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Structure Determination of a Monohydrated Piperazinium Hydrogenphosphate

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Abstract

Piperazinium hydrogenphosphate monohydrate, $(N_2C_4H_{12}).HPO_4.H_2O$, is composed of a network of isolated PO_4 tetrahedra and organic six-membered rings of piperazine. The two amino groups of the piperazine are protonated and the rings are stacked along the [010] direction. The stability of the framework is ensured by strong O—H linkages between the O atoms from the PO_4 tetrahedra and H atoms from the diamine and water molecules.

Comment

The $AlPO_4$-*n* family has been studied extensively over a few years. These compounds possess great similarities to the known zeolites whose frameworks are characterized by their large porosities. This property is fundamental for their activity in catalysis or as molecular sieves. One route to the synthesis of microporous structures uses an organic amine as a template. However, Tapp, Milestone & Bibby (1988) noted that this type of preparation sometimes leads to a simple amine phosphate at low temperatures. This paper describes the synthesis and X-ray structure determination of the piperazinium hydrogenphosphate $(N_2C_4H_{12}).HPO_4.H_2O$.

The title compound consists of a framework of isolated PO_4 tetrahedra interleaved with piperazine and H_2O molecules. The two amino groups of each piperazine molecule are protonated giving the formula $(N_2C_4H_{12})^{2+}$. The organic rings thus constituted are stacked perpendicularly to the [010] direction at approximatively the $y = 0$ and $y = \frac{1}{2}$ levels, whereas the PO_4 tetrahedra and the H_2O molecules are at the $y = \frac{1}{4}$ and $y = \frac{3}{4}$ levels (Fig. 1). The P atoms are distributed at one site in a tetrahedral coordination with three P—O bond lengths around 1.52 Å and a fourth of 1.589 (3) Å. This difference has previously been observed in $(C_2H_{10}N_2).Na_2.(HPO_4)_2.6H_2O$ (Averbuch-Pouchot, Durif & Guitel, 1987) and $(C_2H_{10}N_2).HPO_4$ (Averbuch-Pouchot & Durif, 1987); in each case it is characteristic of the existence of an $(HPO_4)^{2-}$ hydrogenphosphate species. No residual electronic density is detected in the vicinity of the O(4) atom in $(N_2C_4H_{12}).HPO_4.H_2O$, but to satisfy at the electroneutrality of the compound it is reasonable to locate a hydroxyl group at this site. Each O atom [except O(4)] of the PO_4 tetrahedra makes two strong O—H linkages with distances ranging from 1.72 (4) to 1.92 (6) Å; this network of hydrogen bonds ensures the stability of the framework.

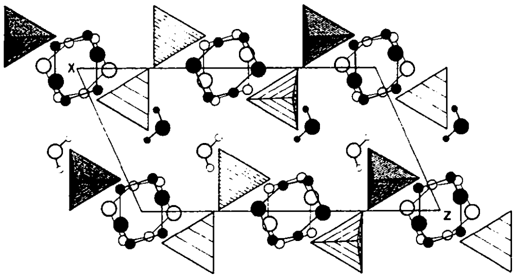

Fig. 1. Projection of $(N_2C_4H_{12}).HPO_4.H_2O$ along [010]; the piperazine rings and the H_2O molecules are drawn.

Experimental

Crystal data

$C_4H_{12}N_2^{2+}.HO_4P^{2-}.H_2O$
$M_r = 202.16$
Monoclinic
$P2_1/c$
$a = 6.4176\ (9)$ Å
$b = 12.296\ (2)$ Å
$c = 12.215\ (2)$ Å
$\beta = 114.30\ (1)°$
$V = 878.5\ (3)$ Å^3
$Z = 4$
$D_x = 1.529$ Mg m^{-3}

Mo *Kα* radiation
$\lambda = 0.71069$ Å
Cell parameters from 34 reflections
$\theta = 15$-$16°$
$\mu = 0.294$ mm^{-1}
$T = 293$ K
Plate
0.228 × 0.171 × 0.027 mm
Colorless

Data collection

Stoe Siemens AED-2 diffractometer
ω/θ scans
Absorption correction: none
4232 measured reflections
4232 independent reflections
891 observed reflections [$I > 3.0\sigma(I)$]

$R_{\rm int} = 0.0$
$\theta_{\rm max} = 27.5°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity variation: 2.1%

Refinement

Refinement on F
Final $R = 0.039$
$wR = 0.034$
891 reflections
155 parameters
All H-atom parameters refined, U(H) with a common value
$w = 2.0811/[\sigma^2(F) + 0.0002F^2]$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) (P, O) and Cromer & Mann (1968) (C, H, N)

Data collection: Stoe *DIF4*. Cell refinement: Stoe *DIF4*. Data reduction: Stoe *REDU4*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985), option *TREF*. Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	$U_{\rm eq}$
P	0.7918 (2)	0.2453 (1)	0.1453 (1)	0.0177 (6)
O1	0.7896 (5)	0.1255 (2)	0.1767 (2)	0.026 (2)
O2	0.9947 (5)	0.3039 (2)	0.2396 (3)	0.035 (2)
O3	0.7778 (4)	0.2604 (2)	0.0197 (2)	0.029 (2)
O4	0.5664 (5)	0.2987 (3)	0.1454 (3)	0.037 (3)
O5	0.4182 (5)	0.2209 (3)	0.3018 (3)	0.043 (3)
N1	0.9090 (6)	0.0815 (3)	0.4095 (3)	0.022 (3)
N2	0.9839 (7)	0.9607 (3)	0.1062 (3)	0.028 (3)
C1	0.8225 (8)	0.9225 (4)	−0.0127 (4)	0.028 (3)
C2	0.7699 (8)	−0.0141 (4)	0.4130 (4)	0.025 (4)
C3	1.2060 (9)	0.9927 (4)	0.1062 (4)	0.028 (3)
C4	1.1552 (7)	0.0542 (4)	0.4615 (4)	0.024 (3)

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

P—O3	1.511 (2)	N2—C3	1.479 (7)
P—O2	1.519 (2)	C1—C3$^{\rm ii}$	1.50 (1)
P—O1	1.524 (2)	O1—H2$^{\rm iii}$	1.89 (4)
P—O4	1.589 (3)	O1—H11	1.92 (6)
N1—C2	1.487 (7)	O2—H13$^{\rm iv}$	1.72 (4)
N1—C4	1.479 (6)	O2—H8$^{\rm v}$	1.79 (4)
C2—C4$^{\rm i}$	1.489 (5)	O3—H14$^{\rm vi}$	1.80 (3)
N2—C1	1.471 (9)	O3—H5$^{\rm vi}$	1.89 (6)
O2—P—O3	112.70 (3)	O1—P—O2	112.20 (3)
O1—P—O3	111.80 (3)	O4—P—O2	107.50 (3)
O4—P—O3	106.00 (3)	O1—P—O4	107.30 (3)

Symmetry code: (i) $2-x, -y, 1-z$; (ii) $2-x, 2-y, -z$; (iii) $x, y-1, z$; (iv) $1+x, y, z$; (v) $2-x, y-\frac{1}{2}, \frac{1}{2}-z$; (vi) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

In order to synthesize a fluoro-aluminophosphate with a microporous framework, $(N_2C_4H_{12}).HPO_4.H_2O$ was prepared hydrothermally from a 1:2:2:5:80 mixture of Al_2O_3, P_2O_5, NH_4F, piperazine and water. The resultant product was heated at 443 K for 3 d in an autoclave under autogenous pressure then filtered, washed and finally dried in air. A fragment of a colorless crystal without definite faces was isolated and its quality tested with Laue photographs.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71053 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1011]

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(4*S*,5*S*)-4-Methyl-5-phenyl-1,3-oxazolidin-2-one

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Abstract

Ring closure of methyl (2*S*,3*R*)-*N*-[(3-hydroxy-3-phenyl)-2-propyl]carbamate by treatment with *p*-toluenesulfonyl chloride and pyridine leads to the title compound in which the oxazolidinone ring is puckered with C5 out of the plane by 21.56°.

Comment

In our studies of the stereoselective conversion of (-)-(1*R*,2*S*)-norephedrine into (+)-(1*S*,2*S*)-2-amino-1-phenylpropanol, we found that (2*S*,3*R*)-*N*-[(3-hydroxy-

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