DTA studies of the glycerol-water system

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The X-ray diffraction results reported in our original paper are incorrect due to an error in specimen-to-film distance. Using the correct distance (3.7 cm), recalculated spacings and line intensities closely match the known diffraction spectra of hexagonal and cubic ice.

The phase which was a bitrarily referred to as "Type I crystals" we now find similar to hexagonal ice (Form I_h)¹. Our "Type II crystals" appear to be a mixture of the less stable cubic ice (Form I_c), plus a lesser proportion of hexagonal ice, and were

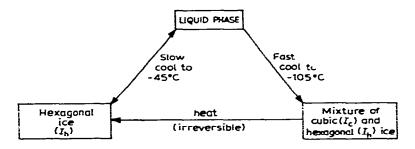


Fig. 5. Phase changes observed in 43.7 wt % glycerol by X-ray diffraction.

TABLE /II

LOW TEMPERATURE X-RAY DIFFRACTION 43.7 wt % glycerol

Slow cool to $-45^{\circ}C$		Fast cool to $-105^{\circ}C$		Hexagonal ice In at – 183°C		Cubic ice I _c at – 183°C	
d _{iki} (Å)	I _{rei}	$d_{kel}(\mathbf{A})$	Irei	$\frac{1}{10} u_1 = 103 C$		$T_c u_1 = 165 C$	
				$d_{hkl}(\vec{A})$	Irei	d <u>mi</u> (Å)	Irei
3.91	100	3.91	30	3.90	100		
3.67	35	3.70	100	3.66	100	3.68	100
3.45	45	3.49	5	3.4	80		
2.68	10			2.67	35		
2.25	25	2.26	25	2.25	90	2.25	40
2.08	17			2.07	60		
1.95	<1			1.95	5		
1.92	3	1.92	7	1.92	50	1.92	25
1.89	<1			1.89	5		

only observed to form when the one solution examined at that time (43.7 wt % glycerol) was rapidly quenched. Since the three diffraction lines of the diamond cubic lattice exactly coincide with hexagonal lines, it is difficult to positively identify the lesser amounts of the cubic form in the presence of predominantly hexagonal ice².

Figure 5 and Table III from our cited publication are reproduced in corrected form herewith.

The line previously reported at 7.73 Å was part of a halo caused by scattering from the Mylar window of the cold chamber and should be ignored. The 4.51 Å line was due to copper beta-radiation.

REFERENCES

I J. E. Bertie, L. D. Calvert and E. Whalley, J. Chem. Phys., 38 (1963) 840.

2 L. G. Dowell and A. P. Rinfret, Nature, 188 (1960) 1144.

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Announcement

Twenty-eighth Annual Calorimetry Conference

The Calorimetry Conference (U. S. A.) will hold its twenty-eighth annual meeting at the College of the Holy Cross, Worcester, Massachusetts, U. S. A. on June 13–15, 1973. The conference will be a joint meeting with the North American Thermal Analysis Society. The Calorimetry Conference will be concerned with all aspects of calorimetry including both the development of calorimetric equipment and the applications of calorimetry to the whole range of the physical and biological sciences. The conference is also concerned with ancillary subjects — thermometry, temperature control, electrical measurements, standard calorimetric samples, data treatment, compilations of thermodynamic data, publication policies, etc.

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