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Bis(2-methylimidazolium) hydroxodiphosphatoaluminium

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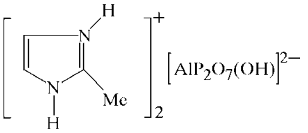
The title compound, $(C_4H_7N_2)_2[AlP_2O_7(OH)]$, is a one-dimensional extended-chain aluminophosphate prepared by a solvothermal synthesis from an alcohol system. The infinite $[AlP_2O_7(OH)]^{2-}$ chains composed of AlO_4, $PO_2(=O)_2$ and $PO_2(=O)(OH)$ tetrahedra are linked *via* hydrogen bonds to the 2-methylimidazolium cations.

Comment

Recent studies of the synthesis of crystalline aluminophosphates have revealed the diversity of their topology (*e.g.* Yu *et al.*, 2001). Such crystalline aluminophosphates are classified into one-, two- and three-dimensional structural groups according to the way in which the coordination polyhedra around the Al and P atoms are linked. An infinite one-dimensional polyhedral chain composed of corner-shared four-membered rings is one of the common structural units found in a variety of one-dimensional aluminophosphates. $[NH_3(CH_2)_2NH_3](H_3O)[AlP_2O_8]$ (Wang *et al.*, 1990), $[CH_3(CH_2)_5NH_3][AlP_2O_6(OH)_2]$ (Jones *et al.*, 1990), $[NH_3(CH_2)_2NH_3](NH_4)[AlP_2O_8]$ (Gao *et al.*, 1996) and $[NH_3(CH_2)_2NH_3]_{0.5}[NH_3(CH_2)_3NH_3]_{0.5}[AlP_2O_8]$ (Sugiyama *et al.*, 1999) are included in this category. The title compound, $(CH_3C_3H_4N_2)_2[AlP_2O_7(OH)]$, (I), is another example of this structural group, as well as the first example with a cyclic amine template.



(I)

The structure of (I) is composed of $Al3O_4$, $P1O_2(=O)_2$ and $P2O_2(=O)(OH)$ tetrahedra, forming infinite one-dimensional $[AlP_2O_7(OH)]^{2-}$ chains (Fig. 1 and Table 1). These chains run parallel to the *a* axis and are joined together along the *b* and *c* axes by the 2-methylimidazolium cations. The $Al3O_4$ tetrahedra share four O atoms with the $P1O_2(=O)_2$ and $P2O_2(=O)(OH)$ tetrahedra, with Al3—O distances ranging from 1.723 (2) to 1.745 (2) Å. The $P1O_2(=O)_2$ and $P2O_2(=O)(OH)$ tetrahedra each share two O atoms with the $Al3O_4$ tetrahedra, and the short P—O distances to the terminal O atoms [P1—O1 = 1.507 (2) Å, P1—O2 = 1.523 (2) Å and P2—O5 = 1.481 (2) Å] suggest their double-bond character. The longest P—O distance [P2—O8 = 1.565 (2) Å] indicates the nature of the terminal hydroxy group. Hydrogen bonds (Table 2) are found between atoms N11, N13, N21 and N23 of the protonated 2-methylimidozole group and the three terminal atoms O1, O2 and O5. The O8—H8 hydroxy group also forms hydrogen bonds with terminal atom O2. The present study offers a limited discussion of the bond distances for C—C and C—N pairs; nevertheless, the configuration obtained is enough to confirm the geometry of the protonated 2-methylimidazole group, including the hydrogen bonding in the title compound.

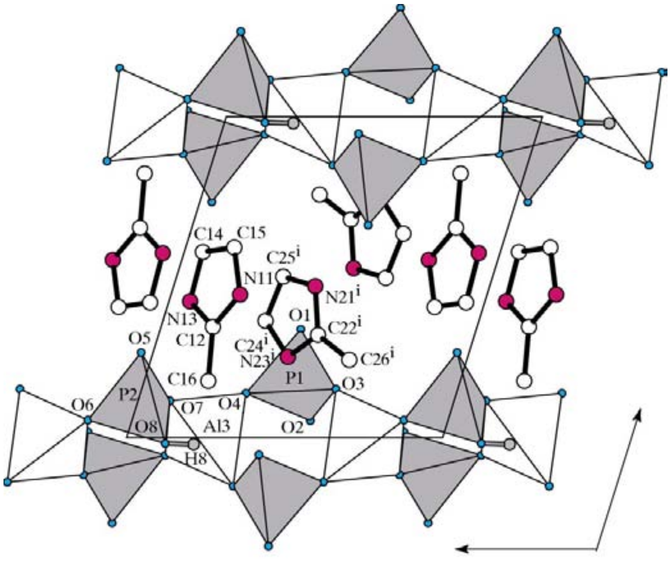


Figure 1
The crystal structure of (I). The AlO_4 and PO_4 tetrahedra are shown in white and gray, respectively. Aliphatic H atoms have been omitted for clarity. [Symmetry code: (i) $1 - x, 1 - y, 1 - z$.]

Experimental

Aluminium triisopropoxide (1.0 g) was dispersed in triethylene glycol (8 ml) with stirring, and then 2-methylimidazole (2.02 g) was added to form a slurry. H_3PO_4 (2.04 ml, 85 wt%) was added dropwise to the above reaction mixture. The resulting gel was sealed in a Teflon-lined stainless steel autoclave and heated at 453 K for 4 d. The resulting crystals of (I) were washed with distilled water and dried at 323 K. In order to compensate the negative charge of the infinite $[AlP_2O_7(OH)]^{2-}$ chains, the template agent is suggested to be protonated. The total weight loss of 48.9% from 353 to 1073 K, observed by thermogravimetric analysis, corresponds to the calculated weight fraction for $2C_4H_6N_2\cdot1.5H_2O$ (49.8%).

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Crystal data

$(C_4H_7N_2)_2[AlP_2O_7(OH)]$
$M_r = 384.16$
Triclinic, $P\bar{1}$
$a = 8.4789\ (17)$ Å
$b = 9.5233\ (11)$ Å
$c = 10.3607\ (17)$ Å
$\alpha = 108.811\ (9)°$
$\beta = 92.172\ (13)°$
$\gamma = 105.561\ (13)°$
$V = 755.8\ (2)$ Å^3
$Z = 2$
$D_x = 1.688$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.3–19.3°$
$\mu = 0.39$ mm^{-1}
$T = 298\ (2)$ K
Elongated prism, colorless
$0.20 \times 0.07 \times 0.04$ mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.929$, $T_{max} = 0.980$
4693 measured reflections
4417 independent reflections
3125 reflections with $I > 2\sigma(I)$

$R_{int} = 0.043$
$\theta_{max} = 30.0°$
$h = -11 \rightarrow 0$
$k = -12 \rightarrow 13$
$l = -14 \rightarrow 14$
3 standard reflections every 50 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.130$
$S = 1.03$
4417 reflections
209 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.3936P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

The hydroxy H atom in the infinite chain was positioned from a difference Fourier map (O—H = 0.91 Å) and the remaining aliphatic H atoms were placed in idealized positions (C—H = 0.93 and 0.96 Å). All H atoms were constrained to ride on their parent atoms. Isotropic displacement parameters for all H atoms were assumed to be equal and were refined [$U_{iso}(H) = 0.072\ (4)$ Å^2].

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *SHELXL*97 (Sheldrick, 1997); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97; molecular graphics: *ATOMS* (Dowty, 1997); software used to prepare material for publication: *SHELXL*97.

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Table 1
Selected interatomic distances (Å).

P1—O1	1.507 (2)	N11—C12	1.337 (4)
P1—O2	1.523 (2)	N11—C15	1.380 (4)
P1—O4	1.544 (2)	C12—N13	1.329 (4)
P1—O3	1.558 (2)	C12—C16	1.476 (4)
P2—O5	1.481 (2)	N13—C14	1.370 (4)
P2—O6	1.535 (2)	C14—C15	1.352 (4)
P2—O7	1.543 (2)	N21—C22	1.324 (4)
P2—O8	1.565 (2)	N21—C25	1.373 (4)
Al3—O4	1.723 (2)	C22—N23	1.334 (4)
Al3—O6^{ii}	1.732 (2)	C22—C26	1.475 (5)
Al3—O3^{iii}	1.736 (2)	N23—C24	1.381 (5)
Al3—O7	1.745 (2)	C24—C25	1.346 (5)

Symmetry codes: (ii) $2 - x, -y, 1 - z$; (iii) $1 - x, -y, 1 - z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O8—H8$\cdots$O2^{iii}	0.91	1.66	2.563 (3)	169
N11—H11$\cdots$O1^{iv}	0.86	1.86	2.711 (3)	169
N13—H13$\cdots$O5	0.86	1.81	2.650 (3)	166
N21—H21$\cdots$O1	0.86	1.88	2.728 (3)	169
N23—H23$\cdots$O2^{v}	0.86	1.89	2.740 (3)	168

Symmetry codes: (iii) $1 - x, -y, 1 - z$; (iv) $x, y, z - 1$; (v) $x, 1 + y, z$.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1178). Services for accessing these data are described at the back of the journal.

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m334 Junya Takashima *et al.* • $(C_4H_7N_2)_2[AlP_2O_7(OH)]$ *Acta Cryst.* (2004). C**60**, m333–m334