

# *N*-(2-Hydroxyethyl)ethylenediammonium hydrogenphosphate monohydrate

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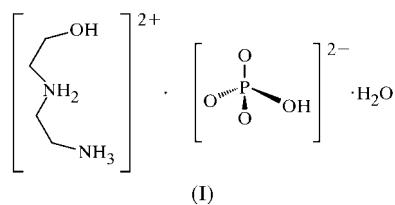
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The title compound,  $C_4H_{14}N_2O^{2+} \cdot HPO_4^{2-} \cdot H_2O$ , contains alternating interleaved layers of hydrogenphosphate and *N*-(2-hydroxyethyl)ethylenediammonium moieties. The water molecules are associated with channel-like voids in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

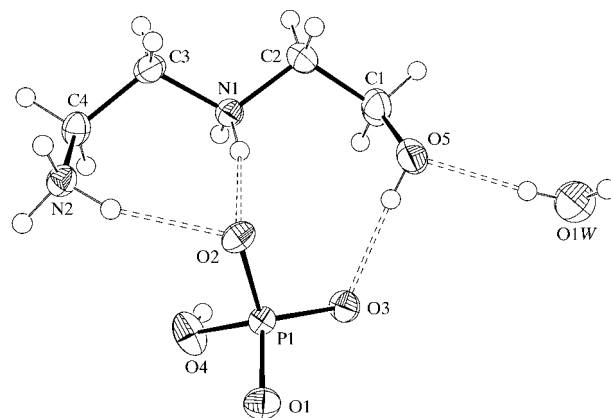
## Comment

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).

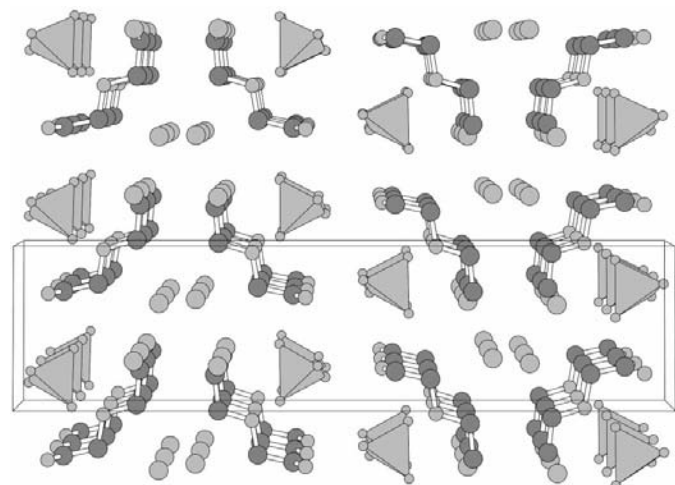


The structure of (I) (Fig. 1) consists of a molecular network. Both amino groups of the *N*-(2-hydroxyethyl)ethylenediammonium moiety are protonated, resulting in a divalent species. The *N*-(2-hydroxyethyl)ethylenediammonium cation exhibits a *gauche* conformation and bond distances within the cation 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,

**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].

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.

## Experimental

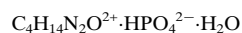
$H_3PO_4$  (0.814 ml, 12 mmol) (aqueous, 85% w/w) was added dropwise to an aqueous solution of ethylene glycol (20%, 20 ml) with *N*-(2-hydroxyethyl)ethylenediamine (1.012 ml, 10 mmol) and the resulting solution was stirred for 2 h at 323 K. The mixture was then left to crystallize at room temperature. 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


 $M_r = 220.17$ 

 Monoclinic,  $P2_1/c$ 
 $a = 7.0863$  (3) Å

 $b = 28.4885$  (11) Å

 $c = 4.8336$  (2) Å

 $\beta = 94.874$  (1)°

 $V = 972.27$  (7) Å<sup>3</sup>
 $Z = 4$ 
 $D_x = 1.504$  Mg m<sup>−3</sup>

 Mo  $K\alpha$  radiation

Cell parameters from 5486

reflections

 $\theta = 2.9$ – $32.5^\circ$ 
 $\mu = 0.29$  mm<sup>−1</sup>
 $T = 293$  (2) K

Chunk, colourless

 $0.46 \times 0.30 \times 0.19$  mm

### Data collection

Bruker SMART 1000 CCD area-detector diffractometer

 $\omega$  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  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.016$ 
 $\theta_{\text{max}} = 32.5^\circ$ 
 $h = -10 \rightarrow 10$ 
 $k = -43 \rightarrow 34$ 
 $l = -6 \rightarrow 7$ 

### 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  $P = (F_o^2 + 2F_c^2)/3$ 
 $(\Delta/\sigma)_{\text{max}} < 0.001$ 
 $\Delta\rho_{\text{max}} = 0.34$  e Å<sup>−3</sup>
 $\Delta\rho_{\text{min}} = -0.25$  e Å<sup>−3</sup>

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, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O4—H4 $\cdots$ O1 <sup>i</sup>	0.80 (3)	1.82 (3)	2.5947 (11)	163 (3)
O5—H5 $\cdots$ O3	0.77 (2)	1.86 (2)	2.6228 (13)	174 (2)
O1W—H1A $\cdots$ O5	0.83 (3)	1.95 (3)	2.7731 (15)	172 (2)
O1W—H1B $\cdots$ O1W <sup>ii</sup>	0.83 (2)	2.00 (2)	2.8227 (14)	170 (2)
N1—H111 $\cdots$ O2	0.881 (16)	1.898 (17)	2.7674 (12)	169 (2)
N1—H112 $\cdots$ O2 <sup>i</sup>	0.832 (18)	1.905 (18)	2.7046 (12)	161 (2)
N2—H211 $\cdots$ O1 <sup>iii</sup>	0.920 (18)	1.846 (18)	2.7643 (13)	175 (2)
N2—H212 $\cdots$ O3 <sup>iv</sup>	0.876 (18)	1.875 (18)	2.7412 (12)	169 (2)
N2—H213 $\cdots$ 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: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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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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