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A new amine phosphate templated by 1,4-diaminocyclohexane

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The title compound, cyclohexane-1,4-diammonium bis(dihydrogenphosphate), $C_6H_{16}N_2^{2+}\cdot 2H_2PO_4^-$, was synthesized using the hydrothermal method. It crystallizes with one anion and half a dication in the asymmetric unit; the latter lies on a centre of inversion. It has an inorganic network built up from $(H_2PO_4)^-$ tetrahedra linked together *via* strong hydrogen bonds to form infinite $(H_2PO_4)^-$ sheets lying parallel to the (100) plane. The diprotonated amine molecules link the layers together, assembling a three-dimensional hydrogen-bond network.

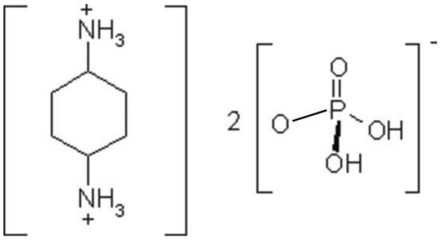
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.104
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Organic cation phosphates have been investigated with respect to their crystal structure, various properties and potential applications (Soumhi *et al.*, 2001; Oliver *et al.*, 1995). In our laboratory, we have been studying the M(Zn,Co,Zr,Ti)–HF–H_3PO_4–amine–solvent system, and some amine phosphates and metallic amine phosphates have been characterized (Dakhlaoui *et al.*, 2005*a*,*b*; Dakhlaoui *et al.*, 2005). In the title compound, (I), the asymmetric unit contains one $(H_2PO_4)^-$ phosphate group and a half of the diprotonated amine molecule (Fig. 1). The cation lies on a centre of inversion.



(I)

The geometrical features of the phosphate group are quite regular (Table 1); the average bond length is 1.539 (1) Å for P—O, and the longest bond lengths are P1—O1 = 1.573 (1) Å and P1—O2 = 1.572 (2) Å, corresponding to the protonated O atoms; the average O—P—O bond angle is 109.41 (8)°. The structure of (I) consists of infinite macroanionic $(H_2PO_4)^-$ sheets separated by the organic cations (Fig. 2). Cohesion of this structure is ensured by hydrogen bonds (Table 2) involving the diprotonated amine molecules and the phosphate groups. Within the inorganic layer the (H_2PO_4) phosphate groups are linked together *via* a strong hydrogen bond [O2···O4^{i} = 2.585 (2) Å] to form dimers [symmetry code: (i) $2 - x$, $-y$, $1 - z$]. Every dimer is linked with four neighbours *via* the [O1···O3^{ii}= 2.545 (2) Å; symmetry code: (ii) $2 - x$,

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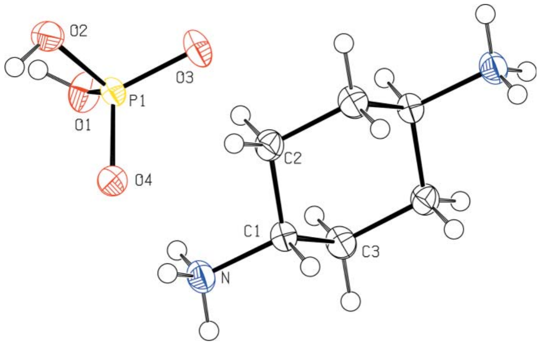

Figure 1
A plot of (I), with displacement ellipsoids shown at the 50% probability level. The non-labelled atoms within the cation are related to the labelled atoms through a inversion centre [symmetry code $(1 - x, 1 - y\ 1 - z)$]. H atoms are shown as spheres of arbitrary radius.

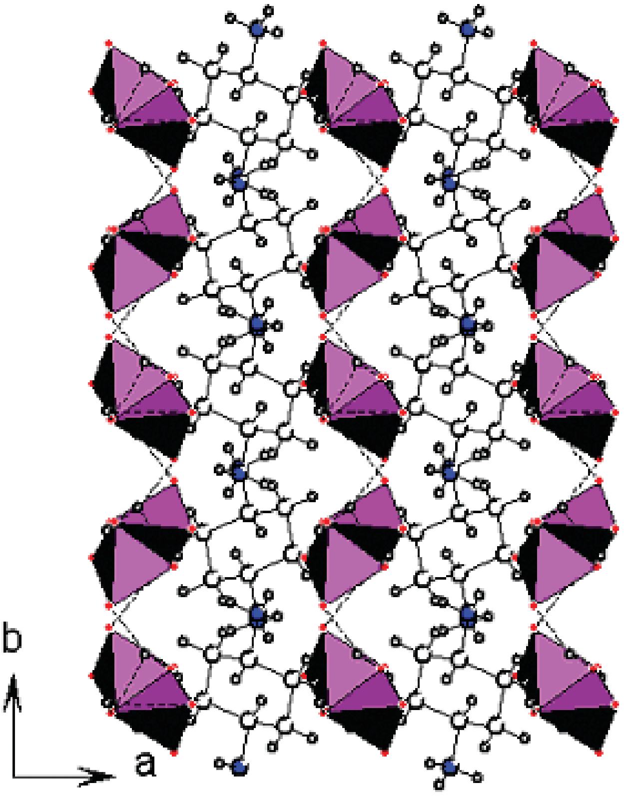

Figure 2
Projection of (I) along the *c* axis, showing the hydrogen-bonding interactions between organic cations and the $(H_2PO_4)^-$ anions.

$y - \frac{1}{2}, \frac{3}{2} - z]$ hydrogen bond to form layers parallel to the (100) plane (Fig. 3). Rings of six phosphate groups assemble in these inorganic layers.

Experimental

The title compound was prepared by hydrothermal synthesis under autogenous pressure. The starting mixture of phosphoric acid (0.6 ml), 1,4-diaminocyclohexane (0.56 g) and water (8 ml) in the molar ratio (2:1:80) was placed in a Teflon-lined stainless steel autoclave and then heated at 443 K for five days. The transparent plate-shaped crystals obtained were filtered off, washed with ethanol and dried at room temperature.

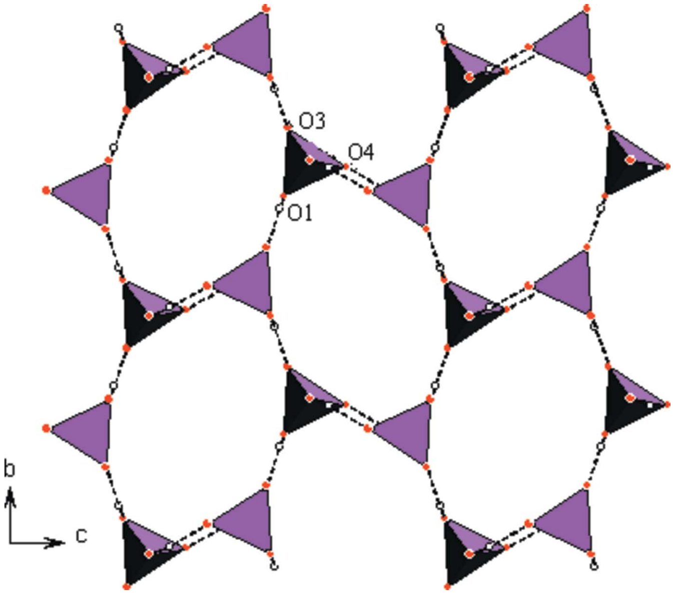

Figure 3
Projection of (I) along the *a* axis, showing the infinite $(H_2PO_4)^-$ sheets.

Crystal data

$C_6H_{16}N_2^{2+}\cdot 2H_2O_4P^-$
$M_r = 310.18$
Monoclinic, $P2_1/c$
$a = 6.298\ (3)$ Å
$b = 8.606\ (2)$ Å
$c = 11.824\ (7)$ Å
$\beta = 101.84\ (2)^\circ$
$V = 627.2\ (5)$ Å^3
$Z = 2$
$D_x = 1.642$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.38$ mm^{-1}
$T = 293\ (2)$ K
Plate, colourless
$0.24 \times 0.13 \times 0.05$ mm

Data collection

Siemens AED2 diffractometer
$2\theta/\omega$ scans
Absorption correction: analytical (*SHELXS76*; Sheldrick, 1976)
$T_{min} = 0.992$, $T_{max} = 0.996$
1775 measured reflections
1771 independent reflections
1480 reflections with $I > 2\sigma(I)$
$R_{int} = 0.003$
$\theta_{max} = 30.0^\circ$
3 standard reflections every 120 reflections intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.104$
$S = 1.09$
1668 reflections
122 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.3355P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.011$
$\Delta\rho_{max} = 0.63$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—O4	1.5022 (15)	P1—O3	1.5089 (13)
O4—P1—O3	114.95 (8)	O4—P1—O1	108.56 (9)
O4—P1—O2	110.57 (8)	O3—P1—O1	107.36 (8)
O3—P1—O2	108.33 (7)	O2—P1—O1	106.72 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0*A*···O4	0.87 (3)	1.93 (3)	2.797 (2)	175 (3)
O2—H2···O4^{i}	0.78 (3)	1.81 (3)	2.585 (2)	174 (3)
O1—H1···O3^{ii}	0.88 (4)	1.67 (4)	2.545 (2)	176 (4)
N—H0*C*···O3^{iii}	0.94 (3)	1.88 (3)	2.811 (2)	171 (2)
N—H0*B*···O1^{iv}	0.88 (3)	2.10 (3)	2.937 (2)	160 (2)

Symmetry codes: (i) $-x+2, -y, -z+1$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $-x+1, -y, -z+1$.

All H atoms were located in difference maps and their individual positional and isotropic displacement parameters were refined without constraints; C—H = 0.88 (2)–0.98 (2) Å.

Data collection: *STADI4* (Stoe & Cie, 1998); cell refinement: *STADI4*; data reduction: *X-RED* (Stoe & Cie, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Brandenburg, K. (2001). *Sci. Comput. World*, **57**, 28–30.

Dakhlaoui, A., Leblanc, M. & Smiri, L. S. (2005). *Anal. Sci.* **21**, 1–2.

Dakhlaoui, A., Maisonneuve, V., Leblanc, M. & Smiri, L. S. (2005*a*). *J. Solid State Chem.* **178**, 1880–1887.

Dakhlaoui, A., Maisonneuve, V., Leblanc, M. & Smiri, L. S. (2005*b*). *Phosphorus Sulfur Silicon*, **180**, 2573–2586.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Oliver, S., Kuperman, A., Coombs, N., Lough, A. & Ozin, G. A. (1995). *Nature*, **378**, 47–50.

Sheldrick, G. M. (1976). *SHELXS76*. Cambridge University Press, England.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Soumhi, E. H., Saadoune, I. & Driss, A. (2001). *J. Solid State Chem.* pp. 364–369.

Stoe & Cie (1998). *STADI4* (Version 1.07) and *X-RED* (Version 1.10). Stoe & Cie, Darmstadt, Germany.