

salts will provide confirmation of this prediction, as well as revealing coupling between molecular geometry of the more strongly interacting groups.

We are grateful to the SERC and IL-L for the provision of reactor facilities, and to R. L. Beddoes for assistance with X-ray work.

References

- ALLEN, F. H. & KIRBY, A. J. (1984). *J. Am. Chem. Soc.* **106**, 6197–6200.
 BARTON, D. H. R., HEAD, A. J. & MAY, P. J. (1957). *J. Chem. Soc.* p. 935.
 BÜRGI, H.-B. & DUNITZ, J. D. (1983). *Acc. Chem. Res.* **16**, 153–161.

- CERNIK, R. V., CRAZE, G.-A., MILLS, O. S. & WATT, I. (1982). *J. Chem. Soc. Perkin Trans. 2*, pp. 361–367.
 CERNIK, R. V., CRAZE, G.-A., MILLS, O. S., WATT, I. & WHITTLETON, S. N. (1984). *J. Chem. Soc. Perkin Trans. 2*, pp. 685–690.
 CRAZE, G.-A. & WATT, I. (1981). *J. Chem. Soc. Perkin Trans. 2*, pp. 175–184.
 ERMER, O., MASON, S. A., ANET, F. A. L. & MIURA, S. S. (1985). *J. Am. Chem. Soc.* **107**, 2330–2334.
 JOHNSON, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 KOESTER, L. & RAUCH, H. (1981). *Summary of Neutron Scattering Lengths*. IAEA Contract 2517/RB. Vienna: International Atomic Energy Agency.
 STEIGERWALD, M. L., GODDARD, W. A. & EVANS, D. A. (1979). *J. Am. Chem. Soc.* **101**, 1994–1999.
 WILLIAMS, I. H. (1983). *Theochem.* **105**, 105–117.
 ZEYEN, C. M. E., CHAGNON, R., DISDIER, F. & MORIN, H. (1984). *Rev. Phys. Appl.* **19**, 789–791.

Acta Cryst. (1989). **C45**, 428–430

Structure of Bis(ethylenediammonium) *cyclo*-Tetraphosphate Monohydrate

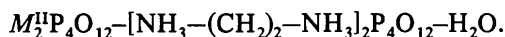
BY M. T. AVERBUCH-POUCHOT, A. DURIF AND J. C. GUITEL

Laboratoire de Cristallographie, Centre National de la Recherche Scientifique, Laboratoire associé à l'Université J. Fourier, 166 X, 38042 Grenoble CEDEX, France

(Received 12 August 1988; accepted 5 October 1988)

Abstract. $[\text{C}_2\text{H}_{10}\text{N}_2]_2[\text{P}_4\text{O}_{12}]\cdot\text{H}_2\text{O}$, $M_r = 458.13$, orthorhombic, *Pcca* (D_{2h}^8), $a = 13.168$ (9), $b = 8.599$ (6), $c = 15.152$ (10) Å, $V = 1704$ (3) Å³, $Z = 4$, D_m not measured, $D_x = 1.786$ g cm⁻³, $\lambda(\text{Ag K}\alpha) = 0.5608$ Å, $\mu = 2.76$ cm⁻¹, $F(000) = 952$, $T = 293$ K, final R value 0.033 for 1983 independent reflexions. Layers ($z \sim 0$ and 0.5) of the P_4O_{12} ring anions and water molecules alternate with corrugated layers of ethylenediammonium dications ($z \sim 0.25$ and 0.75). The two independent ethylenediammonium groups, the P_4O_{12} ring anion and the water molecule have twofold symmetry.

Introduction. The present description of the crystal structure of ethylenediammonium *cyclo*-tetraphosphate monohydrate is part of an investigation of the system

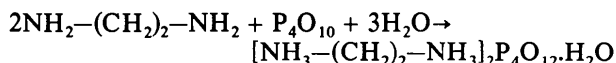


Some compounds belonging to such systems have previously been described. Up to now they correspond to three different structure types.

The first, common to a series of salts: $M^{\text{II}}[\text{NH}_3-(\text{CH}_2)_2-\text{NH}_3]_3\text{P}_4\text{O}_{12}\cdot 14\text{H}_2\text{O}$ has been determined by using the copper salt (Averbuch-Pouchot, Durif & Guitel, 1989). The second is represented by the calcium salt: $\text{Ca}[\text{NH}_3-(\text{CH}_2)_2-\text{NH}_3]_2\text{P}_4\text{O}_{12}\cdot 15/2\text{H}_2\text{O}$

(Averbuch-Pouchot, Durif & Guitel, 1988). The third has recently been described for the strontium salt: $\text{Sr}[\text{NH}_3-(\text{CH}_2)_2-\text{NH}_3]_2\text{P}_4\text{O}_{12}\cdot 5\text{H}_2\text{O}$ (Bagieu-Beucher, Durif & Guitel, 1988).

Experimental. To an aqueous solution of ethylenediamine, kept at a temperature $T < 278$ K, is slowly added an amount of P_4O_{10} corresponding to the reaction:



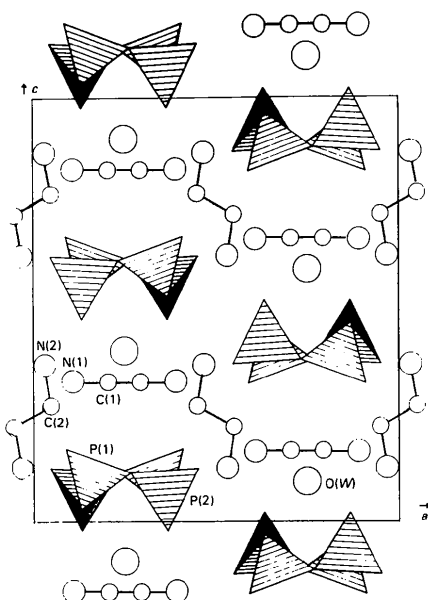
After evaporation for some days at room temperature orthorhombic plates of the title compound appear in the solution.

Density not measured. Prism fragment: $0.34 \times 0.32 \times 0.32$ mm. Philips PW 1100 diffractometer, graphite monochromator. Systematic absences: $0kl$: $l = 2n$; $h0l$: $l = 2n$; $hk0$: $h = 2n$. 16 reflexions ($11 < \theta < 12^\circ$) for refining unit-cell dimensions. ω scan, scan width: 1.20° , scan speed: $0.03^\circ \text{ s}^{-1}$, total background measuring time: 10 s. 2608 reflexions measured ($3 < \theta < 27.5^\circ$), $h, k, l, h_{\text{max}} = 22, k_{\text{max}} = 14, l_{\text{max}} = 26$.

Two intensity and orientation reference reflexions (006 and 820) every two hours, no significant variation. Lorentz and polarization corrections, no absorp-

Table 1. Final atomic coordinates and B_{eq} values for the title compound, with e.s.d.'s in parentheses
$$B_{eq} = \frac{4}{3} \sum_i \sum_j a_i \cdot a_j \beta_{ij}$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
P(1)	0.14816 (4)	0.18830 (6)	0.08985 (4)	1.716 (7)
P(2)	0.36982 (4)	0.15350 (6)	0.06238 (4)	1.667 (7)
O(L12)	0.2665 (1)	0.1756 (2)	0.11715 (9)	1.87 (2)
O(L21)	0.1329 (1)	0.0282 (2)	0.0345 (1)	1.88 (2)
O(E11)	0.0899 (1)	0.1815 (2)	0.1733 (1)	2.59 (3)
O(E12)	0.1306 (1)	0.3203 (2)	0.0292 (1)	2.99 (3)
O(E21)	0.1326 (1)	0.2411 (2)	0.4783 (1)	2.39 (3)
O(E22)	0.4526 (1)	0.1825 (2)	0.1274 (1)	2.46 (3)
O(W)	0.25	0.5	0.4043 (2)	2.69 (4)
N(1)	0.3912 (1)	0.9705 (2)	0.3332 (1)	2.23 (3)
N(2)	0.4677 (2)	0.4877 (3)	0.3715 (2)	3.27 (4)
C(1)	0.2940 (2)	0.0571 (3)	0.3312 (2)	2.09 (4)
C(2)	0.0499 (2)	0.5091 (3)	0.2750 (2)	3.25 (5)

Fig. 1. Projection of the atomic arrangement of $[\text{NH}_3-(\text{CH}_2)_2-\text{NH}_3]_2\text{P}_4\text{O}_{12} \cdot \text{H}_2\text{O}$ along the b axis. The PO_4 tetrahedra are simply denoted by the names of the central phosphorus atoms.

tion correction. Unit weights. Crystal structure solved by direct methods with *MULTAN77* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1977). H atoms located from difference-Fourier syntheses. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. Final refinement with 1983 reflexions ($I > 4\sigma I$). Final $R = 0.033$ ($wR = 0.035$), $S = 1.033$. Max. $\Delta/\sigma = 0.01$. Max. peak height in the final difference Fourier 0.375 e \AA^{-3} . The final R value = 0.044 for the complete set of 2608 independent reflexions. No extinction correction. Scattering factors for neutral atoms and f' , f'' from *International Tables for X-ray Crystallography* (1974). Enraf-Nonius (1977) *SDP* used for all calculations. Computer used: MicroVAX II.

Table 2. Main interatomic distances (\AA) and bond angles ($^\circ$) in the atomic arrangement of the title compound

The P_4O_{12} ring anion					
P(1)O_4 tetrahedron					
P(1)	O(L12)	O(L21)	O(E11)	O(E12)	
O(L12)	1.616 (1)	2.500 (2)	2.477 (2)	2.550 (2)	
O(L21)	$\overline{101.31}$ (8)	1.616 (1)	2.542 (2)	2.495 (2)	
O(E11)	106.15 (9)	$\overline{110.24}$ (9)	1.480 (2)	2.542 (3)	
O(E12)	111.26 (9)	107.67 (9)	$\overline{118.8}$ (1)	1.472 (2)	
P(2)O_4 tetrahedron					
P(2)	O(L12)	O(L21)	O(E21)	O(E22)	
O(L12)	1.605 (1)	2.521 (2)	2.549 (2)	2.457 (2)	
O(L21)	$\overline{103.33}$ (8)	1.609 (1)	2.452 (2)	2.546 (2)	
O(E21)	111.57 (9)	$\overline{105.15}$ (9)	1.477 (2)	2.571 (2)	
O(E22)	105.04 (9)	110.48 (9)	$\overline{120.1}$ (1)	1.490 (2)	
P(1)—P(2) 2.9632 (7) P(2)—P(1)—P(2) 87.71 (2)					
P(1)—P(2) 2.9576 (8) P(1)—P(2)—P(1) 90.02 (2)					
P(1)—O(L12)—P(2) 133.9 (1)					
P(1)—O(L21)—P(2) 133.0 (1)					
The ethylenediammonium groups					
C(1)—N(1)	1.478 (3)	C(2)—N(2)	1.479 (4)		
C(1)—C(1)	1.514 (3)	C(2)—C(2)	1.517 (4)		
C(1)—C(1)—N(1)	109.9 (2)	C(2)—C(2)—N(2)	111.0 (2)		
The hydrogen-bond network					
	O(N)—H	H...O	O(N)—O	O(N)—H...O	
	(\AA)	(\AA)	(\AA)	($^\circ$)	
N(1)—H(1N1)	O(E11)	0.91 (3)	1.85 (3)	2.759 (3)	177 (3)
N(1)—H(2N1)	O(E22)	0.91 (3)	1.91 (3)	2.804 (3)	168 (3)
N(1)—H(3N1)	O(E21)	0.77 (3)	2.10 (3)	2.864 (3)	171 (3)
N(2)—H(1N2)	O(E22)	0.88 (3)	1.97 (3)	2.809 (3)	160 (3)
N(2)—H(2N2)	O(W)	0.83 (3)	2.13 (3)	2.912 (2)	157 (3)
N(2)—H(3N2)	O(E12)	0.93 (3)	2.22 (3)	3.092 (3)	156 (3)
	O(E12)	0.77 (3)	2.28 (4)	2.900 (2)	138 (4)
O(W)—H(W)	O(E21)	0.77 (3)	2.36 (4)	2.921 (2)	131 (4)
	H(W)—O(W)—H(W)			95 (3)	

Discussion. Table 1 lists the final atomic coordinates while Fig. 1 gives a projection of the atomic arrangement along the b axis.*

The structure can be described by a succession of layers perpendicular to the c axis. The P_4O_{12} ring anions and the water molecules alternate in layers located at $z \approx 0.0$ and 0.5 . The P_4O_{12} ring anion has twofold symmetry and so is built by only two independent PO_4 tetrahedra. The water molecule is also located on a twofold axis. The two independent ethylenediammonium groups are located between these layers and form corrugated sheets at $z \approx 0.25$ and 0.75 , one of the organic groups being parallel to the sheet, the second perpendicular. The two independent ethylenediammonium dications have twofold symmetry. Fig. 1, a projection along the b axis, shows this arrangement, while Table 2 gives the geometrical

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51471 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

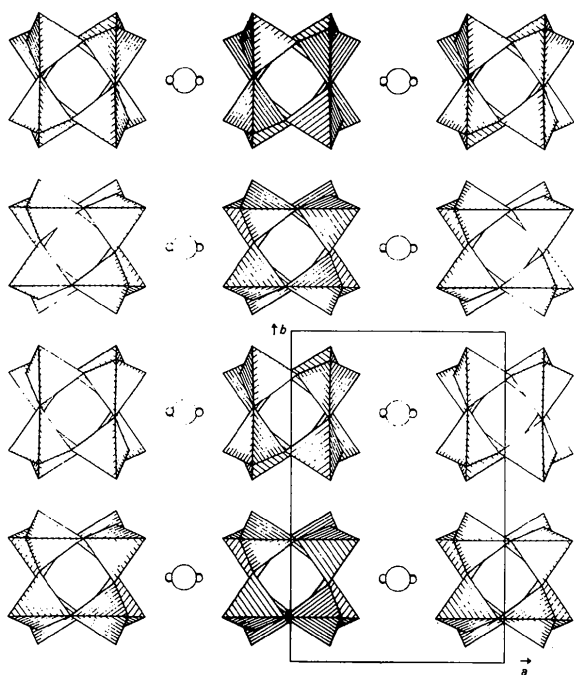


Fig. 2. Projection along the c axis showing the respective organization of the P_4O_{12} ring anions and water molecules along the twofold axes parallel to c .

features of the P_4O_{12} ring anion and of the organic groups. A three-dimensional network of hydrogen bonds connecting the NH_3 radicals and water molecules to the external oxygen atoms of the phosphoric ring anion is reported in Table 2. It should be noticed that the hydrogen bond connecting the hydrogen atom of the water molecule to the phosphoric group is bifurcated (Table 2). Fig. 2 reports the respective arrangement of the arrays of water molecules and P_4O_{12} groups along the twofold axes parallel to c .

References

- AVERBUCH-POUCHOT, M. T., DURIF, A. & GUITEL, J. C. (1988). *Acta Cryst.* **C44**, 1189–1191.
 AVERBUCH-POUCHOT, M. T., DURIF, A. & GUITEL, J. C. (1989). *Acta Cryst.* To be published.
 BAGIEU-BEUCHER, M., DURIF, A. & GUITEL, J. C. (1988). *Acta Cryst.* **C44**, 2063–2065.
 Enraf–Nonius (1977). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERQ, J.-P. & WOOLFSON, M. M. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Acta Cryst. (1989). **C45**, 430–433

Z Méthylamino- α Nitro- β Stilbène

PAR P. DUBOIS ET R. CEOLIN

*Laboratoires de Chimie analytique et de Chimie physique, Faculté de Pharmacie,
2 bis Boulevard Tonnellé, 37042 Tours CEDEX, France*

ET N. RODIER

*Laboratoire de Chimie minérale, Faculté des Sciences pharmaceutiques et biologiques,
5 Rue J.-B. Clément, 92296 Châtenay-Malabry CEDEX, France*

(Reçu le 25 mai 1988, accepté le 18 août 1988)

Abstract. $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_2$, $M_r = 254.3$, orthorhombic, $P2_12_12_1$, $a = 6.983(2)$, $b = 10.702(1)$, $c = 17.297(4)$ Å, $V = 1292.6(8)$ Å³, $Z = 4$, $D_x = 1.307$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.083$ mm⁻¹, $F(000) = 536$, $T = 294(1)$ K, $R = 0.032$ for 786 independent reflections. The molecule exhibits a *cis* configuration. To avoid steric hindrance, the two phenyl groups are twisted out of the plane of the ethylene group. A strong intramolecular N–H...O hydrogen bond [2.579(3) Å] forms a pseudo-six-membered ring which is approximately planar. A delocalized

orbital spreads out from the N atom of the amino group to the nitro group. This orbital does not interact with the π -electron systems of the phenyl rings.

Introduction. Les études réalisées sur les amino-1 nitro-2 stilbènes par les méthodes spectrographiques classiques (UV, IR, RMN du proton) ont été l'objet d'interprétations divergentes. Ainsi, selon Freeman & Emmons (1956), lorsque l'amine est primaire ou secondaire, ces composés possèdent une liaison hydrogène intramoléculaire. Par contre, selon Belon & Perrot