

Table 1. Positional parameters with e.s.d.'s in parentheses

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_i \mu_i^* a_j^* a_i a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
O1	0.5670 (3)	0.3476 (3)	-0.0204 (1)	4.41 (5)
O2	0.5848 (3)	0.4782 (3)	0.0784 (1)	3.74 (4)
O3	0.6600 (4)	0.0844 (3)	0.1333 (1)	5.21 (6)
O4	0.4695 (4)	0.2573 (4)	0.1725 (1)	6.40 (7)
C1	0.6645 (5)	0.4639 (5)	-0.0561 (2)	4.70 (8)
C2	0.5524 (5)	0.5977 (5)	-0.0826 (2)	5.21 (9)
C3	0.4550 (5)	0.6809 (5)	-0.0293 (2)	5.15 (9)
C4	0.3597 (5)	0.5544 (5)	0.0095 (2)	4.60 (8)
C5	0.4750 (5)	0.4169 (4)	0.0299 (2)	4.00 (7)
C6	0.6945 (4)	0.3575 (4)	0.1041 (1)	3.34 (6)
C7	0.5927 (5)	0.2308 (4)	0.1406 (2)	3.82 (7)
C8	0.5978 (7)	-0.0496 (5)	0.1670 (2)	7.1 (1)
C9	0.8199 (4)	0.4429 (4)	0.1480 (1)	3.15 (6)
C10	0.7595 (5)	0.5343 (5)	0.1987 (1)	4.39 (8)
C11	0.8721 (6)	0.6135 (5)	0.2379 (2)	5.35 (9)
C12	1.0448 (5)	0.6024 (5)	0.2277 (2)	5.06 (9)
C13	1.1054 (5)	0.5110 (5)	0.1773 (2)	4.64 (8)
C14	0.9925 (4)	0.4320 (4)	0.1374 (2)	3.80 (7)

Table 2. Bond distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

O1—C1	1.438 (7)	C4—C5	1.511 (8)
O1—C5	1.405 (7)	C6—C7	1.522 (8)
O2—C5	1.430 (7)	C6—C9	1.523 (8)
O2—C6	1.421 (6)	C9—C10	1.392 (7)
O3—C7	1.322 (7)	C9—C14	1.376 (8)
O3—C8	1.454 (8)	C10—C11	1.375 (9)
O4—C7	1.198 (7)	C11—C12	1.38 (1)
C1—C2	1.514 (9)	C12—C13	1.386 (9)
C2—C3	1.523 (9)	C13—C14	1.384 (8)
C3—C4	1.519 (9)		
C1—O1—C5	113.7 (4)	C7—C6—C9	110.3 (4)
C5—O2—C6	113.2 (4)	O3—C7—O4	123.7 (6)
C7—O3—C8	117.2 (5)	O3—C7—C6	110.6 (5)
O1—C1—C2	111.5 (5)	O4—C7—C6	125.7 (6)
C1—C2—C3	110.2 (5)	C6—C9—C10	119.8 (6)
C2—C3—C4	109.8 (5)	C6—C9—C14	120.5 (5)
C3—C4—C5	111.7 (5)	C10—C9—C14	119.7 (6)
O1—C5—O2	111.9 (5)	C9—C10—C11	120.0 (7)
O1—C5—C4	113.2 (5)	C10—C11—C12	120.4 (6)
O2—C5—C4	107.6 (5)	C11—C12—C13	119.7 (7)
O2—C6—C7	110.6 (5)	C12—C13—C14	120.1 (6)
O2—C6—C9	107.6 (4)	C9—C14—C13	120.0 (6)

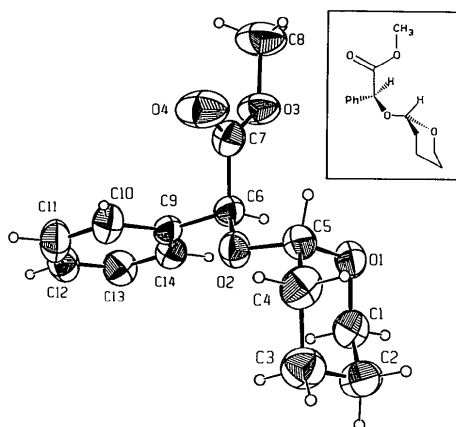


Fig. 1. ORTEP (Johnson, 1965) view of the title compound.

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Structure of ϵ -Ammoniohexanoic Acid Dihydrogenphosphate

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Abstract. (5-Carboxypentyl)ammonium dihydrogenphosphate, $\text{C}_6\text{H}_{14}\text{NO}_2^+ \cdot \text{H}_2\text{PO}_4^-$, $M_r = 229.17$, monoclinic, $I2/a \pm (x, y, z; \frac{1}{2}-x, y, -z)$, $a = 15.108 (3)$, $b = 4.636 (1)$, $c = 28.819 (3) \text{\AA}$, $\beta = 98.62 (1)^\circ$, $V =$

$1996 (4) \text{\AA}^3$, $Z = 8$, $D_x = 1.526 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{\AA}$, $\mu = 2.7 \text{ cm}^{-1}$, $F(000) = 976$, $T = 292 \text{ K}$, $R = 0.039$ for 1682 observed reflections with $I > 3\sigma(I)$. The protonated ϵ -aminohexanoic acid and the

Table 1. Positional and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
P	0.10847 (4)	0.1442 (1)	0.30791 (2)	0.0193 (1)
O(1)	0.1659 (1)	0.2846 (4)	0.35231 (5)	0.0287 (4)
O(2)	0.0458 (1)	0.3910 (3)	0.28499 (6)	0.0327 (4)
O(3)	0.0490 (1)	-0.0904 (3)	0.32334 (5)	0.0239 (4)
O(4)	0.1694 (1)	0.0509 (4)	0.27475 (5)	0.0337 (4)
O(5)	0.0612 (1)	-0.3080 (4)	0.40665 (5)	0.0387 (5)
O(6)	0.1444 (2)	0.0620 (4)	0.43589 (6)	0.0514 (6)
C(1)	0.1088 (2)	-0.1691 (5)	0.44104 (7)	0.0299 (6)
C(2)	0.1172 (2)	-0.3243 (6)	0.48702 (8)	0.0361 (6)
C(3)	0.1279 (2)	-0.1346 (6)	0.53001 (7)	0.0329 (6)
C(4)	0.1397 (2)	-0.3102 (6)	0.57531 (7)	0.0337 (6)
C(5)	0.1399 (2)	-0.1279 (5)	0.61909 (7)	0.0297 (6)
C(6)	0.1478 (2)	-0.3096 (5)	0.66291 (7)	0.0285 (6)
N	0.1457 (1)	-0.1336 (4)	0.70587 (6)	0.0269 (5)

Table 2. Interatomic distances (\AA), angles ($^\circ$), torsional angles ($^\circ$), and hydrogen-bond geometry (\AA , $^\circ$)

ϵ -Ammoniohexanoic acid				
O(5)—C(1)	1.305 (3)	O(5)—C(1)—O(6)	123.0 (3)	
O(6)—C(1)	1.218 (3)	O(5)—C(1)—C(2)	113.5 (3)	
C(1)—C(2)	1.496 (3)	O(6)—C(1)—C(2)	123.5 (2)	
C(2)—C(3)	1.508 (3)	C(1)—C(2)—C(3)	115.6 (2)	
C(3)—C(4)	1.527 (3)	C(2)—C(3)—C(4)	112.2 (3)	
C(4)—C(5)	1.518 (3)	C(3)—C(4)—C(5)	113.5 (3)	
C(5)—C(6)	1.508 (4)	C(4)—C(5)—C(6)	112.0 (3)	
C(6)—N	1.487 (3)	C(5)—C(6)—N	112.4 (2)	
O(5)—C(1)—C(2)—C(3)	-150.3 (2)	C(2)—C(3)—C(4)—C(5)	-173.4 (2)	
O(6)—C(1)—C(2)—C(3)	31.0 (4)	C(3)—C(4)—C(5)—C(6)	177.6 (2)	
C(1)—C(2)—C(3)—C(4)	-177.7 (2)	C(4)—C(5)—C(6)—N	-178.5 (2)	
Dihydrogenphosphate ion				
P—O(1)	1.575 (1)	O(1)—P—O(2)	105.39 (9)	
P—O(2)	1.566 (2)	O(1)—P—O(3)	109.65 (8)	
P—O(3)	1.519 (2)	O(1)—P—O(4)	108.88 (9)	
P—O(4)	1.487 (2)	O(2)—P—O(3)	107.43 (9)	
		O(2)—P—O(4)	109.55 (9)	
		O(3)—P—O(4)	115.5 (1)	
D—H...A	Position of A	D...A	H...A	D—H...A
O(1)—H(1)...O(6)	x, y, z	2.685 (2)	1.94 (3)	171 (4)
O(2)—H(2)...O(3)	x, 1 + y, z	2.644 (2)	1.94 (4)	162 (4)
O(5)—H(O)...O(3)	x, y, z	2.585 (2)	1.68 (3)	166 (3)
N—H(1N)...O(3)	-x, -y, 1 - z	3.113 (2)	2.26 (3)	160 (3)
N—H(2N)...O(4)	x, $\frac{1}{2}$ - y, $\frac{1}{2}$ + z	2.756 (2)	1.81 (4)	168 (3)
N—H(3N)...O(4)	$\frac{1}{2}$ - x, y, 1 - z	2.895 (2)	2.07 (3)	151 (3)

dihydrogenphosphate ion crystallize in the molar ratio 1:1, and are connected through a network of hydrogen bonds. The carboxylic group and the O—P—OH fragment form an eight-membered hydrogen-bonded ring P(O)OH...O(HO)C [P—O(3)⁻ 1.519 (2), P—O(1)H 1.575 (1) \AA , O—P—O 109.65 (8) $^\circ$; C(1)—O(6) 1.218 (3), C(1)—O(5)H 1.305 (3) \AA , O—C—O 123.0 (3) $^\circ$; O(1)...O(6) 2.685 (2), O(5)...O(3) 2.585 (2) \AA]. In addition, the charged O(3) atom participates in hydrogen bonding with the OH group of a neighbouring H_2PO_4^- anion [O(3)...O(2)(x, 1 + y, z) 2.644 (2) \AA]. The phosphoryl O(4) atom approaches two H atoms from two protonated amino groups [O(4)...N(x, $\frac{1}{2}$ - y, $\frac{1}{2}$ + z) 2.756 (2), O(4)...N($\frac{1}{2}$ - x, y, 1 - z) 2.895 (2) \AA]. The third H atom from the amino group is close to an adjacent O(3) atom [N...O(3)(-x, -y, 1 - z) 3.113 (2) \AA].

Experimental. The compound was obtained, among other products, by treatment of polycaproyamide with concentrated orthophosphoric acid. Needle-shaped transparent crystals suitable for X-ray study were recrystallized from ethanol. D_m was not determined. The cell dimensions and intensity data were measured on an Enraf-Nonius CAD-4 diffractometer (graphite monochromator, Mo $K\alpha$ radiation) using a crystal with approximate dimensions $0.25 \times 0.3 \times 0.08$ mm. Cell constants were determined from a least-squares fit of 22 reflections with $20.4 < \theta < 21.7^\circ$. 2783 reflections were collected [$\omega/2\theta$ scan, speed $1-7^\circ \text{ min}^{-1}$, $\Delta\omega = (0.8 + 0.4 \tan\theta)^\circ$] with $\sin\theta/\lambda < 0.660 \text{ \AA}^{-1}$ (h 0 to 19, k 0 to 6, l -37 to 37). Three standard reflections were monitored every 2 h. As the intensity variation was less than 1% no decay correction was applied. Lorentz and polarization corrections were used but no correction for absorption was applied. Although a symmetrically independent region of the reciprocal space was examined, the data-collection routine measured twice the $h0l$ reflections. After averaging the duplicates 2685 unique reflections with $R_{\text{int}} = 0.020$ remained and were used in further calculations. 1003 reflections were considered unobserved [$I < 3\sigma(I)$]. The initial structural model was obtained by MULTAN11/82 (Main *et al.*, 1982) with E magnitudes calculated for a hypothetical composition $\text{C}_3\text{H}_8\text{N}_2\text{O}_6\text{P}$ ($Z = 8$). An E map calculated from the best combined figure of merit (= 2.811) solution revealed two distinct molecules which initially were ascribed the compositions PO_4 and C_8 . This model was further refined by full-matrix least squares on F . Analysis of geometry and atomic thermal parameters in the C_8 molecule after a few cycles of refinement allowed the carboxylic group to be identified. Subsequent refinements and difference Fourier syntheses gave positions of the H atoms. Finally, analysis of the hydrogen-bonding scheme allowed the terminal protonated $-\text{NH}_3^+$ group to be distinguished from a possible methyl group. All H atoms were included in the refinements with fixed $U = 0.0507 \text{ \AA}^2$. Final $R = 0.039$, $wR = 0.048$ and $S = 1.430$; weight w defined as

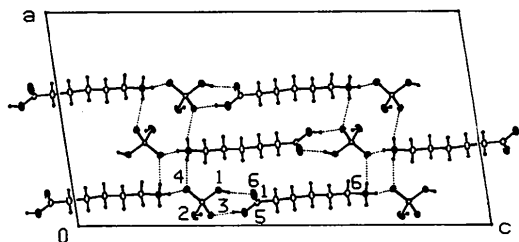


Fig. 1. Part of the structure projected onto the ca plane with simplified atomic numbering. The thermal parameters are at 30% probability and the O and N atoms are highlighted. Hydrogen bonds are marked by dotted lines.

$4F_o^2/[\sigma(F_o^2)]^2$. Max. $\Delta/\sigma = 0.106$; max. residual density $0.22 \text{ e } \text{Å}^{-3}$. No correction for secondary extinction was applied. Atomic scattering factors and anomalous-dispersion coefficients were used as quoted in the *SPD/PDP* software package (Enraf-Nonius, 1985) operating on a PDP11/44 computer. Final fractional coordinates for non-H atoms are given in Table 1.* The interatomic distances and angles are summarized in Table 2. The atom-numbering and hydrogen-bonding schemes are shown in Fig. 1.

Related literature. Products of the reaction of orthophosphoric acid with mono-, oligo- and polymeric caprolactam are of interest to industry (Mladenov & Vladkova, 1977). They have been characterized by X-ray powder and infrared spectroscopy methods (Mladenov, Vladkova, Fakirov & Mitzulov, 1978), by morphological and kinetic studies (Mladenov, Vladkova & Fakirov, 1980; Mladenov, Vladkova,

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54411 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Tzenkova & Fakirov, 1980), and by thermal analysis (Vladkova, 1981). A powder pattern of the title compound has been evaluated and submitted to the PDF database (Macíček, 1991).

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Structure of 1',1'''-Dimethyl-1,1''-tetramethylenedi(4,4'-bipyridinium) Tetraperchlorate

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Abstract. $\text{C}_{26}\text{H}_{30}\text{N}_4^{4+} \cdot 4\text{ClO}_4^-$, $M_r = 796.36$, orthorhombic, $Pbca$, $a = 9.791(2)$, $b = 15.908(4)$, $c = 21.288(5) \text{ Å}$, $V = 3315.7 \text{ Å}^3$, $Z = 4$, $D_x = 1.595 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71073 \text{ Å}$, $\mu =$

4.399 cm^{-1} , $F(000) = 1640$, $T = 295(1) \text{ K}$, $R = 0.052$ for 2072 unique observed reflections with $I > 3.0\sigma(I)$ and 226 parameters. 1',1'''-Dimethyl-1,1''-tetramethylenedi(4,4'-bipyridinium) belongs to the vio-