# The $Fe^{2+}_3(H_2O)_n[PO_4]_2$ Homologous Series. II. The Crystal Structure of $Fe^{2+}_3(H_2O)[PO_4]_2$

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#### Abstract

The synthetic compound  $Fe^{2}s\{H_2O\}[PO_3]_2$  has a 9.431(1)Å, b 10.066(1)Å, c 8.040(1)Å, b 117.632(7)°,  $P2_1/a$ , Z = 4. Its structure, refined to R(hkl) = 0.068 for 2457 independent reflections, consists of complex open framework formed by edge- and corner-linking of distorted Fe(1) and Fe(3) octhedra(a, of distorted Fe(2) polyhedra of five-fold oxygen coordination, and of PO, tetrahedra. Average bond distances are Fe(1)–O, 2.16 Å; Fe(2)–O, 2.08 Å; Fe(3)–O, 2.15 Å; P(1)–O, 1.55 Å; and P(2)–O, 1.54 Å. Unusual features of the structure include a Fe(2)–Fe(2)° O(6)–O(8) 2.41 Å shared edge; and a Fe(2)–Fe(2)° O(4)–O(4)° 2.63 Å shared edge. It is proposed that a bounded oxidative sequence may exist for the crystal, where Fe(1)\* and  $H_2O$  — OH —. The limiting formula for a stable oxidized crystal would be Fe\*\*, Fe\*\*(OH)[PO\_4].

### Introduction

The homologous series  $Fe^2 +_3(H_2O)_n[PO_4]_2$  includes several compounds where all water molecules and all phosphate oxygens are also bound to the transition metal. Well-characterized natural compounds include vivianite and metavivianite (n=8), luddamite (n=4), phosphoferrite (n=3), sarcopside and graftonite (n=0). In addition, several recently synthesized compounds have been brought to our attention, including the subject of this study, where n=1.

#### Experimental

Single crystals of  $Fe^{2+}_{3}(H_{2}O)[PO_{4}]_{2}$  were synthesized by Dr. E. Mattievich at 230°C and 400 bars pressure, using a hydrothermal technique with synthetic vivanite as starting material. Dr. Mattievich informs us that wet chemical analyses closely conform with the ideal formula and that Mössbauer resonance studies have been performed on the compound.

Preliminary single crystal Weissenberg and precession photographs establish the space group and cell parameters, the latter refined by twelve reflections on the Picker four-circle automated diffractometer. The results are a 9.431(1) Å, b 10.066(1) Å, c 8.040(1) Å,  $\beta$  117.632(7)°, space group  $P2_1/a, Z=4$ . A powder pattern, indexed with the aid of the single crystal data, appears in Table 1.

A superior single crystal measuring 0,09 mm along

a, 0.11 mm along b, and 0.04 mm along c was selected for intensity measurements. The compound is pale green in color, with crystals tabular parallel to (001) and striated parallel to [100]. Figure 1 features an idealized sketch of typical development of the crystals.

Successive shells of reflections were gathered to sin  $\theta/\lambda=0.8$  on the Picker diffractometer with MoKe<sub>1</sub> radiation and graphite monochromator. Full scans ranged from 2.4° at the lower levels to 2.8° at high angles with a scan speed of 2°/minute. Twenty-second background measurements were taken on each side of the peek. The data were processed to obtain F(obs) after applying an absorption correction by the Gaussian integral method described by Burnham (1966). All 2457 independent reflections were accepted for the ensuing study.

#### Structure Determination and Refinements

The Patterson synthesis, P(uvw), revealed that all metals reside in general positions, requiring coodinate determination for Fe(1), Fe(2), Fe(3), P(1), and P(2). Several minimum functions provided unambiguous location of the metals which afforded sufficient scattering matter for a  $\beta$ -general synthesis as described by Ramachandran and Srinivasan (1970). The ensuing map revealed all non-hydrogen atom locations without difficulty. Full-matrix least-squares refinement, including

Full-matrix least-squares refinement, including scale factor, non-hydrogen atomic coordinates and isotropic thermal vibration parameters with 2457 in-

TABLE 1. Fe, (H,O)[PO,], Powder Data\*

								_
I/Io	d(obs)	d(cale)	hkl	I/Io	d (obs)	d(cale)	hk1	
70	5.810	5.814	011	20	2,611	2,616	230	
2.0	4.146	4.155	Ĭ21	5	2.571	2.573	213	
40	4.097	4.100	111	40	2.512	2.517	040	
20	3.723	3.730	<b>1</b> 12	5	2.469	2.471	122	
10	3.553	3.561	002	20	2,435	2,437	320	
100	3.351	3.357	012	5	2.378	2.384	313	
15	3.030	3.035	031	30	2.324	2.328	I23	
20	2,984	2,992	311	10	2.039	2.045	410	
15	2,903	2.911	211	20	2.003	2.000	204	
10	2.722	2.729	112	10	1.894	1.896	123	
15	2.680	2.687	131	10	1.833	1.835	324	
45	2.655	2.659	321	10	1.801	1.805	124	
45	2.055	2.005	361		1.672	1.675	242	
				15	T*0/2	1.0/5	242	

<sup>\*</sup>Powder diffractogram, 19/minute, CuK $\alpha$  radiation, Si (a = 5.4301 Å) internal. Indices are based on strong single crystal reflections and cell parameters stated in the text.

dependent reflections converged to  $R(hkl) = \sum |F(obs)| - |F(calc)|/\sum |F(obs)| = 0.068$ . For the 1569 reflections exceeding three times the estimated background error, R(hkl) = 0.039. Estimated standard errors in distances are Me-O  $\pm$  0.004 Å and O-O'  $\pm$  0.006 Å. Scattering curves for Fe<sup>1+</sup>, P<sup>2</sup>, and O-C'  $\pm$  0.006 Å. Scattering curves for Fe<sup>1+</sup>, P<sup>2</sup>, and O-C' derives from the subset of Corper and Mann

# Description of the Structure

Topology and Geometry

The crystal structure of Fe<sup>2+</sup><sub>3</sub>(H<sub>2</sub>O)[PO<sub>4</sub>]<sub>2</sub> is complex, and bears no obvious relationship with any

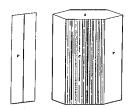


Fig. 1. Typical crystal of Fe<sup>1+</sup>, (H<sub>2</sub>O)[PO<sub>4</sub>], showing the forms c[001], v[011] and s[201]. A. Plan; B. Clinographic projection.

On-0 derive from the tables of Cromer and Mann (1968).

Final atomic coordinate parameters appear in Table 2, and the structure factor data are presented in Table 3. In the distribution of the homologous series  $fe^{+}_{a}(H_{a}O)_{b}(PO_{a})_{b}$ , all n-water molecules are aquated (are found as ligand groups) to the transition metals—the coordinated (= M) and one five coordinated (= S)—and nine oxygen-ligand vertices,  $\phi$ , per formula unit. Characteristic of the homologous series  $fe^{+}_{a}(H_{a}O)_{b}(PO_{a})_{b}$ , all n-water molecules are aquated (are found as ligand groups) to the transition metals—the coordinated (= M) and one five coo on and all oxygens associated with the [PO<sub>4</sub>]? ligand (= Op) are also bound to transition metals.

In a detailed study on this homologous series cer-

tain common characteristics among these structures were emphasized (Moore, 1971). As n decreases, the degree of octahedral condensation or polymerization increases: for n = 8 (vivianite), the octahedral com-

TABLE 2. Atomic Coordinate and Isotropic Thermal

	VIDIATION I	arameters for i	res(triso)(rot);	
Atom	×	У	z	B(Å <sup>2</sup> )
Fe(1)	0.0315(1)	0.1359(1)	0.4130(1)	0.82(1)
Fe(2)	.4386(1)	.8771(1)	.0749(1)	.69(1)
Fe(3)	.2813(1)	.1241(1)	.2628(1)	.61(1)
P(1)	.6740(1)	.0873(1)	.3786(2)	,49 (2)
P(2)	.1343(1)	.8320(1)	.0262(2)	,55(2)
0(1)	.8035(4)	.1876(4)	,3950(5)	.72(5)
0(2)	.7485(4)	,0292 (4)	,5172(S)	.67(5)
o(3)	.5430(4)	.1556(4)	.4110(5)	.78(5)
0 (4)	.5993(4)	.0308(4)	.1747 (5)	.79(5)
0 (5)	0188(4)	.8713(4)	1491(5)	.84(5)
0(6)	.2533(4)	.9497(4)	,1113(5)	.78(5)
0(7)	_0909 (4)	.7740(4)	.1723(5)	.91(5)
0(8)	,2342(4)	.7328(4)	-,0255(S)	.B1(5)
OM	.0715(4)	.0825(4)	,6840(S)	.98(5)

\*Estimated standard errors refer to the last digit

<sup>&</sup>lt;sup>1</sup>To receive a copy of Table 3, order document number AM-75-003-A from the Business oflice, Mineralogical Society of America, 1909 K Street, N.W., Washington, D.C. 20006. Please remit \$1.00 for the microfiche.

bination includes the edge-sharing dimer  $M_2\phi_{10}$  + the monomer  $M\phi_0 = M_3\phi_{10}$ , which is equivalent to the expression  $\phi_{10} = (H_2O)_a + (Op)_b$ . For n = 4 (Judlamite) the cluster formula is  $M_3\phi_{12}$  and the structure contains corner-linked chains of  $M_3\phi_1$ . linear edge-sharing trimers bridged by the [PO<sub>4</sub>] tetrahedra. For n = 3 (phosphoferrite), these linear edge-sharing trimers  $M_3\phi_1$ , fuse by further edge- and corner-sharing to form  $M_3\phi_1$ , sheets. For n = 0, one of the dimorphs is sarcopside (cf Moore, 1972) which is an ordered derivative of the olivine structure type. With the cluster formula  $M_3\phi_3$ , we observe the same linear edge-sharing trimers as the structural motif. Since Fe<sup>1+</sup>, (H<sub>2</sub>O)[PO<sub>4</sub>]<sub>2</sub> is compositionally wedged in between, we had anticipated that it, too, would possess these linear edge-sharing trimers

The Fe<sub>3</sub>(H<sub>2</sub>O)[PO<sub>4</sub>]<sub>2</sub> structure not only reveals the absence of such linear trimers, but also provides two kinds of oxygen coordination about the transition metals: two distorted octahedra and one very distorted tetragonal pyramid! In addition, this awkward structure has resisted any obvious vehicle of description, and we despaired of finding a suitable projection until we selected a projection down a' = a - c, showing the plane  $b \times c' \sin \beta'$ , where c' = a + c. A slab of the structure, where the transition metal coordinates are bounded by  $4 \le x' \le \frac{1}{2}$ , is provided in Figure 2. This projection, which features the transition metal polyhedra only, has advantages in that all three non-equivalent metals can be referred to a common level in x'. At  $x' = \frac{1}{2}$ , the polyhedra are stippled, revealing a chain which runs parallel to  $c' \sin \beta'$  with a crankshaft motif. Equivalent chains are further

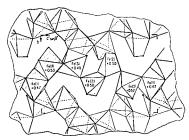


Fig. 2. Polyhedral diagram of the Fe-O arrangement between  $N \leq x' \leq N$ , where a' = a = c and c' = a + c. The chain-like character is shown by stipping. Solid disks denote linkages of corners above and below this slab.

linked above and below to form a highly complex polyhedral framework. The Fe(1)-O octahedron shares three of its edges; one with Fe(1), namely OW'-OW, and two with Fe(3)'s, namely O(1)-O(3)'l and O(2)'-O(5)'. This immediate neighborhood is the 3e(2)-cluster of Moore (1974a). The Fe(3)-O octahedron shares two edges with Fe(1) octahedra. Considering only the octahedra, this is the 2<sub>1</sub>(2) configuration of Moore (1974a). The Fe(2)-O distorted tetragonal pyramids share a mutual edge O(4)-O(4)' whose center is the center of inversion. In addition, an unanticipated feature, the edge O(6)-O(8) is shared with the P(2)O, tetrahedron. We feel it necessary and desirable to point out the edge-sharing neighborhoods, for they have considerable implications in homonuclear electron transfer-absorption where the probability of transfer is increased as the distance between metal centers is decreased.

As yet, no theory predicts or explains a priori the existence of such an unusual structure. In many respects, the structure is reminiscent of the complex graftonite structure. Although sarcopside is a limiting hexagonal close-packed oxygen structure, the high-temperature dimorph graftonite bears no obvious relationship with it. This unusual structure, as shown by Calvo (1968), possesses three non-equivalent metal positions, two five-coordinated and one seven-coordinated. Despite attempts at several projections, no obvious relationship to Fe<sub>3</sub>(H<sub>2</sub>O)[PO<sub>3</sub>], no edges in graftonic are shared between the PO<sub>4</sub> tetrahedra and the five-coordinated polyhedra. It is possible that these unusual structures can be rationalized via some spherepacking theory.

# Bond Distances and Angles

Polyhedral interatomic distances are listed in Table 4. The averages for the Fe<sup>2+</sup>-O octahedral and P-O tetrahedral distances are within the range reported for numerous well-refined structures. The polyhedral interatomic angles in Table 5 reveal rather severe distortions for the octahedra, ranging from O(1)<sup>3</sup>-Fe(3)-O(3) 76.9° to O(2)<sup>3</sup>-Fe(1)-O(3)<sup>3</sup> 116.8°.

The Fe(2)-O polyhedron possesses five vertices, but its angular distortions are so severe that distinction between tetragonal pyramidal and trigonal bipyramidal coordination is not possible. According to the interatomic angle distribution for these two kinds of polyhedra in Stephenson and Moore (1968), the Fe(2)-O polyhedron resembles a trigonal bipyramid with the angle O(4)-Fe(2)-O(8) 170.0° (deviating by 10° from the ideal 180° angle), but most

TABLE 4. Polyhedral Interatomic Distances for Fe<sub>3</sub>(H<sub>2</sub>O)[PO<sub>4</sub>]<sub>2</sub>†

F2(1)	Fe(2)				Fe(3)		P(1)		
Fe(1) -0(5) <sup>1</sup> / <sub>1</sub> " -0(3) ii " -0" " -0(1) " -0(2) i " -0(2) average  O(1) -0(3) ii OW -0(1).	2.070 2.092 2.101 2.156 2.383 2.161 2.731 2.731 2.749 2.887 2.951 3.103 3.133 3.153 3.470 3.626 3.041	Fe(2) " " avera	-0(7) ii -0(6) -0(4) i -0(4) i -0(8) age	1.987 2.036 2.052 2.086 2.243 2.081	Fe(3) " " " aver		2.083 2.135 2.140 2.145 2.206 2.211 2.153	P(1)-0(1) " -0(3) " -0(2) " -0(4) average 0(1)-0(4) 0(2)-0(4) 0(3)-0(4)	1.542 1.543 1.544 1.561 1.548 2.485 2.517 2.536
ON -0(1) 0(2) -0(5) i OW -0(5) i OW -0(2) i OW -0(2) i O(3) i -0(5) i OW -0(3) i OW -0(1) i OW -0(3) ii O(1) -0(5) i O(2) i -0(3) ii average		749° 0(6) 847 0(4) 887 0(7) 924 0(4) 951, 0(4) 103 0(4) 143 0(4) 143 0(4) 1470 average	-0(8)i -0(4)i -0(8)ii -0(7)i -0(6) -0(6) -0(8)ii -0(7)ii	2.413† 2.627√ 2.981 3.069 3.167 3.197 3.364 3.514 3.041	0(1)ii 0(2)ii 0(1)ii 0(6) 0(6)ii 0(1)ii 0(2)ii 0(2)i 0(5)i 0(3) aver	-0(8)11 -0(5)1 -0(2)1 -0(3)1 -0(5)1 -0(6)111 -0(8) -0(3) -0(6)	2.749° 2.935 2.947 2.975 3.031 3.084 3.100 3.100 3.108 3.318 3.391 3.037	0(1)-0(3) 0(2)-0(3) 0(1)-0(2) average P(2) P(2)-0(7) "-0(5) "-0(6) "-0(8) average	2.538 2.540 2.543 2.526 1.529 1.530 1.555 1.556
					OW	0(7) 0(7)	2.72 3.43	0(6) -0(8) 0(5) -0(7) 0(6) -0(7) 0(5) -0(8) 0(7) -0(8) 0(5) -0(6) average	2.413 2.496 2.532 2.535 2.555 2.571 2.517

† i = -x, -y, -z; ii = 1/2+x, 1/2-y, z; iii = 1/2-x, 1/2+y, -z applied to coordinate in Table 2. 
• Octahedral shared edges. 

† Fe(2)-Fe(2) shared edge. VFe(2)-Fe(2)' shared edge.

of the remaining distances more closely resemble the tetragonal pyramid. The Fe(2)-O 2.08 Å average can be compared with the M(2)-O 2.14 and M(3)-O 2.05 Å averages reported by Calvo (1968) for the distance of the longer averages through partial solutions.

TABLE 5. Polyhedral Interatomic Angles for Fe<sub>3</sub>(H<sub>2</sub>O)[PO<sub>4</sub>],

		E								
Fe(1)		Fe(2)		Fe(3)	P(1)					
0(1) 1 -0(3) ii 0(5) i -0Wi 0(2) i -0Wi 0(2) i -0Wi 0(2) i -0(5) i 0(2) i -0(5) i 0(3) ii -0(5) i 0(3) ii -0(5) i 0(3) ii -0(5) i 0(3) ii -0(5) i 0(2) i -0(3) ii	79.11° 79.14 79.84 79.89 80.89 85.12 87.32 87.62 90.32 105.15 111.02	0(6) i -0(8) 0(4) i 1-0(4) 0(7) i -0(8) 0(7) i -0(8) 0(4) i -0(6) 0(4) i -0(6) 0(4) i -0(6) 0(4) i -0(6) 0(4) i -0(6) 0(4) i -0(8) 0(4) i -0(8)	68.47° 78.85 89.40 98.94 101.57 101.75 101.92 119.30 136.86 170.00	0(1) ii -0(3) i	20.20	0 (1) -0 (4) 0 (2) -0 (4) 0 (3) -0 (4) 0 (1) -0 (3) 0 (2) -0 (3) 0 (1) -0 (2) P(2) 0 (6) -0 (8) 0 (5) -0 (7) 0 (5) -0 (8) 0 (7) -0 (8) 0 (7) -0 (8)	106.40° 108.29 109.58 110.73 110.73 110.98 101.76° 109.38 110.39 110.46 111.83			

Severe cation-cation repulsion across shared edges results in some very short distances; thus O(6)-O(8), the edge shared by Fe(2) and P(2), equals 2.41 Å, whereas O(4)-O(4)', which is shared by two Fe(2), equals 2.63 Å. These distances are the shortest for their polyhedra. Short distances between octahedral shared edges also occur, with the exception of OW-OW 3.10 Å. This latter distance reflects the long Fe(1)-OW 2.38 Å bond.

Hydrogen Bonds and Electrostatic Valence Balances

The OW molecule can donate two hydrogen bonds with the most likely acceptor being O(7), which coordinates to P(2) and Fe(2) only. Accepting  $\xi = \pm 1/6$  for the hydrogen bond strength as suggested by Baur (1970), a model can be found where O(7) accepts one strong and one weak hydrogen bond: OW  $\cdot$  O(7) 2.72Å and OW-O(7) 3.43 Å with the angle O(7)-OW-O(7) 100.1°. Two strong hydrogen bonds would result in a very nearly saturated O(7) anion, since  $\Sigma = 5/4 \pm 2/5 \pm 1/6 \pm 1/6 = 1.98$ . Since Fe(2)-O(7) and P(2)-O(7) are the shortest distances for their polyhedra, the presence of one feeble bond is more likely.

The remaining anions deviate only slightly from saturation by cations and accordingly show no systematic deviations in their Me-O bond distances. All oxide anions associated with Fe(1) are slightly un-

Fig. 3. The links between the Fe atoms where edges are shared. This diagram follows from Figure 2. Arrows, pointing in the plane and above or below, denote edge-links.

Severe cation-cation repulsion across shared edges sults in some very short distances; thus O(6)-O(8), edge shared by Fe(2) and P(2), equals 2.41 Å, site discussed below.

# A Proposed Oxidation Sequence for Fe2+,(H2O)[PO4]2

Moore (1971) has pointed out that only cer-in compositions in the homologous series tain Fe2+3(H2O), [PO4]2 are capable of continuous oxidation of the metals without destruction of the structure. The compositions are limited by the condition of maintenance of local electrostatic neutrality of cations about anions during oxidation. Permissible bonded units include H<sub>2</sub>O-bridged ferrous ions (as in edge-sharing Fe<sup>2+</sup> octahedra) where Fe<sup>2+</sup> (OH<sub>2</sub>)<sub>2</sub>Fe<sup>2+</sup> can be continuously oxidized to hydroxide-bridged ferric ions—Fe<sup>2+</sup>(OH)<sub>2</sub>Fe<sup>3+</sup>. On the other hand, hydroxide-bridged ferrous ions, Fe<sup>2+</sup>(OH)<sub>2</sub>Fe<sup>2+</sup>, lead to extreme oxygen undersaturation. Analogously, such species as Fe<sup>3+</sup>(OH) or Fe<sup>3+</sup>(OH<sub>2</sub>)Fe<sup>3+</sup> also lead to local charge balance difficulties and are, accordingly, not observed. Of the homologous series, only n =3 is capable of continuous oxidation to a stable ferric end-member since permissible end-member combinations are preserved. Thus, the series Fe<sup>2+</sup><sub>3</sub> (H<sub>2</sub>O)<sub>3</sub>[PO<sub>4</sub>]<sub>2</sub> (phosphoferrite) — Fe<sup>2+</sup><sub>3</sub>(OH)<sub>3</sub>[PO<sub>4</sub>]<sub>2</sub> (kryzhanovskite) is observed in Nature; the others decompose to yield essentially amorphous ferric equivalents after progressive and complete oxidation (Moore, 1971). A more recent study on pure synthetic phosphoferrite and its ferric equivalent has established beyond doubt the existence of both ferrous and ferric end-member compositions belonging to the same structure type when the hydrogen atom positions are excluded (Moore, 1974b).

Since Fe<sup>2+</sup>(OH<sub>2</sub>)<sub>3</sub>Fe<sup>2+</sup> ions occur in Fe<sup>2+</sup><sub>3</sub>(H<sub>2</sub>O) [PO<sub>4</sub>]<sub>3</sub>, we propose that a bounded mixed valence composition can occur as a stable crystal. The mid-point of the OW-OW' edge shared between two Fe(1) atoms is an inversion center. This suggests that an upper bound for a stable oxidized equivalent would be Fe<sup>2+</sup><sub>2</sub>Fe<sup>2+</sup>(OH)[PO<sub>4</sub>]<sub>3</sub>, where Fe(1) is completely oxidized.

If such an oxidative sequence does indeed exist, then mixed-valence transfer and intense pleochroism should occur with maximum absorption along the Fe(1)\*-Fe(3)\*- direction. Unique Fe-Fe separations across the shared edges are Fe(1)-Fe(1) 3.25 Å, Fe(1)-Fe(3) 3.11 and 3.20 Å, and Fe(2)-Fe(2) 3.20 Å. We provide a skeleton of Fe-Fe' connections and distances across shared edges in Figure 3. These distances are similar to those found between the

shared edge in vivianite, suggesting that mild oxidation of  $Fe^{z+}_3(H_2O)[PO_4]_2$  would result in intense pleochroism, similar to that observed for vivianite.

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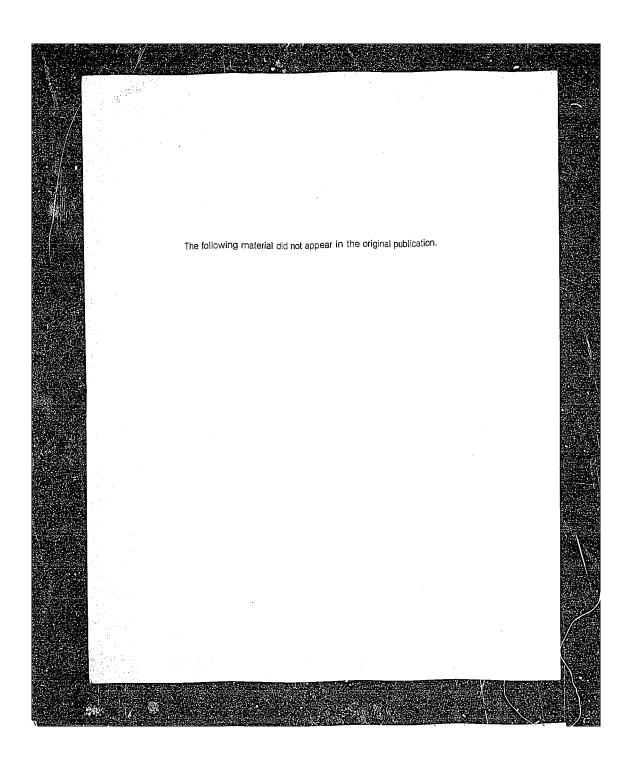
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PAUL BRIAN MOORE and TAKAHARU ARAKI: The  $\operatorname{Fe}_3^{2+}(\operatorname{H}_2\operatorname{O})_n[\operatorname{PO}_4]_2$  homologous series. II. The crystal structure of  $\operatorname{Fe}_3^{2+}(\operatorname{H}_2\operatorname{O})[\operatorname{PO}_4]_2$ . Published in *The American Mineralogist*, Volume 60 (May-June, 1975).

TABLE 3. Calculated and Observed Structure Factors for  $\mathrm{Fe_3(H_2O)LPO_4J_2}$ 

H K L FO FC 0 6 2 52.9 51.2 0 6 3 11.5 10.1 0 0 1 16.7 18.5 0 6 4 29.3 27.0 0 0 0 2 63.1 70.7 0 6 5 29.6 30.1 0 0 0 3 49.0 53.7 0 6 6 9.7 10.5 0 0 4 36.7 39.8 0 6 7 30.0 30.8 0 0 0 5 49.7 49.2 0 6 8 13.1 10.3 0 0 6 93.5 98.5 0 6 9 8.7 4.7 0 0 0 7 26.5 25.8 0 7 1 30.9 29.3 0 0 8 15.2 15.4 0 7 2 37.0 38.0 0 0 9 64.4 66.6 0 7 3 35.4 35.8 0 0 0 10 9.1 11.1 0 7 4 5.3 3.4 0 1 1 95.7 97.8 0 7 5 17.9 19.4 0 1 2 193.8 188.1 0 7 6 7.9 5.5 0 1 3 3.0 2.4 0 7 7 2 5.0 23.3 0 1 4 99.8 103.5 0 7 8 45.0 45.3 10.1 117.8 0 1 5 13.9 11.1 0 7 9 51.3 51.5 0 1 6 12.1 12.2 0 8 0 130.1 117.8 0 1 9 11.5 9.5 0 8 2 16.4 14.5 0 1 1 10.4 17 0 1 10.4 14.5 0 1 1 10.4 17.0 0 1 10.4 14.5 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 1 10.4 17.0 0 8 4 14.8 15.6 0 1 10.4 17.0 0 8 4 14.8 15.6 0 1 10.5 0 1 10.4 17.0 0 8 4 14.8 15.6 0 1 10.5 0 10.0 0 8 1 19.0 13.0 1 10.5 0 10.0 0 8 1 19.0 13.0 1 10.5 0 10.0 0 8 1 19.0 13.0 1 10.5 0 10.0 0 8 1 19.0 13.0 1 10.5 0 10.0 0 1 10.0 0 1 1.7 0 8 4 14.8 15.6 0 10.0 0 1 10.0 0 1 1.7 0 8 4 14.8 15.6 0 10.0 0 1 10.0 0 1 1.7 0 8 4 14.8 15.6 0 10.0 0 1 10.0 0 1 1.7 0 8 4 14.8 15.6 0 10.0 0 1 10.0 0 1 1.7 0 8 4 14.8 15.6 0 10.0 0 1 10.0 0 1 10.0 0 1 1.7 0 8 4 14.8 15.6 0 10.0 0 1 10.0 0 1 10.0 0 1 1.7 0 8 4 14.8 15.6 0 10.0 0 1	
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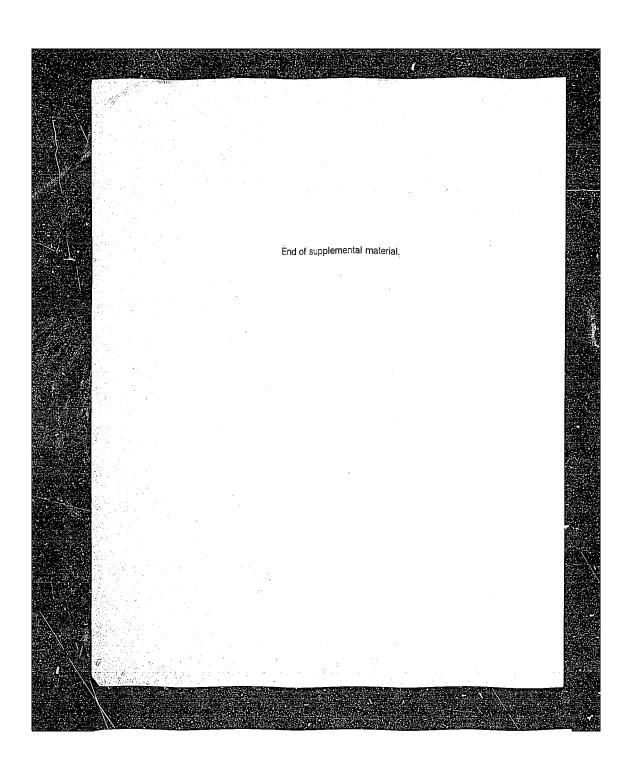
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# Palermoite, $SrLi_2[Al_4(OH)_4(PO_4)_4]$ : Its Atomic Arrangement and Relationship to Carminite, $Pb_2[Fe_4(OH)_4(AsO_4)_4]$

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#### Abstract

Palermoite,  $SrLi_1[Al_1(OH),(PO)_1]$ , space group Imcb, a 11.556(5), b 15.847(7), c 7.315(4) Å, Z = 4, is structurally related to carminite,  $Pb_1[Fe_1(OH),(AsO_1)_1]$ , R(hkl) = 0.090 for 1471 non-equivalent reflections. Both contain the same  $[M^{**}_{\bullet}(OH),(TO)_{\bullet}]^{\bullet}$  octahedral and tetrahedral slabs oriented parallel to [010]. They are distinguished by the link to symmetry-equivalent slabs across the glides at b = 1/4. The space group Amaa for carminite shares the same subgroup (Pmaa) with palermoite. In both structures, the octahedra form chains of edge-linked dimers which are corner-linked to symmetry-equivalent dimers resulting in the composition  $M^{2*}_{\bullet}(O)_1(OH)_{\bullet}$ . One-eighth of the tetrahedral orygens are not bonded to the octahedra

Polyhedral interatomic averages are VIIISr-O 2.62 Å, VIAI-O 1.91 Å, IVLi-O 2.13 Å, IVP(1)-O 1.53 Å and IVP(2)-O 1.54 Å. Local isomorphism of all atoms excepting Li and Pb(2) occurs: the Li atoms are split into twice the equipoint rank number as Pb(2) and possess lower point symmetry.

# Introduction

Palermoite occurs locally in moderate abundance as small striated prismatic colorless crystals at its type locality, the Palermo No, I pegmatite, near North Groton, New Hampshire. It was originally described by Mrose (1953), who proposed the formula (Li, Na),SrAl<sub>8</sub>(PO<sub>4</sub>)<sub>4</sub>(OH)<sub>6</sub>. Z = 2, and the unit cell parameters a 7.31 Å, b 15.79 Å, c 11.53 Å with the space grapu Immm. Another chemical analysis by Frondel and Ito (1965) ied to the proposed formula (Li, Na),(Sr, Ca) Al<sub>4</sub>(PO<sub>4</sub>)<sub>4</sub>(OH)<sub>6</sub>. Z = 4, with the refined cell parameters a 7.315(4), b 15.849(9), c 11.556(6) Å. Meanwhile, Strunz (1960) proposed an isotypic relationship between palermoite and carminite, PbFe<sup>3+</sup><sub>2</sub>(ASO<sub>4</sub>)<sub>3</sub>(OH)<sub>6</sub>. To reconcile the rather complex formula of Mrose and the similarity in the crystal cell parameters between palermoite and carminite, he proposed the formula SrAl<sub>2</sub>(PO<sub>4</sub>)<sub>2</sub>(OH).

Despite similarities in the cell dimensions, we were puzzled by the difference between the body-centered cell for palermoite and the end-centered cell for carminite. Atomic positions based on the crystal structure analysis of carminite by Finney (1963) could not be isomorphically transformed into the palermoite

cell since the space groups are neither isomorphic nor is one a subgroup of the other.

#### Experimental

Palermoite single crystals collected at the type locality by P.B.M. were submitted to single crystal X-ray study. In addition, a qualitative electron probe scan detected Sr, Al, P, and only minor Ca (< 1%). The extinction criteria, from films and single crystal diffractometer, suggested the space groups 12cb or Imcb, in disagreement with Immm proposed by Mrose (1953). Doubly terminated crystals and the three-dimensional crystal structure analysis support the centrosymmetric space group Imcb.

Refinement of the cell parameters on a PICKER.

Refinement of the cell parameters on a Picker automated diffractometer afforded a 11.556(5), b 15.847(7), c 7.315(4) Å. We selected the standard axial convention for the orthorhombic system to which the space group Imab conforms and accepted the cell contents  $4[SrLi_2Al_4(OH)_4(PO_4)_4]$ . Other salient details: graphite monochromatized  $MoKa_1$  radiation  $(\lambda = 0.7093 \text{ Å})$ ; maximum  $\sin \theta/\lambda = 0.80$ ; twenty second background counting times; scan rate  $1.0^9$ /minute; half angle scan  $1.8^9$ . The thick prismatic crystal, of maximum dimension 0.12 mm, was not corrected for absorption. The equivalent reflec-

TABLE, I. Palermoite: Atomic Coordinate and Isotropic Thermal Vibration Parameters\*

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Atom	Point group	Hilt.	×	у	z	B(Å <sup>2</sup> )	Atom	Point	ault.	×	у	z	c(l?)			
Sr Pb(1)	222 222	4	0.2500 .2500	0.5000 .5000	0.5000 .5000	0,80(2)	P(2) As(1)	n.	8	.0000	.4576(1) .457	.7731(2) .760	.50(2)			
L( Pb(2)	m 2/m	8	.5000 .5000	.2851(11) .2500	.2723(21) .2500	2.20(24)	n(3) 0(3)	e e	8	.0000	,3593(3) ,386	.6107(6) .606	.98(7)			
Al Te	1	16 16	.1307(1) .136	.3727(1) .378	.1374(2) .131	.50(Z)	0(4) 0(2)	m m	8	.0000	.4014(3) .405	.9502(6) .968	.76(6)			
P(1) As(2)	2	8	.2500 .250	.2922(1) .289	.5000 .500	.45(2)	0(5) 0(1)	1	16 16	.1083(3) .115	.4857(2) .482	.2275(5) .260	.69(4)			
0(1) 0(5)	1	16 16	.1438(3) .145	.2532(2) .272	.0387(4) 013	.75(5)	OH(1) OH(1)	E E	8	.0000	,3363(3) ,334	.2606(6) .241	.62(6)			
D(2) D(4)	1	15 16	.2257(3) .241	.3515(2) .348	,3389 (4) ,326	.57(4)	OH(2) OH(2)	2 2	8	.2500 .250	.4117(3) .430	.0000	per (c)			

Estimated standard errors refer to the last digit, the stonic parameters of carminite in Pinney (1961), appropriately recrienced, are shown for comparison.

tions (hkl) and (hkl) were averaged, yielding 1471 independent F (obs), which were obtained through standard computational procedures.

## Determination and Refinement of the Structure

Three-dimensional Patterson synthesis indicated strong vector densities at 0 v C, 1/2 v 0; 0 v 1/2; and 1/4 v 1/4. Symmetry restrictions led to rapid determination of the Sr, Al. F(1), and P(2) positions. The  $\beta$ - and  $\gamma$ 'syntheses of Ramachandran and Srinivasan (1970) led to unambiguous resolution of all non-hydrogen stums.

hydrogen atoms. Four cycles of full-matrix, least-squares refinement based on isotropic thermal vibration parameters, full site occupancies, secondary extinction correction with  $c_0=0.428\times 10^{-6}$  (Zachariasen, 1968) and anomalous dispersion correction for Sr, Al, and P led

$$R(hkl) = \frac{\sum ||F(\text{obs})| - |F(\text{calc})||}{\sum |F(\text{obs})|} = 0.090$$

for all 1471 reflections. The final atomic coordinates and the isotropic thermal vibration parameters are presented in Table 1. Table 2 lists the structure factor data.

# Description of the Structures

The formula SrLi<sub>3</sub>[Al<sub>4</sub>(OH)<sub>4</sub>(PO<sub>4</sub>)<sub>4</sub>] (here idealized) proposed by Frondel and Ito (1965) is confirmed. Although the structures of palermoite and carminite are closely related, the two crystals do not exhibit an isotypic relation. Figure 1 features the symmetry

diagrams of the space groups Imcb (palermoite) and Amaa (carminite) down the c axis in the conventional b>a>c orthorhombic setting. The essential difference between the two is a c glide at b=1/4 in

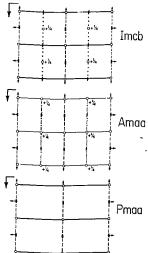


FIG. 1. Symmetry diagrams of the space groups Imcb. Amaa, and Pmaa in standard cell orientation (down the caxis). The first two groups apply to palervoite and carminite respectively. The third group is the subgroup common to the first two.

<sup>&</sup>lt;sup>1</sup> To obtain a copy of Table 2, order document number AM-75-003-B from the Business Office, Mineralogical Society of America, 1909 K Street, N.W., Washington, D. C. 20006, Please remit \$1.00 for the microfiche.

palermoite which is replaced by a n glide at b = 1/4 in freedom in the atomic coordinates. Pb(2) possesses carminite. Accordingly, the screw axis at (0 1/4 z) and the inversion at (1/4 1/4 1/4) in *Imcb* is translated by (1/4 0 0) to create *Amaa*. The two-fold rotations at (1/4 y 0); (1/4 0 z); (x 0 0); the a-glide at c = 0; the a-glide at b = 0; the mirror plane at a = 0; and the inversion at (000) remain invariant in the two and the inversion at (too) remain invariant in the two space groups. This corresponds to the mutual subgroup *Pmov* (D<sub>2n</sub>), shown at the bottom in Figure 1.

The palermoite and carminite structures (Fig. 2)

both contain the same [Ma+4(OH)4(TO4)4]4contain the same [m] AO(1)A(7)A(7) other tashedral (M) and tetrahedral (T) slab oriented parallel to (010). The structures are distinguished by the links to symmetry equivalent slabs across the c glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at b = 1/4 in the former and the n glide at a and a are a are a and a are a and a are a and a are a and a are a are a and a are a and a are a are a and a are a and a are a and a are a and a are a are a and a are a are a and a are a and a are a and a are a and a are a are a and a are a and a are a are a and a are a and a are a are a and a are a are a are a and a are a and a are a are a are a are a are a and a are a are a are a are a are a and a are a are a are a are a are a and a are a are a a 1/4 in the latter. In fact, taking the symmetry operations which are invariant in the two space groups, the pairs of linked slabs between 1/4 < b < 3/43/4 are isomorphous in the two structures. For this reason, the atomic coordinates for palermoite and carminite in Table I are compared within this bound. It is seen that all the parameters and their equipoint rank numbers and point symmetries are similar, with the exception of Li in palermoite and Pb(2) in carminite. The Li atom possesses an equipoint rank number of 8, point symmetry m, and two degrees of

rank number 4, point symmetry 2/m, and no degrees of freedom.

Palermoite and carminite contain the same type of M-O octahedral chain. In palermoite it consists of an edge-linked dimer which is corner-linked at the same level to symmetry equivalent dimers (see Fig. 2). The level to symmetry equivalent dimers (see Fig. 2). The chains, which run parallel to the a axis, have composition  $Al_2(Op)_7(OH)_2$ , where Op are oxygens that belong to PO<sub>4</sub> tetrahedra. One eighth of the tetrahedral oxygens, namely O(3) in both structures, do not bond to the trivalent octahedrally coordinated cations. The points of condensation of the octahedral chains include OH(1), OH(2), and O(4) in palermoite, each of equipoint rank number 8.

Remaining in the structures are pockets at (1/4 1/2 1/2); and (1/2 1/4 1/4), each of equipoint rank number 4. Both non-equivalent polyhedra are distorted cubes and accommodate Pb(1) and Pb(2) in carminite. Although Sr in palermoite is isomorphic to Pb(1) in carminite, the Li atoms in palermoite are split into two equivalences, the coordination polyhedron being a distorted tetrahedron. The polyhedral environments about Pb(2) and Li are shown as projections down the a axis in Figure 3. These regions are non-isomorphic as they do not

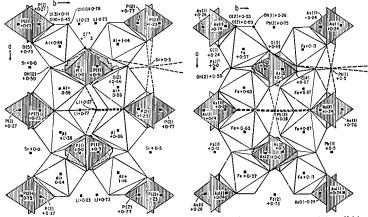
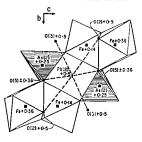


Fig. 2. Polyhedral diagrams of palermoite (left) and carminite (right) structures down the c axis. The outline  $a \times b/2$  is shown. Heights are given as fractional coordinates in z. The Pb-, Sr-, and Li-O bonds are dashed in

possess the same point symmetry in the two structures.

One of the curious features of the two structures is the observation that the "X" position at (1/2 1/4 1/4) in palermoite possesses a coordination polyhedron similar to that in carminite, both being distorted cubes. Figure 4 provides the bond distances for "X" in palermoite and Pb(2) in carminite. The position "X", however, still has equipoint rank number 3 since its point symmetry remains m and to afford a carminite-like composition would require disordered half-occupied sites over the Pb(2) positions. Since the environments about the X and Pb(2) sites are non-isomorphic, the energetic relationships between the two structures are probably dictated by the charges and ionic radii of the cations competing for the X and Pb(2) environments, as the distinctions in the bonding over the rest of the structures are small.



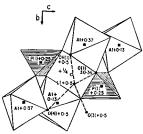
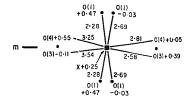


Fig. 3. The polyhedral environments of Pb(2) in carminite (top) and Li in palermoite (bottom) down the a axis. The Pb(2) atom resides at (1/2 1/4 1/4). In palermoite, the locus at (1/4 1/4 1/4) is drawn as a circle.



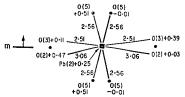


FIG. 4. The "X"-O bonds and Pb(2)-O bonds in palermoite (top) and carminite (bottom). The symmetry elements (m and 2m respectively) are shown on the left and apply to the loci of "X" and Pb(2). Bond distances are specified.

We propose that palermoite and carminite structure ideals with compositions  $X(1)X(2)M_1(OH)_2$ .  $(TO_4)_2$  are combinatorial polymorphs in the sense defined by Moore (1975).

## **Bond Distances**

Table 3 lists the anions and the coordinating cations in rows and columns. In this manner, the valence bond strengths can be conveniently tabulated and deviations from bond distance averages can be related to deviations in bond strength sums.

One problem immediately arises regarding the coordination of lithium. Although its nearest neighborhood defines a distorted trigonal bipyramid, the valence balances suggest that the true coordination is distorted tetrahedral, the polyhedral vertices including an OMAL and CASA and the coordination is distorted tetrahedral, the polyhedral vertices including an OMAL and CASA and the CASA and the

including one OH(1), one O(3), and two O(1) atoms. Interatomic distances are given in Table 4. The Li-O tetrahedral distances range from 1.94 to 2.29 Å, the fifth distance Li-O(4) = 2.46 Å suggesting that the tetrahedral coordination is more likely. We note in addition that a distorted trigonal bipyramid would require that the O(3)-O(4) edge be shared between Li and P(2). The additional distances which obtain from the distorted trigonal bipyramidal model are listed

TABLE 3. Palermoite: Electrostatic Bond Strengths and Their Sums about the Anions\*

									Me	-0	
Anion	Sr <sup>2+</sup>	Li <sup>1+</sup>	Al <sup>3+</sup>	P <sup>5+</sup>	H(d)	Н(а)	Σ	Sr	Li	Al	P
0(1) 0(2)	2/8	1/4	3/6 3/6	5/4 5/4			2.00 2.00	+	+	-	0
0 (3) 0 (4) 0 (5)	2/8	1/4	3/6+3/6 3/6	5/4 5/4 5/4		1/6 ½(1/6)	1.67 2.25 2.08	D	-	+	+ 0
OH(1)		1/4	3/6+3/6 3/6+3/6		5/6 5/6		2.08 1.83		-	_=	

<sup>\*</sup>He-O refers to distances which are greater than (+), less than (-), or within (0)  $2\sigma$  of the interatomic error referred to the polyhedral average. H(d) = hydrogen donor, H(a) = hydrogen bond acceptor.

parenthetically in Table 4. The Sr-O polyhedron is a distorted cube with distances between 2.59 and 2.65 Å. The remaining polyhedral distance averages are typical, with the 1.91 Å average for Al-O and 1.53 and 1.54 Å averages for P(1)-O and P(2)-O respectively.

Table 4 also reveals the effects of polyhedral edgesharing. The SrO<sub>4</sub> cube, for example, shares two

TABLE 4. Palermoite: Interatomic Distances

	Al	P(1)		Sr	
Al -0!( -0!( - 0!( - 0( - 0( - 0( average	2) 1.815(5)Å 1) 1.850(5) 2)i 1.868(4) 1) 1.884(4) 5) 1.928(4)	2 P(1) -0(2) 2 -0(1) average 1 0(2) -0(2)iii 2 0(2) -0(1)iii 1 0(1) -0(1)iii	1.534(4) 1.535(4) 1.534 2.423(4) <sup>+</sup> 2.517(4) 2.519(4)	# Sr - 0(5) i 4 - 0(2) average 2 0(2) - 0(5) i 4 0(2) - 0(5) i 4 0(5) i - 0(2) iv 2 0(5) i - 0(5) vii	
OH(1) - O( OH(2) - O( OH(2) - O(	1) 2.596 (6) 1 5) 2.613 (6)	2 0(1) -0(2) average P(2)	2,526(4) 2,505	average Li	3.062
OH(2) - 0( O(2)i-OH( O(2)i-OH( OH(1) - 0( O(4) - 0( O(2)i- 0(	(1) 2.669 (6) (2) 2.671 (6) (1) 2.681 (6) (5) 2.688 (6) (5) 2.732 (6) (1) 2.771 (4)	1 P(2) -0(3) 2 -0(5) iv 1 -0(4) average	1.506(5) 1.540(4) 1.572(5) 1.539	l Li -OH(1) ii l - O(3) 2 - O(1) ii (1 - O(")	1.94(2) 2.00(2) 2.29(2) 2.46(2)) 2.13
0(1) - 0( 0H(2) - 0( average		1 0(3) -0(4) v 1 0(5) iv-0(5) iv 2 0(3) -0(5) iv 2 0(4) -0(5) iv average	2.484(7)** 2.502(4) 2.507(6) 2.540(6) 2.513	(1 0(3) - 0(4) i.i. 2 0H(1) ii. 0(1) ii. 2 0(1) ii. 0(3) ix 1 0(1) ii. 0(3) ix 1 0H(1) ii. 0(3) (1 0H(1) ii. 0(4) (2 0(1) i. 0(4) (4) (4) i. 0(4)	2.484(7)* 2.596(6)* 3.110(6) 3.323(4) 3.891(7)
OH(1)0(	(3) 2.749 (7)	44.7484		(1 0H(1) ii - 0(4) (2 0(1) ii - 0(4)	4.014(7)) 4.316(6))

<sup>\*</sup>Edge sharing between Al-Al octahedra; \*between Sr-P polyhedra; \*between Sr-Al polyhedra; \*hetween Li-Al polyhedra; \*Thetween Li-Al polyhedra.  $i=1/2-x,\ 1/2-y,\ 1/2-z;\ ii=x,\ 1/2-y,\ 1/2+z;\ iii=1/2-x,\ y,\ -z;\ iv=x,\ -y,\ -z;\ v=-x,\ -y,\ -z;\ v=1/2-x,\ -y,\ z;\ vii=1/2-x,\ 1/2-y,\ 1/2+z;\ viii=1/2-x,\ 1/2-y,\ 1/2-z;\ ix=-x,\ 1/2-y,\ 1/2+z,\ viii=1/2-x,\ 1/2-y,\ 1/2-z;\ ix=-x,\ 1/2-y,\ 1/2+z,\ viii=1/2-x,\ 1/2-y,\ 1/2-z;\ ix=-x,\ 1/2-y,\ 1/2-z,\ 1/2-x,\ 1/2-y,\ 1/2-z,\ 1/2-x,\ 1/2-x,\$ 

edges with the P(1)O4 tetrahedron and four edges with the AlO<sub>6</sub> octahedra. These smaller more tightly bound polyhedra geometrically restrict the O-O' shared edges such that the shortest SrO<sub>6</sub> edge distances are associated with P(1)O<sub>4</sub> and AlO<sub>6</sub> respectively. The OH(1)-O(4) shared edge between AlO<sub>6</sub> octahedra is 2.49 Å and can be compared with the O(9)-O(9) shared edge distances (= 2.40 Å) in the structurally related bjarebyite (Moore and Araki,

Finney (1963) noted a short OH(2)-OH(2) = 2.44± 0.13 distance although that distance does not correspond to any cation-oxygen polyhedral edge. In palermoite, the OH(2)-OH(2)<sup>v1</sup> = 2.80 Å distance is considerably longer.

The proposed hydrogen bonds involve OH(1)...

O(3) = 2.75 Å and OH(2)-O(5) = 2.84 Å distances. Since O(5) resides in a general position, the hydrogen bond to it is on the average half-occupied. Deviations from average bond lengths can be roughly correlated with degree of undersaturation or oversaturation of cations about anions (Table 3). Thus, O(3), with  $\Sigma =$ 2.25, are all longer than average. OH(1), with  $\Sigma$  = 2.08, has shorter than average bonds, however. At present, we cannot offer a satisfactory explanation for this discrepancy.

## Acknowledgments

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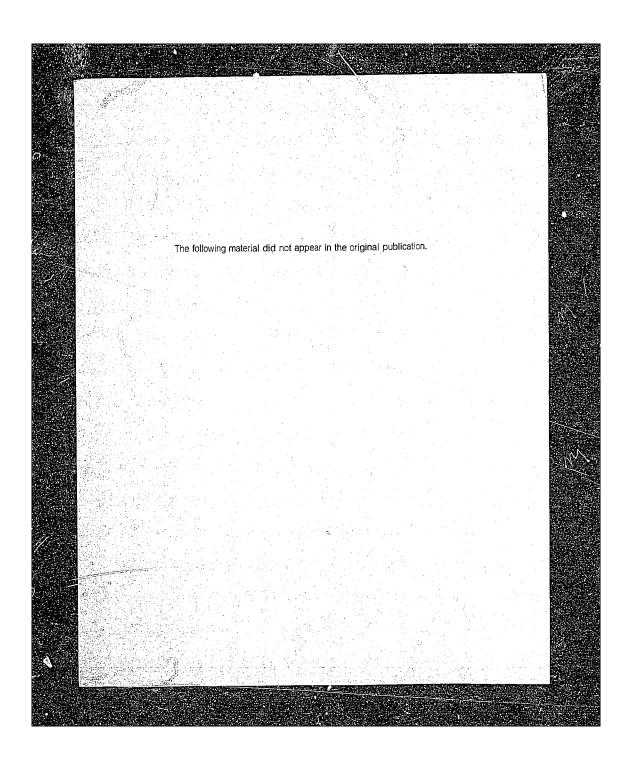
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AM-75-003-B

PAUL BRIAN MOORE and TAKAHARU ARAKI: Palermoite,  $SrLi_2[A1_4(OH)_4(PO_4)_4]$ : Its atomic arrangement and relationship to carminite,  $Pb_2[Fe_4(OH)_4(AsO_4)_4].$ 

Published in The American Mineralogist, Volume 60 (May-June, 1975).

TABLE 2. Palermoite. Observed and Calculated Structure Factors

			7										V 7/	
						90 GS/A	ekijer		234047		an in the state of			27)
	Н	K	L FC	FC	-					Н	ĸ		FO	FC
		24	0 101.6		10 12	8	0	78.8 77.7	84•4 66•4	18	0 · 25	0	21.1 27.9	20.4 14.3
	4	24 22	0 59.7	53.3	14 16	8	0	11.4 25.1	11.8 27.6	2	25 24	1	34.0 12.2	32•1 3•2
	2	22	0. 11.6	11.6	0	6	0	106.9	118.0	<u>*</u> 3 5	24	1	12.3	20.6
	6	22 22	0 31.7		2	6	0	69.5	132.2 72.6	. 0	23	- <u>1</u>	38.0 11.9	33.5 12.8
		22 20	0 48.7		6 8	6	0	169.1 61.5	166.6 62.6	2	23	<u>1</u>	21.9 37.7	32•2 29•2
	2	20 20	0 45.6	45.8	10 12	6	0	17.9 22.0	16.1 20.0	6 1	23 22	<u>1</u>	34.2 11.5	26.2 3.1
	6	20	0 49.5	45.8	14	6	0	61.7	68.6	3	22	1	11.7	7.6
		20 20	0 43.3 0 12.3	49.2 11.4	16 0	6 4	0	12.0 26.3	19.6 40.6	5 7	22 22	1	20.1 32.9	13.0 33.4
1.	0	1 8 1 8	0 28.3	31.2	2	4	0	96.1 18.6	102.6	9	22 21	1	12.6	16.1 12.6
4 .	4	18	0 10.3	70.3	8	4	0	124.7	17.5 126.2	2	21	1	30.4	25.9
		L 8 L 8	0 18.5	13.7 33.1	10	4	0	16.7 56.3	19.3 60.5	- 4	21	$-\frac{1}{1}$	31.0	32.6
		L 8 L 8	0 11.8	9.7 51.8	12 14	4	0	121.9 95.4	125.2 102.0	<u>8</u>	21	<u>1</u>	12.1	10.8 8.4
	0 1	6	0 42.5 0 23.9	31.5	16	4	0	11.9	13.6	11_	20	1	31.0	42.8
		l6 l6	0 73.0 0 131.0	69.9 130.6	18	2	0	12.7 87.4	32.0 107.7	3 5	20 20	1	40.7 20.0	44.2 22.1
	6 ]	6	0 45.8	46.8	4	2	0	103.0 78.3	109.6	7	20	1	24.9 41.1	24.0 42.1
	10 1		0 28.1 0 77.1	30.0 77.6	8	2	ō	78.2	68.2 83.8	11	20	1	43.7	47.9
			0 69.1 0 29.8	59•2 30•9	10 12	2	0	89.4 52.1	81.2 61.1	0_ 2	19	- <u>1</u>	153.0 104.5	153.0 104.2
	0 1	4	0 95.5	92.8	14 16	2	0	39.0 39.1	37.2 48.8	4	19 19	- <u>1</u>	41.7 66.1	37.6 66.3
	4 1	4	0 54.8	208.2 51.0	18	2	0	47.0	37.8	8	19	1	110.7	111.3
			0 174.5 0 59.8	186.6 57.0	2 4	0	0	120.0 275.1	125-0 259.3	10 12	19 19	1	97.3 36.2	91.5 34.5
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