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Note

Investigations on the phase behavior of "Pagodan"

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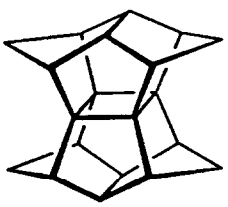
"Pagodan" (**1**), the $(CH)_{20}$ polycycle synthesized by the group in Freiburg, was studied to obtain detailed information about its phase behavior. These investigations were of special interest because a phase transition from a crystalline to a plastic crystalline state of pagodan could be presumed by its nearly spherical molecular form and the high melting point of 516 K published earlier [1]. The phase behavior of pagodan was measured with high pressure DTA in the temperature range 293–550 K at normal pressure and at pressures up to 190 MPa. The apparatus and the measuring method have been described elsewhere [2, 4].



Undecacyclo[9.9.0.$0^{1,5}$.$0^{2,12}$.$0^{2,18}$.$0^{3,7}$.$0^{6,10}$.$0^{8,12}$.$0^{11,15}$.$0^{13,17}$.$3^{16,20}$]eicosan (**1**).

Figure 1 shows two typical thermograms of pagodan at 0.1 and 43 MPa that have been obtained in the present measurements.

Figure 2 shows the pressure–temperature (p–T) phase diagram obtained from the measurements.

The results given in Fig. 2 demonstrate that the predicted phase behavior

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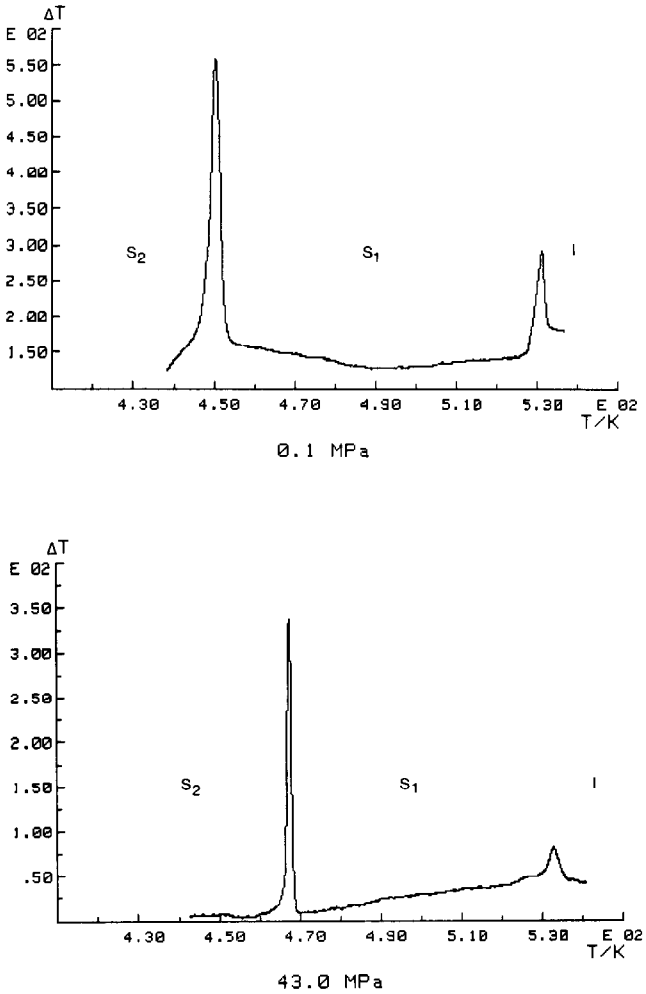

Fig. 1. Original thermograms of pagodan at 0.1 and 43.0 MPa.

could be confirmed. A second solid phase (s_1) has been found, which must be attributed to a plastic crystalline state, because, for example, of the relatively high enthalpy change accompanying the s_2–s_1 transformation compared to that of the melting transition (see Fig. 1). Extrapolation of the s_2–s_1 and s_1–l transition lines in Fig. 2 result in a triple point s_1–s_1–l at approximately 215 MPa and 545 K; at higher pressures the s_1 phase is no longer stable.

The melting point of $T^*(s_1\text{–}l) = (529.5 \pm 0.5)$ K found during the present measurements is distinctively higher than the melting point of 516 K reported in an earlier paper [1]. This discrepancy is probably caused by sublimation effects that were avoided by the use of special measuring cells in the present work [3].

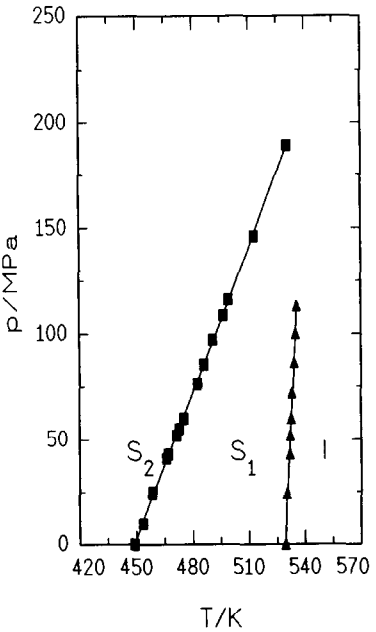

Fig. 2. Phase diagram of pagodan.

ACKNOWLEDGMENT

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