du niobium pur puisque les distances Nb–Nb sont normalement supérieures à 2,5 Å (2,54 Å dans $Nb_5Si_3\beta$) dans ces types d'alliages.

Ces distances métal–métal (2,48 Å) de la structure de $Nb_{78}Fe_{40}Si_{80}$ sont par ailleurs très voisines de celles caractérisant les chaînes Fe–Fe de $Nb_4Fe_4Si_7$ (2,49 Å) ou Cr–Cr de Nb_4CrSi_3 (2,47 Å).

Donc, à partir d'une occupation des sites Fe(3) et Fe(6) en fer pur, correspondant à la formule $Nb_{76}Fe_{42}Si_{80}$ l'alliage au niobium doit être limité et le domaine d'homogénéité ne s'étend pas bien au-delà de la composition des cristaux analysés, comme le confirment les résultats de l'étude cristallographique sur poudres, et la deuxième composition limites $Nb_{80}Fe_{38}Si_{80}$, doit correspondre pour les deux sites concernés par les substitutions Nb–Fe à un taux d'alliage d'environ 50%.

Les auteurs remercient le Dr H. D. Flack (Université de Genève) pour les conseils qu'il leur a donnés lors de la mise en œuvre du programme *CAMEL JOCKEY* sur l'ordinateur Iris 80 de Nancy.

Références

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.

Flack, H. D. (1975). *J. Appl. Cryst.* **8**, 520–521.

Henoc, J. (1974). *Application du Programme CØR à la Microanalyse par Sonde Electronique.* Document PEC n° 176, CNET.

Jeitschko, W., Jordan, A. G. & Beck, P. A. (1969). *Trans. Metall. Soc. AIME*, **245**, 335–339.

Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J. P. (1978). *MULTAN 78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. de York, Angleterre, et Louvain-la-Neuve, Belgique.

Parthé, E., Lux, B. & Nowotny, H. (1955). *Monatsh. Chem.* **86**, 859–867.

Steinmetz, J., Albrecht, J. M., Zanne, M. & Roques, B. (1975). *C. R. Acad. Sci. Sér. C*, **281**, 831–833.

Steinmetz, J., Malaman, B., Albrecht, J. M. & Roques, B. (1976). *C. R. Acad. Sci. Sér. C*, **282**, 823–825.

Steinmetz, J. & Steinmetz, P. (1979). A paraître.

Acta Cryst. (1979). B**35**, 2514–2518

Hydrogen Bonding in $MgHPO_4.3H_2O$ (Newberyite)

By F. Abbona,* R. Boistelle and R. Haser

Centre de Recherche sur les Mécanismes de la Croissance Cristalline, CNRS, Centre Scientifique de St Jérôme, 13397 Marseille CEDEX 4, France

(*Received* 31 *May* 1979; *accepted* 9 *July* 1979)

Abstract

The structure of pure synthetic crystals of $MgHPO_4.3H_2O$ (newberyite) [a = 10·203 (3), b = 10·678 (4), c = 10·015 (3) Å, Z = 8, space group *Pbca*] was refined to R = 0·047 for 1137 non-zero reflections measured on an automatic diffractometer with Cu $K\alpha$ radiation. All H atoms were located on a difference map and included in the refinement. The structure can be described as layers parallel to (010), built up by Mg octahedra sandwiched between phosphate tetrahedra, both running along [001]. These layers are only connected by hydrogen bonds involving the water molecules. The hydrogen-bond system is discussed and the functions of the different water molecules are described. One of them forms a bifurcated hydrogen bond.

Introduction

$MgHPO_4.3H_2O$ is found as a mineral (newberyite) and as a constituent of human and animal calculi. The significance of its presence in human urinary stones is still controversial: the question is whether newberyite has its primary origin in stones or, as is more probable, it is a decomposition product of struvite ($MgNH_4PO_4.6H_2O$), with which it is usually associated (Sücker, 1963; Sutor, 1968; Whitaker, 1968). In felines, on the contrary, newberyite appears to be deposited as such (Sutor, Wooley & Jackson, 1970).

The crystal structure of newberyite, as regards the heavy atoms, was determined by Sutor (1967) on a small elongated fragment of natural crystal from Skipton Caves (Victoria, Australia), but no attempt was made to locate the H atoms; nevertheless some hydrogen bonds were suggested on the basis of O···O contacts.

---

* On leave from Istituto di Mineralogia e Cristallografia dell'Università, Via S. Massimo 22, 10123 Torino, Italy.

0567-7408/79/112514-05$01.00

In our research programme on calculi growth, we are investigating the structural and genetic relationships between struvite and other crystalline components of calculi, among them newberyite. That implies knowledge of the surface structure of many crystal faces and the characterization of the bond system in the structure; for newberyite, therefore, the exact localization of the H atoms is of prime importance.

As large single crystals of newberyite were obtainable, we succeeded in locating the H atoms by X-ray methods. We present here the results of our work, which has been carried out on synthetic pure crystals, with a discussion of the hydrogen-bond system; the work also represents a contribution to the crystal chemistry of acidic magnesium orthophosphates.

Experimental

Synthetic crystals of newberyite can be obtained by a variety of methods (De Schulten, 1903). By a new method, which involves mixing aqueous solutions of magnesium chloride or sulphate with ammonium dihydrogenphosphate at 310 K, and adjusting the pH to about 5·5 with ammonium hydroxide, we prepared large colourless pseudo-octahedral single crystals. The crystal habit is characterized by the large development of the {111} bipyramid, and by the occurrence of small or very small faces belonging to the forms {100}, {010}, {101} and {102}.

Weissenberg and precession photographs and single-crystal diffractometry confirmed orthorhombic symmetry with space group *Pbca*. Cell parameters [a = 10·203 (3), b = 10·678 (4), c = 10·015 (3) Å] were obtained by least squares from 25 centred reflections, measured on a diffractometer (Cu $K\alpha$ radiation). They are in excellent agreement with those measured using very pure crystals from Mono Lake (California), but smaller than those of impure crystals from Skipton Caves (Australia), where 0·36 wt.% Mn probably replaces Mg (Cohen & Ribbe, 1966; Sutor, 1967); a is more sensitive to the replacement than the other two cell constants.

Other data are: $Z = 8$, $V = 1091\cdot11$ Å^3, $M_r = 174\cdot34$, $D_o = 2\cdot123$ (De Schulten, 1903), $D_c = 2\cdot122$ Mg m^{-3}, $F(000) = 720$, $\mu(\text{Cu } K\alpha) = 5\cdot5$ mm^{-1}.

The crystal used for data collection was shaped into a sphere of 0·4 mm diameter. The intensities were collected on an Enraf–Nonius CAD-4 diffractometer with the θ–2θ scan mode, a scan width of 0·9° and a scan speed of 2° min^{-1}. Cu $K\alpha$ radiation with a Ni filter was used to record reflections up to $2\theta = 175°$. 2450 reflections (*hkl* and $\bar{h}kl$) were measured. A periodic check of several standard reflections showed no significant intensity variation. Equivalent intensities were merged into a set of 1230 independent reflections. The internal consistency index $R_I = \sum (I - \bar{I})/\sum I$, where $\bar{I}$ is the weighted mean of *hkl* and $\bar{h}kl$ intensities, was 3·6%.

All data were corrected for absorption (sphere approximation), Lorentz and polarization effects. The e.s.d. of each intensity, $\sigma(I)$, was calculated according to the method of Stout & Jensen (1968). On the basis of a $2\sigma(I)$ test, 1137 reflections were considered observed. Scattering factors are from Moore (1963). Real and imaginary dispersion corrections were applied for P and Mg (Cromer & Waber, 1974).

Refinement

Refinement was started with atomic parameters reported by Sutor (1967). The initial $R = (\sum ||F_o| - |F_c||)/\sum |F_o| = 0\cdot11$. During these calculations we observed that the strongest reflections were significantly affected by secondary extinction. We excluded these from the refinement and Fourier syntheses. In the final structure factor calculation these intensities were corrected, according to $I_{\text{corr}}/I_{\text{obs}} = k(1 + 2gI_{\text{corr}})$. k and g, determined by least squares from the plot, are respectively 1·02 and 3×10^{-5} e^{-2}. Full-matrix least-squares refinement was based on F and the function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w(F) = 1/\sigma^2(F)$. For the nine non-hydrogen atoms, which were assigned anisotropic thermal parameters, the refinement converged to $R = 0\cdot056$ and $R_w = 0\cdot065$. At this stage a difference map revealed the positions of the seven H atoms. Further refinement was carried out with H atoms included, their positional and isotropic thermal parameters being varied simultaneously with all the non-hydrogen atom parameters. The final R was 0·047 ($R_w = 0\cdot060$) and 0·058 for all observed data (strong intensities corrected for secondary extinction); a dif-

Table 1. *Fractional atomic coordinates* ($\times 10^4$; *for* H $\times 10^3$) *with significant figures of the e.s.d.'s in parentheses*

	x	*y*	*z*
P	−89·9 (7)	1328·7 (7)	1537·2 (8)
Mg	2969·4 (9)	2466·3 (9)	862·0 (9)
O(3)	−884 (3)	1996 (2)	2578 (3)
O(4)	−514 (2)	1617 (2)	98 (2)
O(5)	1392 (2)	1535 (2)	1681 (2)
O(6)	−341 (3)	−128 (2)	1783 (3)
W(7)	1671 (3)	3129 (3)	−614 (3)
W(8)	2500 (3)	4125 (3)	1894 (3)
W(9)	3539 (3)	924 (3)	−319 (3)
H(1)	174 (5)	310 (5)	−161 (5)
H(2)	80 (5)	290 (4)	−53 (4)
H(3)	278 (5)	485 (5)	167 (5)
H(4)	175 (4)	429 (4)	232 (4)
H(5)	313 (8)	88 (8)	−97 (8)
H(6)	351 (5)	−4 (5)	−4 (6)
H(7)	−16 (6)	−62 (7)	98 (6)

ference map was now featureless. Final positional parameters are given in Table 1.*

Description and discussion of the structure

Coordination polyhedra

The P—O and O—O distances and O—P—O angles within the phosphate tetrahedron are given in Table 2 or in Fig. 1; for clarity the atom numbering is the same as that of Sutor (1967), except for the O atoms of water molecules which are indicated as *W*. Our values are very close to those found by Sutor (1967). The three distortion indices (Baur, 1974) for the P—O and O—O distances and the O—P—O angles (0·018, 0·0076, 0·029, respectively) correspond to the average values for acidic orthophosphates (0·017, 0·011, 0·025, respectively).

Even the Mg—O and O—O distances and the angles O—Mg—O within the Mg octahedron (Table 3, Fig. 2)* are very close to those reported by Sutor (1967); the difference between the average values is 0·24%.

Hydrogen bonding

The geometrical features of the H bonds are shown in Table 4; distances and angles involving the H atoms are satisfactory, taking into account that X-ray diffraction shortens the O—H distance. The acidic H(7) atom is attached to O(6), as Sutor (1967) suggested, and forms a H bond with O(4,2),* which is 1·67 Å

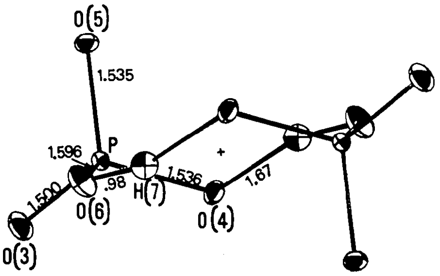

Fig. 1. Hydrogen bonding of H(7) between the centrosymmetrically related PO_4 groups (+ denotes the centre of symmetry).

Table 2. *Interatomic distances* (Å) *and* O—P—O *angles* (°) *within the* PO_4 *group*

The e.s.d.'s are 0·002 and 0·003 Å for the P—O and O—O distances respectively and 0·1° for the angles.

P—O(3)	1·500	P—O(5)	1·535
P—O(4)	1·536	P—O(6)	1·596
Average	1·542		
O(3)—O(4)	2·544	O(4)—O(5)	2·511
O(3)—O(5)	2·538	O(4)—O(6)	2·520
O(3)—O(6)	2·467	O(5)—O(6)	2·509
Average	2·515		
O(3)—P—O(4)	113·8	O(4)—P—O(5)	109·7
O(3)—P—O(5)	113·5	O(4)—P—O(6)	107·1
O(3)—P—O(6)	105·6	O(5)—P—O(6)	106·5
Average	110·4		

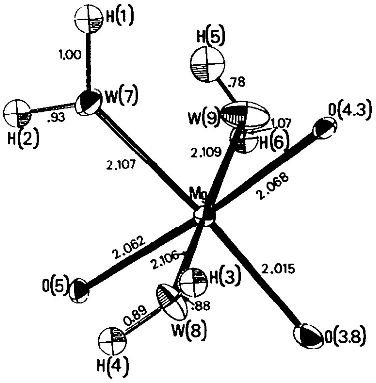

Fig. 2. Projection along [0$\bar{1}$0] showing the Mg-atom environment.

Table 3. *Interatomic distances* (Å) *and* O—Mg—O *angles* (°) *in the* Mg *coordination polyhedron*

The e.s.d.'s are 0·002 and 0·003 Å for Mg—O and O—O distances, respectively, and 0·1° for the angles.

Mg—O(3,8)	2·015	O(3,8)—Mg—*W*(8)	87·8
Mg—O(4,3)	2·068	O(3,8)—Mg—*W*(9)	94·6
Mg—O(5)	2·062	O(3,8)—Mg—O(5)	91·4
Mg—*W*(7)	2·107	O(3,8)—Mg—O(4,3)	92·6
Mg—*W*(8)	2·106	*W*(7)—Mg—O(5)	87·2
Mg—*W*(9)	2·109	*W*(7)—Mg—*W*(9)	92·4
Average	2·078	*W*(7)—Mg—*W*(8)	85·3
		W(7)—Mg—O(4,3)	89·1
O(3,8)—*W*(8)	2·858	*W*(8)—Mg—O(5)	91·9
O(3,8)—*W*(9)	3·032	*W*(8)—Mg—O(4,3)	90·0
O(3,8)—O(5)	2·918	*W*(9)—Mg—O(5)	93·5
O(3,8)—O(4,3)	2·952	*W*(9)—Mg—O(4,3)	84·4
W(7)—O(5)	2·875	Average	90·0
W(7)—*W*(9)	3·043		
W(7)—*W*(8)	2·856	O(5)—Mg—O(4,3)	175·7
W(7)—O(4,3)	2·930	*W*(7)—Mg—O(3,8)	172·9
W(8)—O(5)	2·995	*W*(8)—Mg—*W*(9)	174·0
W(8)—O(4,3)	2·953		
W(9)—O(5)	3·039		
W(9)—O(4,3)	2·807		
Average	2·938		

---

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34603 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Atoms are labelled as follows: a single number (or no number) denotes an atom of the asymmetric unit; a second number, ranging from 2 to 8, indicates positions: (2) $\bar{x},\bar{y},\bar{z}$; (3, 4) $\pm(\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$; (5, 6) $\pm(\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z)$; (7, 8) $\pm(\frac{1}{2}-x, \bar{y}, \frac{1}{2}+z)$. Translations are represented by Roman numbers: (I) $-c$; (II) $-b$.

distant from H(7). This H bond, which deviates significantly from linearity [O(6)–H(7)···O(4,2) = 162°], is the strongest H bond in the structure, and corresponds to the typical distance for this kind of bond, 2·615 Å (Fig. 1).

As expected, the largest P–O distance within the PO_4 group corresponds to the short H–O bond. All the H atoms of water molecules are engaged in H bonds of different strengths; *W*(7) and *W*(8) form H bonds only with O atoms of the PO_4 group, whereas *W*(9) is hydrogen bonded only to O atoms of the water molecules. *W*··· *W* contacts range from 2·986 to 3·118 Å, indicating weak H bonds, while the O–*W* contacts range from 2·690 to 2·845 Å corresponding to medium–strong hydrogen bonds.

Besides the two H bonds shown in Table 4, *W*(7) is connected to O(3,3) by H(1) [H(1)···O(3,3) = 2·611 Å] but this contact cannot be considered as a H bond, since H(1) is already engaged in a strong H bond with O(5,6)$^{\rm I}$. Moreover O(3,3)···H(1)–*W*(7) = 116° is too far from the linearity condition and normal geometry of a water molecule (Ferraris & Franchini-Angela, 1972).

W(8) forms two H bonds, one for each H atom: H(3) to O(5,4) (*W*···O = 2·819 Å) and H(4) to O(6,5) (*W*···O = 2·690 Å). H(3) is also in close contact with O(6,4) and *W*(9,4) at 2·616 and 2·652 Å respectively; even if the *W*···O distances are acceptable for H bonds [*W*(8)···O(6,4) = 3·009; *W*(8)···*W*(9,4) = 3·118 Å], the contacts cannot be considered as H bonds, since the corresponding donor–H–acceptor angles are 108 and 114°, respectively. Moreover O(6,4) and *W*(9,4) are 1·40 and 2·33 Å out of the water-molecule plane.

The water molecule *W*(9) is near three water molecules, *W*(7,4)$^{\rm II}$, *W*(8,4)$^{\rm II}$ and *W*(8,6)$^{\rm I}$, at similar distances, *W*(9)··· *W*(8,6)$^{\rm I}$ = 2·986, *W*(9)··· *W*(7,4)$^{\rm II}$ = 3·007, *W*(9)···*W*(8,4)$^{\rm II}$ = 3·118 Å; with the first, *W*(8,6)$^{\rm I}$, H(5) forms one H bond. As the distances H(6)··· *W*(7,4)$^{\rm II}$ and H(6)··· *W*(8,4)$^{\rm II}$ are smaller than 2·4 Å, the sum of the corresponding van der Waals radii, H(6) is thought to form a bifurcated H bond of approximately the same strength with each of these two water molecules, even though one of them is quite out of the *W*(9) plane [*W*(7,4)$^{\rm II}$ is 0·44 and *W*(8,4)$^{\rm II}$ 1·73 Å from the plane, with *W*(9)–H(6)··· *W*(7,4)$^{\rm II}$ = 148 and *W*(9)–H(6)··· *W*(8,4)$^{\rm II}$ = 126°].

The H-bond system found in newberyite excludes the possibility, suggested by Sutor (1967), of H bonds within the Mg octahedron.

$CaHAsO_4.3H_2O$ is isostructural with newberyite (Catti & Ferraris, 1973). The H bonding involving *W*(7) and *W*(8) is the same as that of *W*(3) and *W*(1), respectively, in $CaHAsO_4.3H_2O$. An anomaly occurs in the third water molecule, *W*(9) in newberyite and *W*(2) in the arsenate: whereas H(5) in newberyite forms a H bond with *W*(8,6)$^{\rm I}$, in the arsenate the corresponding H(5) atom is thought to be almost free from H bonding; the other H atom in newberyite, H(6), forms a bifurcated H bond, while the corresponding H atom in the arsenate, H(4), forms a single H bond.

Crystal packing

The structure consists of alternating P and Mg strata parallel to (100) (Sutor, 1967), but it may be also described in terms of (010) layers (Fig. 3). These layers are built up by Mg octahedra sandwiched between phosphate tetrahedra, running along [001]. Three O atoms of each PO_4 tetrahedron are nearly parallel to (010), whereas the fourth points up and down alternately along [001]. Within the (010) layer the three O atoms of the PO_4 group are connected along [100] and [001] to as many Mg atoms forming a network of strong bonds. Additional interactions in the (010) plane are provided by H bonds between the water molecules and the O atoms in the [100] and [001] directions. Between the (010) layers only H bonds are established, because of the different roles of water molecules. *W*(7) forms its H bonds within the (010) layer, connecting the polyhedra along [100] and [001]. *W*(8) ties one Mg octahedron of the layer to two PO_4 groups of the facing layer, whereas *W*(9) forms one of its H bonds within the (010) Mg octahedra of the facing layer.

Electrostatic balance

Bond-strength values were obtained by applying the universal third-row relationship $s = (R/1{\cdot}622)^{-4{\cdot}29}$ (Brown & Shannon, 1973) for P–O and Mg–O bonds and the *s versus* O···O distance curve (Brown, 1976) for H bonds; the results are summarized in Table 5.

Table 4. *Interatomic distances* (Å) *and angles* (°) *in hydrogen bonds*

The e.s.d.'s are 0·05 Å and 5° for distances and angles involving H atoms.

AB	*BC*	*AC*	*ABC*	*A* *B* *C* *D* *E*	*CE*	*CD*	*DE*	*CDE*	*BCD*
1·794	1·00	2·747	158	O(5,6)$^{\rm I}$···H(1)–*W*(7)–H(2)···O(4)	2·845	0·93	2·01	148	98
1·963	0·88	2·819	164	O(5,4)···H(3)–*W*(8)–H(4)···O(6,5)	2·690	0·89	1·80	171	102
2·229	0·78	2·986	164	*W*(8,6)$^{\rm I}$···H(5)–*W*(9)–H(6)···*W*(7,4)$^{\rm II}$	3·007	1·07	2·04	148	98
				···*W*(8,4)$^{\rm II}$	3·118	1·07	2·37	125	
				P–O(6)–H(7)···O(4,2)	2·615	0·98	1·67	162	111

Table 5. *Electrostatic balance*

	O(3)	O(4)	O(5)	O(6)	$W(7)$	$W(8)$	$W(9)$	$\sum s_i$
P	1·40	1·26	1·27	1·07				5·00
Mg	0·40	0·35	0·36		0·32	0·33	0·32	2·08
H(1)			0·18		0·82			1·00
H(2)		0·14			0·86			1·00
H(3)			0·15			0·85		1·00
H(4)				0·22		0·78		1·00
H(5)						0·07	0·93	1·00
H(6)					0·04	0·03	0·93	1·00
H(7)		0·28		0·72				1·00
$\sum s_i$	1·80	2·03	1·96	2·01	2·04	2·06	2·18	

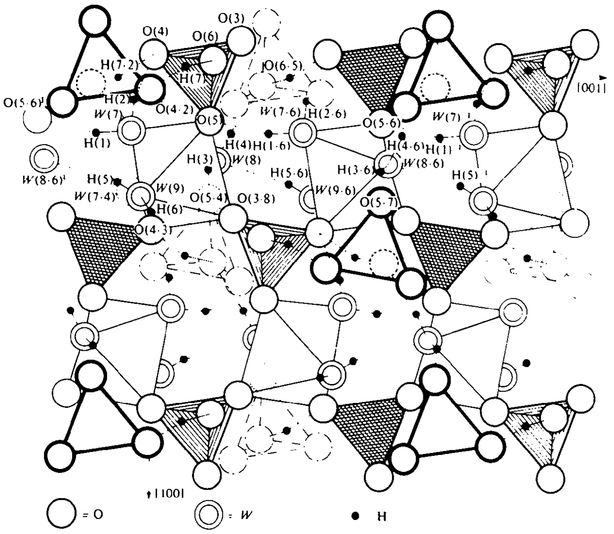

Fig. 3. Hydrogen-bond network in newberyite; projection along [0$\bar{1}$0].

The electrostatic balance for O atoms corresponds to the expected values according to the Pauling rules except for O(3) and $W(9)$, which are underbonded (s_i = 1·80) and overbonded (s_i = 2·18) respectively. The P—O(3) distance (1·500 Å) is one of the shortest found in orthophosphate groups. The underbonding of the O atom, which is usually observed in these cases, suggests some limited validity of Brown & Shannon's formula for short or very short bond distances. O(3) is the only O atom which is not an acceptor of a H bond; it has the strongest bond with P and Mg.

The difference in bond distances between Mg—O (average: 2·048 Å) and Mg—W (average: 2·107 Å) (Table 3) is due to the underbonding of O atoms [especially O(3)], which demands an approach of these atoms to the P and Mg atoms with respect to the water molecules, which exchange a small bond valence through their generally long H bonds.

The authors thank J. P. Astier for technical assistance, Professor G. Ferraris for his interest and criticism, and NATO for financial support (Grant 1438).

References

Baur, W. H. (1974). *Acta Cryst.* **B30**, 1195–1215.
Brown, I. D. (1976). *Acta Cryst.* **A32**, 24–31.
Brown, I. D. & Shannon, R. D. (1973). *Acta Cryst.* **A29**, 266–282.
Catti, M. & Ferraris, G. (1973). *Acta Cryst.* **B29**, 90–96.
Cohen, L. H. & Ribbe, P. H. (1966). *Am. Mineral.* **51**, 1755–1765.
Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV. Birmingham: Kynoch Press.
De Schulten, M. A. (1903). *Bull. Soc. Fr. Minéral. Cristallogr.* **26**, 24–29.
Ferraris, G. & Franchini-Angela, M. (1972). *Acta Cryst.* **B28**, 3572–3583.
Moore, F. H. (1963). *Acta Cryst.* **16**, 1169–1175.
Stout, G. H. & Jensen, L. H. (1968). *X-ray Structure Determination.* New York: Macmillan.
Sücker, I. (1963). *Naturwissenschaften*, **50**, 499–500.
Sutor, D. J. (1967). *Acta Cryst.* **23**, 418–422.
Sutor, D. J. (1968). *Nature* (*London*), **218**, 295.
Sutor, D. J., Wooley, S. E. & Jackson, O. F. (1970). *Res. Vet. Sci.* **11**, 298–299.
Whitaker, A. (1968). *Mineral. Mag.* **36**, 820–824.

Acta Cryst. (1979). B**35**, 2518–2522

The Structure of Potassium Tantalum Fluoride Oxide $K_{12}Ta_{15\cdot5}F_{18\cdot5}O_{35\cdot5}$

By A. Boukhari, J. P. Chaminade, M. Pouchard and M. Vlasse

Laboratoire de Chimie du Solide du CNRS, Université de Bordeaux I, *351 cours de la Libération,*
33405 *Talence CEDEX, France*

(*Received* 18 *April* 1979; *accepted* 9 *July* 1979)

Abstract

The structure of $K_{12}Ta_{15\cdot5}F_{18\cdot5}O_{35\cdot5}$ has been determined and refined in space group $P\bar{6}$ to an R of 0·063. It can be described as a three-dimensional framework formed by the interpenetration of two distinct sublattices with the formulations $[Ta_5X_{17}]_{3n}$ and $[Ta_{0\cdot5}X_3]_n$ (X = O, F). The $[Ta_5X_{17}]_{3n}$ skeleton is formed entirely of

0567-7408/79/112518-05$01.00