The cyclopentene rings are non-planar and have a partly opened envelope conformation. Calculations for relevant planes are given in Table 3. The flap angles, β, for the two crystallographically non-equivalent cyclopentene rings are 26 and 19° respectively for rings defined by C(1) to C(5) and C(6) to C(10). These values are comparable with the angle of 21·9 (2)° in perfluorocyclopentene (Chang & Bauer, 1971) but smaller than that of 29 (1)° in cyclopentene (Davis & Muecke, 1970). Average corrected bond lengths for the cyclopentene rings are similar to those of perfluorocyclopentene (shown in square brackets); –C=C– = 1·326 (5) [1·342], =C–C– = 1·497 (4) [1·510 (9)], –C–C– = 1·524 (4) [1·539 (13) Å]. The C–F lengths are comparable with those in perfluoro-1,2-3,4-5,6-triethanobenzene (Cobbledick & Einstein, 1976). There are no particularly short intermolecular contacts.

We acknowledge the support of the National Research Council of Canada and thank Dr W. R. Cullen for his gift of the crystals.

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Acta Cryst. (1977). B**33**, 2342–2344

2-Formylamino-2-oxo-5,5-dimethyl-1,3,2-dioxaphosphorinane

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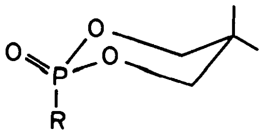
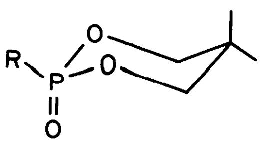
(*Received* 20 *December* 1976; *accepted* 18 *March* 1977)

Abstract. $C_6H_{12}NO_4P$, M_r = 193·1, orthorhombic, *Pbca*, a = 10·17 (1), b = 10·01 (1), c = 18·36 (2) Å, Z = 8, D_c = 1·372, D_m = 1·360 g cm^{-3}, μ(Cu $K\alpha$) = 24·7 cm^{-1}, λ = 1·5418 Å. The structure was solved by the heavy-atom method and refined to R = 0·08. The molecule has the chair configuration with the phosphoryl O atom equatorial. This atom forms a hydrogen bond to the amide group of an adjacent molecule.

Introduction. Most of the information on the preferred configuration of the P substituents in the chair form of 2-R-2-oxo-1,3,2-dioxaphosphorinanes suggests that the molecule with the O atom equatorial (I*a*) rather than axial (I*b*) is thermodynamically more stable (White, McEwen, Bertrand & Verkade, 1971). However, when R = NMe_2 this O is axial (Wadsworth, 1977), although it is equatorial when R = NHPh (Cameron & Karolak-Wojciechowska, 1976). The present structure, where R = NHCHO, has been examined to see if a strong electronegative group at the N atom has any influence on the P configuration.



(I*a*)



(I*b*)

The compound was prepared by A. Zwierzak (Technical University, Łodz). The crystal system and cell dimensions were determined from Weissenberg photographs; systematic absences 0*kl*, $k = 2n + 1$; *h*0*l*, $l = 2n + 1$; *hk*0, $h = 2n + 1$ uniquely determined the space group *Pbca*. 1030 independent reflexions were visually estimated from equi-inclination Weissenberg

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photographs ($h0l$–$h8l$) and for interlayer scaling (0–1kl). The data were corrected for Lorentz and polarization effects and the interlayer scale factors were calculated by the method of Rollett & Sparks (1960).

The position of the P atom was determined from an unsharpened three-dimensional Patterson function and the locations of the other non-hydrogen atoms were found from a subsequent F_o map. Refinement of this model with isotropic thermal parameters converged at $R = 0{\cdot}12$. A difference Fourier synthesis calculated at this stage showed the positions of the H atoms bonded to the N and carbonyl C atoms. The methylene H atom positions were calculated and all these H atoms with isotropic temperature factors were included in a full-matrix refinement where the other atoms had anisotropic temperature factors; the weights for the individual reflexions were given by $w = (|F_o| + 0{\cdot}057F_o^2)^{-1}$. The refinement finally converged at $R = 0{\cdot}08$. Sheldrick's (1976) system on the Dalhousie University CDC 6400 computer was used for the calculations.

The final atomic parameters are in Table 1.* Table 2 lists the interatomic distances and interbond angles.

Table 1. *Atomic coordinates* (×10^4)

	x	y	z
P(1)	1814 (2)	2009 (2)	675 (1)
N(1)	3016 (5)	3191 (7)	711 (3)
O(1)	2169 (6)	826 (7)	253 (4)
O(2)	1438 (5)	1641 (6)	1479 (3)
O(3)	642 (5)	2778 (5)	343 (3)
O(4)	4485 (7)	1972 (7)	1369 (3)
C(1)	771 (7)	2685 (9)	1910 (4)
C(2)	−490 (7)	3116 (8)	1517 (4)
C(3)	−120 (7)	3734 (9)	809 (4)
C(4)	−1464 (9)	2018 (11)	1429 (6)
C(5)	−1102 (11)	4291 (11)	1991 (6)
C(6)	4216 (8)	3024 (10)	1042 (5)
H(1)	3190	4010	390
H(2)	4972	3705	1077
H(13)	418	4527	897
H(23)	−929	3975	537
H(11)	576	2351	2413
H(21)	1380	3479	1965

Table 2. *Interatomic distances* (Å) *and interbond angles* (°)

A prime indicates the equivalent position $\frac{1}{2} - x, \frac{1}{2} + y, z$.

P(1)–N(1)	1·702 (5)	O(1)–P(1)–O(2)	111·6 (3)
P(1)–O(1)	1·461 (6)	O(1)–P(1)–O(3)	112·7 (3)
P(1)–O(2)	1·570 (5)	O(2)–P(1)–O(3)	107·5 (3)
P(1)–O(3)	1·544 (4)	P(1)–N(1)–C(6)	124·8 (4)
N(1)–C(6)	1·373 (9)	P(1)–N(1)–H(1)	131·0
N(1)–H(1)	1·025	C(6)–N(1)–H(1)	101·4
O(2)–C(1)	1·476 (8)	N(1)–H(1)···O(1′)	145·2
O(3)–C(3)	1·499 (8)	P(1′)–O(1′)···H(1)	132·0
O(4)–C(6)	1·243 (9)	P(1)–O(2)–C(1)	116·8 (3)
C(1)–C(2)	1·533 (9)	P(1)–O(3)–C(3)	119·5 (3)
C(2)–C(3)	1·488 (9)	O(2)–C(1)–C(2)	109·3 (4)
C(2)–C(4)	1·488 (10)	C(1)–C(2)–C(3)	108·5 (5)
C(2)–C(5)	1·589 (11)	C(1)–C(2)–C(4)	113·6 (5)
		C(1)–C(2)–C(5)	106·2 (4)
N(1)···O(1′)	2·775 (7)	C(3)–C(2)–C(4)	112·4 (4)
N(1)–P(1)–O(1)	114·0 (2)	C(3)–C(2)–C(5)	105·6 (4)
N(1)–P(1)–O(2)	107·5 (2)	C(4)–C(2)–C(5)	110·2 (4)
N(1)–P(1)–O(3)	103·0 (2)	O(3)–C(3)–C(2)	110·3 (5)
		N(1)–C(6)–O(4)	120·8 (6)

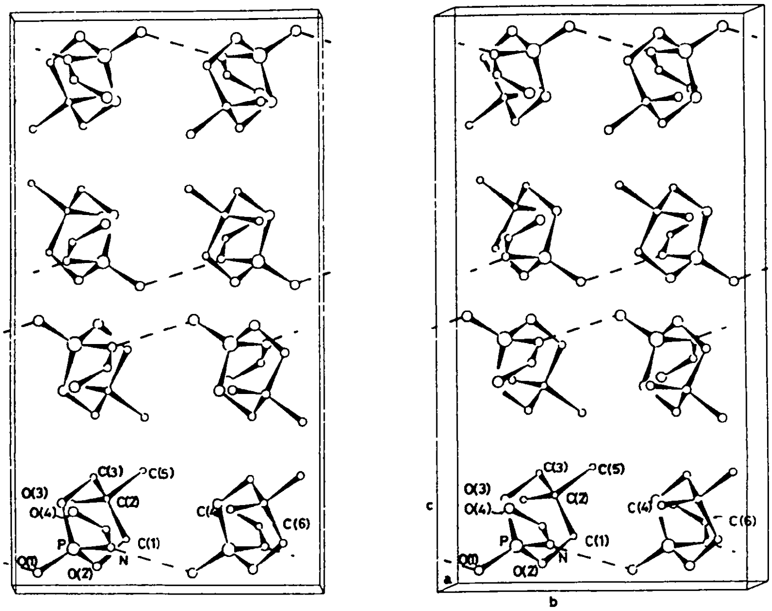

Fig. 1. A stereoscopic projection of the unit cell.

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32575 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Fig. 1 [drawn by *STRPL* (Cameron, 1973)] gives the packing of the molecules in the unit cell.

Discussion. The crystal is constructed from molecules with the formula I*a* (R = NHCHO), which are linked together to form chains parallel to **b** with a hydrogen bond between the phosphoryl oxygen atom O(1) and the amide nitrogen atom N(1). The chains stack side by side along **a** with the carbonyl group of one molecule fitting between the two methyl groups of a molecule in the adjacent stack. Along **c** the non-bonding contact between the stacks is made with the methyl and methylene groups of the phosphorinane ring (Fig. 1).

The phosphoryl O is equatorial, so the configuration at the P is apparently uninfluenced by the amido group. The P–O, P–N and N–C bond lengths of 1·461 (6), 1·702 (5) and 1·373 (9) Å can be compared with 1·476 (10), 1·649 (10) and 1·424 (9) Å in phenylaminophosphorinane (I*a*, R = NHPh) in which there is also a P=O···H–N hydrogen bond (Cameron & Karolak-Wojciechowska, 1976). The significant lengthening of only the P–N bond in the present compound is probably caused by the strong electron-withdrawing carbonyl group bonded to the N atom.

The configuration and dimensions of the dioxaphosphorinane ring are normal. The angle between the planes through P(1), O(2), O(3) and O(2), O(3), C(1) and C(3) is 32·1° and the angle between this second plane and that through C(1), C(2) and C(3) is 65·3°. The corresponding interplane angles in (I*a*) (R = NHPh) are 34·2 and 55·6°. The two P–O single bonds of 1·570 (6) Å [O(2)] and 1·544 (6) Å [O(3)] are not significantly different and are within the normal range (1·522–1·584 Å) for this type of compound (Gałdecki & Karolak-Wojciechowska, 1973). There is considerable spread in the C–C interatomic distances, particularly the C–Me, 1·488 (11) Å [C(4)] and 1·589 (10) Å [C(5)]. The methyl groups do not pack very closely (Fig. 1) and from the Fourier map it is clear that the methyl C atoms are strongly vibrating with ill defined positions.

We thank the Polish Ministry of Education and Technology for a scholarship to JK and the University of Dalhousie for computing facilities.

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Acta Cryst. (1977). B33, 2344–2346

1,10-Phenanthrotricyclo[4.1.1.0^{2,7}]heptene

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Abstract. $C_{18}H_{12}$. Orthorhombic, *Iba*2, a = 16·34 (1), b = 18·64 (1), c = 7·884 (5) Å, D_c = 1·26 g cm^{-3}. Z = 8. Full-matrix least-squares refinement gave R = 0·067 for 74 parameters and 409 significant reflexions [$I > 2\sigma(I)$]. The molecular geometry is normal, and the packing similar to that for 1,8-naphthotricyclo[4.1.1.0^{2,7}]heptene.

Introduction. The reaction of benz[*de*]anthracenyl anion with methylene chloride/*n*-butyllithium (Pagni, Burnett & Hazell, 1977) yielded 4,5-benzocyclohepta[1,2,3-*de*]naphthalene and an isomeric compound. NMR showed the compound to possess a bicyclobutane structure and to be either 1,10-phenanthrotricyclo[4.1.1.0^{2,7}]heptene (I) or 1,9-anthrotricyclo-