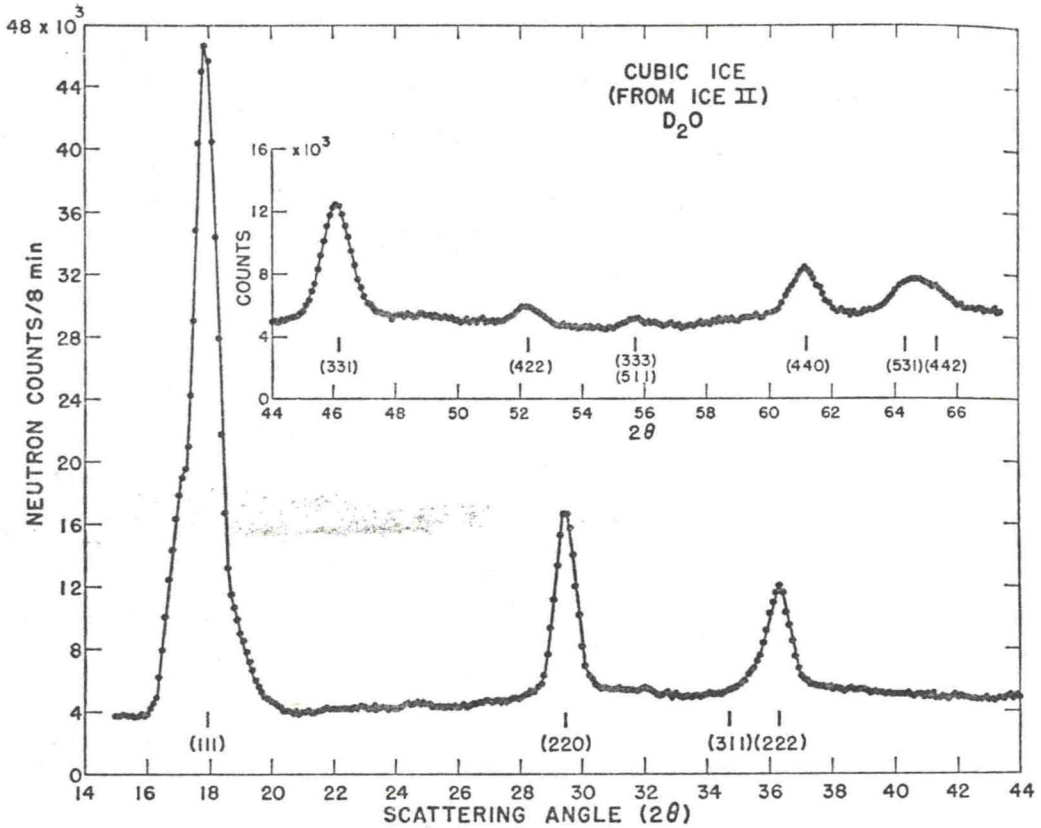

FIG. 1. Neutron-diffraction spectrum of ice Ic at 80°K prepared from ice II.

parameter $a_0 = 6.358 \pm 0.004$ Å at −185°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 g-cm^{-3} which is identical within experimental error to that of ice Ih.[14]

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.[9] The high-pressure ices II, V, and IX were prepared in the Be–Cu pressure vessel described previously,[15] and transformed to the cubic form for this study. Because of the large spin-incoherent neutron scattering of protons, it was necessary to use D_2O samples to achieve spectra with acceptable peak-to-background ratios. The temperatures at which the transitions were made were 166°K for ices V and IX, and 177° for ice II. A liquid-nitrogen-cooled *n*-pentane bath was used. Some of the transformations were made in small glass bottles placed dire in the constant-temperature bath, but none of the were in direct contact with the *n*-pentane. In other ca the Ti–Zr neutron-diffraction sample-holder lid unscrewed to allow for the expansion of one of the hi pressure ices, and this container was placed in an op topped, thin-walled copper sleeve immersed in t *n*-pentane bath. The sample temperature was monito with a copper–constantan thermocouple.

Neutron-diffraction spectra of liquid-nitrog quenched solutions of $FeCl_2$ and KCl were taken examine the reported induced formation[10,11] of ice I Analytical-grade reagents were used to make the 0.47 $FeCl_2$ and 0.4*N* KCl solutions in D_2O. Droplets of mm in diameter were dispensed from a capillary tip a quenched in liquid nitrogen. These small ice pellets w ground in a polished steel mortar under liquid nitroge This same procedure had been used in the samp preparation of the high-pressure ices without bringi about a phase change. The powdered ice was transferre to the sample holder under liquid nitrogen.

A characteristic feature in the preparation of ice I 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[5] and for th transformation of both vitreous ice[6] and the high pressure ices.[12] The impurity in the cubic-ice sample ha

[14] S. W. Rabideau, E. D. Finch, G. P. Arnold, and A. L. Bowman, J. Chem. Phys. 49, 2514 (1968), Paper I of this series.
[15] E. D. Finch, S. W. Rabideau, R. G. Wenzel, and N. Nereson, J. Chem. Phys. 49, 4361 (1968), Paper II of this series.