

FIG. 4. Cole-Cole plots for hexagonal ice prepared (left) by freezing liquid water and (right) below -100°C by depressurizing ice II. Semicircles are drawn to suggest the extent of departure from single relaxation time behavior.

time was some 40 times smaller than the initial value at -86.5°C .

This drift tended to interfere with attempts to measure the pressure coefficient of the relaxation time. Values of the activation volume were nevertheless estimated to be $1.8 \pm 0.6 \text{ cm}^3/\text{mole}$ at -87°C and, for a sample relaxing some 15 times as fast as the Auty-Cole rate, $0.6 \pm 0.5 \text{ cm}^3/\text{mole}$ at -40°C .

Effect of the I \rightarrow II \rightarrow I Cycle on the Relaxation Times

Several experiments were made to determine the effect on relatively rapidly relaxing samples of conversion to II and back to I at a number of fixed temperatures, including some so high that Ic was probably never present. Samples of Ih in the parallel-plate cell were first annealed at -30°C and 110 bar

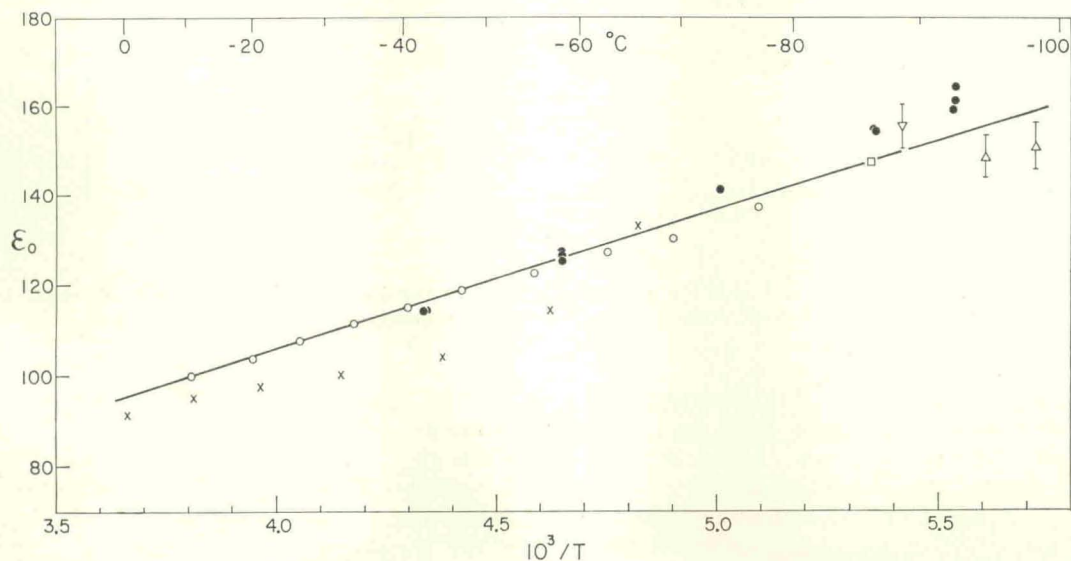


FIG. 5. Static permittivities of hexagonal ice: \times , data of Auty and Cole¹; \circ , sample frozen from liquid; otherwise samples prepared from ice II or IX in the coaxial cell.

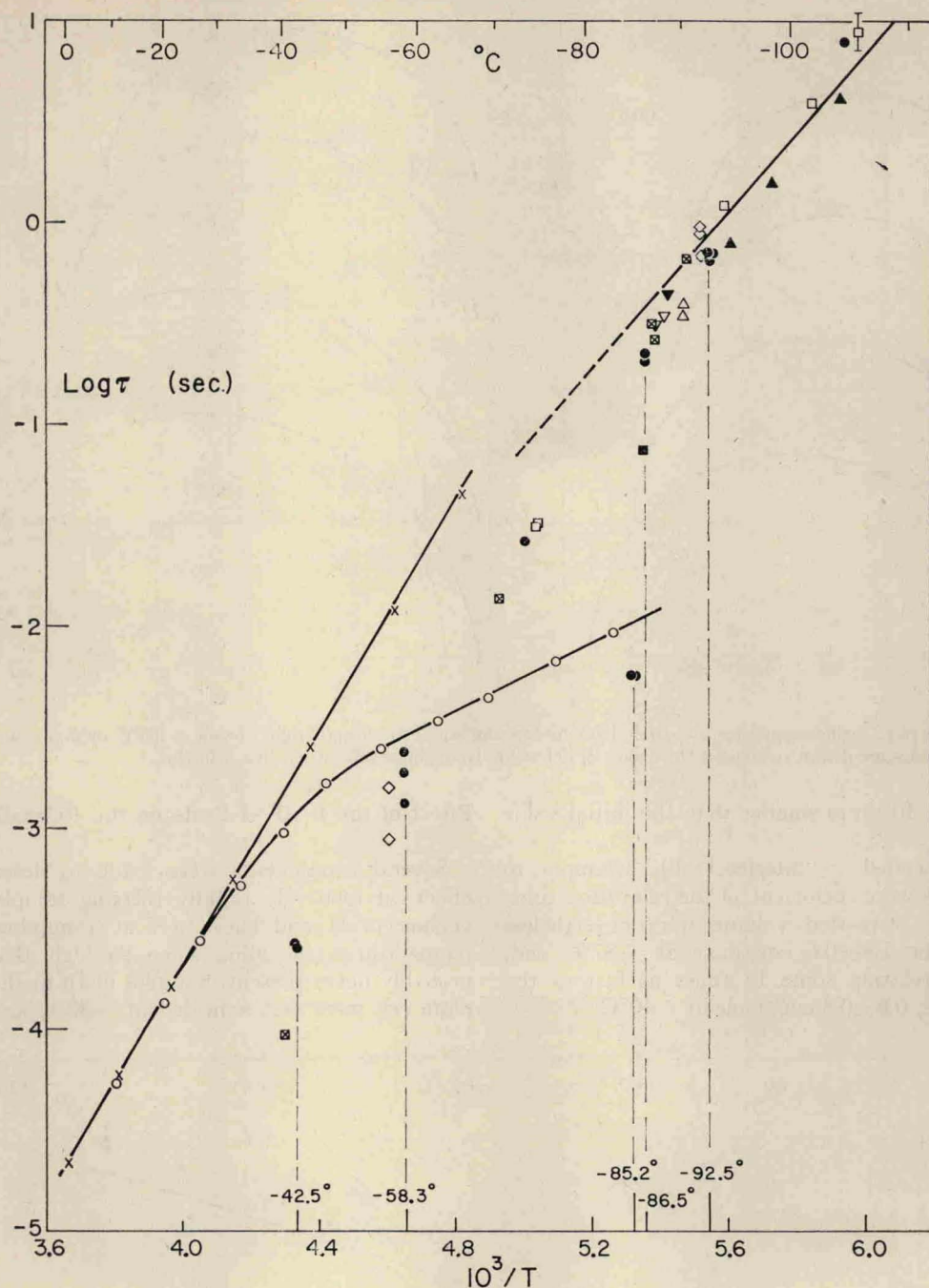


FIG. 6. Dielectric relaxation times: \times and \circ as in Fig. 5, remaining points for samples prepared from high-pressure forms in coaxial cell (solid symbols) or parallel-plate cell (open symbols).

for 24 h. The temperature was then lowered to the desired value where it was held fixed during the following sequence of operations. Initial dielectric measurements were made at 110 bar, the pressure was raised to $2\frac{3}{4}$ kbar for at least 12 h to ensure complete conversion to II, the sample was reconverted to I, and new measurements were made at 110 bar after an equilibration period of 20 min and again after

12 h. The relaxation times measured at three temperatures are given in Table I.

The newly transformed samples at -51 and -70°C were undoubtedly Ih. At -92°C the lifetime of Ic is ≈ 100 min,^{11,21} and the sample measured shortly after the transformation was likely Ic or a mixture of Ic and Ih.

It is apparent that the transformation sequence