

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U _{eq}
P1	0.38412 (5)	0.57986 (5)	0.25518 (4)	0.02344 (14)
P2	0.22845 (5)	0.29839 (5)	0.34538 (4)	0.02411 (14)
P3	0.09604 (6)	0.37352 (5)	0.13570 (4)	0.02651 (14)
OE11	0.4015 (2)	0.71604 (15)	0.33127 (11)	0.0356 (3)
OE12	0.5045 (2)	0.58629 (15)	0.16791 (11)	0.0333 (3)
OL12	0.3877 (2)	0.43657 (14)	0.32636 (11)	0.0339 (3)
OL13	0.19577 (15)	0.53501 (13)	0.19889 (10)	0.0276 (3)
OE21	0.2976 (2)	0.17137 (15)	0.37985 (11)	0.0368 (3)
OE22	0.1117 (2)	0.3539 (2)	0.41437 (12)	0.0432 (4)
OL23	0.1481 (2)	0.25726 (14)	0.22033 (10)	0.0320 (3)
OE31	-0.0831 (2)	0.3637 (2)	0.14078 (14)	0.0486 (4)
OE32	0.1714 (2)	0.3528 (2)	0.03005 (11)	0.0425 (4)
OW1	0.1433 (2)	0.4755 (2)	0.62967 (15)	0.0477 (4)
OW2	0.2338 (2)	0.9106 (2)	0.24067 (15)	0.0502 (4)
N1	-0.3981 (2)	0.3511 (2)	0.05530 (14)	0.0285 (3)
N2	0.6437 (2)	0.2481 (2)	0.44668 (15)	0.0332 (4)
N3	0.0938 (2)	0.2556 (2)	-0.19197 (15)	0.0352 (4)
C1	0.5011 (2)	0.8048 (2)	-0.0692 (2)	0.0300 (4)
C2	-0.4445 (2)	0.0801 (2)	-0.0031 (2)	0.0327 (4)
C3	-0.2707 (3)	0.1220 (2)	0.4425 (2)	0.0322 (4)
C4	-0.2664 (3)	0.0595 (2)	0.3246 (2)	0.0312 (4)
C5	0.8238 (3)	0.9326 (2)	0.3199 (2)	0.0325 (4)
C6	0.8248 (3)	0.8724 (2)	0.2006 (2)	0.0338 (4)

Table 2. Selected geometric parameters (Å, °)

P1—OE11	1.4727 (13)	P2—P3	2.874 (1)
P1—OE12	1.4796 (13)	OW1—H1W1	0.88 (3)
P1—OL12	1.5993 (13)	OW1—H2W1	0.75 (4)
P1—OL13	1.6031 (13)	OW2—H1W2	0.88 (3)
P2—OE22	1.4700 (14)	OW2—H2W2	0.86 (4)
P2—OE21	1.4755 (13)	N1—C1 ⁱ	1.481 (2)
P2—OL23	1.5964 (13)	N2—C3 ⁱⁱ	1.487 (2)
P2—OL12	1.6098 (13)	N3—C6 ⁱⁱⁱ	1.482 (3)
P3—OE31	1.462 (2)	C1—C2 ^j	1.506 (3)
P3—OE32	1.4712 (14)	C2—C2 ^v	1.520 (4)
P3—OL13	1.6174 (13)	C3—C4	1.508 (3)
P3—OL23	1.6193 (13)	C4—C5 ^v	1.519 (3)
P1—P2	2.857 (1)	C5—C6	1.512 (3)
P1—P3	2.885 (1)		
OE11—P1—OE12	118.42 (8)	OE32—P3—OL23	107.35 (8)
OE11—P1—OL12	109.44 (8)	OL13—P3—OL23	100.17 (6)
OE12—P1—OL12	108.34 (8)	P1—OL12—P2	125.82 (8)
OE11—P1—OL13	107.31 (7)	P1—OL13—P3	127.26 (8)
OE12—P1—OL13	109.81 (7)	P2—OL23—P3	126.68 (8)
OL12—P1—OL13	102.34 (7)	P1—P2—P3	60.46 (2)
OE22—P2—OE21	120.48 (9)	P1—P3—P2	59.46 (2)
OE22—P2—OL23	110.06 (8)	P2—P1—P3	60.06 (2)
OE21—P2—OL23	107.92 (8)	H1W1—OW1—H2W1	103 (4)
OE22—P2—OL12	109.95 (9)	H1W2—OW2—H2W2	100 (3)
OE21—P2—OL12	106.08 (8)	N1 ⁱ —C1—C2 ^j	110.5 (2)
OL23—P2—OL12	100.44 (7)	C1 ⁱ —C2—C2 ^v	111.8 (2)
OE31—P3—OE32	122.31 (9)	N2 ⁱⁱ —C3—C4	111.7 (2)
OE31—P3—OL13	106.68 (8)	C3—C4—C5 ^v	111.8 (2)
OE32—P3—OL13	109.29 (8)	C6—C5—C4 ⁱⁱⁱ	110.3 (2)
OE31—P3—OL23	108.81 (8)	N3 ⁱⁱⁱ —C6—C5	112.4 (2)

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1+x, y, z$; (iii) $1-x, 1-y, -z$; (iv) $-1-x, -y, -z$; (v) $x-1, y-1, z$; (vi) $x-1, y, z$; (vii) $1+x, 1+y, z$.

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
OW1—H1W1...OE22	0.88 (3)	1.87 (4)	2.737 (2)	171 (3)
OW1—H2W1...OE22 ⁱ	0.75 (4)	2.24 (4)	2.946 (2)	158 (4)
OW2—H1W2...OE21 ⁱⁱ	0.88 (3)	1.87 (3)	2.750 (2)	178 (2)
OW2—H2W2...OE11	0.86 (4)	1.88 (4)	2.732 (2)	169 (3)
N1—H1N1...OE12 ⁱⁱⁱ	0.88 (2)	1.91 (3)	2.780 (2)	170 (2)
N1—H2N1...OE12 ^{iv}	0.88 (3)	2.05 (3)	2.906 (2)	163 (2)
N1—H3N1...OE31	0.90 (2)	1.82 (3)	2.710 (2)	168 (2)
N2—H1N2...OE11 ^v	0.93 (3)	1.82 (3)	2.734 (2)	166 (2)
N2—H2N2...OW1 ^v	0.89 (3)	2.02 (3)	2.907 (3)	172 (2)

N2—H3N2...OE21	0.88 (3)	1.97 (3)	2.820 (2)	165 (2)
N3—H1N3...OW1 ^{vi}	0.94 (3)	2.09 (3)	2.982 (3)	159 (2)
N3—H2N3...OE32	0.93 (3)	1.84 (3)	2.759 (2)	169 (2)
N3—H3N3...OW2 ^{vii}	0.89 (3)	1.89 (3)	2.757 (2)	163 (2)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $x, 1+y, z$; (iii) $x-1, y, z$; (iv) $-x, 1-y, -z$; (v) $1-x, 1-y, 1-z$; (vi) $x, y, z-1$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1152). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,6-Hexanediammonium cyclo-Hexaphosphate Hexahydrate

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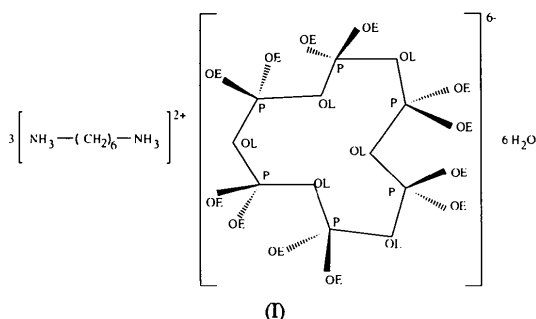
Abstract

The atomic arrangement of the title compound, 3C₆H₁₈N₂²⁺·P₆O₁₈⁶⁻·6H₂O, is described as a stacking of P₆O₁₈ groups and organic entities. The stability of such an arrangement results from a network of weak hydro-

gen bonds $[N(O)-H \cdots O]$ connecting organic groups and water molecules to the external O atoms of the ring anions.

Comment

The *cyclo*-hexaphosphate class has been investigated little when compared, for example, with the body of knowledge collected on condensed phosphates containing the P_2O_7 , P_3O_9 or P_4O_{12} groups (Durif, 1995). Among the few structurally well characterized compounds, a small number of *cyclo*-hexaphosphates including organic cations are known. All were synthesized by Boullé's process (Boullé, 1938). The previously reported $(N_3C_4H_{16})_2P_6O_{18} \cdot 2H_2O$ (Gharbi, Jouini & Durif, 1995) and the title compound were synthesized with the aid of the ion-exchange resins. This work describes the structural investigation of 1,6-hexanediammonium *cyclo*-hexaphosphate hexahydrate, (I).



This atomic arrangement, depicted in Fig. 1 by a projection along the c axis, can be described as a simple stacking of P_6O_{18} groups and organic entities. Both of the $P_6O_{18}^{6-}$ anions and one of the organic cations are located around the $(0, 1/2, 1/2)$ and the $(1/2, 0, 0)$ inversion centers of the triclinic cell, respectively, and are interconnected along the a axis by hydrogen bonds from one water molecule. Connection by hydrogen bonds, along the b and c axes, from the remaining two organic groups and the water molecules gives rise to a three-dimensional network. All hydrogen bonds establishing the cohesion between the different components of the structure are rather weak, with $O(N) \cdots O$ distances ranging from 2.715 (4) to 3.019 (3) Å (Blessing, 1986; Brown, 1976). The main geometric features of a phosphoric ring anion $[P_nO_{3n}]^{n-}$ are the $P \cdots P$ distances and the $P-O-P$ and $P \cdots P \cdots P$ angles. The observed $P \cdots P$ distances are quite similar whatever the n value. The $P-O-P$ angles also exhibit values typical for $[P_nO_{3n}]^{3-}$ anions. The situation is very different when the observed $P \cdots P \cdots P$ angles in $[P_6O_{18}]^{6-}$ ($n = 6$) are compared with those of $[P_3O_9]^{3-}$ ($n = 3$) and $[P_4O_{12}]^{4-}$ ($n = 4$). For this purpose the $P \cdots P \cdots P$ angles do not depart significantly from their ideal values of $60 \pm 2^\circ$ for $n = 3$ and $90 \pm 4^\circ$ for $n = 4$. However, for $n = 6$ we observe, in most of the available structural

data, very large deviations from the ideal value of 120° . The average $P \cdots P \cdots P$ angle in (I) is 116.96° with values ranging from $107.68 (4)$ to $125.55 (4)^\circ$. It should be noted that these large deviations are commonly observed in *cyclo*-hexaphosphates with a low local symmetry ring ($\bar{1}$) like that in the title compound. Details of the main geometrical features of the anionic group and the organic cation are reported in Table 2. The $(N-C, C-C)$ distances and $(N-C-C, C-C-C)$ angles are similar to those previously reported for condensed phosphates with organic cations. Thus, they are within the ranges $1.481 (3)$ – $1.522 (3)$ Å and $110.2 (2)$ – $115.4 (2)^\circ$, respectively.

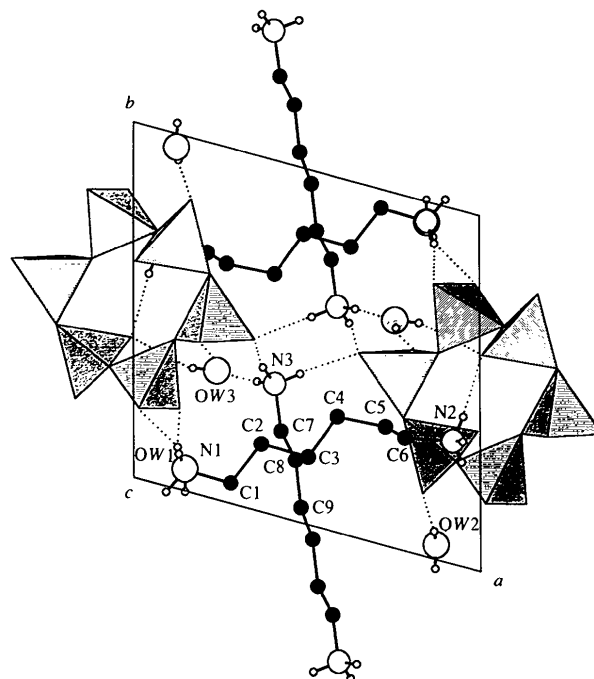


Fig. 1. Projection along the $[001]$ direction of the P_6O_{18} groups, in polyhedral representation and of the organic entities. By order of decreasing sizes, circles represent water molecules, N and C atoms. Hydrogen bonds are denoted by full and dotted lines.

Experimental

The title compound was synthesized by the action of *cyclo*-hexaphosphoric acid on an aqueous solution of 1,6-diaminohexane. The acid was produced from $Li_6P_6O_{18}$ solution through the use of ion-exchange resins. Colourless single crystals appeared as thick prisms after some days of evaporation of the solution at room temperature.

Crystal data

$3C_6H_{18}N_2^{2+} \cdot P_6O_{18}^{6-} \cdot 6H_2O$
 $M_r = 936.59$
 Triclinic
 $P\bar{1}$
 $a = 11.085 (3)$ Å
 $b = 11.678 (4)$ Å
 $c = 8.995 (4)$ Å

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 25 reflections
 $\theta = 10$ – 14°
 $\mu = 0.349$ mm $^{-1}$
 $T = 293 (2)$ K

$\alpha = 112.98 (3)^\circ$
 $\beta = 87.85 (3)^\circ$
 $\gamma = 104.90 (2)^\circ$
 $V = 1033.4 (6) \text{ \AA}^3$
 $Z = 1$
 $D_x = 1.505 \text{ Mg m}^{-3}$
 $D_m = 1.50 \text{ Mg m}^{-3}$
 D_m measured by pycnometry
 (toluene as pycnometric
 liquid)

Data collection

Enraf-Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 none
 3885 measured reflections
 3633 independent reflections
 3088 observed reflections
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0066$
 $\theta_{\text{max}} = 24.96^\circ$
 $h = -13 \rightarrow 13$
 $k = -13 \rightarrow 12$
 $l = 0 \rightarrow 10$
 1 standard reflection (031)
 frequency: 120 min
 intensity decay: 0.64%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0282$
 $wR(F^2) = 0.0954$
 $S = 0.955$
 3633 reflections
 377 parameters
 All H-atom parameters
 refined
 $w = 1/[\sigma^2(F_o^2) + (0.0317P)^2 + 1.2088P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.299 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.351 \text{ e \AA}^{-3}$
 Extinction correction:
 SHELXL93 (Sheldrick,
 1993)
 Extinction coefficient:
 0.0199 (21)
 Atomic scattering factors
 from *International Tables*
 for *Crystallography* (1992,
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
P1	0.77327 (5)	0.51804 (5)	0.32682 (6)	0.0200 (2)
P2	0.94537 (5)	0.72793 (5)	0.58775 (6)	0.0198 (2)
P3	1.11003 (5)	0.69600 (5)	0.80720 (6)	0.0207 (2)
OE21	0.9848 (2)	0.80500 (15)	0.4864 (2)	0.0286 (4)
OE31	1.1673 (2)	0.8253 (2)	0.9318 (2)	0.0365 (4)
OE11	0.8081 (2)	0.5705 (2)	0.2019 (2)	0.0343 (4)
OE12	0.65042 (15)	0.5213 (2)	0.3982 (2)	0.0310 (4)
OL12	0.88159 (15)	0.58147 (14)	0.4713 (2)	0.0259 (4)
OE22	0.8679 (2)	0.7745 (2)	0.7235 (2)	0.0336 (4)
OE32	1.0066 (2)	0.6084 (2)	0.8524 (2)	0.0318 (4)
OL23	1.06986 (14)	0.7092 (2)	0.6464 (2)	0.0275 (4)
OL13	0.77927 (14)	0.37080 (14)	0.2615 (2)	0.0262 (4)
OW1	1.1497 (3)	1.0606 (3)	0.9380 (3)	0.0673 (8)
OW2	1.1266 (3)	0.9602 (3)	1.2572 (3)	0.0528 (6)
OW3	0.7579 (2)	0.6400 (2)	-0.0444 (3)	0.0454 (5)
N1	1.1498 (2)	1.0582 (2)	0.6350 (3)	0.0316 (5)
N2	1.0765 (2)	0.6564 (2)	0.1892 (3)	0.0265 (4)
N3	0.5907 (2)	0.6337 (2)	0.7192 (3)	0.0289 (4)
C1	1.2811 (2)	1.0553 (3)	0.6018 (3)	0.0340 (6)
C2	0.6307 (2)	0.8117 (2)	0.3332 (3)	0.0318 (5)
C3	0.4944 (2)	0.8117 (2)	0.3578 (3)	0.0321 (5)
C4	0.5904 (2)	0.3232 (2)	0.6849 (3)	0.0332 (6)
C5	1.2706 (2)	0.6682 (3)	0.3294 (3)	0.0298 (5)
C6	1.2148 (2)	0.6845 (3)	0.1897 (3)	0.0285 (5)
C7	0.5736 (3)	0.7609 (3)	0.7409 (3)	0.0333 (6)
C8	0.5312 (3)	0.8302 (3)	0.9036 (3)	0.0354 (6)
C9	1.4834 (3)	1.0403 (3)	1.0825 (3)	0.0398 (6)

Table 2. Selected geometric parameters (\AA , $^\circ$)

P1—OE11	1.473 (2)	N2—C6	1.483 (3)
P1—OE12	1.487 (2)	N3—C7	1.482 (3)
P1—OL13	1.604 (2)	C1—C2 ⁱⁱ	1.505 (4)
P1—OL12	1.609 (2)	C2—C3	1.519 (3)
P2—OE22	1.472 (2)	C3—C4 ⁱⁱⁱ	1.518 (4)
P2—OE21	1.497 (2)	C4—C5 ⁱ	1.522 (3)
P2—OL23	1.591 (2)	C5—C6	1.517 (3)
P2—OL12	1.599 (2)	C7—C8	1.496 (4)
P3—OE31	1.476 (2)	C8—C9 ^{iv}	1.518 (4)
P3—OE32	1.488 (2)	C9—C9 ^v	1.506 (5)
P3—OL13 ⁱ	1.599 (2)	P1—P2	2.932 (2)
P3—OL23	1.604 (2)	P2—P3	2.926 (1)
N1—C1	1.481 (3)	P1—P3 ⁱ	2.927 (1)
OE11—P1—OE12	119.65 (10)	OL13 ⁱ —P3—OL23	98.31 (9)
OE11—P1—OL13	110.62 (10)	P2—OL12—P1	132.11 (10)
OE12—P1—OL13	106.18 (9)	P2—OL23—P3	132.71 (11)
OE11—P1—OL12	110.35 (10)	P3 ⁱ —OL13—P1	132.11 (10)
OE12—P1—OL12	108.71 (10)	P2—P1—P3 ⁱ	107.68 (4)
OL13—P1—OL12	99.36 (9)	P1—P2—P3	125.55 (4)
OE22—P2—OE21	118.37 (10)	P2—P3—P1 ⁱ	117.65 (4)
OE22—P2—OL23	111.61 (10)	N1—C1—C2 ⁱⁱ	111.9 (2)
OE21—P2—OL23	106.36 (9)	C1 ⁱⁱ —C2—C3	113.3 (2)
OE22—P2—OL12	110.96 (10)	C4 ⁱⁱⁱ —C3—C2	112.3 (2)
OE21—P2—OL12	108.81 (9)	C3 ⁱⁱⁱ —C4—C5 ⁱ	115.4 (2)
OL23—P2—OL12	98.89 (9)	C6—C5—C4 ⁱ	111.4 (2)
OE31—P3—OE32	118.15 (11)	N2—C6—C5	111.5 (2)
OE31—P3—OL13 ⁱ	107.21 (10)	N3—C7—C8	113.1 (2)
OE32—P3—OL13 ⁱ	110.82 (9)	C7—C8—C9 ^{iv}	110.2 (2)
OE31—P3—OL23	109.51 (10)	C9 ^v —C9—C8 ^{iv}	113.7 (3)
OE32—P3—OL23	110.97 (10)		

Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $2 - x, 2 - y, 1 - z$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $2 - x, 2 - y, 2 - z$; (v) $3 - x, 2 - y, 2 - z$.

Table 3. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1N1...OE21 ⁱ	0.89 (3)	2.09 (3)	2.946 (3)	159 (3)
N1—H2N1...OW1	0.93 (4)	1.84 (4)	2.715 (4)	157 (3)
N1—H3N1...OE21	0.91 (4)	1.96 (4)	2.862 (3)	171 (3)
N2—H1N2...OE32 ⁱⁱ	0.85 (3)	2.02 (3)	2.865 (3)	170 (3)
N2—H2N2...OE32 ⁱⁱⁱ	0.87 (4)	2.10 (4)	2.953 (3)	166 (3)
N2—H3N2...OE21	0.88 (4)	2.00 (4)	2.876 (3)	172 (3)
N3—H1N3...OW3 ^{iv}	0.89 (3)	1.96 (3)	2.840 (3)	175 (2)
N3—H2N3...OE12 ^v	0.88 (3)	2.01 (3)	2.787 (3)	147 (3)
N3—H3N3...OE12	0.91 (3)	1.92 (3)	2.801 (3)	163 (3)
OW1—H1W1...OE31	0.77 (4)	2.02 (5)	2.784 (4)	173 (4)
OW1—H2W1...OE22 ^{vi}	0.70 (5)	2.26 (5)	2.930 (4)	163 (5)
OW2—H1W2...OE22 ^{vi}	0.79 (4)	2.23 (4)	3.019 (3)	178 (3)
OW2—H2W2...OE31	0.81 (4)	2.01 (5)	2.814 (3)	171 (4)
OW3—H1W3...OE11	0.73 (4)	2.04 (4)	2.758 (3)	168 (4)
OW3—H2W3...OE32 ⁱⁱⁱ	0.81 (4)	2.19 (4)	2.943 (3)	156 (4)

Symmetry codes: (i) $2 - x, 2 - y, 1 - z$; (ii) $2 - x, 1 - y, 1 - z$; (iii) $x, y, z - 1$; (iv) $x, y, 1 + z$; (v) $1 - x, 1 - y, 1 - z$; (vi) $2 - x, 2 - y, 2 - z$.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989).
 Cell refinement: *CAD-4 Software*. Data reduction: *MolEN*
 (Fair, 1990). Program(s) used to solve structure: *SHELXS86*
 (Sheldrick, 1990). Program(s) used to refine structure:
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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1153). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Dihydrooxazinone

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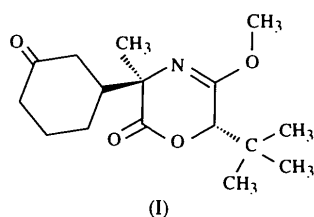
(Received 3 January 1996; accepted 18 March 1996)

Abstract

(3*RS*,6*SR*,1'*RS*)-6-*tert*-Butyl-3,6-dihydro-5-methoxy-3-methyl-3-(3-oxocyclohexyl)-2*H*-1,4-oxazin-2-one, C₁₆H₂₅NO₄, is an intermediate in the stereoselective synthesis of α -methylated amino acids. Its structure agrees well with those of other dihydrooxazinones. The heterocyclic ring is almost planar while the side chains exhibit the expected staggered conformations.

Comment

The stereospecific synthesis of dihydrooxazinones is a versatile synthetic route to enantiomerically pure α -methylated amino acids formed upon hydrolysis (Maywald, 1987). In order to study the influence of the size and nature of the substituents on the conformation of the heterocyclic ring, several dihydrooxazinones with an isopropyl group at C(6) have been investigated (Bolte, 1995). We have determined the structure of a racemic derivative, (I), with a tertiary butyl group replacing the isopropyl moiety.



The bond lengths and angles agree well with the other dihydrooxazinones studied so far (Bolte, 1995). In contrast to lactides (1,4-dioxane-2,5-diones) in which N(4) is replaced by an O atom (Bolte, Beck, Nieger & Egert, 1994), the heterocyclic ring is almost planar ($\sigma = 0.04 \text{ \AA}$). The cyclohexanone ring adopts a chair conformation and is oriented such that the five-atom chain from C(2) to C(5') is fully extended.

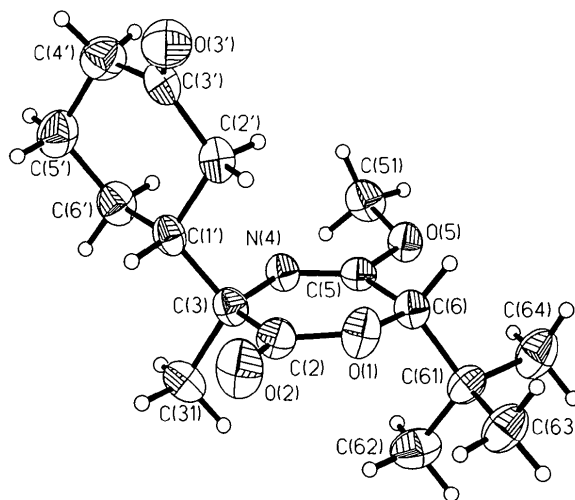


Fig. 1. Molecular structure of (I) showing 40% probability displacement ellipsoids.

Experimental

The compound was provided by Professor U. Schöllkopf (University of Göttingen) and recrystallized from cyclohexane.

Crystal data

C₁₆H₂₅NO₄
M_r = 295.4
 Triclinic
P $\bar{1}$
a = 6.806 (1) Å
b = 8.166 (1) Å
c = 15.568 (2) Å
 α = 100.69 (1)°
 β = 94.99 (2)°
 γ = 103.29 (1)°
V = 819.8 Å³
Z = 2
D_x = 1.197 Mg m⁻³
D_m not measured

Mo *K* α radiation
 λ = 0.71073 Å
 Cell parameters from 50 reflections
 θ = 10–12.5°
 μ = 0.08 mm⁻¹
T = 293 K
 Block
 0.6 × 0.4 × 0.1 mm
 Colourless

Data collection

Stoe–Siemens four-circle diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 3763 measured reflections
 2882 independent reflections
 1947 observed reflections
 $[F > 3\sigma(F)]$

*R*_{int} = 0.025
 θ_{max} = 25°
h = -8 → 7
k = -9 → 9
l = -4 → 18
 3 standard reflections monitored every 100 reflections
 intensity decay: none