ISIS Experimental Report

Rutherford Appleton Laboratory

Title of Experiment:	Equation of state of ice VI	**Local Contact:**	M. G. Tucker
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Experimental Team:	A.D. Fortes, I. G. Wood, L. H. Norman, M. G. Tucker	**Date of Experiment:**	18-21/02/2010 04/05/2010

Introduction: Ice VI is a tetragonal high-pressure polymorph of solid H_2O which is likely to be an important rock-forming mineral in many icy planetary bodies: inside an object the size of Ganymede or Titan, there may be a layer of ice VI up to 400-500 km deep [2-4]. Ice VI may also be formed by impact shock metamorphism on icy planetary surfaces and could persist metastably on geological timescales at low ambient temperatures (< 100 K) [5]. It has recently been established that ices VI and VII are able to accommodate small quantities of NaCl or LiCl in their structures, with a marked effect upon their thermoelastic properties [7,8]. In order to quantify the effect of occluded ions on the the properties of ice VI it is necessary to determine the thermoelastic properties of the pure phase. Moreover, ice VI is a common accessory phase that forms in many of our high-pressure studies of aqueous systems of planetary interest [e.g., 9]. Given its near ubiquity in these studies, a good equation of state would provide us with an in situ pressure marker of comparable incompressibility to the solids under investigation.

Experimental method: A small ball of silica wool, containing a pellet of Pb foil, was soaked with liquid D_2O and then sealed between encapsulated TiZr gaskets using the Paris-Edinburgh press under a load of ~6 tons. Warming and cooling of the specimen were achieved using the new variable temperature insert. The sample temperature was reduced to 240 K, resulting in formation of a mixture of ice phases II and III. Upon compression in 2 ton increments, ice V was formed and then the desired phase, ice VI.

We carried out two such loadings, separated by several months. The P-T paths followed in each loading are shown in Figure 1. In the first loading we employed the V-T insert to collect data on warming and cooling of the sample under constant load. In the second loading we cooled the fresh ice VI sample to 120 K and made isothermal measurements up to ~2.7 GPa. In both loadings, problems were encountered with refilling of the dewar feeding the V-T insert, which resulted in uncontrolled warming of the specimen whilst it existed metastably in the stability field of ice VIII, resulting in a phase transformation to ice VIII.

Results: In addition to the data collected from our two P-E cell loadings, we incorporated an ice VI isothermal compression dataset (295 K) acquired in May 2005 by Bill Marshall. Together, these three loadings provide good coverage of the P-T stability of ice VI (Figure 1). The unit-cell volumes have been fitted with a Murnaghan equation of state, in which the temperature dependence of V_0 at zero pressure is described by a second order polynomial $(V_{0,T}) = V_{0,0}+aT+bT^2$, and the zero-P temperature dependencies of K and K′ are linear in T. This P-V-T equation of state gives an excellent fit to the combined datasets (Figure 2), albeit with a smaller value of K′ than expected from published ultrasonic measurements. The equation of state parameters from

this fit, referenced to P = 0.6 GPa and T = 120 K are, $V_{0.6,120}$ = 218.0(1)Å^3, a = 0.022(2)Å^{3}K^{-1}, b = 0.00011(1)Å^{3}K^{-2}, $K_{0.6,120}$ = 22.4(7) GPa, dK/dT = -22(8) MPa K^{-1}, $K'_{0.6,120}$ = 2.0(7), dK′/dT = 0.007(9). Further work on characterising the thermoelastic anisotropy of the ice VI is continuing.

Figure 1: Loci of datasets acquired in P-T space across the water-ice phase diagram. Roman numerals indicate the stability of fields of various high-pressure ice polymorphs.
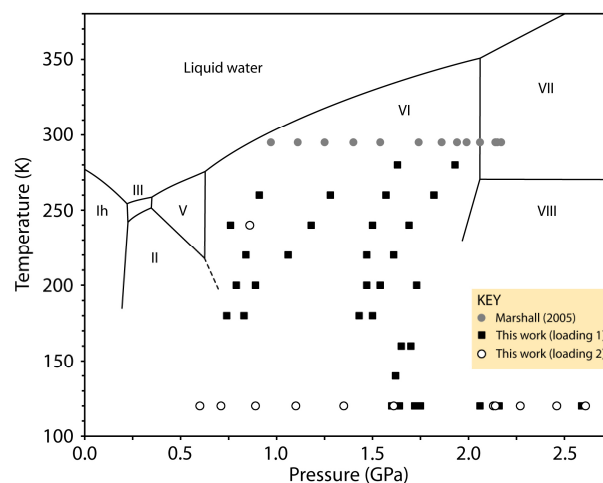
Figure 2: P-V-T surface fitted to the ice VI unit-cell volume data.
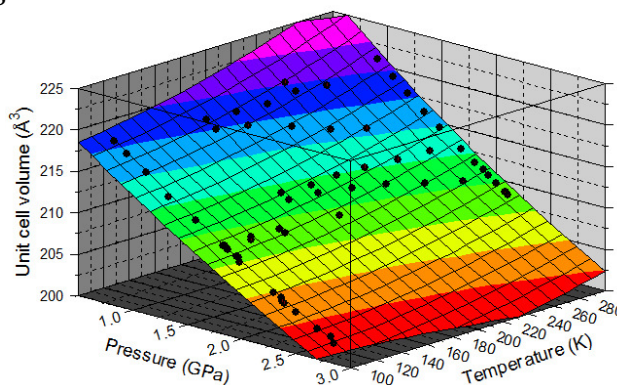
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