



Fig. 1. Neutron-diffraction spectrum of ice Ic at 80°K prepared from ice II.

parameter  $a_0 = 6.358 \pm 0.004 \text{ \AA}$  at  $-185^\circ\text{C}$  from x-ray studies of polycrystalline material. With eight molecules in the unit cell, this lattice dimension (with the latest values of the fundamental constants) leads to a calculated density of  $0.931 \text{ g-cm}^{-3}$  which is identical within experimental error to that of ice Ih.<sup>14</sup>

#### EXPERIMENTAL

Although ice Ic can be prepared by the low-temperature deposition of water vapor, only relatively small quantities can conveniently be prepared in this manner. Since about 10 g of ice were needed for a neutron-diffraction sample, use was made of the fact that all high-pressure ices first transform to ice Ic.<sup>9</sup> The high-pressure ices II, V, and IX were prepared in the Be-Cu pressure vessel described previously,<sup>15</sup> and transformed to the cubic form for this study. Because of the large spin-incoherent neutron scattering of protons, it was necessary to use  $\text{D}_2\text{O}$  samples to achieve spectra with acceptable peak-to-background ratios. The temperatures at which the transitions were made were  $166^\circ\text{K}$  for ices V and IX, and  $177^\circ$  for ice II. A liquid-nitrogen-cooled *n*-pentane bath was used. Some of the transfor-

mations were made in small glass bottles placed directly in the constant-temperature bath, but none of the bottles were in direct contact with the *n*-pentane. In other cases the Ti-Zr neutron-diffraction sample-holder lid was unscrewed to allow for the expansion of one of the high-pressure ices, and this container was placed in an open-topped, thin-walled copper sleeve immersed in the *n*-pentane bath. The sample temperature was monitored with a copper-constantan thermocouple.

Neutron-diffraction spectra of liquid-nitrogen-quenched solutions of  $\text{FeCl}_2$  and  $\text{KCl}$  were taken to examine the reported induced formation<sup>10,11</sup> of ice Ic. Analytical-grade reagents were used to make the 0.47M  $\text{FeCl}_2$  and 0.4N  $\text{KCl}$  solutions in  $\text{D}_2\text{O}$ . Droplets of 1 mm in diameter were dispensed from a capillary tip and quenched in liquid nitrogen. These small ice pellets were ground in a polished steel mortar under liquid nitrogen. This same procedure had been used in the sample preparation of the high-pressure ices without bringing about a phase change. The powdered ice was transferred to the sample holder under liquid nitrogen.

A characteristic feature in the preparation of ice Ic is that the pure cubic phase is not formed by any of the reported methods. This appears to be true for the low-temperature deposition of water vapor<sup>5</sup> and for the transformation of both vitreous ice<sup>6</sup> and the high-pressure ices.<sup>12</sup> The impurity in the cubic-ice sample has

<sup>14</sup> S. W. Rabideau, E. D. Finch, G. P. Arnold, and A. L. Bowman, *J. Chem. Phys.* **49**, 2514 (1968), Paper I of this series.

<sup>15</sup> E. D. Finch, S. W. Rabideau, R. G. Wenzel, and N. Nereson, *J. Chem. Phys.* **49**, 4361 (1968), Paper II of this series.