

Table 3. Main interatomic distances (Å) and bond angles (°) in HPO₄(NH₃)₂(CH₂)₂

PO ₄ tetrahedron				
P	O(1)	O(2)	O(3)	O(4)
O(1)	1.590 (2)	2.459 (3)	2.540 (3)	2.523 (3)
O(2)	104.3 (1)	1.525 (2)	2.532 (3)	2.535 (3)
O(3)	108.8 (1)	111.8 (1)	1.533 (2)	2.512 (3)
O(4)	108.4 (1)	112.7 (1)	110.7 (1)	1.521 (2)

$$\overline{P-O} = 1.542 (2)$$

NH₃-(CH₂)₂-NH₃ group

N(1)-C(1)	1.469 (4)	N(1)-C(1)-C(2)	112.1 (3)
C(1)-C(2)	1.507 (4)	C(1)-C(2)-N(2)	109.6 (2)
C(2)-N(2)	1.486 (4)		

Hydrogen bonds

	(O,N)-H	H...O	(O,N)-O	∠(O,N)-H...O
O(1)-H...O(4)	0.71 (6)	1.90 (6)	2.578 (3)	160 (7)
N(1)-H(1N1)...O(4)	0.82 (4)	1.93 (4)	2.747 (4)	173 (4)
N(1)-H(2N1)...O(2)	0.88 (5)	1.85 (5)	2.719 (4)	167 (5)
N(1)-H(3N1)...O(3)	0.91 (4)	2.00 (5)	2.906 (4)	176 (4)
N(2)-H(1N2)...O(3)	0.90 (4)	1.91 (5)	2.810 (4)	178 (4)
N(2)-H(2N2)...O(2)	1.08 (5)	1.65 (5)	2.720 (4)	176 (4)
N(2)-H(3N2)...O(3)	0.94 (5)	1.90 (5)	2.836 (4)	172 (4)

groups. In both structures one observes a layer arrangement: planes of HXO₄ tetrahedra alternate with planes of (CH₂)₂(NH₃)₂²⁺ groups, as depicted in Figs. 1 and 2.

Another common feature for these two arrangements is the internal repartition of the HXO₄²⁻ tetrahedra in their planes; they are associated in pairs forming H₂X₂O₈ clusters with rather short X-X distances (P-P = 4.847, As-As = 4.994 Å). The two HXO₄ groups in such a cluster are linked by a double hydrogen bridge.

In the case of the phosphorus compound the (NH₃)₂(CH₂)₂ entities are strongly pseudocentrosymmetric while they are centrosymmetric in the arsenic compound. This implies the existence of two crystallographically independent (NH₃)₂(CH₂)₂ units.

Table 4. Main interatomic distances (Å) and bond angles (°) in HAsO₄(NH₃)₂(CH₂)₂

AsO ₄ tetrahedron				
As	O(1)	O(2)	O(3)	O(4)
O(1)	1.678 (3)	2.647 (4)	2.775 (4)	2.751 (4)
O(2)	101.7 (2)	1.734 (3)	2.793 (5)	2.763 (5)
O(3)	111.9 (2)	110.2 (2)	1.672 (3)	2.763 (5)
O(4)	111.1 (2)	109.1 (2)	112.2 (2)	1.657 (3)

$$\overline{As-O} = 1.685 (3)$$

NH₃-(CH₂)₂-NH₃ groups

C(1)-C(1)	1.496 (8)	C(2)-C(2)	1.516 (9)
C(1)-N(1)	1.479 (5)	C(2)-N(2)	1.482 (6)
C(1)-C(1)-N(1)	111.3 (4)	C(2)-C(2)-N(2)	110.6 (5)

Hydrogen bonds

	(O,N)-H	H...O	(O,N)-O	∠(O,N)-H...O
O(2)-H...O(3)	0.64 (6)	2.03 (6)	2.634 (5)	158 (8)
N(1)-H(1N1)...O(3)	0.64 (6)	2.14 (6)	2.744 (5)	157 (7)
N(1)-H(2N1)...O(1)	0.82 (6)	1.99 (6)	2.805 (5)	170 (5)
N(1)-H(3N1)...O(1)	0.88 (5)	1.92 (5)	2.788 (5)	168 (5)
N(2)-H(1N2)...O(4)	0.76 (8)	1.98 (8)	2.718 (5)	162 (8)
N(2)-H(2N2)...O(1)	0.77 (5)	2.14 (6)	2.820 (5)	148 (5)
N(2)-H(3N2)...O(4)	0.88 (6)	1.81 (6)	2.686 (5)	170 (5)

Tables 1 and 2* report the final atomic coordinates, while Tables 3 and 4 give the main interatomic distances, bond angles and details of the hydrogen-bond scheme.

* Lists of structure factors, anisotropic thermal parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44090 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1987). **C43**, 1896-1898

Structure of Disodium Ethylenediammonium Bis[monohydrogentetraoxophosphate(V)] Hexahydrate

BY M. T. AVERBUCH-POUCHOT, A. DURIF AND J. C. GUITEL

Laboratoire de Cristallographie, Centre National de la Recherche Scientifique, Laboratoire associé à l'USTMG, 166X, 38042 Grenoble CEDEX, France

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Abstract. C₂H₁₀N₂²⁺·2Na⁺·2HPO₄²⁻·6H₂O, *M_r* = 408.15, monoclinic, *P*2₁/*c*, *a* = 11.699 (9), *b* = 10.164 (9), *c* = 6.835 (4) Å, β = 105.00 (5)°, *V* = 785 (2) Å³, *Z* = 2, *D_x* = 1.727 Mg m⁻³, λ(Mo Kα) = 0.7107 Å, μ = 0.415 mm⁻¹, *F*(000) = 428, *T* = 295 K, final *R* = 0.022 for 1823 independent reflexions. HPO₄²⁻ and (CH₂)₂(NH₃)₂²⁺ groups alternate in planes perpendicular to the *a* axis. In these planes, HPO₄²⁻

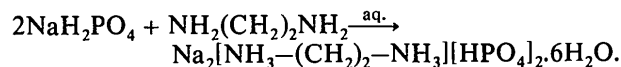
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groups link together to form $(P_2O_8H_2)^{4-}$ clusters with an internal P—P distance of 4.320 Å. Na atoms and water molecules are located between these planes, separated by a distance of 11.7 Å. Sodium coordination is a distorted octahedron made of five water molecules and one O atom. The potassium salt is isotypic with the title compound.

Introduction. During an investigation of the interaction of ethylenediamine with various acidic monophosphates, we generally observed the formation of $[(CH_2)_2(NH_3)_2]^{2+} \cdot [HPO_4]^{2-}$, a very stable compound (Averbuch-Pouchot & Durif, 1987). In some cases, the formation of more complex compounds was observed. The title compound is an example of such a compound, characterized during the investigation of the ethylenediamine— NaH_2PO_4 system.

Experimental. Crystals of the title compound are prepared by slow evaporation at room temperature of an aqueous solution of the two components in a proper ratio. Schematically the reaction can be written:



If an excess of ethylenediamine is slowly added to the solution precipitation occurs immediately. The polycrystalline precipitate so obtained is then purified by recrystallization in water. The process for the preparation of the isotypic potassium salt is identical.

Density not measured. Monoclinic stout prism: $0.32 \times 0.32 \times 0.24$ mm. Nonius CAD-4 diffractometer. Graphite monochromator. Systematic absences: $0k0, k = 2n; h0l, l = 2n$. 24 reflexions ($11.5 < \theta < 16.20^\circ$) for refining unit-cell dimensions. ω scan. 2586 reflexions measured ($3 < \theta < 30^\circ$), $\pm hkl, h_{max} = 15, k_{max} = 14, l_{max} = 9$. Scan width 1.20° , scan speed $0.02-0.04^\circ s^{-1}$, background measuring time 15–30 s. Two orientation (441 and $4\bar{4}\bar{1}$) and two intensity (620 and $44\bar{1}$) reference reflexions: no significant variations. Lorentz and polarization corrections, no absorption correction.

Crystal structure solved by direct methods with *MULTAN* (Main, Woolfson & Germain, 1971); anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. Unit weights. Final refinement with 1823 reflexions ($F > 2\sigma_F$). Final $R = 0.022$ ($wR = 0.024$). Extinction coefficient refined: 6.43×10^{-6} (Stout & Jensen, 1968). $S = 0.416$. Max. $\Delta/\sigma = 0.29$ (extinction coefficient). Max. peak height in final difference Fourier synthesis $0.260 e \text{ \AA}^{-3}$. The final R value = 0.033 for the complete set of independent reflexions (2268 reflexions). H atoms located by difference Fourier map and refined isotropically. Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974).

Enraf–Nonius (1977) *SDP* employed for all calculations. Computer used: VAX 780.

Discussion. Table 1* reports the final atomic coordinates. Fig. 1 is a projection of the atomic arrangement along the c axis.

* Lists of structure factors, anisotropic thermal parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44093 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic coordinates and B_{eq} or B_{iso} for $Na_2[(CH_2)_2(NH_3)_2][HPO_4]_2 \cdot 6H_2O$

	$B_{eq} = \frac{4}{3} \sum_i \sum_j a_i \cdot a_j \beta_{ij}$			$B_{eq}/B_{iso} (\text{\AA}^2)$
	x	y	z	
Na	0.59327 (5)	0.13069 (5)	0.10285 (8)	2.23 (1)
P	0.19048 (3)	0.01690 (3)	0.09303 (5)	1.486 (5)
O(1)	0.27422 (8)	0.0182 (1)	0.3038 (1)	2.14 (2)
O(2)	0.12632 (8)	0.87853 (9)	0.0688 (2)	2.54 (2)
O(3)	0.09597 (8)	0.12196 (9)	0.0723 (2)	2.42 (2)
O(4)	0.25655 (8)	0.4721 (1)	0.4311 (1)	2.11 (2)
O(W1)	0.49542 (8)	0.9641 (1)	0.2335 (2)	2.51 (2)
O(W2)	0.31084 (8)	0.80010 (9)	0.5560 (2)	2.30 (2)
O(W3)	0.6006 (1)	0.7512 (1)	0.4911 (2)	3.01 (2)
N	0.12995 (9)	0.3850 (1)	0.0602 (2)	1.79 (2)
C	0.0048 (1)	0.4260 (1)	0.0063 (2)	1.88 (2)
H	0.054 (1)	0.883 (2)	0.019 (3)	3.6 (4)
H(1W1)	0.565 (2)	0.022 (2)	0.728 (3)	4.1 (4)
H(2W1)	0.469 (2)	0.412 (2)	0.195 (3)	4.7 (5)
H(1W2)	0.752 (1)	0.236 (2)	0.462 (2)	2.9 (4)
H(2W2)	0.707 (2)	0.363 (2)	0.025 (3)	3.8 (4)
H(1W3)	0.648 (2)	0.810 (2)	0.012 (3)	3.7 (4)
H(2W3)	0.370 (2)	0.301 (2)	0.919 (3)	5.7 (5)
H(1N)	0.831 (1)	0.916 (2)	0.317 (3)	3.0 (4)
H(2N)	0.829 (1)	0.914 (2)	0.528 (3)	3.2 (4)
H(3N)	0.130 (1)	0.302 (2)	0.066 (2)	3.3 (4)
H(1C)	0.033 (1)	0.616 (2)	0.122 (2)	2.2 (3)
H(2C)	0.028 (1)	0.609 (2)	0.893 (2)	2.6 (3)

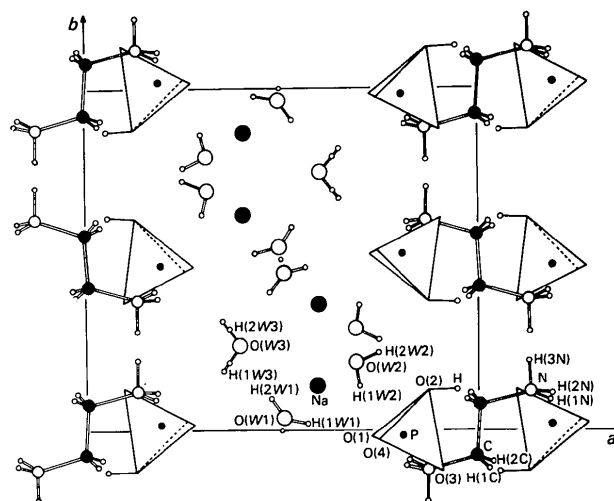


Fig. 1. Projection along the c axis of the atomic arrangement of $Na_2[(CH_2)_2(NH_3)_2][HPO_4]_2 \cdot 6H_2O$. Two O(W3) water molecules are superimposed in projection.

Table 2. *Main interatomic distances (Å), bond angles (°) and hydrogen-bond scheme (Å, °) in $Na_2[(CH_2)_2(NH_3)_2][HPO_4]_2 \cdot 6H_2O$*

PO ₄ tetrahedron				
P	O(1)	O(2)	O(3)	O(4)
O(1)	1.518 (1)	2.480 (2)	2.501 (2)	2.504 (2)
O(2)	106.21 (9)	1.583 (1)	2.501 (2)	2.501 (2)
O(3)	110.96 (9)	107.53 (8)	1.517 (1)	2.513 (2)
O(4)	111.61 (8)	107.95 (9)	112.25 (9)	1.509 (1)
P-P		4.320 (2)		
NaO ₆ octahedron				
Na-O(4)	2.439 (2)	Na-O(W2)	2.416 (2)	
Na-O(W1)	2.346 (2)	Na-O(W2)	2.456 (2)	
Na-O(W1)	2.461 (2)	Na-O(W3)	2.511 (2)	
NH ₃ -(CH ₂) ₂ -NH ₃ group				
N-C	1.475 (3)	N-C-C	110.5 (2)	
C-C	1.509 (4)			
Hydrogen bonds				
	O-H		O-O	O-H...O
	N-H	H...O	N-O	N-H...O
O(2)-H...O(3)	0.82 (3)	1.71 (3)	2.529 (2)	174 (3)
N-H(1N)...O(4)	0.90 (3)	1.83 (2)	2.732 (2)	176 (3)
N-H(2N)...O(1)	0.91 (3)	2.00 (3)	2.901 (2)	174 (3)
N-H(3N)...O(3)	0.85 (3)	1.87 (3)	2.707 (2)	168 (3)
O(W1)-H(1W1)...O(1)	0.83 (3)	1.99 (3)	2.806 (2)	168 (3)
O(W1)-H(2W1)...O(W3)	0.77 (4)	2.10 (4)	2.859 (2)	170 (4)
O(W2)-H(1W2)...O(2)	0.80 (3)	2.08 (3)	2.839 (2)	158 (3)
O(W2)-H(2W2)...O(1)	0.84 (3)	1.94 (3)	2.773 (2)	172 (3)
O(W3)-H(1W3)...O(4)	0.82 (3)	1.97 (3)	2.787 (2)	172 (3)
O(W3)-H(2W3)...O(1)	0.80 (4)	2.20 (4)	2.922 (2)	152 (4)
H(1W1)-O(W1)-H(2W1)	107 (3)	H(1W3)-O(W3)-H(2W3)	102 (3)	
H(1W2)-O(W2)-H(2W2)	101 (3)			

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Monopotassium Phosphoenolpyruvate: New Diffractometer Data

BY T. LIS

Instytut Chemii, Uniwersytet, 50-383 Wrocław, Poland

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Abstract. Potassium 2-(phosphonoxy)propenoate, $C_3H_4O_6P \cdot K^+$, $M_r = 206.14$, orthorhombic, $Pbca$, $a = 14.534$ (8), $b = 13.863$ (6), $c = 6.883$ (3) Å, $V = 1387$ Å³, $Z = 8$, $D_m = 1.96$, $D_x = 1.97$ Mg m⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.97$ mm⁻¹, $F(000) = 832$, $T = 294$ (2) K, final $R = 0.042$ for 1821 non-zero reflexions. The value of b differs significantly from that reported by Hosur & Viswamitra [*Acta Cryst.* (1981), B37, 839-843]. The length of the P-O(ester) bond is 1.622 (2) Å, the P-O(H) bond is 1.545 (2) Å.

Introduction. During statistical analysis of the geometry of the monophosphate group in organic esters (Starynowicz & Lis, 1985; Starynowicz, Lis &

The HPO₄ and organic groups alternate in planes perpendicular to the a axis. The HPO₄ groups are associated in pairs forming centrosymmetric finite clusters $[H_2P_2O_8]^{4-}$, the two tetrahedra being linked by hydrogen bonds, not drawn in Fig. 1 but reported in Table 2. In such a group the P-P distance is relatively short (4.320 Å). In these same planes ethylenediamine is present as the zwitterion, $(CH_2)_2(NH_3)_2^{2+}$. This group is centrosymmetrical.

Between these planes and separated by a distance of 11.70 Å are located the Na atoms and the six water molecules. Na atoms have a sixfold coordination formed by five water molecules and one O atom.

Main interatomic distances, bond angles and hydrogen bonds are reported in Table 2. The potassium salt is isotopic with the following unit-cell dimensions: $a = 11.77$, $b = 10.62$, $c = 6.93$ Å, $\beta = 102.68^\circ$.

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Weichsel, 1986; Starynowicz, 1986) it was found that in monopotassium phosphoenolpyruvate (Hosur & Viswamitra, 1981) the P-O(H) distance does not differ from other P-O(terminal) distances. Since this is unexpected it was decided to reinvestigate this structure.

Experimental. Crystals of monopotassium phosphoenolpyruvate were obtained from an aqueous solution at room temperature of the commercially available compound (Boehringer). An almost parallelepipedal fragment 0.2 × 0.5 × 0.4 mm was cut from a large crystal; preliminary examination by rotation and Weissenberg photographs. Syntex P₂ diffractometer