

DIFFERENTIAL SCANNING CALORIMETRY (DSC) AT HIGH PRESSURE

R. SANDROCK, U. WENZEL, H. ARNTZ, and G.M. SCHNEIDER

Department of Chemistry, University of Bochum, 4630 Bochum, F.R.G.

On the basis of a high-pressure apparatus described by M. Kamp-  
hausen<sup>1,2,3</sup>, a DSC calorimeter for pressures up to 4000 bar and in  
the temperature range 100 to 300 K was constructed by H. Arntz<sup>4,5,6</sup>.  
The apparatus has been described in detail elsewhere<sup>4,6</sup>.

Recent results obtained by U. Wenzel are presented on the plastic  
crystal 2-chloro-2-methylpropane at pressures up to 1500 bar. At  
normal pressure two solid-solid phase transitions of the crystal  
are known. During the investigations a new pressure-induced solid  
phase was found at pressures above 800 bar. From the transition  
enthalpies and the p-T data of the coexistence lines transition  
entropies and transition volumes can be calculated.

In order to extend the ranges of temperature and pressure to higher  
values a new high-pressure twin DSC calorimeter was constructed.  
It works in the temperature range 300 to 600 K and at pressures up  
to 6000 bar. The pressure is generated by a hand pump and is trans-  
mitted by silicon oil. In spite of the similarity of the general  
set up some principal changes of the apparatus were necessary be-  
cause of the higher values of temperature and pressure. The pressure  
vessels are shrunked cylinders made of the nickel alloy Inconel  
718. The set up of the calorimeter is given and the procedure of  
temperature calibration and enthalpy determination described.

Preliminary results are reported concerning measurements on the plastic crystal diamantane and the high pressure phase of chain extended polyethylene<sup>8</sup>. The hexagonal phase, first reported by Bassett<sup>9</sup>, could be confirmed.

## REFERENCES

- 1 M. Kamphausen, Rev. Sci. Instrum. 46 (1975) 668.
- 2 M. Kamphausen, Dissertation, University of Bochum, F.R.G., 1976.
- 3 M. Kamphausen and G.M. Schneider, Thermochemica Acta 22 (1978) 371.
- 4 H. Arntz, Rev. Sci. Instrum. 51 (1980) 965.
- 5 H. Arntz and G.M. Schneider, Faraday Discussions 69 (1980) 139.
- 6 H. Arntz, Dissertation, University of Bochum, F.R.G., 1980.
- 7 U. Wenzel, Diplom thesis, University of Bochum, F.R.G., in preperation.
- 8 R. Sandrock, Dissertation, University of Bochum, F.R.G., in preperation.
- 9 D.C. Bassett, Polymer 17 (1976) 460.