Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Ti1—O1 | 1.822 (3) | Ti1—O2 | 1.789 (3) |
| Ti1—O3 | 2.138 (3) | Ti1—O4 | 1.994 (4) |
| Ti1—O7 | 2.092 (3) | Ti1—O10 | 2.001 (4) |
| Ti2—O1$^{i}$ | 1.810 (3) | Ti2—O2 | 1.836 (4) |
| Ti2—O12 | 1.980 (4) | Ti2—O13 | 2.092 (3) |
| Ti2—O15 | 2.068 (4) | Ti2—O16 | 2.006 (4) |
| C1—C2 | 1.548 (8) | C3—C4 | 1.543 (6) |
| C5—C6 | 1.537 (9) | C7—C8 | 1.528 (9) |
| C1—O3 | 1.278 (6) | C3—O7 | 1.283 (6) |
| C5—O12 | 1.281 (5) | C7—O16 | 1.300 (7) |
| C1—O5 | 1.229 (6) | C3—O8 | 1.232 (5) |
| C5—O11 | 1.211 (8) | C7—O18 | 1.219 (8) |
| C2—O4 | 1.290 (6) | C4—O10 | 1.305 (5) |
| C6—O13 | 1.283 (7) | C8—O15 | 1.266 (7) |
| C2—O6 | 1.229 (8) | C4—O9 | 1.214 (7) |
| C6—O14 | 1.239 (6) | C8—O17 | 1.223 (8) |
| O1—Ti1—O2 | 98.9 (1) | O1—Ti1—O10 | 91.4 (2) |
| O1—Ti1—O7 | 165.2 (2) | O1—Ti1—O4 | 102.8 (2) |
| O1—Ti1—O3 | 87.1 (1) | O2—Ti1—O7 | 92.7 (1) |
| O2—Ti1—O10 | 100.0 (2) | O2—Ti1—O3 | 170.6 (2) |
| O2—Ti1—O4 | 94.8 (2) | O3—Ti1—O4 | 76.7 (2) |
| O4—Ti1—O7 | 85.3 (1) | O4—Ti1—O10 | 157.7 (1) |
| O3—Ti1—O7 | 82.7 (1) | O3—Ti1—O10 | 87.0 (1) |
| O7—Ti1—O10 | 77.4 (1) | O2$^{i}$—Ti2—O1 | 99.1 (2) |
| O2—Ti2—O13 | 88.7 (1) | O2—Ti2—O15 | 165.6 (2) |
| O2—Ti2—O12 | 99.6 (2) | O2—Ti2—O16 | 90.3 (2) |
| O1$^{i}$—Ti2—O16 | 100.0 (1) | O1$^{i}$—Ti2—O12 | 91.9 (2) |
| O1$^{i}$—Ti2—O13 | 167.8 (2) | O1$^{i}$—Ti2—O15 | 90.6 (1) |
| O13—Ti2—O12 | 77.6 (1) | O13—Ti2—O15 | 83.5 (1) |
| O13—Ti2—O16 | 89.2 (1) | O12—Ti2—O15 | 90.6 (2) |
| O12—Ti2—O16 | 163.2 (1) | O15—Ti2—O16 | 77.5 (2) |
| O5—C1—O3 | 126.3 (5) | O8—C3—O7 | 127.2 (5) |
| O11—C5—O12 | 125.4 (6) | O16—C7—O18 | 125.5 (7) |
| O5—C1—C2 | 120.7 (5) | O8—C3—C4 | 119.4 (5) |
| O11—C5—C6 | 121.8 (4) | O18—C7—C8 | 121.3 (6) |
| O3—C1—C2 | 113.0 (4) | O7—C3—C4 | 113.4 (3) |
| O12—C5—C6 | 112.8 (5) | O16—C7—C8 | 113.2 (5) |
| O4—C2—O6 | 125.1 (6) | O10—C4—O9 | 125.6 (5) |
| O13—C6—O14 | 126.5 (6) | O15—C8—O17 | 126.1 (7) |
| O6—C2—C1 | 121.4 (5) | O9—C4—C3 | 121.5 (3) |
| O14—C6—C5 | 119.7 (6) | O17—C8—C7 | 120.1 (5) |
| O4—C2—C1 | 113.6 (5) | O10—C4—C3 | 113.0 (4) |
| O13—C6—C5 | 113.8 (4) | O15—C8—C7 | 113.8 (5) |

Symmetry code: (i) $-x, 1-y, -z$.

Preliminary Weissenberg photographs were used to check the quality of the chosen crystal and to determine the unit-cell dimensions. The structure was solved by direct methods using the *SHELXTL-Plus* program system (Sheldrick, 1990). All non-H atoms were located by difference synthesis. The refinement of the population parameters of the disordered water molecules was carried out by fixing the temperature factors at a reasonable value. In the final stages, the temperature factors were refined isotropically. Chemical analysis clearly indicates two water molecules in the formula unit. Omitting O23 and O26, both of which have an occupation factor of 0.1, leads to an increase of the $R$ factor from 0.0541 to 0.0578 and $\Delta F$ then shows two maxima of 1.59 and 1.48 e Å$^{-3}$ close to O21 and O22. Refinement was by full-matrix least-squares methods. The weighting scheme shows no significant dependence on $(\sin\theta/\lambda)$ and $F_o/F_{o\,\max}$.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71743 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1038]

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# Microcrystal Structure Determination of $AlPO_4$-CHA Using Synchrotron Radiation

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## Abstract

Data were recorded from a very small crystal of $AlPO_4$-CHA [trialuminum morpholinium fluoride tris(phosphate), $C_4H_{10}NO^+.Al_3(PO_4)_3F^-$] using synchrotron radiation. The aluminophosphate framework forms a three-dimensional network of channels in which F atoms form bridges between octahedral Al atoms. The N atom of the template (morpholine) is hydrogen bonded to framework O atoms.

## Comment

Knowledge of the structure of zeolitic materials is often required in order to rationalize their ion-exchange, molecular sieving and catalytic properties. Synthetic zeolites are frequently available only as polycrystalline powders and so powder diffraction is the common method of structure determination. Data collection using high-intensity radiation sources and very small single crystals has also been shown to be viable, and structures of aluminophosphates and other materials have been obtained (Cheetham, Harding, Haggitt, Mingos & Powell, 1993; Harding, Kariuki, Mathews, Smith & Braunstein, 1994; Cheetham, Harding, Rizkallah, Kaučič & Rajić 1991; Helliwell *et al.*, 1993).

The asymmetric unit of the framework is composed of one octahedrally coordinated Al atom, two tetrahedrally coordinated Al atoms and three tetrahedral P atoms. The octahedral Al1 is coordinated to four O and two F atoms. The framework forms eight-ring channels parallel to the *a*, *b* and *c* axes and six-ring channels along [111], as depicted in Fig. 2. The figure also shows the F bridge between two symmetry-related Al atoms. The structure-type code is CHA (Meier & Olson, 1987), the same as chabazite (Smith, Rinaldi & Dent Glasser, 1963). Bridging by F draws the Al atoms closer together leading to distortion of the trigonal symmetry of chabazite. The template ions reside at the intersections of the six- and eight-ring channels. The template N atom is suitably located for hydrogen bonding with the framework [3.31 (5) Å from O2 and 2.82 (5) Å from O9].

The morpholine molecules in the cages were also located in an independent synchrotron powder diffraction investigation of the material (Simmen, 1992). The Rietveld refinement of the structure using these high-resolution powder data produced results in agreement with ours.

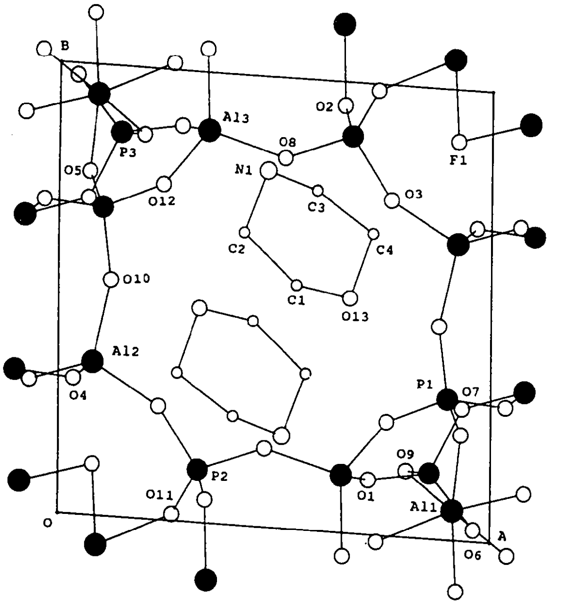

Fig. 1. A unit cell of $AlPO_4$-CHA showing the atom numbering.

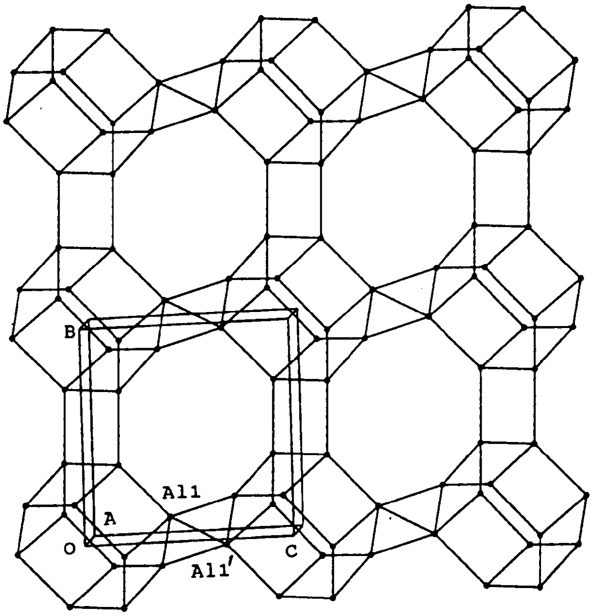

Fig. 2. The strucure of $AlPO_4$-CHA showing only the Al and P atoms for clarity.

## Experimental

The sample was provided by L. McCusker (ETH, Zurich, Switzerland) and had been prepared by Guth (1989) from a mixture with the molar ratios $1.5C_4H_{10}NO{:}Al_2O_3{:}P_2O_5{:}HF{:}100H_2O$. This mixture, with a pH between 7.0 and 7.5, was placed in an autoclave, kept at 473 K for 10 d, and then filtered.

*Crystal data*

$C_4H_{10}NO^+.Al_3(PO_4)_3F^-$
$M_r = 472.98$
Triclinic
$P\bar{1}$
$a = 9.333\ (11)$ Å
$b = 9.183\ (8)$ Å
$c = 9.162\ (7)$ Å
$\alpha = 88.45\ (6)°$
$\beta = 102.57\ (2)°$
$\gamma = 93.76\ (4)°$
$V = 764.7\ (13)$ Å$^3$

$Z = 2$
$D_x = 2.054$ Mg m$^{-3}$
Synchrotron radiation
$\lambda = 0.895\ (5)$ Å
Cell parameters from 50 reflections
$\mu = 0.63$ mm$^{-1}$
$T = 293\ (2)$ K
Block
0.035 × 0.020 × 0.015 mm
Transparent

*Data collection*

Enraf-Nonius FAST diffractometer
611 measured reflections
590 independent reflections
289 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.04$
$\theta_{\rm max} = 30.86°$
$h = -7 \rightarrow 6$
$k = -5 \rightarrow 9$
$l = 0 \rightarrow 10$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.081$
$wR$(all reflections) = 0.2097
$S$(all reflections) = 0.838
590 reflections
67 parameters
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.1306P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.120$
$\Delta\rho_{\rm max} = 0.390$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.383$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and isotropic displacement parameters* (Å$^2$)

| | $x$ | $y$ | $z$ | $U_{\rm iso}$ |
|---|---|---|---|---|
| Al1 | 0.9124 (11) | 0.0651 (9) | 0.3633 (7) | 0.0170 (10) |
| Al2 | 0.0767 (11) | 0.3415 (9) | 0.8690 (8) | 0.0170 (10) |
| Al3 | 0.3448 (11) | 0.8728 (9) | 0.1061 (7) | 0.0170 (10) |

| | | | | |
|---|---|---|---|---|
| P1 | 0.8999 (9) | 0.3125 (9) | 0.1192 (7) | 0.0170 (10) |
| P2 | 0.3218 (10) | 0.1185 (9) | 0.8729 (7) | 0.0170 (10) |
| P3 | 0.1411 (10) | 0.8546 (9) | 0.3311 (7) | 0.0170 (10) |
| F1 | 0.9192 (21) | 0.8875 (19) | 0.4878 (14) | 0.0228 (15) |
| O1 | 0.7149 (25) | 0.1209 (21) | 0.7279 (17) | 0.0228 (15) |
| O2 | 0.6609 (26) | 0.9471 (23) | 0.9697 (18) | 0.0228 (15) |
| O3 | 0.7682 (24) | 0.7471 (22) | 0.1386 (18) | 0.0228 (15) |
| O4 | 0.0354 (25) | 0.3027 (22) | 0.0368 (17) | 0.0228 (15) |
| O5 | 0.0722 (24) | 0.7623 (24) | 0.7350 (19) | 0.0228 (15) |
| O6 | 0.9613 (26) | 0.0273 (22) | 0.7321 (18) | 0.0228 (15) |
| O7 | 0.9291 (25) | 0.2953 (24) | 0.7220 (18) | 0.0228 (15) |
| O8 | 0.5223 (26) | 0.8249 (22) | 0.1495 (18) | 0.0228 (15) |
| O9 | 0.8055 (25) | 0.1458 (23) | 0.4969 (19) | 0.0228 (15) |
| O10 | 0.1199 (24) | 0.5246 (22) | 0.8592 (18) | 0.0228 (15) |
| O11 | 0.2641 (26) | 0.0150 (24) | 0.7495 (18) | 0.0228 (15) |
| O12 | 0.2408 (26) | 0.7476 (23) | 0.9864 (18) | 0.0228 (15) |
| C1 | 0.5505 (64) | 0.5414 (56) | 0.7447 (43) | 0.139 (9) |
| C2 | 0.4293 (52) | 0.6513 (72) | 0.7134 (54) | 0.139 (9) |
| C3 | 0.5960 (58) | 0.7546 (49) | 0.5746 (46) | 0.139 (9) |
| C4 | 0.7256 (50) | 0.6670 (57) | 0.6562 (50) | 0.139 (9) |
| N1 | 0.4833 (57) | 0.7920 (58) | 0.6562 (55) | 0.139 (9) |
| O13 | 0.6734 (62) | 0.5202 (51) | 0.6797 (47) | 0.139 (9) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Al1—O5$^{i}$ | 1.81 (3) | Al1—O6$^{iv}$ | 1.87 (2) |
| Al1—O11$^{ii}$ | 1.86 (2) | Al1—O9 | 1.93 (2) |
| Al1—F1$^{iii}$ | 1.88 (2) | Al1—F1$^{v}$ | 1.96 (2) |

| | |
|---|---|
| Al(tetrahedral)—O (mean) | 1.72 (2) |
| Al(tetrahedral)—O (range) | 1.69 (2)-1.78 (2) |
| P—O (mean) | 1.53 (2) |
| P—O (range) | 1.46 (2)-1.62 (2) |

| | | | |
|---|---|---|---|
| O5$^{i}$—Al1—O11$^{ii}$ | 100.4 (10) | F1$^{iii}$—Al1—O9 | 85.3 (8) |
| O5$^{i}$—Al1—F1$^{iii}$ | 92.8 (8) | O11$^{ii}$—Al1—F1$^{v}$ | 87.0 (10) |
| O5$^{i}$—Al1—O6$^{iv}$ | 94.4 (10) | F1$^{iii}$—Al1—F1$^{v}$ | 79.7 (6) |
| O11$^{ii}$—Al1—O6$^{iv}$ | 97.5 (8) | O6$^{iv}$—Al1—F1$^{v}$ | 84.9 (9) |
| O5$^{i}$—Al1—O9 | 92.9 (9) | O9—Al1—F1$^{v}$ | 86.8 (9) |
| O11$^{ii}$—Al1—O9 | 89.5 (9) | | |

| | |
|---|---|
| O—Al(tetrahedral)—O (range) | 106.4 (10)-112.6 (9) |
| O—P—O (range) | 104.0 (10)-115.2 (13) |

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, -y, 1-z$; (iii) $2-x, 1-y, 1-z$; (iv) $2-x, -y, 1-z$; (v) $x, y-1, z$; (vi) $x, 1+y, z$; (vii) $1-x, 1-y, 2-z$; (viii) $1-x, 2-y, 1-z$; (ix) $x-1, y, z$; (x) $x, y, z-1$; (xi) $1+x, y, z$; (xii) $x, y, 1+z$.

The crystal was mounted on a strand of glass wool (1 mm long) attached to a stronger glass fibre. Data were recorded on workstation 9.6 at the SRS, Daresbury, England. Crystal to detector distance was 40 mm and the detector angle ($2\theta$) was $-28°$. Frames of 0.2° were accumulated for 25-35 s at 2 GeV and 187 mA. A collimator of diameter 0.2 mm was used. The incident beam intensity was monitored continuously and intensity data were corrected for decrease with time. The images were processed offline by *MADNES* (Messerschmidt & Pflugrath, 1987) using a profile-fitting procedure characterized by *EVAL* = 6. Data collection, cell refinement and data reduction were performed using *MADNES*. After refinement of the framework, calculation of the difference map revealed the atoms of the template ion as distinct peaks. Assignment of the F site was based on chemical and geometrical grounds as well as spectroscopic evidence and confirmed by refinement of site occupancy. All atoms were refined isotropically with one temperature factor for Al and P, one for framework O and F and another for non-H template atoms (H-atom $U_{iso}$ was 1.2 times this value). In the last cycles of refinement, the template was refined as a rigid body. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1992). Molecular graphics: *DTMM* (Crabbe & Appleyard, 1991). Software used to prepare material for publication: *SHELXL*92.

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Lists of structure factors and H-atom coordinates have been deposited with the IUCr (Reference: LI1095). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Tetracarbonyl[1-phenyl-*N*-(2-pyridylmethylidene)ethylamine-*N*,*N'*]molybdenum(0)

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## Abstract

The Mo atom in the title complex, $[Mo(CO)_4(C_{14}H_{14}N_2)]$ (I), is in a distorted octahedral environment. The Mo—C distances *trans* to each N atom [1.944 (5), 1.955 (4) Å] are significantly shorter than those *cis* to both N atoms [2.031 (5), 2.045 (4) Å]. The carbonyl groups *cis* to both N atoms are bent away from the nitrogen-donor ligand with Mo—C—O angles of 172.1 (4) and 173.9 (4)°.