

those in (II) (1.728 and 1.914 Å respectively). Within the cyclopropylidene ring the three C—C bonds range from 1.507 (27)–1.557 (24) Å (av. 1.529 Å) and probably reflect the three different steric environments of each ring C. The average C—C distance of 1.529 Å is somewhat larger than the three equal bond values in cyclopropane (1.509 Å) (Bastiansen, Fritsch & Hedberg, 1964). The C(3)—C(4)(methyl) distance of 1.486 (28) Å is similar to the ring C—methyl value of 1.51 Å in dimethyl 1,1'-dimethylbicyclopropyl-2,2'-dicarboxylate (Jongsma & Van der Meer, 1971).

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## Microporous Aluminophosphate Number 17 with Encapsulated Piperidine, Topological Similarity to Erionite

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**Abstract.** Aluminiumphosphate–piperidinehydrate,  $\text{Al}_{18}\text{P}_{18}\text{O}_{72}\cdot 4(\text{C}_5\text{H}_{11}\text{N}\cdot\text{H}_2\text{O})$ ,  $M_r = 2608$ , hexagonal,  $P6_3/m$ ,  $a = 13.2371$  (9),  $c = 14.7708$  (10) Å,  $V = 2241.4$  Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.93$  g cm<sup>-3</sup>,  $\lambda(\text{Cu K}\alpha) = 1.5418$  Å,  $\mu = 61$  cm<sup>-1</sup>,  $F(000) = 1312$ ,  $T \sim 295$  K,  $R = 0.086$  for 1146 diffractions. An aluminophosphate gel, produced from hydrated alumina and phosphoric acid, was digested with piperidine ( $\text{C}_5\text{H}_{10}\text{NH}$ ) for 1 week at 473 K to form  $\text{AlPO}_4\text{-17}$ . Al and P atoms alternate near the tetrahedral nodes of the erionite framework. An extraframework oxygen species in a partially occupied site bridges between a pair of Al atoms in the opposing 6-rings of each cancrinite cage. Each of the two ellipsoidal cavities contains two piperidine species. The nature of the charge on the extraframework oxygen species and the piperidine species is discussed.

**Introduction.** A new class of microporous materials was synthesized from aluminophosphate gels using a wide variety of organic amines and quaternary ammonium cations as templates (Wilson, Lok, Messina,

Cannan & Flanigen, 1982, 1983). Fourteen out of the twenty new materials are microporous after calcination to remove encapsulated organic material, and the tetrahedral frameworks with alternating Al and P include structural analogs of the aluminosilicate zeolites erionite ( $\text{AlPO}_4\text{-17}$ ) and sodalite ( $\text{AlPO}_4\text{-20}$ ). The novel framework of  $\text{AlPO}_4\text{-5}$  encloses non-connecting parallel channels bounded by 12-rings, and the as-synthesized material (Wilson, Lok & Flanigen, 1982) contains tetrapropylammonium hydroxide species in a tripod configuration (Bennett, Cohen, Flanigen, Pluth & Smith, 1983).

Four aluminophosphates prepared in the presence of an amine contain Al in 4-, 5- or 6-coordination with O. In two of them, the amine apparently fragmented during crystallization of the aluminophosphate framework. The potential template diaminebutane yielded  $\text{AlPO}_4\text{-15}$ ,  $\text{NH}_4\text{Al}_2(\text{OH})(\text{H}_2\text{O})(\text{PO}_4)_2\cdot 2\text{H}_2\text{O}$ , which has a tetramer of edge-sharing  $\text{AlO}_4(\text{OH})_2$  and vertex-sharing  $\text{AlO}_4(\text{OH})(\text{H}_2\text{O})$  octahedra sharing vertices with  $\text{PO}_4$  tetrahedra to form a framework 3-connected

at (OH) and 2-connected at O (Pluth, Smith, Bennett & Cohen, 1984; Parise, 1984b). Another aluminophosphate, AlPO<sub>4</sub>-21 (Bennett, Cohen, Artioli, Pluth & Smith, 1985; Parise & Day, 1985), contains three types of tetrahedral P, one type of tetrahedral Al, and two types of trigonal-bipyramidal Al. The other aluminophosphates contain intact templates: AlPO<sub>4</sub>-12 (Parise, 1984a) and AlPO<sub>4</sub>-EN3 (Parise, 1984c). In all seven of the listed AlPO<sub>4</sub> structures, each Al atom shares the four surrounding O atoms with an adjacent PO<sub>4</sub> tetrahedron of the 4-connected AlPO<sub>4</sub> framework. In addition, some of the Al atoms are bonded to either one or two oxygen species at a longer distance. These extra oxygen species are not part of the tetrahedral framework. The 5- or 6-coordinated Al atoms are called trigonal-bipyramidal or octahedral, but the surrounding oxygen species are not equivalent.

We now describe the crystal structure of the as-synthesized precursor to AlPO<sub>4</sub>-17, a structural analog of erionite except for unusual bridging of an oxygen species between Al atoms of opposing 6-rings in the cancrinite cages.

**Experimental.** An aluminophosphate gel, produced from hydrated alumina and phosphoric acid, was mixed with piperidine (C<sub>5</sub>H<sub>10</sub>NH) and held at 473 K for 1 week. The product consists of hexagonal needles of AlPO<sub>4</sub>-17 mixed with AlPO<sub>4</sub>-cristobalite and AlPO<sub>4</sub>-15. Bulk chemical analysis of pure AlPO<sub>4</sub>-17 preparations with various templates (*R*) of size similar to that of piperidine yield a typical unit-cell composition of Al<sub>18</sub>P<sub>18</sub>O<sub>72</sub>·4*R*·*x*H<sub>2</sub>O, where *x* is commonly 8 or more.

An hexagonal prism (0.018 × 0.16 mm) of AlPO<sub>4</sub>-17 was coated with oil to control humidity, and mounted on an automatic Picker four-circle diffractometer with Krisel automation. Refinement of 20 diffractions (49 < 2θ < 68°), each the average of the automatic centering of the 8 possible settings, gave the cell parameters. A total of 8077 measured intensities were averaged to produce 1304 merged intensities [*R*(*F*) = 0.038], with 1146 unique (158 < 4σ<sub>*F*</sub>), maximum sinθ/λ = 0.58 Å<sup>-1</sup>, data-collection range *h* ± 14, *k* ± 14, *l* + 17, and maximum intensity variation of 3 standard reflections 0.6%. An absorption correction gave transmission factors 0.82–0.91. Refinement began with parameters from erionite (Gard & Tait, 1973). The space group *P*6<sub>3</sub>/*mmc* for disordered Al and Si in erionite was modified to *P*6<sub>3</sub>/*m* for ordered Al and P in AlPO<sub>4</sub>-17 (Cox & Bennett, 1983). Three sites SiAl(1), SiAl(2) and O(1) in erionite split into two sites P(1) and Al(1), P(2) and Al(2), and O(1) and O(1'), respectively, while the positions O(2)–O(6) remain equivalent. Reasonable distances and angles were obtained in the refinement, and a difference-Fourier synthesis yielded a diffuse region of electron density in the cages, and a well defined peak inside the hexagonal prism. Each diffuse region was consistent with the

presence of a piperidine ring C<sub>5</sub>N in the expected stoichiometry, but the distinction between C and N would violate the hexagonal symmetry. Individual positions could not be located and the diffuse maxima of the electron density were approximated by two fixed positions C(1) and C(2) with a common displacement parameter. The well defined peak in a 6(*h*) position lies at ~2.0 Å to a pair of adjacent Al atoms, which is consistent with occupancy by an oxygen species; refinement yielded an occupancy of 4 oxygens in the sixfold site. The inability to model properly the disordered piperidine molecule results in a poor match of low-angle diffractions and is reflected in the high final *R* value. The final least-squares refinement\* used *F*'s with *w* = σ<sub>*F*</sub><sup>-2</sup>: *R* = 0.086, *wR* = 0.081, *S* = 4.7; maximum Δ/σ = 0.005; maximum and minimum height on final difference-Fourier synthesis 1.0 and -0.8 e Å<sup>-3</sup>; no correction for secondary extinction; neutral scattering factors (*International Tables for X-ray Crystallography*, 1974); computer programs *SHELX76* (Sheldrick, 1976), *ORFFE* (Busing, Martin & Levy, 1964), *ORTEP* (Johnson, 1965) plus local data reduction. Final atomic coordinates and displacement factors are given in Table 1 and interatomic distances and angles in Table 2.

**Discussion.** The topology of the erionite framework is described in Gard & Tait (1971), Meier & Olson (1971) and Smith (1976). After calcination, the neutral framework of AlPO<sub>4</sub>-17 undoubtedly matches that of erionite except for strict alternation of Al and P over the tetrahedral nodes (Cox & Bennett, 1983). Before calcination, the as-synthesized AlPO<sub>4</sub>-17 contains a modified erionite framework together with encapsulated piperidine species. P and Al alternate around the framework. All the P and Al atoms are tetrahedrally bonded to the framework O atoms, each of which is bonded to one P and one Al. Four O atoms of type O(7) were determined to be close to the twelve Al(1) sites, and each of these extraframework O atoms is bonded to two Al(1). The remaining four Al(1) atoms are not bonded to O atoms of type O(7). For convenience, Fig. 1 shows full occupancy of the six positions of site O(7), but only four are occupied. No evidence was found for symmetry lower than hexagonal, and it is presumed that there is no long-range order of the four occupants of O(7). Because of the disorder, the centroid of the electron density for Al(1) is the average for different types of coordinated Al, and it is impossible to determine the exact geometry of the coordination polyhedra. However, the secondary

\* Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42564 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

coordination with O(7) has distorted the tetrahedron around Al(1) towards a distorted trigonal bipyramid.

What type of oxygen and piperidine species occur in the as-synthesized  $\text{AlPO}_4\text{-17}$ ? A conclusive answer cannot be given from the X-ray diffraction evidence, but one plausible interpretation of the data is that the species are  $\text{OH}^-$  and  $\text{C}_5\text{H}_{10}\text{NH}_2^+$  which are expected to coexist in equilibrium with  $\text{C}_5\text{H}_{10}\text{NH}$  and  $\text{H}_2\text{O}$  at the pH of the synthesis. This is consistent with the refined

Table 1. Atomic populations, positions and displacements of  $\text{AlPO}_4\text{-17}$

Fractional coordinates are multiplied by  $10^4$ .  $U_{\text{eq}}(\text{\AA}^2)$  is defined as:

$$\frac{10^3}{3} \sum_{i=1}^3 \sum_{j=1}^3 U_{ij} a_i^* a_j^* (\mathbf{a}_i \cdot \mathbf{a}_j).$$

	x	y	z	$U_{\text{eq}}$
P(1)*	9989 (2)	2369 (2)	938 (2)	25 (1)
P(2)	5675 (3)	9079 (3)	2500	18 (1)
Al(1)	7708 (2)	9983 (2)	1180 (2)	35 (1)
Al(2)	999 (3)	4214 (3)	2500	24 (2)
O(1)	365 (6)	3435 (5)	1529 (4)	38 (3)
O(1')	6373 (5)	9669 (5)	1649 (4)	27 (3)
O(2)	826 (5)	1910 (5)	1053 (5)	40 (3)
O(3)	1432 (5)	2640 (6)	6244 (4)	40 (4)
O(4)	2773 (6)	30 (6)	9970 (4)	42 (4)
O(5)	2468 (7)	4693 (7)	2500	33 (5)
O(6)	4584 (7)	9171 (4)	2500	26 (4)
O(7)†	1721 (12)	1876 (12)	2500	44 (8)
C(1)‡	3770	7930	9599	419 (17)
C(2)‡	4590	7490	9920	419 (17)

\* In the erionite structure P(1), Al(1); P(2), Al(2); O(1), O(1') are equivalent.

† Only 4.14 (14) of six sites are occupied.

‡ C(1) and C(2) are disordered (fixed in refinement).

Table 2. Interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

	Centroid	Riding	Centroid	Riding
P(1)—O(1)	1.516 (7)	1.531 (8)	P(2)—O(1')	1.524 (6)
P(1)—O(2)	1.515 (9)	1.531 (9)	P(2)—O(5)	1.541 (12)
P(1)—O(3)	1.512 (9)	1.528 (9)	P(2)—O(6)	1.509 (11)
P(1)—O(4)	1.518 (7)	1.538 (8)	Mean	1.525
Mean	1.515	1.532		1.536
Al(1)—O(1')	1.744 (7)	1.744 (7)*	Al(2)—O(1)	1.721 (6)
Al(1)—O(2)	1.732 (8)	1.735 (8)	Al(2)—O(5)	1.718 (10)
Al(1)—O(3)	1.731 (7)	1.733 (8)	Al(2)—O(6)	1.711 (11)
Al(1)—O(4)	1.811 (8)	1.828 (8)	Mean	1.718
Al(1)—O(7)	2.059 (5)	2.078 (7)		1.727
Mean	1.815	1.824		
O(1)—P(1)—O(2)	110.1 (4)		O(1')—P(2)—O(1')	111.2 (4)
O(1)—P(1)—O(3)	108.2 (4)		2 O(1')—P(2)—O(5)	110.2 (4)
O(1)—P(1)—O(4)	106.8 (5)		2 O(1')—P(2)—O(6)	108.5 (4)
O(2)—P(1)—O(3)	108.3 (4)		O(5)—P(2)—O(6)	108.1 (4)
O(2)—P(1)—O(4)	111.8 (4)			
O(3)—P(1)—O(4)	111.5 (4)		O(1)—Al(2)—O(1)	112.9 (4)
			2 O(1)—Al(2)—O(5)	110.1 (4)
			2 O(1)—Al(2)—O(6)	107.8 (4)
O(1')—Al(1)—O(2)	121.5 (4)		O(5)—Al(2)—O(6)	107.9 (5)
O(1')—Al(1)—O(3)	115.0 (4)			
O(1')—Al(1)—O(4)	93.5 (3)		P(1)—O(1)—Al(2)	150.7 (6)
O(1')—Al(1)—O(7)	83.5 (4)		P(2)—O(1')—Al(1)	137.0 (5)
O(2)—Al(1)—O(3)	119.5 (4)		P(1)—O(2)—Al(1)	145.1 (4)
O(2)—Al(1)—O(4)	97.8 (4)		P(1)—O(3)—Al(1)	147.2 (5)
O(2)—Al(1)—O(7)	80.2 (5)		P(1)—O(4)—Al(1)	140.1 (6)
O(3)—Al(1)—O(4)	98.9 (3)		P(2)—O(5)—Al(2)	145.5 (6)
O(3)—Al(1)—O(7)	86.4 (6)		P(2)—O(6)—Al(2)	169.4 (6)
O(4)—Al(1)—O(7)	174.7 (6)		Al(1)—O(7)—Al(1)	142.6 (7)

\* Not defined.

populations for O(7), C(1) and C(2) in Table 1 [O(7),  $4.14 \pm 0.14$ ; one-third of C(1) and C(2), 4]. Assignment of  $\text{H}_2\text{O}$  or  $\text{O}^{2-}$  to site O(7) is less plausible because of problems in simultaneous satisfaction of constraints from the population factors and charge balance.

All distances and angles in Table 2 are generally reasonable when account is taken of riding motion and disorder. Lists of Al—O and P—O distances in aluminophosphate materials are given in Bennett, Cohen, Artioli, Pluth & Smith (1985). The occupancy of O(7) affects all the adjacent atoms; indeed, the displacement ellipsoid for Al(1) is elongated towards O(7) in response to the averaging of more than one centroid of thermal motion, while that of O(7) is elongated in the perpendicular direction. In  $\text{AlPO}_4\text{-17}$ , the distances from Al(1) and Al(2) to the O atoms shared with an adjacent P atom range from 1.71 to 1.74  $\text{\AA}$ , compared with 1.72 to 1.75  $\text{\AA}$  for Al(1)—O<sub>4</sub>, and 1.78–1.89  $\text{\AA}$  for Al(2)—O<sub>4</sub>(OH) and Al(3)—O<sub>4</sub>(OH), in  $\text{AlPO}_4\text{-21}$ . The distance from Al(1) to the partly occupied site O(7) of  $\text{AlPO}_4\text{-17}$  is 2.06  $\text{\AA}$ , but the real distance might be smaller because the observed position of Al(1) is the average for one-third occupancy of a tetrahedron and two-thirds occupancy of a non-regular trigonal bipyramid. The Al(2)—OH and Al(3)—OH distances (1.94 and 1.83  $\text{\AA}$ ) in trigonal bipyramids of  $\text{AlPO}_4\text{-21}$  are considerably smaller than the apparent distance of 2.06  $\text{\AA}$  in  $\text{AlPO}_4\text{-17}$ , and a considerable displacement (0.1–0.2  $\text{\AA}$ ) of Al(1) would be needed from the averaged position in Table 1 to obtain a comparable Al(1)—OH distance. In the octahedra of  $\text{AlPO}_4\text{-15}$ , the OH group is bonded to two Al(1) and one Al(2) at distances of 2.03, 2.05 and 2.20  $\text{\AA}$ , whereas the O of a water molecule lies at 1.94  $\text{\AA}$  to Al(2). Because bond lengths are not determined just by the first neighbors, and because there is no theory which can predict bond lengths to 0.01  $\text{\AA}$  in complex framework structures, further discussion of the Al—O distances in  $\text{AlPO}_4\text{-17}$  is unwarranted.

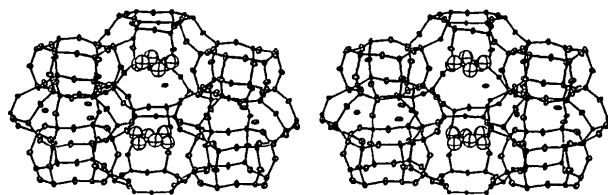


Fig. 1. Stereoplot of the  $\text{AlPO}_4\text{-17}$  structure at the 30% probability level. The isotropic displacement parameters of C(1) and C(2) were reduced from 0.42 to 0.25 to reduce overlap: it should be emphasized that the accuracy of these positions is poor, and that there is disorder between the five C and one N of each piperidine ring. H atoms were not located. Only two out of the three positions for O(7) in each cancrinite cage are occupied.

Magic-angle-spinning nuclear-magnetic-resonance spectra for <sup>27</sup>Al in as-synthesized AlPO<sub>4</sub>-17 contain two peaks (Blackwell & Patton, 1984). The peak showing a chemical shift of 31 p.p.m. with respect to aqueous Al(NO<sub>3</sub>)<sub>3</sub> is attributed to tetrahedrally coordinated Al [six Al(2) atoms and four Al(1) atoms], and the peak at 14 p.p.m. to trigonal-bipyramidal Al [eight Al(1) atoms]. Although the height of the first peak is less than that of the second peak, its area could be greater if the peak is asymmetric like those for AlPO<sub>4</sub>-5 and 11 (Blackwell & Patton's Fig. 2). Furthermore, the peak area may not be related linearly to the number of atoms if the bonding characteristics differ. The greater enhancement by cross-polarization from <sup>1</sup>H of the peak at 14 p.p.m. than the one at 31 p.p.m. is consistent with the proposal that O(7) represents a hydroxyl species. The additional spectra of calcined and rehydrated AlPO<sub>4</sub>-17 can be interpreted in terms of removal of the hydroxyl species and the piperidine upon calcination, and adsorption of H<sub>2</sub>O near the Al(1) atom upon hydration. Further X-ray structure determinations of calcined and hydrated specimens are needed to provide a detailed interpretation.

In conclusion, the subtle modification of the erionite structure type to accommodate a non-framework hydroxyl or H<sub>2</sub>O into the coordination group of framework Al atoms is a further illustration of the fascinating properties of the new family of synthetic aluminophosphates. In addition to the changes of Al coordination, there are important phenomena related to the organic compounds used as possible templates. Whereas in AlPO<sub>4</sub>-21 (Bennett *et al.*, 1985), the organic species (*N,N,N',N'*-tetramethyl-1,3-propanediamine) is fissioned into three parts, in AlPO<sub>4</sub>-17 the piperidine has apparently reacted with a water molecule to yield a piperidinium ion and a hydroxyl, which then interacts with the aluminophosphate framework.

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## X-ray Structure and Thermal Motion of Barium *N*-Dithiocarboxylatoglycinate Trihydrate

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**Abstract.** Ba<sup>2+</sup>·C<sub>3</sub>H<sub>3</sub>NO<sub>2</sub>S<sub>2</sub><sup>-</sup>·3H<sub>2</sub>O, *M<sub>r</sub>* = 340·6, triclinic, *P* $\bar{1}$ , *a* = 7·906 (1), *b* = 7·905 (1), *c* = 8·000 (1) Å,  $\alpha$  = 96·34 (1),  $\beta$  = 100·08 (1),  $\gamma$  = 93·54 (1)°, *V* = 487·6 (1) Å<sup>3</sup>, *Z* = 2, *D<sub>m</sub>* = 2·31 (1), *D<sub>x</sub>* = 2·320 Mg m<sup>-3</sup>,  $\lambda$ (Mo *K*α) = 0·7107 Å,  $\mu$  = 4·47 mm<sup>-1</sup>, *F*(000) = 324, room temperature, final

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