FIG. 1. Neutron-diffraction spectrum of polycrystalline D_2O ice II at 80°K.

from the pressure vessel. Because it has been reported that if the temperature is much above $-78°C$ ice III can form from ice Ih even in the region where ice II is the stable phase,[1,7] it was necessary to try to obtain confirmation that ice II was indeed the product of the transformation. This was in part accomplished by an experimental determination of the density of H_2O ice by measuring the buoyant force exerted on the coarsely divided powder as it was immersed in liquid nitrogen (later confirmed by neutron diffraction results). The ice was suspended at the end of the beam of an analytical balance with a fine wire supporting a glass vial immersed in a Dewar of liquid nitrogen. The experimental value of the ice II density was within 1% of the calculated value[6] of 1.17 g cm^{-3}. It must be pointed out that the use of density as a means of distinguishing between ices II and III is somewhat marginal because of the rather small difference in the density which is found for these ices. Ice III (equivalent to ice IX) has a density of 1.14[12] g cm^{-3}.

A procedure has been described[4] for the preparation of ice II by the decompression of ice V at $-35°C$. As suggested in this method, liquid water was cooled to $-35°C$ under a pressure of 5 kbar. After ice V formed, the pressure was lowered to about 3 kbar and held at this value for about 1 h. Upon examination of the quenched sample in the neutron diffractometer, it was determined that ice II had not formed, but instead, a spectrum corresponding to that of ice V was obtained. The rate of transformation of ice V to ice II has not been exhaustively examined. Since Wilson et al.[4] have reported the formation of ice II in this way, and suggest it as a more convenient way than the compression of ice Ih, we simply wish to point out that under some circumstances the rate of transformation of ice V to ice II may be slow.

[12] W. B. Kamb and S. K. Datta, Nature 187, 140 (1960).

The grinding of the powdered ice and loading of the neutron-diffraction sample holder under liquid nitrogen, as well as the characteristics of the neutron diffractometer and its calibration, have been described previously.[ ]

EXPERIMENTAL RESULTS

The neutron-diffraction spectrum of polycrystalline D_2O ice II obtained at 80°K is shown in Fig. 1. As described in the Experimental section, the ice II was made by the transformation of ice Ih under pressure. Hexagonal ice was present as an impurity of at most a few percent. This is shown by the appearance of weak peaks at diffraction angles of 2θ equal to approximately 16.8° and 29.4°. The presence of these peaks is indicated as much by the asymmetry which they impart to the nearby ice II peaks as by their own intensities. Not only is it logical to expect the impurity to be ice Ih since the ice II was made by the transformation of this ice, but also the positions of the lines can be indexed as (100) and (110) hexagonal-ice peaks. These are lines of major intensities in the hexagonal-ice spectrum. Because the peak intensities, centroids, and half-widths of the lines in the hexagonal ice spectrum had been determined in this work under similar experimental conditions, it was possible to subtract the contribution of an amount of hexagonal ice which would produce the expected Gaussian line shape for the (300) ice II peak. This hexagonal-ice background was subtracted throughout the entire range of 2θ covered to obtain the corrected ice II diffraction pattern. From the care which was taken to keep the ice II sample at or within a few degrees of liquid-nitrogen temperature during the grinding and loading operations, as well as during the spectrum-recording period, it appears that the hexagonal ice which was present in the sample was not the result of the transformation of ice II to the hexagonal form. Since the ice II was made by the phase trans-

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