

to pressures of about 3 GPa, and down to liquid helium temperatures. We present here the results of high pressure powder measurements on the three D<sub>2</sub>O ice phases that have interpenetrating sublattices (ices VI, VII and VIII). The mode of ordering of tetragonal ice VIII (SG: I4<sub>1</sub>/amd) is clearly established, and no evidence is found of partial ordering as the temperature is raised. Ice VII is accurately cubic (SG: Pn3m) with D<sub>2</sub>O molecules disordered around their center of mass; there is no evidence of partial ordering at any temperature. The water molecule geometry is normal in both phases, and the hydrogen-bonded first neighbour in ice VIII is confirmed to be more distant than the first non-bonded neighbour. The transition temperature between the two phases occurs at 263 ± 2 K, some 11 K lower than expected. Hydrogen bond lengths in both phases are equal at the transition. Although the ice VI data is less good, we can see no evidence of the antiferroelectric ordering proposed by Kamb from work on recovered samples. Our results are consistent with thermodynamic measurements indicating disorder in ice VI at 193 K. We conclude that the orientational ordering behaviour of these high pressure ices is simpler than thought previously. For our ice VI result to be consistent with those of Kamb, his antiferroelectric ordering must be an artifact of the recovery process, thus throwing doubt on the validity of structural ordering data obtained on samples recovered to ambient pressure.

04.6-7 HYDROGEN-BOND RESEARCH USING THE CAMBRIDGE STRUCTURAL DATABASE. Robin Taylor and Olga Kennard, Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, U.K.

Analysis of data in the Cambridge Structural Database provides detailed information about the basic factors governing hydrogen bond geometry. H...A distances can be correlated with the nature and crystallographic environment of the donor and acceptor groups - for example, R<sub>3</sub>N<sup>+</sup>H...O bonds are found to be shorter than H<sub>3</sub>N<sup>+</sup>-H...O bonds. D-H...A angles have an energetic preference for linearity or near-linearity, particularly when the H...A distance is short. There is a slight preference for hydrogen bonding in the directions of sp<sup>2</sup> lone pairs, but it is arguable whether a similar preference exists for sp<sup>3</sup> lone pairs. Donor and acceptor valence bonds appear to be lengthened by hydrogen-bond formation, but this may be an artefact of librational phenomena. Hydrogen bonds involving C-H donors or the 3-centre ('bifurcated') arrangement are more common than previously supposed.

04.6-8 CRYSTAL STRUCTURE OF M<sup>II</sup>H<sub>10</sub>(AsO<sub>4</sub>)<sub>4</sub> COMPOUNDS AND HYDROGEN BONDING (M<sup>II</sup> = Mg, Mn, Ni, Cu and Zn). By M. Chiadmi and D. Tran Qui, Laboratoire de Cristallographie, C.N.R.S., associé à l'U.S.M.G., 166 X, 38042 - Grenoble Cedex, France.

Acidic orthoarsenate M<sup>II</sup>H<sub>10</sub>(AsO<sub>4</sub>)<sub>4</sub> compounds (M<sup>II</sup> = Mg, Mn, Co, Ni, Cu and Zn) have been prepared and single crystal grown. X-ray data show that all the series members belong to space group P1 and have very close unit cell parameters suggesting normally isomorphism between these salts. Crystal structure of CuH<sub>10</sub>(AsO<sub>4</sub>)<sub>4</sub> (a = 5.391(3), b = 7.631(4), c = 8.298(5), α = 105.84(4), β = 97.62(3) and γ = 93.64(10)) has been established including hydrogen positions and refined to a final R factor 3.9 % for 2158 reflections. The structure consists of CuO<sub>6</sub>-AsO<sub>4</sub> chain parallel to a-axis. CuO<sub>6</sub> octahedra and AsO<sub>4</sub> tetrahedra are linked together by oxygen corners sharing. Hydrogen localizations confirm the existence of biacidic and triacidic groups: AsO<sub>2</sub>(OH)<sub>2</sub> and AsO(OH)<sub>3</sub>.

It was also observed that the triacidic AsO(OH)<sub>3</sub> orientation with respect to CuO<sub>6</sub>-AsO<sub>4</sub> chain is significantly different in CuH<sub>10</sub>(AsO<sub>4</sub>)<sub>4</sub> crystal structure and in that of CdH<sub>10</sub>(AsO<sub>4</sub>)<sub>4</sub> (a = 5.69(5), b = 7.42(4), c = 8.60(6), α = 105.17(1), β = 95.13(5), γ = 91.85(5), (Boudjada et al., Acta Cryst. (1980), B36, 133-135)). This structural distortion will be discussed in terms of hydrogen bonding.

04.6-9 HYDROGEN BONDING IN ACID SALTS OF MESO-TARTARIC ACID. By P.F.W. Stouten, A.J.A.R. Blankensteyn & J. Kroon, Laboratorium voor Kristal- en Structuurchemie, Rijksuniversiteit, Padualaan 8, Utrecht, The Netherlands.

In acid salts of dicarboxylic acids H<sub>2</sub>Y we should expect the anion in MHY to have one carboxyl group ionized, the other not. In this case the carboxyl groups are linked by H bonds, which are essentially non-symmetric; they are called B-type bonds [Speakman, Structure and Bonding (1972) 12, 141]. However, frequently another variant is found in which inter-carboxyl H bonds lie across crystallographic symmetry elements; according to Speakman they are classified as A-type bonds.

Acid salts of meso-tartaric acid display a wide variety of H-bond patterns. We already reported the mixed A/B type of KHC<sub>4</sub>H<sub>4</sub>O<sub>6</sub> [Kroon & Kanters, Acta Cryst. (1972) B28, 714] and the B-type acid salt PhCH(NH<sub>2</sub>).C<sub>4</sub>H<sub>5</sub>O<sub>6</sub> [Blankensteyn, Moerman, Ouwkerk & Kroon, Coll. Abstracts E.C.M. 8 (1983) 161].

We now have solved the crystal structure of the superacid salt of composition NaH<sub>3</sub>(C<sub>4</sub>H<sub>4</sub>O<sub>6</sub>)<sub>2</sub>, which also appears to be of a mixed A/B type and that of LiHC<sub>4</sub>H<sub>4</sub>O<sub>6</sub>.H<sub>2</sub>O with unusual cyclic H-bonded carboxyl dimer formation.

NaH<sub>3</sub>(C<sub>4</sub>H<sub>4</sub>O<sub>6</sub>)<sub>2</sub>, monoclinic, P2<sub>1</sub>/c, a=6.514(1), b=9.193(3), c=9.440(3) Å, β=96.38(2)°, v=561.8 Å<sup>3</sup>, Z=2, λ(CuKα)=1.5418 Å, 992 unique reflections [I ≥ 2.5σ(I)], current R=0.065.

LiHC<sub>4</sub>H<sub>4</sub>O<sub>6</sub>.H<sub>2</sub>O, monoclinic, P2<sub>1</sub>/c, a=4.886(1), b=25.361(3), c=5.310(1) Å, β=84.19(1)°, v=654.6 Å<sup>3</sup>, Z=4, λ(CuKα)=1.5418 Å, 1218 unique reflections [I ≥ 2.5σ(I)], current R=0.078.