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N-(2-Hydroxyethyl)ethylenediammonium hydrogenphosphate monohydrate

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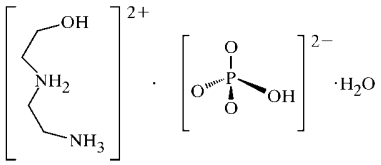
The title compound, $C_4H_{14}N_2O^{2+}\cdot HPO_4^{2-}\cdot H_2O$, contains alternating interleaved layers of hydrogenphosphate and *N*-(2-hydroxyethyl)ethylenediammonium moieties. The water molecules are associated with channel-like voids in the structure and a network of hydrogen bonds stabilizes the crystal packing.

Comment

During the synthesis of metal phosphates templated by organic amines (Cheetham *et al.*, 1999), amine phosphates may occur as unexpected side products and may also act as intermediates in the formation of open-framework structures (Oliver *et al.*, 1998; Neeraj *et al.*, 1999). We describe here the structure of one such amine phosphate, namely the title compound, (I).



(I)

The structure of (I) (Fig. 1) consists of a molecular network. Both amino groups of the *N*-(2-hydroxyethyl)ethylenediammonium moiety are protonated, resulting in a divalent species. The *N*-(2-hydroxyethyl)ethylenediammonium cation exhibits a *gauche* conformation and bond distances within the cation are comparable with those in the neutral molecule coordinated to Cu^{II} and Cd^{II} ions (Yilmaz *et al.*, 2002). One of the phosphate P—O vertices is protonated and shows the expected lengthening relative to the other P—O bonds (Oliver *et al.*, 1998).

The crystal packing in (I) is shown in Fig. 2. The structure contains alternating interleaved layers of anions and cations, with the layers propagating in the (101) plane. The water molecules occupy channel-like voids propagating along [001]. All the H atoms of the ammonium groups form N—H···O hydrogen bonds to neighbouring phosphate O atoms, while the hydroxyl group of the organic species forms hydrogen bonds to the O atoms of both the phosphate ions and water molecules. Adjacent phosphate anions are also linked by P—OH···O hydrogen bonds in the [001] direction.

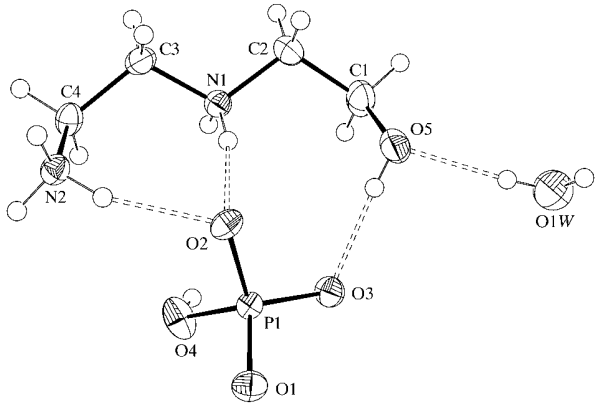


Figure 1
A view of the molecule of (I) with 50% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii and hydrogen bonds are indicated by dashed lines.

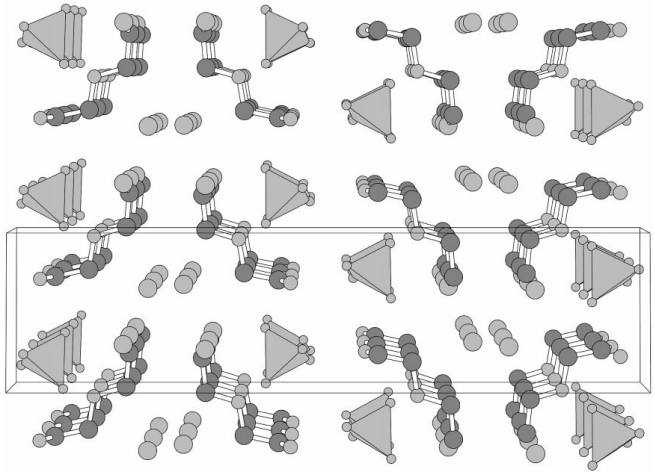


Figure 2
A projection of the structure of (I) along [001].

Experimental

H_3PO_4 (0.814 ml, 12 mmol) (aqueous, 85% *w*/*w*) was added dropwise to an aqueous solution of ethylene glycol (20%, 20 ml) with *N*-(2-hydroxyethyl)ethylenediamine (1.012 ml, 10 mmol) and the resulting solution was stirred for 2 h at 323 K. The mixture was then left to crystallize at room temperature. Colourless chunk-type crystals of (I) were formed, and these were washed with a small amount of water and acetone, and dried in air.

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.5144 (8)	N1—C2	1.4875 (15)
P1—O2	1.5402 (7)	N1—C3	1.4933 (13)
P1—O3	1.5247 (8)	N2—C4	1.4857 (13)
P1—O4	1.5862 (8)	C1—C2	1.5178 (17)
O5—C1	1.4179 (16)	C3—C4	1.5162 (16)
C3—N1—C2—C1	−179.52 (10)	C2—N1—C3—C4	174.46 (9)
O5—C1—C2—N1	−71.91 (14)	N1—C3—C4—N2	82.13 (11)

Crystal data

$C_4H_{14}N_2O^{2+}\cdot HPO_4^{2-}\cdot H_2O$
M_r = 220.17
Monoclinic, $P2_1/c$
a = 7.0863 (3) Å
b = 28.4885 (11) Å
c = 4.8336 (2) Å
β = 94.874 (1)°
V = 972.27 (7) Å^3
Z = 4
D_x = 1.504 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5486 reflections
θ = 2.9–32.5°
μ = 0.29 mm^{-1}
T = 293 (2) K
Chunk, colourless
0.46 × 0.30 × 0.19 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.902, T_{max} = 0.947
9939 measured reflections
3520 independent reflections
3051 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 32.5°
$h = -10 \rightarrow 10$
$k = -43 \rightarrow 34$
$l = -6 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.091
S = 1.10
3520 reflections
162 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.0694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Water, hydroxyl and amine H atoms were found in difference maps and refined freely. H atoms bonded to C atoms were placed in calculated positions (C—H = 0.97 Å) and treated as riding.

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O1^{i}	0.80 (3)	1.82 (3)	2.5947 (11)	163 (3)
O5—H5···O3	0.77 (2)	1.86 (2)	2.6228 (13)	174 (2)
O1W—H1A···O5	0.83 (3)	1.95 (3)	2.7731 (15)	172 (2)
O1W—H1B···O1W^{ii}	0.83 (2)	2.00 (2)	2.8227 (14)	170 (2)
N1—H111···O2	0.881 (16)	1.898 (17)	2.7674 (12)	169 (2)
N1—H112···O2^{i}	0.832 (18)	1.905 (18)	2.7046 (12)	161 (2)
N2—H211···O1^{iii}	0.920 (18)	1.846 (18)	2.7643 (13)	175 (2)
N2—H212···O3^{iv}	0.876 (18)	1.875 (18)	2.7412 (12)	169 (2)
N2—H213···O2	0.897 (16)	1.994 (17)	2.8584 (12)	162 (2)

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1110). Services for accessing these data are described at the back of the journal.

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supporting information

Acta Cryst. (2002). C**58**, o407–o408 [doi:10.1107/S0108270102009629]

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N-(2-hydroxyethyl)ethylenediammonium hydrogenphosphate monohydrate

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$c = 4.8336\ (2)$ Å
$\beta = 94.874\ (1)°$
$V = 972.27\ (7)$ Å^3
$Z = 4$
$F(000) = 472$
$D_x = 1.504$ Mg m^{-3}
Melting point: not measured K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5486 reflections
$\theta = 2.9–32.5°$
$\mu = 0.29$ mm^{-1}
$T = 293$ K
Chunk, colourless
0.46 × 0.30 × 0.19 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
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9939 measured reflections
3520 independent reflections
3051 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 32.5°$, $\theta_{min} = 2.9°$
$h = -10\rightarrow10$
$k = -43\rightarrow34$
$l = -6\rightarrow7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.091$
$S = 1.10$
3520 reflections
162 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: none
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.0694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
P1	0.24386 (3)	0.066713 (9)	0.53198 (5)	0.01795 (7)
O1	0.29299 (11)	0.03924 (3)	0.79651 (15)	0.02687 (16)
O2	0.06383 (10)	0.09652 (3)	0.54860 (15)	0.02467 (15)
O3	0.40488 (10)	0.09761 (3)	0.44708 (16)	0.02603 (16)
O4	0.19514 (14)	0.02854 (3)	0.29789 (17)	0.0344 (2)
H4	0.227 (4)	0.0375 (10)	0.152 (5)	0.090 (8)*
O5	0.32252 (15)	0.18549 (3)	0.3287 (2)	0.0384 (2)
H5	0.346 (3)	0.1595 (7)	0.351 (4)	0.050 (5)*
O1W	0.67791 (16)	0.22440 (5)	0.4570 (2)	0.0462 (3)
H1A	0.572 (4)	0.2120 (9)	0.435 (5)	0.074 (7)*
H1B	0.667 (3)	0.2415 (8)	0.595 (5)	0.069 (7)*
N1	−0.01794 (12)	0.13671 (3)	0.03106 (18)	0.02173 (16)
H111	0.022 (2)	0.1264 (6)	0.198 (3)	0.036 (4)*
H112	0.027 (2)	0.1211 (6)	−0.094 (4)	0.038 (4)*
N2	−0.27371 (12)	0.05690 (4)	0.27835 (19)	0.02369 (17)
H211	−0.278 (2)	0.0247 (6)	0.264 (3)	0.038 (4)*
H212	−0.371 (2)	0.0689 (6)	0.354 (4)	0.040 (4)*
H213	−0.167 (2)	0.0633 (6)	0.385 (3)	0.035 (4)*
C1	0.25351 (17)	0.19171 (5)	0.0471 (3)	0.0361 (3)
H11	0.3124	0.1687	−0.0661	0.053 (5)*
H12	0.2891	0.2227	−0.0138	0.045 (5)*
C2	0.03989 (17)	0.18656 (4)	0.0031 (3)	0.0328 (2)
H21	−0.0190	0.2055	0.1386	0.049 (5)*
H22	−0.0038	0.1979	−0.1804	0.050 (5)*
C3	−0.22630 (14)	0.12816 (4)	−0.0113 (2)	0.0261 (2)
H31	−0.2751	0.1413	−0.1882	0.040 (4)*
H32	−0.2887	0.1438	0.1339	0.052 (5)*
C4	−0.27073 (14)	0.07612 (4)	−0.0070 (2)	0.0252 (2)
H4A	−0.3932	0.0708	−0.1076	0.040 (4)*
H4B	−0.1766	0.0593	−0.1026	0.038 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01798 (11)	0.02074 (13)	0.01533 (11)	−0.00010 (8)	0.00256 (7)	−0.00059 (8)
O1	0.0317 (4)	0.0298 (4)	0.0188 (3)	0.0015 (3)	0.0009 (3)	0.0046 (3)

O2	0.0181 (3)	0.0311 (4)	0.0249 (3)	0.0035 (3)	0.0023 (2)	−0.0001 (3)
O3	0.0202 (3)	0.0264 (4)	0.0324 (4)	−0.0010 (3)	0.0074 (3)	0.0029 (3)
O4	0.0533 (5)	0.0300 (4)	0.0213 (4)	−0.0118 (4)	0.0108 (3)	−0.0072 (3)
O5	0.0470 (5)	0.0253 (4)	0.0400 (5)	−0.0064 (4)	−0.0119 (4)	0.0015 (3)
O1W	0.0382 (5)	0.0562 (7)	0.0438 (6)	−0.0040 (5)	0.0006 (4)	−0.0047 (5)
N1	0.0220 (4)	0.0214 (4)	0.0215 (4)	0.0001 (3)	0.0009 (3)	−0.0001 (3)
N2	0.0200 (4)	0.0274 (5)	0.0238 (4)	−0.0008 (3)	0.0027 (3)	0.0011 (3)
C1	0.0363 (6)	0.0342 (6)	0.0368 (6)	−0.0120 (5)	−0.0026 (5)	0.0073 (5)
C2	0.0357 (6)	0.0215 (5)	0.0397 (6)	−0.0021 (4)	−0.0048 (5)	0.0045 (4)
C3	0.0212 (4)	0.0288 (5)	0.0279 (5)	0.0027 (4)	−0.0001 (3)	0.0035 (4)
C4	0.0247 (4)	0.0302 (5)	0.0202 (4)	−0.0049 (4)	−0.0003 (3)	−0.0009 (3)

Geometric parameters (Å, º)

P1—O1	1.5144 (8)	N2—H211	0.920 (18)
P1—O2	1.5402 (7)	N2—H212	0.876 (18)
P1—O3	1.5247 (8)	N2—H213	0.897 (16)
P1—O4	1.5862 (8)	C1—C2	1.5178 (17)
O4—H4	0.80 (3)	C1—H11	0.9700
O5—C1	1.4179 (16)	C1—H12	0.9700
O5—H5	0.77 (2)	C2—H21	0.9700
O1W—H1A	0.83 (3)	C2—H22	0.9700
O1W—H1B	0.83 (2)	C3—C4	1.5162 (16)
N1—C2	1.4875 (15)	C3—H31	0.9700
N1—C3	1.4933 (13)	C3—H32	0.9700
N1—H111	0.881 (16)	C4—H4A	0.9700
N1—H112	0.832 (18)	C4—H4B	0.9700
N2—C4	1.4857 (13)		

O1—P1—O3	113.71 (4)	C2—C1—H11	109.1
O1—P1—O2	111.90 (4)	O5—C1—H12	109.1
O3—P1—O2	109.75 (5)	C2—C1—H12	109.1
O1—P1—O4	105.57 (5)	H11—C1—H12	107.9
O3—P1—O4	109.05 (5)	N1—C2—C1	111.03 (10)
O2—P1—O4	106.51 (5)	N1—C2—H21	109.4
P1—O4—H4	110 (2)	C1—C2—H21	109.4
C1—O5—H5	108.1 (15)	N1—C2—H22	109.4
H1A—O1W—H1B	102 (2)	C1—C2—H22	109.4
C2—N1—C3	114.85 (9)	H21—C2—H22	108.0
C2—N1—H111	109.4 (10)	N1—C3—C4	111.21 (9)
C3—N1—H111	107.9 (10)	N1—C3—H31	109.4
C2—N1—H112	108.6 (12)	C4—C3—H31	109.4
C3—N1—H112	104.2 (11)	N1—C3—H32	109.4
H111—N1—H112	111.7 (15)	C4—C3—H32	109.4
C4—N2—H211	107.4 (10)	H31—C3—H32	108.0
C4—N2—H212	108.4 (11)	N2—C4—C3	113.12 (9)
H211—N2—H212	113.6 (14)	N2—C4—H4A	109.0
C4—N2—H213	112.2 (10)	C3—C4—H4A	109.0

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