

Structure of Ethylenediammonium Bis(dihydrogenmonophosphate)

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(Received 21 June 1988; accepted 12 October 1988)

Abstract. $C_2H_{10}N_2^{2+} \cdot 2H_2PO_4^-$, $M_r = 256.09$, monoclinic, $P2_1/a$, $a = 14.224(3)$, $b = 5.917(1)$, $c = 5.845(1)$ Å, $\beta = 94.96(1)^\circ$, $V = 490.1(1)$ Å³, $Z = 2$, $D_x = 1.735$, $D_m = 1.688(3)$ Mg m⁻³, $\lambda(\text{Ag } K\alpha) = 0.5608$ Å, $\mu = 0.239$ mm⁻¹, $F(000) = 268$, $T = 298$ K, final $R = 0.026$ for 1854 independent reflexions. H_2PO_4 tetrahedra are connected through O—H...O hydrogen bonds so that infinite chains of composition $(H_2PO_4)_n^{n-}$ are formed in the structure, parallel to the a axis. The chains are themselves interconnected by means of N—H...O hydrogen bonds originating from the $(CH_2)_2(NH_3)_2^{2+}$ dication forming a three-dimensional network.

Introduction. During the 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 salt (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— H_3PO_4 system.

The preparation of ethylenediammonium bis(dihydrogenmonophosphate) has been described (Kamoun, Jouini, Kamoun & Daoud, 1988). The present work is devoted to a detailed structural investigation.

Experimental. Crystal size 0.10 × 0.11 × 0.13 mm. Density measured by flotation in bromobenzene. Philips PW 1100 diffractometer, graphite monochromator. ω scan. 3297 reflexions collected between 3 and 30 (θ)°. $\pm h$, $\pm k$, l . $h_{\max} = 25$, $k_{\max} = 10$, $l_{\max} = 10$. Scan width $(1 + 0.2 \tan \theta)^\circ$. Scan speed 0.02° s⁻¹. Total background time from 10 to 106 s. Three intensities and orientation reference reflexions (312, 3 $\bar{1}$ 2, 231) measured every 2 h; no significant intensity variation. Lorentz and polarization corrections. No absorption correction. $R_{\text{int}} = 0.018$, 1854 unique reflexions. Structure solved by classical methods (Patterson and successive Fourier syntheses). H atoms located in Fourier difference map. Anisotropic full-matrix least-

squares refinement (on F) for non-hydrogen atoms, isotropic for H atoms. Unit weights. $\sum \Delta F^2$ minimized. Final refinement cycles with 1854 reflexions corresponding to $F > 1\sigma_F$. Final $R = 0.026$, $wR = 0.031$, $S = 0.411$, max. $\Delta/\sigma = 0.00$. Max. peak height in ΔF : 0.375 e Å⁻³. No extinction correction. Scattering factors and anomalous-dispersion terms taken from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1980) SDP employed for all refinements. Computer used PDP 11/34.

Discussion. Atomic parameters are given in Table 1.* Interatomic distances, bond angles and the hydrogen-bond scheme are in Table 2. The structure is depicted in Fig. 1.

Examination of the results obtained from the crystal structure determination shows clearly a layer arrangement parallel to the a axis: planes of H_2PO_4 tetrahedra alternate with planes of $(CH_2)_2(NH_3)_2$ groups.

The H_2PO_4 tetrahedra are connected together through a double hydrogen bond: O(1)—H(O1)...O(4) and O(2)—H(O2)...O(3) types, forming infinite two-dimensional chains of composition $(H_2PO_4)_n^{n-}$. These chains are located in the (002) planes. The P—P distance between H_2PO_4 tetrahedra: 4.130(1) Å is slightly shorter than those observed in ethylenediammonium monohydrogenphosphate complexes (Averbuch-Pouchot, Durif & Guitel, 1987). This is probably due to the presence of two acidic hydrogen atoms on the PO_4 tetrahedron, which is favourable for the formation of strong hydrogen bonds.

The organic groups, present as the dication $(CH_2)_2(NH_3)_2^{2+}$, are located in the (001) planes. The intermolecular hydrogen-bonding contacts: N—H(1N)...O(4), N—H(2N)...O(1), N—H(3N)...O(3) provide a linkage between the $(CH_2)_2(NH_3)_2^{2+}$ entities and

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51488 (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} values
$$B_{eq} = \frac{1}{3} \sum_i \sum_j a_i \cdot a_j B_{ij}$$

	x	y	z	B_{eq} (\AA^2)
P	0.35958 (2)	0.44848 (6)	0.39159 (6)	1.410 (4)
O(1)	0.14910 (7)	0.6836 (2)	0.5956 (2)	2.09 (2)
O(2)	0.38837 (7)	0.5335 (2)	0.6411 (2)	2.09 (2)
O(3)	0.06530 (6)	-0.0022 (2)	0.7671 (2)	1.86 (2)
O(4)	0.23491 (6)	0.0512 (2)	0.6823 (2)	2.16 (2)
N	0.37437 (8)	0.9063 (2)	0.0060 (2)	2.01 (2)
C	0.04296 (9)	0.5176 (2)	0.0829 (2)	1.87 (2)

Table 2. Principal interatomic distances (\AA) and bond angles ($^\circ$) and details of the hydrogen-bonding scheme

PO_4 tetrahedron				
P	O(1)	O(2)	O(3)	O(4)
O(1)	1.574 (1)	2.521 (2)	2.467 (2)	2.524 (2)
O(2)	106.93 (6)	1.564 (1)	2.538 (1)	2.467 (1)
O(3)	106.58 (6)	111.74 (6)	1.503 (1)	2.524 (1)
O(4)	110.13 (6)	107.06 (6)	114.20 (6)	1.504 (1)

Min. P-P	4.130 (1)
P-O	1.536 (1)

 $\text{NH}_3-(\text{CH}_2)_2-\text{NH}_3$ group

N-C	1.481 (2)
C-C	1.508 (3)
C-C-C	109.7 (1)

Hydrogen bonds

	(O,N)-H	H...O	(O,N)-O	(O,N)-H...O
O(1)-H(1)...O(4)	0.74 (2)	1.81 (2)	2.524 (2)	168 (2)
O(2)-H(2)...O(3)	0.74 (2)	1.83 (2)	2.538 (1)	177 (3)
N-H(1N)...O(4)	0.92 (2)	1.91 (2)	2.757 (2)	152 (2)
N-H(2N)...O(1)	0.83 (2)	2.08 (2)	2.892 (2)	163 (2)
N-H(3N)...O(3)	0.90 (2)	2.02 (2)	2.854 (2)	153 (2)

$(\text{H}_2\text{PO}_4)_n^{n-}$ chains belonging to two successive (002) planes.

All these hydrogen bonds (O-H...O and N-H...O types) give rise to a three-dimensional network in the structure and add stability to this compound.

Acta Cryst. (1989), **C45**, 482-485

Structure of 1,4-Phenylenebis(3,3-diphenylketenimine)

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(Received 10 June 1988; accepted 4 October 1988)

Abstract. 1,4-*N,N'*-(2,2'-Diphenylvinylidene)phenylenediamine, $\text{C}_{34}\text{H}_{24}\text{N}_2$, $M_r = 460.58$, triclinic, $P\bar{1}$, $a = 7.928$ (7), $b = 9.474$ (6), $c = 9.599$ (4) \AA , $\alpha = 72.18$ (4), $\beta = 74.98$ (6), $\gamma = 68.84$ (6) $^\circ$, $V = 631.1$ (9) \AA^3 , $Z = 1$, $D_x = 1.212$ g cm^{-3} , $\lambda(\text{Mo K}\alpha) =$

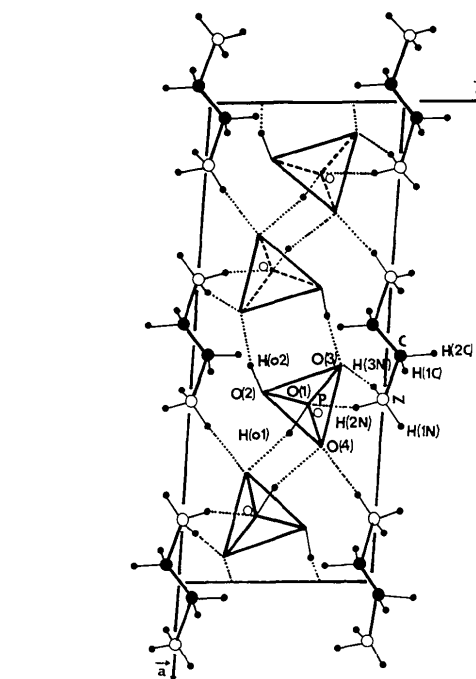


Fig. 1. Projection along the b axis of the atomic arrangement of $\text{NH}_3-(\text{CH}_2)_2-\text{NH}_3 \cdot (\text{H}_2\text{PO}_4)_2$.

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