

# "High Pressure at Low Temperatures"



The first coolable high-pressure DSC 204 HP *Phoenix*<sup>®</sup>

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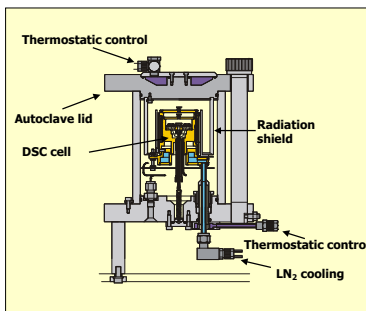


Fig. 1: Design of the coolable DSC 204 HP *Phoenix*<sup>®</sup>

Figure 1 depicts a cross section of the measuring cell of the coolable DSC 204 HP *Phoenix*<sup>®</sup> with surrounding autoclave. With the help of liquid nitrogen cooling, temperature programs (fig. 2a) can be realized which until now had been reserved for unpressurized DSC instruments. The heating and cooling rates achieved as well as the maximum and minimum temperatures are significantly influenced by the atmospheres used (fig. 2b). Since for oxidation reactions, the oxygen not only serves for the generation of pressure but is also a reaction partner of the expected solid-gas reaction, regulation of the pressure and gas flow must be particularly precise (fig. 2c). Pressure and gas flow represent the concentration of the gaseous oxygen reactand and can therefore ultimately influence the reaction process.

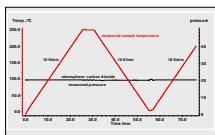


Fig. 2a: Temperature program in CO<sub>2</sub>

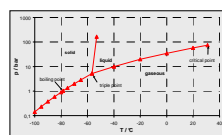


Fig. 2b: Phase diagram of CO<sub>2</sub>

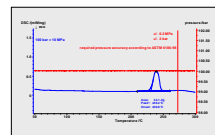


Fig. 2c: Accuracy of the pressure control

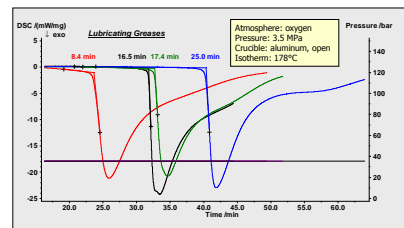


Fig. 3: Determination of the O.I.T for different lubricating greases at 3.5 MPa (35 bar) oxygen

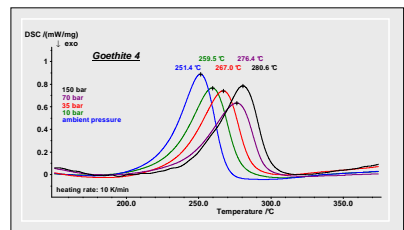


Fig. 4: Influence of the pressure on the dehydration of nanocrystalline geothite ( $\alpha$ -FeOOH, 0.1+0.01+0.01  $\mu$ m)

By means of Differential Scanning Calorimetry (DSC), accelerated aging tests can be carried out under an increased oxygen pressure (mostly 3.5 MPa) on hydrocarbons such as oils, fats, waxes and polymers (fig. 3). The Oxidation Induction Time (O.I.T) indicates the point in time at which the exothermal combustion of the hydrocarbons begins. The higher this time value is, the more oxidation-resistant the substance.

The interest in using inert gases lies mainly in investigating the influence of pressure on the calorific effects of the sample. Both decomposition reactions and dehydration reactions are considerably influenced by the pressure applied. With increasing pressure, the release of gaseous reaction products is shifted to higher temperatures (fig. 4) or entirely suppressed within the temperature range investigated (fig. 5).

Also for the glass transition of amorphous substances, changes in behavior under an increased pressure can be detected (fig. 6). For investigations on polyvinyl acetate (fig. 7) and tomato powder (fig. 8), the cooling potential of the DSC 204 HP *Phoenix*<sup>®</sup> is an important pre-requisite since the glass transition steps are expected to be close to room temperature.

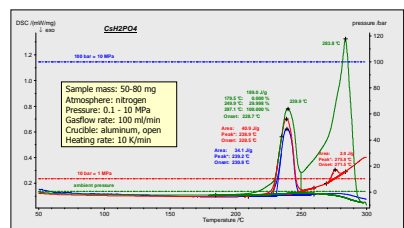


Fig. 5: Influence of the pressure on the dehydration of Csh<sub>2</sub>PO<sub>4</sub>

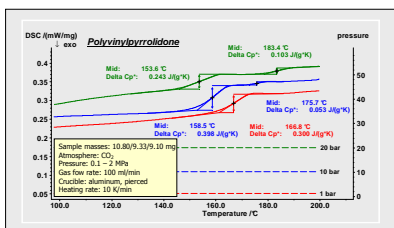


Fig. 6: Influence of the pressure on the glass transition of second heating of polyvinyl pyrrolidone in a CO<sub>2</sub> atmosphere

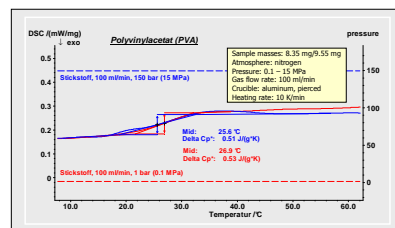


Fig. 7: Influence of the pressure on the glass transition of polyvinyl acetate (PVA)

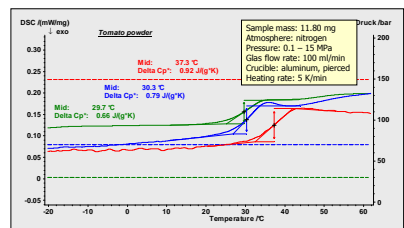


Fig. 8: Influence of the pressure on the glass transition of tomato powder