There are certain restrictions for combustible and toxic gases

Pressure vessel	
Made of stainless steel	1.4435 (AISI/SAE: 316 L, UNS: S 31603)

Calorimetric information		
Sensor type	FRS5	HSS7
Sensor material	Ceramic	
Number of thermocouples	56	120
Signal time constant	1.7 s	3.9 s
Measurement range at 100 °C	± 350 mW	± 160 mW
Digital resolution	16.8 million points	

Sampling	
Sampling rate	max. 50 points per second (freely selectable)

Software	
Software	STAR® software 9.30 or later

Approvals

IEC/EN 61010-1:2001, IEC/EN 61010-2-010-2003
CAN/CSA C22.2 No. 61010-1-04
UL Std. No. 61010A-1

EN 61326-1:2006 (class B)
EN 61326-1:2006 (Industrial environments)
FCC, Part 15, class A
AS/NZS CISPR 22, AS/NZS 61000.4.3

Markings and approvals:

CE Conformity mark
CB
CSA

Low Voltage Directive 2006/95/EC
Electromagnetic compatibility (EMC) 2004/108/EC
Pressure Equipment Directive 97/23/EG

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