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Structure of SAPO-31 Refined from Single-Crystal Diffraction Data: Substitution of P by Si Established by Diffraction Methods

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Abstract

SAPO-31 is a silicoaluminophosphate with an idealized composition $Al_9P_8SiHO_{36}$ and a crystal structure close to $AlPO_4$-31, a microporous substance of zeolite framework topology ATO, $M_r = 1095.72$, rhombohedral, hexagonal setting, space group $R\bar{3}$, $a = 20.839$ (2), $c = 5.041$ (1) Å, $V = 1886$ (1) Å^3, $Z = 2$, $D_x = 1.93$ Mg m^{-3}, μ(Mo $K\alpha$) = 0.72 mm^{-1}, λ(Mo $K\alpha$) = 0.71069 Å, $F(000) = 1081$, 2440 single crystal X-ray intensities measured, 391 averaged unique intensities, refinement on $|F|$, $R = 0.051$, $wR = 0.018$, including all unobserved and weak F_{hkl}. A previous refinement based on synchrotron X-ray powder diffraction data for $AlPO_4$-31 is confirmed with much higher precision. The Si atoms in SAPO-31 replace statistically part of the P atoms in the framework. This type of substitution has thus been established by X-ray diffraction methods for the first time in a microporous silicoaluminophosphate.

Introduction

The synthetic compound $AlPO_4$-31 is one of a series of microporous aluminophosphates that are in many ways analogous to zeolites, but have a neutral framework. This type of compound is usually crystallized in the presence of so-called template molecules, in the present case of di-*n*-propylamine [DPA, $(C_3H_7)NH(C_3H_7)$]. The crystal structure of calcined $AlPO_4$-31 was solved from synchrotron X-ray

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powder diffraction data by Bennett & Kirchner (1992). In a calcined sample the template molecules are removed by appropriate heat treatment. The topology of the framework corresponds to the new zeolite structure type named ATO (Meier & Olson, 1992). We succeeded in growing single crystals of SAPO-31 (Si-doped $AlPO_4$-31 or silicoaluminophosphate) sufficiently large to allow a single-crystal study of the material. Our sample was not calcined, thus the DPA molecules were still present in the micropores of the framework.

Experimental

Colorless crystals of SAPO-31 were crystallized hydrothermally following the procedure claimed by Kornatowski, Finger, Baur & Rozwadowski (1993), which is similar to that described by Kornatowski & Finger (1990) and Finger & Kornatowski (1990) for materials with frameworks of AFI (Heier & Olson, 1992) topology. The reaction gel was prepared by reacting the following components: solution A, aluminium oxide hydrate solution with about 2.3 wt% Al_2O_3 stirred for 12 h with Ludox AS40 (40 wt% SiO_2), and solution B, 85% H_3PO_4 diluted with H_2O, cooled down to ~273 K and reacted under stirring with di-n-propylamine (DPA) to yield a clear solution (pH 3.5–4.0). Solution B was dropped into solution A under vigorous stirring and the reaction mixture was stirred for another 3 h, while the pH remained at the values as for solution B. The gel was then poured into a Teflon-lined stainless steel autoclave and crystallized at 463 K for 48–60 h. The reagents were used in amounts yielding a final reaction gel of the formal molar composition $1Al_2O_3$.-$1.04P_2O_5.aSiO_2.1.72DPA.bH_2O$, where $a \approx 0.2$ and $b \geq 500$. If the crystals of SAPO-31 would incorporate into the framework as much Si as was present in the reaction gel the approximate framework composition of our SAPO-31 phase should be $Al_9P_8SiHO_{36}$. Hydrogen is assumed to be present in order to provide charge balance. The actual ratio of Al:P:Si as determined by an ICP (inductively coupled plasma) analysis was 9.08:8.06:0.86. The analysis of another much smaller sample synthesized in parallel in another autoclave yielded 8.94:7.94:1.12. These results agree very well with the composition expected from the reaction gel. We have no information on the amount of DPA or water in our sample, but both are presumably present. The amount of pore space would accommodate at most 1.5 DPA molecules per unit cell. This would correspond to a content of 6.5 wt% DPA.

The unit-cell constants were determined at room temperature on the basis of 25 precisely measured reflections (Table 1). Three-dimensional X-ray diffraction data were collected on an Enraf–Nonius

Table 1. $Al_9P_8SiHO_{36}$-31 crystal data, data collection and R values

The values of M_r, D_x and μ do not contain a contribution from the DPA or water molecules.

Chemical formula	$Al_9P_8SiHO_{36}.xDPA.yH_2O$
Crystal system	Trigonal
Space group	$R\bar{3}$
a (Å)	20.839 (2)
c (Å)	5.014 (1)
Range of 2θ of reflections for cell-constant determination ( )	21 to 32 and -21 to -32
V (Å^3)	1886 (1)
Z	2
M_r	1095.72
D_x (Mg m^{-3})	1.93
Scan	ω
λ(Mo $K\alpha$) (Å)	0.71069
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.48
μ(Mo $K\alpha$) (mm^{-1})	0.72
Size of crystal (μm)	$100 \times 30 \times 30$
Number of I_{hkl} measured	2440
Number of unique F_{hkl} used in refinement (NREF) (including all weak and unobserved F_{hkl})	391
Standard reflections	2 every 8 h
Variation of standards	Maximally $\pm 1.0\%$
R(internal) based on F^2	0.068
$h_{min},h_{max}, k_{min},k_{max}, l_{min},l_{max}$	$-16,16, -16,16, -4,4$
Number of variables (NVAR)	67
$R = \Sigma[F_o| - F_c|]/\Sigma|F_o$	0.051
$wR = [\Sigma w(F_o - F_c)^2/\Sigma wF_o^2]^{1/2}$	0.018
$GoF = [\Sigma w(F_o - F_c)^2/(NREF - NVAR)]^{1/2}$	1.99

CAD-4 diffractometer at room temperature on a single crystal (an elongated hexagonal prism) within a complete sphere of reflection. Because of the small size of the crystal and the small value of μ, an absorption correction was not needed. The starting parameters for the refinement were the positional coordinates determined by Bennett & Kirchner (1992) for $AlPO_4$-31. The only clue we had for the location of the DPA molecules in the pores is a very broad and ill-defined maximum visible in a difference synthesis within the large tubular space surrounded by a 12-ring (see Fig. 1). The difference synthesis was based on structure factors from a refinement using only framework atoms. Attempts to locate C or N atoms belonging to the template molecule failed. Since DPA is basically a chain molecule $[(C_3H_7)NH(C_3H_7)]$, of a length of slightly more than 10 Å, this means that it must be statistically distributed over the length of the tubular pore (repeat distance in the c direction of 5.014 Å) of SAPO-31, without having a preferred location. After some appropriate experimentation by trial and error we modeled this electron cloud by choosing the three largest maxima, using the scattering factors of carbon, assigning a displacement factor of 0.20 Å^2 and refining their coordinates and the population factors. The weighted R factor without the three C positions was 0.039%, and with them was 0.018%. The refinement was uneventful and allowed the determination of anisotropic displacement param-

eters (for results see Tables 2 and 3*). Because of the good experience we had with using scattering factors for half-ionized atoms in the case of natrolite (Stuckenschmidt, Joswig & Baur, 1993), the scattering factors for O^{-1}, $Al^{1.5+}$ and P were taken or interpolated from the values listed in the *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 73–75). Si was not introduced explicitly in the structural model. The population factors of the P and Al positions were refined in order to find the location of silicon which in principle could replace either of these atoms. The population factor of the P position depends also slightly on the scattering factor used. The low population factor for the P site is an indication that the Si atoms scattering less share their positions statistically with the P atoms. If conversely Si atoms were partly replacing the Al atoms, the population factor for the Al site should be greater than one. In fact it is (insignificantly) smaller than one. The final value of wR is extremely low if one considers that all possible structure factors of low intensity were included in the least-squares calcula-

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* Lists of observed and calculated structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71660 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH0036]
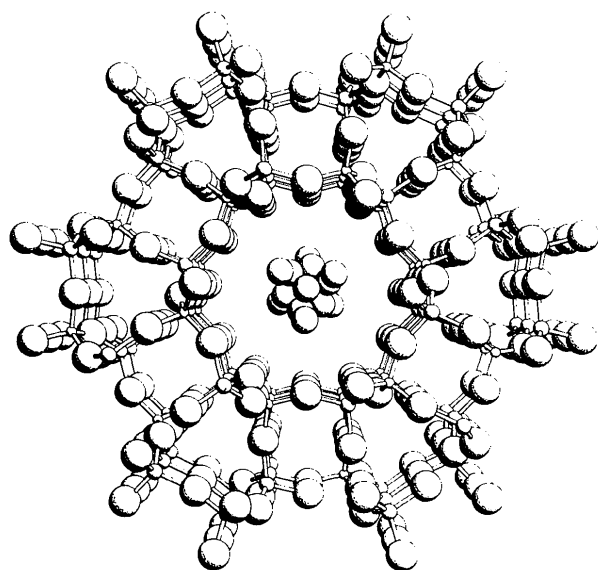

Fig. 1. Perspective view of SAPO-31 parallel to *c* down the tubular opening lined by 12-rings. The 4-rings and the very distorted 6-rings are arranged around the tubular opening. The smallest spheres correspond to the P atoms, the medium sized spheres to Al, the largest to the O atoms; the spheres in the center of the 12-ring indicate the C-atom positions modeling the DPA molecule [drawing prepared using *ATOMS* (Dowty, 1993)].

Table 2. *SAPO*-31, *coordinates,* U_{eq} (Å²) *and population factors*

(*a*) This work, space group $R\bar{3}$ [refinement with *CRYLSQ* (Olthof-Hazekamp, 1990); (*b*) DLS-simulated structure in space group $R\bar{3}m$ [using *RERIET* (Kassner, 1993)]. Multiplicity = mult., Wyckoff letter = Wyck., site symmetry = symm., population factors = pop.

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	mult. Wyck.	symm.	x	y	z	U_{eq}	pop.
Al	*a* 18(*f*)	1	0.61736(8)	0.08455(7)	0.0381(3)	0.0239(9)	0.984(7)
T(1)	*b* 36(*i*)	1	0.6156	0.0861	0.0765	- -	—
P	*a* 18(*f*)	1	0.47126(8)	0.08774(7)	0.1138(3)	0.0166(9)	0.926(7)
T(1)'	*b* 36(*i*)	1	0.4705	0.0861	0.0765	·	—
O(1)	*a* 18(*f*)	1	0.55134(15)	0.10711(17)	0.1317(6)	0.038(2)	1.0
O(1)	*b* 18(*h*)	.*m*	0.5548	0.1096	0.1401	·	—
O(2)	*a* 18(*f*)	1	0.42390(16)	0.00893(16)	0.0133(6)	0.042(2)	1.0
O(2)	*b* 18(*f*)	.2	0.4249	0	0		—
O(3)	*a* 18(*f*)	1	0.44586(17)	0.09533(16)	0.3880(7)	0.042(2)	1.0
O(3)	*b* 18(*f*)	.2	0.4338	0.1005	0.3333	-·	
O(4)	*a* 18(*f*)	1	0.46468(16)	0.14061(16)	0.9208(5)	0.042(2)	1.0
O(4)	*b* 18(*g*)	.2	0.4680	0.1346	0.8333	—	—
C(1)	*a* 18(*f*)	1	0.046(2)	0.013(3)	0.111(8)	[0.2]	0.46(2)
C(2)	*a* 18(*f*)	1	0.064(3)	0.029(4)	0.388(12)	[0.2]	0.25(2)
C(3)	*a* 6(*c*)	3	0	0	0.30(3)	[0.2]	0.33(5)

Table 3. *SAPO*-31, *bond lengths* (Å) *and angles* (°) *and comparison with other* P—O *and* Al—O *bond lengths*

These were calculated using *SADIAN*90 (Baur & Kassner, 1991) (see text also).

	Al—O			P—O
Al—O(3)	1.705 (3)		P—O(3)	1.510 (2)
Al—O(2)	1.710 (3)		P—O(1)	1.510 (3)
Al—O(1)	1.727 (2)		P—O(2)	1.518 (3)
Al—O(4)	1.726 (2)		P—O(4)	1.523 (3)
Mean	1.717		Mean	1.515

	O—Al—O	O—O		O—P—O	O—O
O(3)—O(2)	110.8 (1)	2.812 (3)	O(1)—O(1)	108.4 (2)	2.450 (3)
O(3)—O(1)	109.7 (2)	2.806 (4)	O(1)—O(2)	110.0 (2)	2.481 (4)
O(3)—O(4)	109.1 (1)	2.795 (4)	O(1)—O(4)	109.4 (2)	2.475 (3)
O(2)—O(1)	109.6 (1)	2.809 (4)	O(3)—O(2)	110.1 (2)	2.481 (4)
O(2)—O(4)	110.0 (2)	2.815 (4)	O(3)—O(4)	109.9 (2)	2.482 (4)
O(1)—O(4)	107.6 (1)	2.787 (3)	O(2) O(4)	109.1 (2)	2.477 (4)

	P—O—Al	P—Al		P—O—Al	P—Al
P—O(1)—Al	146.7 (2)	3.102 (1)	P—O(2)—Al	165.2 (2)	3.202 (2)
P—O(3)—Al	156.1 (2)	3.145 (2)	P—O(4)—Al	137.0 (2)	3.025 (2)

	Overall mean (Baur, 1981)	Berlinite (Ngo Thong & Schwarzenbach, 1979)	Adjusted for thermal motion as has been observed in SAPO-31	SAPO-31 (this work)
P—O	1.537	1.522	1.504	1.515
Al—O	1.752	1.736	1.718	1.717

tion, even the F_{hkl} derived from the 29 I_{hkl} which were measured with negative intensities were included with zero values (see Kassner *et al.*, 1993).

The following computer programs were used in the course of the work: *CRYLSQ* (Olthof-Hazekamp, 1990), *SADIAN*90 (Baur & Kassner, 1991), *RERIET* (Kassner, 1993), *ORTEP* (Davenport, Hall & Dreissig, 1990) and *ATOMS* (Dowty, 1993).

Results and discussion

The crystal structure of $AlPO_4$-31 as determined by Bennett & Kirchner (1992) from synchrotron X-ray powder diffraction data is verified by our single-crystal diffraction experiment on SAPO-31. All of their descriptions of the topology of the framework of the ATO zeolite structure type are confirmed. However, the improved precision of our single-crystal refinement shows itself in appreciably smaller e.s.d.'s of the parameters (by factors ranging from ten to 20) and by a much smaller spread in the values of the distances Al—O and P—O and of the angles O—Al—O and O—P—O. On average, the atomic positions differ in the two determinations by 0.13 Å, the smallest deviation is observed for Al (0.04 Å), the largest for O(4) (0.21 Å).

Three facts are noteworthy: (a) the unit-cell constants of our as-synthesized material, i.e. a SAPO-31 with the template molecules (di-n-propylamine, DPA) still present in the pores of this molecular sieve, are essentially identical to those of the calcined sample of $AlPO_4$-31 studied by Bennett & Kirchner (1992), where the template had been removed by heat treatment. The values are $a = 20.839$ (2) Å [20.827 (1) Å] and $c = 5.014$ (1) Å [5.003 (1) Å], where the numbers in parentheses refer to the calcined $AlPO_4$-31 sample. (b) We could not model the molecules of DPA within the pores except in a very approximate fashion. Of course one cannot expect an ordered arrangement of molecules, that are of about twice the length of the cell constant of the tubular opening. (c) The triangular distortion of the 6-ring, which is arranged around the 3_1 and 3_2 screw axes (see Fig. 1), that was already indicated by Bennett & Kirchner (1992), is confirmed by our refinement.

The disordered state of the template molecules within the tubular pores indicates that there may be little interaction between the framework and the DPA molecules. Therefore, the cell constants are likely to be little affected by the presence or absence
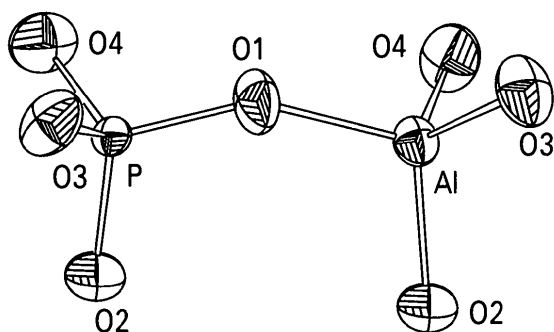

Fig. 2. Displacement ellipsoids of the tetrahedrally coordinated atoms P and Al and of the O atoms in SAPO-31 [drawing prepared using ORTEP (Davenport et al., 1990)].

of the template molecule in the as-synthesized or calcined samples. The small difference in the cell constants between $AlPO_4$-31 and SAPO-31 may then be due to the statistical replacement of part of the P atoms by Si atoms within the framework.

The mean Al—O and P—O distances in molecular sieves of composition $AlPO_4$ are usually shorter than one observes for tetrahedrally coordinated Al and P in general (Baur, 1981; Table 3 and Fig. 2.) They are even shorter than has been observed in the quartz-type modification of $AlPO_4$, in berlinite (Ngo Thong & Schwarzenbach, 1979). This shortening relative to the bond lengths in berlinite must be at least partly related to the large atomic displacement parameters found in many aluminophosphates. Liebau (1984) had discussed this for the analogous case of the SiO_2 modifications. He found that the apparent bond length Si—O is shortened by 0.075 Å for an increase in the displacement factor $B = 1$ Å^2. Therefore, the bond lengths in SAPO-31, where the displacement factors are more than three times as large as in berlinite, should be shorter by about 0.018 Å than those in berlinite (see 'adjusted' column in Table 3). This procedure gives a good agreement for the distances Al—O between the observation in SAPO-31 and the adjusted value for a berlinite assumed to have the same high displacement factors as SAPO-31. However, the observed distance P—O in SAPO-31 is clearly longer than the value estimated from the comparison with the distance in berlinite as adjusted for an assumed high displacement factor. This probably is due to the statistical occupancy of the phosphorus site by silicon, which amounts to about 10%. When we consider that an average Si—O bond is about 0.1 Å longer than a P—O bond, the (P,Si)—O bond length in SAPO-31 has to be reduced by approximately 0.01–1.505 Å in order to be comparable to a pure P—O bond length. Then it is extremely close to the value estimated from the adjustment of the bond length P—O observed in berlinite (1.504 Å, see column 3 at bottom of Table 3).

In principle the Si atoms in a silicoaluminophosphate could substitute either for the Al atoms, or for the P atoms, or else two Si atoms could replace one Al—P pair (only in the latter case would the framework remain charge balanced). All three mechanisms have been discussed in the literature. Chemical analyses and ^{29}Si MAS NMR spectra showed that in SAPO-5 the Si atoms replace the P atoms (Zibrowius et al., 1991). In SAPO-37 (zeolite structure type FAU), the Si atoms were found to replace Al—P pairs (Martens, Janssens, Grobet, Beyer & Jacobs, 1989). In this respect SAPO-31 resembles SAPO-5. Our case of SAPO-31 seems to be the first instance among the microporous silico-aluminophosphates where this type of substitution

has been established by X-ray diffraction methods in conjunction with chemical analyses and without recourse to MAS NMR methods.

If one could synthesize an SiO_2 modification with the structure of SAPO-31 (instead of just doping $AlPO_4$-31 with silicon) there would be no reason for it to crystallize in the same space group as $AlPO_4$-31 and SAPO-31, namely in $R\bar{3}$ since there is no need to distinguish between Al and P. Thus, the symmetry of the compound could be higher and the space group could be $R\bar{3}m$, a supergroup of $R\bar{3}$. We simulated its crystal structure by distance least squares (using *RERIET*, Kassner, 1993) assuming an Si—O distance of 1.60 Å. In this higher symmetry all O atoms would be located on special positions (site symmetry either 2 or *m*), but none of the Si—O—Si angles would of necessity be straight, thus the simulated structure given here (Table 2) is a likely candidate for a SiO_2 modification with the same topology as $AlPO_4$-31.

Concluding remarks

While the single-crystal structure refinement of SAPO-31 is much more precise than the synchrotron X-ray powder diffraction study of Bennett & Kirchner (1992), it still did not prove possible to locate precisely the template molecules in the pores of SAPO-31, because they are highly disordered. The higher precision makes it possible, however, to find that the Si atoms replace partly the P atoms and not the Al atoms in this microporous molecular sieve.

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Structure Refinement of the Icosahedral Quasicrystal $Al_{57}Li_{32}Cu_{11}$

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Abstract

The structure of the icosahedral quasicrystal $Al_{57}Li_{32}Cu_{11}$ has been refined within the superspace formalism using symmetry-adapted surface harmonics for the description of the boundaries of atomic surfaces (occupation domains) in internal space. The refinement process has been performed with a general program, *QUASI*, recently developed for this purpose. Besides published neutron and X-ray diffraction data [de Boissieu, Janot, Dubois, Audier & Dubost (1991). *J. Phys. Condens. Matter*, **3**, 1–25],