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 D2O ice phases that have interpenetrating sublattices (ices VI, VII and VIII). The node of ordering of tetragonal ice VIII (8G: 1T4/and) is clearly established, and no evidence is found of partial ordering as the temperature is raised. Ice VII is accurately cubic (2G: Pm3m) with D2O 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 VII is confirmed to be more distant than the first non-bonded neighbour. The transition temperature between these 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.

Acidic orthoarsenate MIIH4[AsO4]4 compounds (MII = Na, K, Rb, Cs and Tl) 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 CuH4[AsO4]4 (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 2336 reflections. The structure consists of CuO6-AsO4 tetrahedra linked together by oxygen corners sharing. Hydrogen localizations confirm the existence of bidentate and tridentate groups: AsO2(OH)2 and AsO(OH)3.

It was also observed that the triacidic As2O3(0H)3 orientation with respect to CuO6-AsO4 chain is significantly different in CuH4[AsO4]4 crystal structure and that of CuH2[AsO4]2 (a = 5.69(5), b = 7.62(4), c = 8.60(6), α = 103.17(1), β = 98.18(6), γ = 91.85(1)) (Boudewyns et al., Acta Cryst. (1980), 386, 133-135). This structural distortion will be discussed in terms of hydrogen bonding.

04.6-8 CRYSTAL STRUCTURE OF MIIH4[AsO4]4 COMPOUNDS AND HYDROGEN BONDING (MII = Mg, Mn, Ni, Cu and Zn). By M. Chiadini and D. Tran Qui, Laboratoire de Cristallographie, C.N.R.S., ASSOCIÉ à l’S.M.G., 166 X, 88042-Grenoble Cedex, France.

In acid salts of dicarboxylic acids H, Y we should expect the anion in MN 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 A-type bonds [Speekman, 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 Speekman they are classified as B-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 KNH4H3O (Kroon & Kanters, Acta Cryst. (1972) B28, 714) and the B-type acid salt NH4CH(NH3)CO2H4 [Blankensteyn, Hoeman, Overkerk & Kroon, Coll. Abstracts E.C.N. 8 (1983) 161]. We have now solved the crystal structure of the superacid salt of composition NaH2(C4H40S)2, which also appears to be of a mixed A/B type and that of LiH2H4O4H4O with unusual cyclic k-bonded carboxyl dimer formation.