

**Discussion.** Atomic coordinates of non-H atoms are listed in Table 1.\* The bond lengths and angles for non-H atoms are given in Table 2. The molecular geometry depicted in Fig. 1 confirms the results of spectroscopic studies.

The phenyl ring, which is coplanar with the methoxy group, exhibits endocyclic bond angles in the range 118–122°, whereas the bond lengths forming the ring are close to the standard value [mean C<sub>ph</sub>—C<sub>ph</sub> distance 1.386 (3) Å]. The 1,3-biplanar pyran ring, made up of different C—C and C—O single and multiple bonds, assumes an almost perfect skew conformation. The corresponding puckering parameters (Cremer & Pople, 1975) are:  $Q = 0.379$  (4) Å,  $\varphi = 216.0$  (7)° and  $\theta = 115.2$  (6)° [starting from O(6) through C(5), etc. in a clockwise direction]. Atom C(5) has an almost perfect tetrahedral configuration [mean bond angle 109.5 (3.5)°] with C(11) and C(12) methyl groups in pseudo-axial and pseudo-equatorial positions, respectively.

The rather distorted dithiine ring exhibits a state transitional between <sup>4</sup>E-envelope and <sup>4</sup>S<sub>5</sub>-skew forms. This phenomenon can be attributed to the vigorous thermal motions of C(2) and C(3), presumably influenced by the presence of the two bulky S atoms. As the conformational disorder of the dithiine ring could not be resolved, its puckering cannot be discussed in detail. However, the unusually short C(2)—C(3) single bond of 1.358 (8) Å is evidence of this disorder. A similar phenomenon was found in both polymorphs of 2-phenyl-5,6-tetramethylenepyrimidine-4(3H)-thione (Kálmán, Argay, Lázár, Rudisch & Bernáth, 1985).

\* 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 53061 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

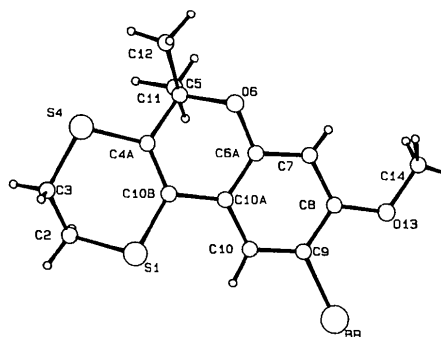


Fig. 1. Perspective view of the molecule showing atomic numbering. The H atoms are shown but not labelled.

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## Structure of 3-(4-Methoxyphenyl)-4-phenyl-4H-1,2,4-triazole

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**Abstract.** C<sub>15</sub>H<sub>13</sub>N<sub>3</sub>O,  $M_r = 251.29$ , triclinic,  $P\bar{1}$ ,  $a = 9.294$  (1),  $b = 11.394$  (4),  $c = 13.375$  (2) Å,  $\alpha = 103.60$  (2),  $\beta = 96.82$  (1),  $\gamma = 107.47$  (2)°,  $V =$

$1285$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.30$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 0.92$  cm<sup>-1</sup>,  $F(000) = 528$ ,  $T = 295$  K, final  $R = 0.045$  for 2311 observed [ $F_o \geq 5\sigma(F_o)$ ] reflections. There are two molecules in the asymmetric unit. The triazole rings are planar with the

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phenyl substituents twisted with respect to these planes. The two molecules in the asymmetric unit are different in the relative twists of the phenyl groups.

**Introduction.** Six 3,4-diaryl-4*H*-1,2,4-triazoles were previously prepared by the interaction of the appropriate diarylimidoyl chloride with diazomethyl-lithium (Kevill & Park, 1978). The assignment of five of these compounds as derivatives of 1,2,4-triazole, rather than of 1,2,3-triazole, was subsequently confirmed by the finding that materials prepared by the reaction of appropriate benzamidrazones with sodium hydride showed identical characteristics (m.p., <sup>1</sup>HMR). The sixth compound, the title compound, was not investigated in the study by Ito, Tanaka & Kakehi (1984) and its assignment as a 1,2,4-triazole is confirmed in the present study.

**Experimental.** Recrystallization from tetrachloromethane-hexane gave white needles, m.p. 414.5–415 K; <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 160.78, 153.02, 144.49, 134.82, 130.05, 129.93, 129.34, 125.77, 118.70, 114.01, 55.26. *D<sub>m</sub>* not determined. Crystal 0.10 × 0.25 × 0.38 mm. Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo Kα. Cell constants from setting angles of 25 reflections ( $\theta > 17^\circ$ ).  $\omega$ -2 $\theta$  scans. Correction for Lorentz-polarization effects.  $\theta_{\max} = 50^\circ$ ; *h* 0 to 11, *k* -13 to 13, *l* -15 to 15. Three standard reflections (400, 050, 007) observed every 3600 s of data-collection time, variation ± 1%. 4494 reflections measured, 2311 independent observed reflections [ $F_o \geq 5\sigma(F_o)$ ]. Structure solved utilizing SHELXS86 (Sheldrick, 1985) direct-methods program. Least-squares refinement with isotropic thermal parameters led to *R* = 0.114. The H atoms were located from a difference Fourier map and refined isotropically. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72, 99, 149), structure refined with SHELX76 (Sheldrick, 1976).  $\sum w(|F_o| - |F_c|)^2$  minimized,  $w = [\sigma(F_o)^2 + 0.016F_o^2]^{-1}$ , 447 parameters varied. *R* = 0.045, *wR* = 0.067, *S* = 0.22.  $\Delta/\sigma$  in final least-squares refinement cycle < 0.01,  $\Delta\rho < 0.2 \text{ e } \text{\AA}^{-3}$  in final difference map.

**Discussion.** Fractional coordinates and *B<sub>eq</sub>* values are given in Table 1,\* bond distances and angles in Table 2, and an ORTEP (Johnson, 1976) drawing in Fig. 1. A cell plot is provided in Fig. 2.

\* Lists of structure factors, anisotropic thermal parameters and least-squares-planes results have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53069 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final fractional coordinates for C<sub>15</sub>H<sub>13</sub>N<sub>3</sub>O

$$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}].$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B<sub>eq</sub></i> (Å <sup>2</sup> )
O(1)	0.1768 (3)	-0.0014 (2)	0.9470 (2)	4.31
N(1)	0.3149 (3)	0.4837 (2)	1.3594 (2)	3.04
N(2)	0.5566 (3)	0.6064 (3)	1.4186 (2)	4.27
N(3)	0.5490 (3)	0.5041 (3)	1.3348 (2)	3.94
C(1)	0.4043 (3)	0.4324 (3)	1.3012 (2)	3.06
C(2)	0.4162 (4)	0.5908 (3)	1.4296 (3)	3.70
C(3)	0.3446 (3)	0.3169 (3)	1.2097 (2)	2.99
C(4)	0.4012 (3)	0.2166 (3)	1.2040 (2)	3.05
C(5)	0.3490 (3)	0.1086 (3)	1.1180 (2)	3.01
C(6)	0.2380 (3)	0.1002 (3)	1.0352 (2)	3.00
C(7)	0.1816 (4)	0.2004 (3)	1.0400 (2)	3.84
C(8)	0.2333 (4)	0.3067 (3)	1.1254 (3)	3.74
C(9)	0.2244 (5)	-0.1105 (4)	0.9427 (3)	5.03
C(10)	0.1519 (3)	0.4345 (3)	1.3553 (2)	2.69
C(11)	0.0823 (4)	0.3076 (3)	1.3495 (2)	3.09
C(12)	-0.0739 (4)	0.2628 (3)	1.3476 (3)	3.48
C(13)	-0.1578 (4)	0.3446 (4)	1.3531 (3)	4.15
C(14)	-0.0865 (4)	0.4712 (4)	1.3608 (3)	4.45
C(15)	0.0687 (4)	0.5169 (3)	1.3609 (3)	3.73
O(2)	0.2271 (3)	0.5040 (2)	-0.0419 (2)	4.43
N(4)	0.3167 (2)	0.9870 (2)	0.3666 (2)	2.55
N(5)	0.5544 (3)	1.1178 (2)	0.4265 (2)	3.33
N(6)	0.5527 (3)	1.0195 (2)	0.3414 (2)	3.10
C(16)	0.4097 (3)	0.9416 (3)	0.3064 (2)	2.62
C(17)	0.4139 (3)	1.0948 (3)	0.4393 (2)	2.92
C(18)	0.3607 (3)	0.8267 (3)	0.2159 (2)	2.66
C(19)	0.4593 (3)	0.8141 (3)	0.1471 (2)	3.11
C(20)	0.4208 (3)	0.7086 (3)	0.0603 (2)	3.49
C(21)	0.2791 (4)	0.6124 (3)	0.0408 (2)	3.24
C(22)	0.1800 (4)	0.6222 (3)	0.1091 (3)	3.53
C(23)	0.2194 (3)	0.7282 (3)	0.1949 (3)	3.12
C(24)	0.3153 (5)	0.4925 (4)	-0.1215 (3)	5.04
C(25)	0.1512 (3)	0.9380 (3)	0.3572 (2)	2.47
C(26)	0.0569 (3)	0.9577 (3)	0.2799 (2)	3.31
C(27)	-0.1009 (4)	0.9099 (3)	0.2707 (3)	3.58
C(28)	-0.1625 (3)	0.8420 (3)	0.3364 (2)	3.25
C(29)	-0.0672 (3)	0.8249 (3)	0.4144 (2)	3.27
C(30)	0.0910 (3)	0.8727 (3)	0.4248 (2)	2.94
H(1)(C(2))	0.390 (3)	0.646 (3)	1.485 (2)	5.2 (7)*
H(1)(C(4))	0.481 (3)	0.224 (3)	1.259 (2)	5.5 (7)*
H(1)(C(5))	0.393 (3)	0.040 (3)	1.115 (2)	4.8 (6)*
H(1)(C(7))	0.113 (3)	0.195 (3)	0.984 (2)	5.0 (7)*
H(1)(C(8))	0.198 (3)	0.377 (3)	1.129 (2)	5.0 (6)*
H(1)(C(9))	0.163 (4)	-0.176 (3)	0.882 (3)	6.5 (8)*
H(2)(C(9))	0.204 (3)	-0.141 (3)	1.004 (2)	6.0 (7)*
H(3)(C(9))	0.333 (4)	-0.090 (4)	0.940 (3)	10 (1)*
H(1)(C(11))	0.143 (3)	0.256 (2)	1.341 (2)	4.5 (6)*
H(1)(C(12))	-0.122 (3)	0.173 (2)	1.337 (2)	4.2 (6)*
H(1)(C(13))	-0.267 (4)	0.311 (3)	1.351 (3)	6.7 (8)*
H(1)(C(14))	-0.151 (3)	0.526 (3)	1.360 (2)	5.2 (7)*
H(1)(C(15))	0.121 (4)	0.603 (3)	1.365 (3)	7.2 (8)*
H(1)(C(17))	0.378 (3)	1.157 (3)	0.493 (2)	5.0 (7)*
H(1)(C(19))	0.559 (3)	0.877 (2)	0.163 (2)	3.2 (5)*
H(1)(C(20))	0.501 (3)	0.710 (2)	0.017 (2)	3.5 (5)
H(1)(C(22))	0.081 (3)	0.557 (3)	0.100 (2)	5.2 (7)*
H(1)(C(23))	0.149 (3)	0.736 (2)	0.239 (2)	4.5 (6)*
H(1)(C(24))	0.250 (5)	0.401 (4)	-0.169 (3)	10 (1)*
H(2)(C(24))	0.418 (4)	0.494 (4)	-0.091 (3)	9 (1)*
H(3)(C(24))	0.328 (4)	0.568 (3)	-0.150 (3)	8.0 (9)*
H(1)(C(26))	0.100 (3)	0.998 (3)	0.232 (2)	5.2 (7)*
H(1)(C(27))	-0.171 (3)	0.922 (3)	0.214 (2)	6.2 (7)*
H(1)(C(28))	-0.272 (3)	0.806 (2)	0.332 (2)	4.2 (6)*
H(1)(C(29))	-0.109 (3)	0.776 (2)	0.458 (2)	4.5 (6)*
H(1)(C(30))	0.159 (3)	0.864 (2)	0.476 (2)	4.2 (6)*

\* Isotropic refinement.

There are two unique molecules in the asymmetric unit. Both molecules are identical except for the relative orientation of the phenyl rings. In both conformations the triazole and phenyl rings are planar to within 0.012 Å. The triazole bond distances and angles are internally consistent, with average values of N(1)—C(1), N(4)—C(16) = 1.372 (7) Å [interior angle at N = 104.5 (3)°]; N(1)—C(2),

Table 2. Bond distances (Å) and angles (°) for  $C_{15}H_{13}N_3O$ 

O(1)—C(6)	1.365 (3)	O(1)—C(9)	1.430 (6)
N(1)—C(1)	1.366 (4)	N(1)—C(2)	1.349 (3)
N(1)—C(10)	1.438 (4)	N(2)—N(3)	1.393 (4)
N(2)—C(2)	1.293 (5)	N(3)—C(1)	1.305 (4)
C(1)—C(3)	1.478 (4)	C(3)—C(4)	1.385 (5)
C(3)—C(8)	1.395 (5)	C(4)—C(5)	1.384 (4)
C(5)—C(6)	1.386 (4)	C(6)—C(7)	1.384 (6)
C(7)—C(8)	1.368 (4)	C(10)—C(11)	1.373 (4)
C(10)—C(15)	1.379 (5)	C(11)—C(12)	1.382 (5)
C(12)—C(13)	1.378 (6)	C(13)—C(14)	1.367 (6)
C(14)—C(15)	1.379 (5)	O(2)—C(21)	1.362 (3)
O(2)—C(24)	1.424 (5)	N(4)—C(16)	1.379 (4)
N(4)—C(17)	1.354 (3)	N(4)—C(25)	1.448 (3)
N(5)—N(6)	1.391 (4)	N(5)—C(17)	1.293 (4)
N(6)—C(16)	1.312 (3)	C(16)—C(18)	1.467 (4)
C(18)—C(19)	1.387 (5)	C(18)—C(23)	1.394 (4)
C(19)—C(20)	1.381 (4)	C(20)—C(21)	1.386 (4)
C(21)—C(22)	1.382 (5)	C(22)—C(23)	1.375 (4)
C(25)—C(26)	1.378 (5)	C(25)—C(30)	1.368 (5)
C(26)—C(27)	1.382 (4)	C(27)—C(28)	1.368 (5)
C(28)—C(29)	1.375 (5)	C(29)—C(30)	1.383 (4)
C(6)—O(1)—C(9)	117.3 (3)	C(1)—N(1)—C(2)	104.2 (2)
C(1)—N(1)—C(10)	129.8 (2)	C(2)—N(1)—C(10)	125.8 (3)
N(3)—N(2)—C(2)	106.3 (2)	N(2)—N(3)—C(1)	107.2 (2)
N(1)—C(1)—N(3)	110.2 (2)	N(1)—C(1)—C(3)	124.8 (2)
N(3)—C(1)—C(3)	124.9 (3)	N(1)—C(2)—N(2)	112.0 (3)
C(1)—C(3)—C(4)	120.6 (3)	C(1)—C(3)—C(8)	121.4 (3)
C(4)—C(3)—C(8)	118.0 (3)	C(3)—C(4)—C(5)	121.6 (3)
C(4)—C(5)—C(6)	119.5 (3)	O(1)—C(6)—C(5)	124.9 (3)
O(1)—C(6)—C(7)	115.8 (3)	C(5)—C(6)—C(7)	119.3 (3)
C(6)—C(7)—C(8)	120.9 (3)	C(3)—C(8)—C(7)	120.8 (4)
N(1)—C(10)—C(11)	119.8 (3)	N(1)—C(10)—C(15)	119.2 (3)
C(11)—C(10)—C(15)	121.0 (3)	C(10)—C(11)—C(12)	118.9 (3)
C(11)—C(12)—C(13)	120.5 (3)	C(12)—C(13)—C(14)	120.1 (3)
C(13)—C(14)—C(15)	120.1 (4)	C(10)—C(15)—C(14)	119.4 (3)
C(21)—O(2)—C(24)	118.7 (3)	C(16)—N(4)—C(17)	104.8 (2)
C(16)—N(4)—C(25)	130.2 (2)	C(17)—N(4)—C(25)	125.0 (3)
N(6)—N(5)—C(17)	106.9 (2)	N(5)—N(6)—C(16)	107.7 (2)
N(4)—C(16)—N(6)	109.2 (2)	N(4)—C(16)—C(18)	126.8 (2)
N(6)—C(16)—C(18)	124.0 (3)	N(4)—C(17)—N(5)	111.5 (3)
C(16)—C(18)—C(19)	118.9 (2)	C(16)—C(18)—C(23)	123.5 (3)
C(19)—C(18)—C(23)	117.7 (2)	C(18)—C(19)—C(20)	122.0 (2)
C(19)—C(20)—C(21)	119.2 (3)	O(2)—C(21)—C(20)	125.0 (3)
O(2)—C(21)—C(22)	115.3 (3)	C(20)—C(21)—C(22)	119.7 (3)
C(21)—C(22)—C(23)	120.5 (3)	C(18)—C(23)—C(22)	120.9 (3)
N(4)—C(25)—C(26)	119.4 (3)	N(4)—C(25)—C(30)	119.5 (3)
C(26)—C(25)—C(30)	121.1 (3)	C(25)—C(26)—C(27)	119.1 (3)
C(26)—C(27)—C(28)	120.3 (3)	C(27)—C(28)—C(29)	120.0 (3)
C(28)—C(29)—C(30)	120.3 (3)	C(25)—C(30)—C(29)	119.2 (3)

N(4)—C(17) = 1.352 (3) Å [interior angle at C = 111.8 (3)°]; C(2)—N(2), C(17)—N(5) = 1.293 (1) Å [interior angle at N = 106.6 (3)°]; N(2)—N(3), N(5)—N(6) = 1.392 (1) Å [interior angle at N(3), N(6) = 107.4 (3)°]; N(3)—C(1), N(6)—C(16) = 1.308 (4) Å [interior angle at C = 109.7 (5)°].

Crystallographic studies of the parent 1,2,4-triazole (Goldstein, Ladell & Abowitz, 1969; Jeffrey, Ruble & Yates, 1983; Toda, Tanaka, Elguero, Nassimbeni & Niven, 1987) have involved the more stable (Bolton, Brown, Burden & Mishra, 1971) 1*H*-1,2,4-triazole tautomer. A trisubstituted 4*H*-1,2,4-triazole derivative, 4-[5-ethyl-4-(4-methoxyphenyl)-4*H*-1,2,4-triazol-3-yl]-*N,N*-dimethylbenzamine, has also been studied (Cotrait, 1986); bond distances and angles within the triazole ring are similar to those of this study.

A derivative of the less stable 4*H*-1,2,4-triazole with the same substitution pattern as the title compound, 4-amino-3-(2-aminophenyl)-4*H*-1,2,4-

triazole, was investigated by Stam & van der Plas (1976). Despite the widely differing substituents in the 4-position (amino *versus* phenyl), the bond lengths and angles within the triazole ring are very similar for the two 3,4-disubstituted-4*H*-1,2,4-triazoles. This is consistent with the claim of a steric inhibition of resonance, associated with the non-coplanarity of the amino and phenyl groups with the triazole ring. Non-coplanarity of the phenyl groups is also observed in each of the two molecular conform-

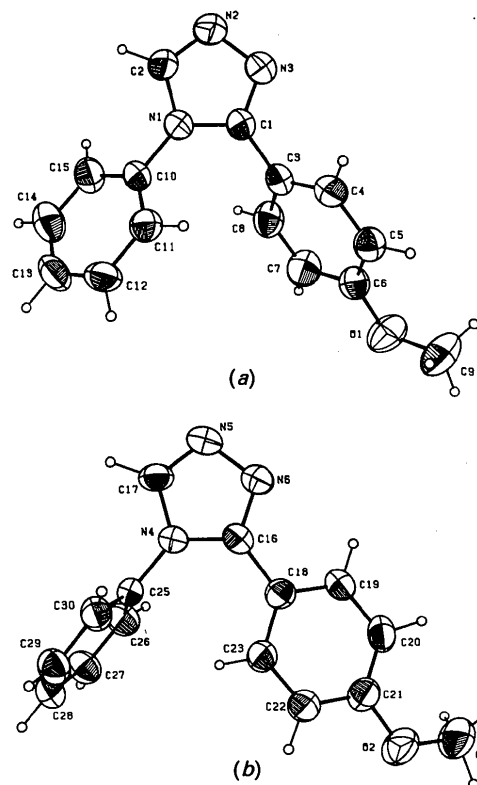


Fig. 1. (a), (b) ORTEP illustrations of the two unique molecules of the title compound.

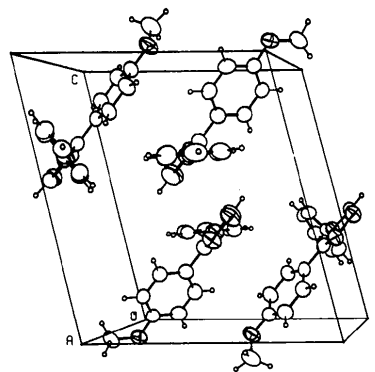


Fig. 2. Cell packing diagram.

mations observed for the title compound. The relative twists of the phenyl rings are different in the two unique conformations. In one conformation of the title compound [N(1)—N(3)] the plane of the triazole ring intersects the plane of the phenyl group at  $46.6^\circ$  and that of the methoxyphenyl group at  $54.6^\circ$ . The two phenyl substituent planes intersect at an angle of  $62.2^\circ$ . In the second conformation the methoxyphenyl ligand is much closer to the triazole plane intersecting at only  $17.2^\circ$ . A corresponding increase in the dihedral angle between the triazole and phenyl ring planes is observed ( $77.4^\circ$ ). The methoxyphenyl and phenyl ring planes intersect at  $73.5^\circ$ .

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## Structure of 2-(3,5-Dimethylphenoxy)-3-(4-methylphenyl)-3,4-dihydronaphth[1,2-*e*]-[1,3,2]oxazaphosphorinine 2-Oxide

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**Abstract.**  $C_{26}H_{24}NO_3P$ ,  $M_r = 429.4$ , monoclinic,  $P2_1/c$ ,  $a = 10.345$  (4),  $b = 20.705$  (2),  $c = 11.272$  (6) Å,  $\beta = 115.2$  (2)°,  $V = 2184.6$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.30$ ,  $D_m = 1.31$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 13.23$  cm<sup>-1</sup>,  $F(000) = 904$ ,  $T = 296$  K,  $R = 0.067$  for 3853 observed reflections. The phosphorine ring adopts a sofa conformation. The other phenyl and aromatic rings are planar. The phosphoryl oxygen O(2) occupies an equatorial position and the atom O(3) occupies an axial position on the phosphorine ring. The dimethylphenoxy and methylphenyl rings are *trans* to each other and they occupy equatorial positions on the phosphorine ring. The P=O(2) distance is  $1.45$  (3) Å. The molecular packing involves van der Waals contacts only.

**Introduction.** In a search for new anti-cancer drugs, synthesis of a large number of phosphorus heterocyclic compounds is reported in the literature and some of these compounds are found to possess significant anti-tumour activity (Chugani Pharmaceutical Co., 1966; Zimmer & Sill, 1964). The confor-

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mations and geometries of 1,3,2-dioxaphosphorinane have been reported by Wade Warrent, Caughlan, Howard Hargis, Yee & Bentrude (1978). The crystal and molecular structure of two derivatives of 1,3,2-benzoxazaphosphorine 2-oxide have been reported by Subramanian, Selladurai & Ponnuswamy (1989) and Selladurai, Subramanian & Nagaraju (1989). In each case the phosphorine ring assumes a conformation midway between a half-chair and a sofa. We now report the X-ray crystallographic results for a third phosphorine 2-oxide which supports the above findings.

**Experimental.** A crystal of dimensions  $0.45 \times 0.55 \times 0.75$  mm grown from ethanol ( $D_m$  by flotation), mounted on an Enraf–Nonius CAD-4 diffractometer, take-off angle  $5^\circ$ , graphite-crystal-monochromated Cu  $K\alpha$  radiation; unit-cell parameters from 25 accurately centered reflections ( $25 < \theta < 35^\circ$ ) by least-squares refinement. Intensities of 4461 reflections ( $-11 < h < 12$ ,  $0 < k < 25$ ,  $0 < l < 13$ ) measured using  $\omega$ - $2\theta$  scan;  $\theta_{\max} = 70^\circ$ . Intensities of two