"High Pressure at Low Temperatures"

The first coolable high-pressure DSC 204 HP Phoenix®

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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 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 reactant and can therefore ultimately influence the reaction process.

Figure 1 depicts a cross section of the measuring cell of the coolable DSC 204 HP Phoenix® 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 reactant and can therefore ultimately influence the reaction process.

Figure 2a: Temperature program in CO₂

Figure 2b: Phase diagram of CO₂

Figure 2c: Accuracy of the pressure control

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

Figure 4: Influence of the pressure on the dehydration of nanocrystalline geothite (α=400 nm, 1.1x10⁻⁴ m/s gas flow rate)

Figure 5: Influence of the pressure on the dehydration of CaH₂PO₄

Figure 6: Influence of the pressure on the glass transition of each second heating of polyvinyl pyrrolidone in a CO₂ atmosphere

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

Figure 8: Influence of the pressure on the glass transition of tomato powder