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Crystal structure studies of mixed system of NaH_2PO_4 and KH_2PO_4 with H_3BO_3

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Abstract. X-ray powder analysis of solid solution of NaH_2PO_4 and KH_2PO_4 with H_3BO_3 was carried out. Both the systems were observed to be tetragonal. The crystallographic data are reported.

Keywords. X-ray diffraction; solid solutions; tetragonal system.

1. Introduction

Sodium and potassium dihydrogen phosphates belong to the family XH_2YO_4 with X as NH_4, Na, K and Y as P, As. All these crystals belong to the tetragonal system (Hellwege 1975). It was reported that NaH_2PO_4 and KH_2PO_4 formed a solid phase when mixed with boric acid (H_3BO_3), the proportions being 108·3 g and 11·2 g for NaDP and 33·7 g and 8·9 g for KDP respectively for 100 ml of saturated aqueous solution (William 1958). Powder X-ray diffraction study was carried out on the binary systems of (i) NaH_2PO_4 (90·62% by weight fraction) and (ii) KH_2PO_4 (79·11% by weight fraction) mixed with H_3BO_3 respectively; the systems being reported (William 1958) for aqueous solubility.

2. Experimental

Crystals were grown from aqueous solutions at room temperature (30°C). The crystals obtained were approximately 2 × 1 × 1 mm and were colourless. In order to determine the lattice parameters of the binary powder X-ray diffraction patterns were taken on a Stifert (German make) diffractometer using CuK_α radiation ($\lambda = 1{\cdot}542$ Å).

3. Results and discussion

The data for the two systems (table 1) give the observed and calculated d (hkl) values for prominent reflections. The agreement of d (obs) and d (cal) was seen to be fairly good. From the data it was inferred that both the binary systems were tetragonal. For NaDP binary system, systematic absence in $00l$ for l odd determine the space group to be either $P4_2/m$ or $P4_2$. For KDP binary system, systematic absences for $0kl$ for $k + l$ odd, hhl for l odd, $h00$ for h odd determine the space group to be either P4/mnc or P4nc. The density of the crystals was measured by the floatation method. The crystal parameters are tabulated in table 2. The densities for the two systems found from the X-ray data and those measured were found to be in good agreement. The number of molecules in unit cell for pure KDP was 4 (Hellwege 1975). The observed number of molecules for binary systems of KDP and NaDP with H_3BO_3 was 3 and 2 respectively

Table 1. X-ray powder data.

2θ (°)	hkl	d(obs) Å	d(cal) Å	2θ (°)	hkl	d(obs) Å	d(cal) Å
(a) $NaH_2PO_4 + H_3BO_3$							
23·54	002	3·724	3·726	56·30	302	1·628	1·626
25·15	111	3·532	3·410	59·30	132	1·554	1·557
29·00	012	3·086	3·086	60·15	204	1·537	1·535
33·45	112	2·657	2·672	62·45	214	1·482	1·477
37·45	120	2·384	2·425	63·30	303	1·466	1·461
45·00	122	2·014	2·032	75·45	314	1·255	1·261
48·15	004	1·885	1·863	78·30	106	1·218	1·210
51·15	104	1·791	1·762	81·00	403	1·187	1·190
54·15	222	1·692	1·704	83·09	413	1·168	1·162
(b) $KH_2PO_4 + H_3BO_3$							
23·15	101	3·829	3·810	45·15	103	1·991	2·012
33·30	112	2·675	2·645	58·15	330	1·584	1·587
37·30	220	2·398	2·384	69·15	430	1·357	1·356
40·15	300	2·240	2·247	79·15	233	1·209	1·208

Table 2. Lattice parameters and crystal data.

	NaDP + boric acid	KDP + boric acid
Crystal system	Tetragonal	Tetragonal
Cell dimensions (Å)		
a	5·42	6·74
c	7·45	6·32
Molecular weight	123·7	108·8
Number of molecules in unit cell (z)	2	3
Observed density (g/cc)	1·833	1·875
Calculated density (from X-ray data) (g/cc)	1·873	1·885
Volume of unit cell ($Å^3$)	242·05	287·50

and was lesser than pure KDP. It may be noted that in the absence of data for pure NaDP, the observed NaDP/boric acid binary system was compared with that of pure KDP system because of the chemical similarity between K and Na atoms. The structure of pure KDP is tetragonal at room temperature (Hellwege 1975). It was found that the structure of binary systems was also tetragonal but for the change in the lattice parameters *a* and *c*. The values of *a* and *c* decreased with addition of H_3BO_3 in comparison with those of pure KDP. Hence the volume of the unit cell decreased resulting in decrease in the number of molecules in the unit for both systems. The decrease of *a* and *c* parameters in both the systems was thus attributed to the addition of H_3BO_3.

3. Conclusions

The powder X-ray diffraction reveals that while both the binary systems NaDP and KDP with H_3BO_3 retained the tetragonal nature, there was a decrease in the unit cell parameters.

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