08.2-9 NON-PERIODIC PATTERSON FUNCTION OF β-AgI. By M. Tokonami and N. Haga. Faculty of Science. University of Tokyo. Tokyo. Japan and K. Koto and A. Yoshiasa. ISIR. Osaka University. Osaka. Japan.

The experimental approach to find out the difference of the correlation of atomic fluctuations between the nearest neighbors from that between the sufficiently distant atoms has been done by means of X-ray single crystal diffractometry. Fourier transform of a square of continuous $|F|$ distribution would give a kind of Patterson function map. which has pseudoperiodicity in the Patterson space. The peaks near the origin in that Patterson map should be very sharp caused by little influence of thermal motion or positional disorder. Whereas. those in the distant region from the origin should correspond to the ordinary Patterson peaks. which are averaged in the unit cell scale. They are considered to be the products of two atoms vibrating independently.

β-AgI is well known as the crystal which has the characteristic diffuse streaks caused by the extreme thermal motion of atoms. The diffuse intensity mostly appears in the plane parallel to $\{10\bar{1}0\}*$ and in the rods parallel to $a*$ and $c*$ axes. This wurtzite-type compound should be the convenient specimen for executing the experiment for the above purpose.

Integrated intensities around the points corresponding to one fifth of the basic reciprocal cell vectors were measured on a four-circle diffractometer using monochromatized Mo $K\alpha$ radiation. The effect of thermal motion on atomic correlations according to distance will be discussed based on the Patterson synthesis of the observed intensity distribution.

interatomic distances and bond angles of $(B_3O_6)^{3-}$ rings are listed in Tables 1 and 2. The $(B_3O_6)^{3-}$ ring is shown in Fig. 1.

The result of structure determination indicates that both $CaB_2(B_3O_6)_2$ and high temperature phase BaB_2O_4 (A. D. Mighell. A. Perloff. S. Block. Acta Cryst. 20, 1966, 819.) are isostructural compounds. The high temperature structure of BaB_2O_4 is stabilized by the substitution of Ca^{2+} for Ba^{2+}. as is the case in $Ba_{1-x}Sr_xB_2O_4$ (x<0.35) solid solution in the BaB_2O_4-SrO system(Wang Guofu, Huang Qingzhen, Liang Jingkui. Acta Chimica Sinica, 42, 6, 1984, 503.). The other isostructural compound $CdB_2(B_3O_6)_2$ (a=b=7.144 Å, c=34.562 Å, Z=6.) had been found by using Cd^{2+}. It can be seen that the high temperature phase structure of BaB_2O_4 is stabilized by the substitution of bivalent cations with smaller ionic radius for Ba^{2+}. But no isostructural compounds can be formed by the substitution of Zn^{2+} and Mg^{2+} with much smaller ionic radius.

Table 1. The final atomic parameters.

Positions		x/a	y/b	z/c
O(1)	36(f)	0.159	0.213	0.0449
O(2)	36(f)	0.420	0.104	0.0403
B	36(f)	0.944	0.162	0.0425
Ba	12(c)	0	0	0.3515
Ca	6(a)	0	0	0.25

Fig. 1. The $(B_3O_6)^{3-}$ ring.
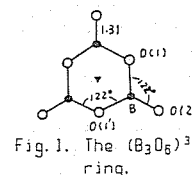
Table 2. The interatomic distances and bond angles.

$(B_3O_6)^{3-}$ Distances		$(B_3O_6)^{3-}$ Angles	
B-O(1)	1.40 Å	B-O(1')-B	122°
B-O(1')	1.39 Å	O(1')-B-O(1)	118°
B-O(2)	1.31 Å	O(1')-B-O(2)	120°
		O(2)-B-O(1)	122°
B-B	2.43 Å	Ba-O and Ca-O distances	
		Ba-O(2)	2.97 Å
O(1)-O(1')	2.37 Å	Ba-O(1)	2.99 Å
O(1)-O(2)	2.36 Å	Ba-O(2')	2.68 Å
O(1')-O(2)	2.35 Å	Ca-O(2)	2.28 Å

08.2-10 CRYSTAL STRUCTURE DETERMINATION OF $CaBa_2(B_3O_6)_2$ BY POWDER DIFFRACTION METHOD. BY Huang Qingzhen, Zhou Zicong, Liang Jingkui.. Fujian Institute of Research on the Structure of Matter, Academia Sinica, China.

The pseudo-binary system BaB_2O_4-CaB_2O_4 has been studied by means of thermal analysis and X-ray diffraction. A compound $CaBa_2(B_3O_6)_2$ has been formed in the system. which melts congruently at 1117±3°C. There exist eutectic horizontals from BaB_2O_4 to $CaBa_2(B_3O_6)_2$ at 1076±3°C and from $CaBa_2(B_3O_6)_2$ to CaB_2O_4 at 1046±3°C. Both interplanar distances and diffraction intensities of powder pattern of $CaBa_2(B_3O_6)_2$ have been collected by Guinier-de Wolff camera and D/max-rA diffractometer respectively. The density measured is ρ=3.64g/cm^3. The IR absorption spectra show that there may be the $(B_3O_6)^{3-}$ rings of boroxol type in $CaBa_2(B_3O_6)_2$. A powder SHG test has been carried out. but no SHG effect has been observed. The indexing of the X-ray powder diffraction pattern indicates that $CaBa_2(B_3O_6)_2$ belongs to the trigonal system with the unit cell dimensions: a=7.157Å, c=35.298Å in hexagonal lattice. There are 6 formula units in one hexagonal unit cell. ρ=3.64g/cm^3. Only the reflections of type $-h+k+l$=3n are observed while the reflections $h0l$ with odd l are absent. This limits the possible space groups to R3c and R$\bar{3}$c. From the fact that no SHG effect can be observed. the space group is assumed to be R$\bar{3}$c. A further structure refinement has been conducted by the comparison between the calculated intensities I_c and the observed intensities I_o. $I_c = M \cdot h \cdot PL \cdot F^2 \exp(-2B\sin^2\theta/\lambda^2)$, where M is the multiplicity factor, PL the Lorentz polarization factor, the atomic scattering factor in F is calculated by relativistic Hartree-Fock wave function(D. T. Cromer, J. T. Waber, "International Table for X-ray Crystallography." Vol. IV, PP. 71, 184. Birmingham, Kynoch, 1974.) refined by anomalous scattering. B, the average temperature factor, and h, the proportional constant. By using $2B$=3.8Å^2, we obtain the refinement result $R = \Sigma|I_c-I_o|/\Sigma I_o$=11% with 75 possible reflections of I_c>0.25 (the strongest one is 100.). The final atomic parameters.

08.2-11 INTERACTION PRODUCTS OF CONDENSED PHOSPHORIC ACIDS WITH VARIOUS AMINES AND AMINO ACIDS. By M.T. Averbuch-Pouchot and A. Durif, Laboratoire de Cristallogaphie, associé à l'U.S.T.M.G., C.N.R.S., 166 X, 38042 Grenoble Cedex (France).

Interactions between amines, amino acids and various condensed phosphoric acids ($H_4P_2O_7$, $H_3P_3O_9$, $H_5P_3O_{10}$, $H_4P_4O_{12}$...) have been investigated. Being given the instability of the condensed phosphoric acids a metathesis reaction between the amine chlorohydrates and the phosphoric silver salts has been used for their preparation. A typical reaction of this kind is :

$$3[(CH_3)_2CH.NH_3]Cl + Ag_3P_3O_9 \rightarrow [(CH_3)_2CHNH_3]_3P_3O_9 + 3AgCl$$
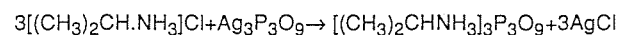
We describe here the first results obtained in the case of the cyclic phosphoric acid : $H_3P_3O_9$.

Formules	a	b	c	β	S.G.	Z
$(CH_3.NH_3)_3P_3O_9$	12.144	15.362	7.203	97.32	$P2_1/n$	4
$(C_2H_5.NH_3)_3P_3O_9$	16.284	19.17	12.425	118.62	C2/c	4
$[(CH_3)_2CH.NH_3]_3P_3O_9$	25.22	12.225	15.451	123.90	C2	8
$(CH_2.COOH.NH_3)_3P_3O_9$	12.31	14.62	10.146	100.40	$P2_1/c$	4

To the difference with previous investigations made with monophosphoric acid : H_3PO_4 the phosphoric group is here a non acidic one : P_3O_9. The three dimensional cohesion is assured by hydrogen bonds between the external oxygen atoms of the P_3O_9 ring anion and the hydrogen of the NH_3 radicals.