been suggested to be hexagonal ice. In the transformation of vitreous ice, a residual of this form was stated to be present, in addition to the ice Ih impurity.

The powder-diffraction spectra were obtained on a neutron diffractometer at the Los Alamos Omega West Reactor. The calibration, resolution, and operation of the diffractometer have been described in Paper I of this series.[15]

EXPERIMENTAL RESULTS

The neutron-diffraction spectrum obtained at 80°K by the transformation of ice II is shown in Fig. 1. The cubic cell lattice parameter, a_0, was found to be 6.353± 0.001 Å by an analysis of the centroids of the first seven peaks at scattering angles (2θ) between 15° and 62°. The calculated positions for this value of a_0 are indicated by the vertical lines placed above the (hkl) symbols in Fig. 1.

As indicated previously,[15] the angular accuracy of the neutron diffractometer and the dispersion were checked with the use of a standard sample of NbO, so that it was possible to determine the extent to which a given line in the ice Ic pattern was broadened.

The asymmetry of the (111) peak in the ice Ic spectrum of Fig. 1 is apparent. In addition to the shoulder on the low-angle side of this peak centroid, there is also broadening throughout the peak with a suggestion of an incipient peak in the region of $2\theta = 19°$. All remaining lines in the spectrum, while reasonably symmetric, are appreciably broadened.

Essentially the same type of diffraction pattern was obtained from the samples prepared by the transformation of ices V and IX. The portion of the spectrum for a scattering angle 2θ of 16° to 20° is shown in Fig. 2. The ice Ic obtained from ice IX shows evidence for a shoulder on the high-angle side of the peak of major intensity, and a clearly resolved peak on the low-angle side. Although the heats associated with the transformations of ices V and IX to ice Ic are only 4 and 11 cal/g, respectively,[12] the possible contributions of these heats to the nature of the final ice Ic product was examined. Approximately 10 g of ice were needed for the neutron-diffraction pattern. In one case, the entire 10-g sample of ice V was placed in a glass bottle and transformed to ice Ic in the constant-temperature *n*-pentane bath. In a second trial, the sample of ice V was divided into six batches of approximately 1½ g and the transformation of each batch was made separately at 166°K. Since there was no detectable difference between the neutron-diffraction spectra obtained in these two ways, it was concluded that the transformation heats contributed little or nothing to the asymmetries observed under the conditions of these experiments.

In an attempt to try to resolve peaks of ice Ic obtained from ice IX into a series of component lines, Fig. 2, use was made of a Gaussian line-fitting program. However, even with as many as six lines in the 2θ region between

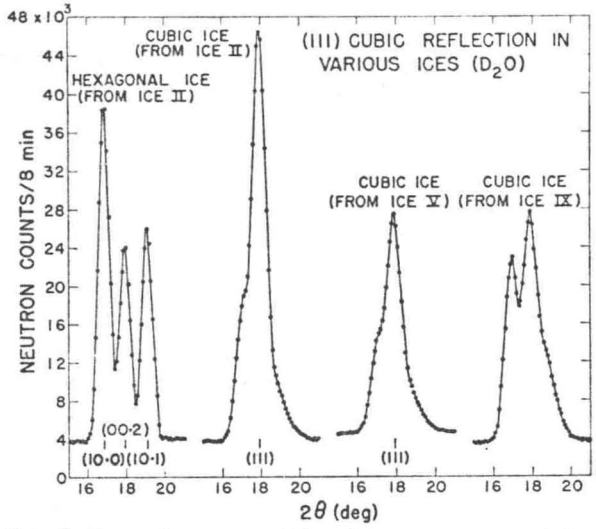

FIG. 2. Composite neutron-diffraction spectrum recorded at 80°K. Ice Ih prepared from ice II, ice Ic made by the transformation of ices II, V, and IX.

16° and 20°, the full widths at half-heights of the peaks were greater than would have been expected from the diffractometer dispersion characteristics. The spectrum of ice Ic from ice II was used for further analysis since this pattern had the most symmetric peaks.

The analysis of the spectrum pattern of ice Ic is made difficult by the presence of hexagonal ice. There are no lines in the cubic pattern that are not also found at essentially identical positions for ice Ih. To be able to determine the appropriate values to be subtracted from an observed possible mixture of ices Ic and Ih, peak intensities, half-widths, and centroids were obtained for ice Ih for the triplet peaks in the portion of the spectrum between 16° and 20°. For ice Ih prepared by the transformation of ice II at −50°C, the half-widths were found to be in accord with expected values for this instrument in this spectral region. Hexagonal indices are indicated for this triplet in Fig. 2. Ice II, prepared as described in Paper II of this series,[16] contained approximately 5% ice Ih. The contribution of this ice Ih impurity to the ice Ic spectrum was subtracted prior to attempting an analysis of the observed ice Ic spectrum. The residual peak widths were considerably in excess of those expected. These excess widths have been interpreted as arising from the small particle sizes of the ices which resulted from the ice II transformation. Although ice Ic was the principal product of this transformation, it appears that a small, additional amount of ice Ih was also formed.

In a series of Mössbauer studies of ferrous ions trapped in ice by the quenching of aqueous ferrous ion solutions in liquid nitrogen, Nozik and Kaplan[10] give convincing evidence for the induced formation of cubic ice. It was of interest to determine whether any evidence could be obtained by neutron-diffraction studies for

[16] R. Brill and A. Tippe, Acta Cryst. 23, 343 (1967).