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° and that of the methoxyphenyl group at 54·6°. The two phenyl substituent planes intersect at an angle of 62·2°. In the second conformation the methoxyphenyl ligand is much closer to the triazole plane intersecting at only 17·2°. A corresponding increase in the dihedral angle between the triazole and phenyl ring planes is observed (77·4°). The methoxyphenyl and phenyl ring planes intersect at 73·5°.

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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. $C_{26}H_{24}NO_3P$, $M_r = 429\cdot4$, monoclinic, $P2_1/c$, $a = 10\cdot345$ (4), $b = 20\cdot705$ (2), $c = 11\cdot272$ (6) Å, $\beta = 115\cdot2$ (2)°, $V = 2184\cdot6$ Å^3, $Z = 4$, $D_x = 1\cdot30$, $D_m = 1\cdot31$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 13\cdot23$ cm^{-1}, $F(000) = 904$, $T = 296$ K, $R = 0\cdot067$ 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-

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 $0\cdot45 \times 0\cdot55 \times 0\cdot75$ mm grown from ethanol (D_m by flotation), mounted on an Enraf–Nonius CAD-4 diffractometer, take-off angle 5°, graphite-crystal-monochromated Cu $K\alpha$ radiation; unit-cell parameters from 25 accurately centered reflections ($25 < \theta < 35°$) by least-squares refinement. Intensities of 4461 reflections ($-11 < h < 12$, $0 < k < 25$, $0 < l < 13$) measured using ω–2θ scan; $\theta_{max} = 70°$. Intensities of two

standard reflections monitored every 100 reflections showed no significant variation; 4241 unique reflections ($R_{int} = 0.00$) out of which 3853 reflections considered observed having $I > 3\sigma(I)$. Lorentz and polarization corrections applied. Structure solved by the direct methods using *SHELXS*86 (Sheldrick, 1986) and refinement was carried out by full-matrix least-squares method using *SHELX*76 (Sheldrick, 1976). All H atoms located from a difference Fourier map and isotropically refined. In the final stage of refinement, weights were introduced resulting in $R = 0.067$, $wR = 0.07$, $w = 0.1629/(\sigma^2|F| + 0.02232|F|^2)$; ratio of final shift to e.s.d. $= 0.09$. Final difference Fourier map featureless with $\Delta\rho$ within ± 0.12 e Å^{-3}. The atomic scattering factors used for all the atoms were as provided in the *SHELX*76 program. Computer programs: *PARST* (Nardelli, 1983) for geometrical calculations, *MOL-DRAW* (Ugliengo, Borzani & Viterbo, 1988) for molecular illustrations. All calculations performed on an VAX 11/780 computer.

Discussion. The atomic parameters with their e.s.d.'s and equivalent isotropic thermal parameters for non-H atoms are given in Table 1.* The molecular diagram with the adopted numbering scheme is shown in Fig. 1. Bond lengths and bond angles are shown in Figs. 2(a) and 2(b), respectively. Selected torsion angles are given in Table 2. The bond length P(1)=O(2) of 1.45 (3) Å agrees with those found in other related phosphorine structures (Subramanian, Selladurai & Ponnuswamy, 1989; Selladurai, Subramanian & Nagaraju, 1989). The average P—O bond length of 1.575 Å is quite similar to the bonds such as P—O in 1,3,2-dioxaphosphorinanyl (Wojciechowska, Wieczorek, Struchkov, Antipn, Mikolajczyk, Kielbasinski & Sut, 1983). The oxaza-phosphorine ring adopts a sofa conformation. The dihedral angle between the planes P(1)—N(5)—C(22) and P(1)—O(4)—C(21)—C(22) is 45.5 (2)° and between P(1)—O(4)—C(21)—C(22) and O(4)—C(12)—C(21) is 1.7 (3)°. The phosphoryl oxygen O(2) occupies an equatorial position and the atom O(3) occupies an axial position on the phosphorine ring. The torsion angle P(1)—N(5)—C(23)—C(28) [61.9 (3)°] indicates that the phenyl substituent on N(5) is rotated by about 92° (angle between phosphorine ring and phenyl ring is 122.7°) and the distance O(2)···C(28) is 3.075 (3) Å. Hence the conformation of the phosphorine

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* Lists of strucure factors, anisotropic thermal parameters, bond lengths and angles involving non-hydrogen atoms, least-squares-planes calculations and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53088 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters and equivalent isotropic thermal parameters of non-H atoms, with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å²)
P(1)	0.1872 (1)	0.3162 (0)	0.2613 (1)	3.9 (0)
O(2)	0.1576 (2)	0.2996 (1)	0.3725 (2)	5.0 (1)
O(3)	0.3229 (2)	0.2812 (1)	0.2606 (2)	4.8 (1)
O(4)	0.2352 (2)	0.3880 (1)	0.2614 (2)	4.8 (0)
N(5)	0.0527 (2)	0.3077 (1)	0.1174 (2)	3.9 (1)
C(6)	0.3528 (3)	0.2158 (1)	0.2959 (3)	4.2 (1)
C(7)	0.4145 (3)	0.2001 (1)	0.4284 (3)	4.4 (1)
C(8)	0.4496 (3)	0.1369 (1)	0.4654 (3)	4.7 (1)
C(9)	0.4226 (3)	0.0908 (1)	0.3689 (3)	5.2 (1)
C(10)	0.3598 (3)	0.1063 (1)	0.2351 (3)	5.0 (1)
C(11)	0.3247 (3)	0.1706 (2)	0.1996 (3)	4.8 (1)
C(12)	0.2024 (3)	0.4249 (1)	0.1485 (2)	3.9 (1)
C(13)	0.2512 (3)	0.4886 (1)	0.1749 (3)	4.7 (1)
C(14)	0.2251 (3)	0.5291 (1)	0.0718 (3)	4.9 (1)
C(15)	0.1502 (3)	0.5074 (1)	-0.0586 (3)	4.4 (1)
C(16)	0.1184 (4)	0.5494 (1)	-0.1666 (4)	5.6 (1)
C(17)	0.0445 (4)	0.5286 (2)	-0.2916 (4)	6.2 (1)
C(18)	0.0025 (4)	0.4645 (2)	-0.3150 (3)	5.8 (1)
C(19)	0.0311 (3)	0.4218 (1)	-0.2142 (3)	4.7 (1)
C(20)	0.1026 (2)	0.4423 (1)	-0.0827 (3)	3.8 (1)
C(21)	0.1289 (2)	0.4008 (1)	0.0264 (2)	3.6 (1)
C(22)	0.0734 (3)	0.3325 (1)	0.0033 (2)	4.2 (1)
C(23)	-0.0560 (3)	0.2605 (1)	0.0930 (2)	3.7 (1)
C(24)	-0.0818 (3)	0.2128 (1)	0.0006 (2)	4.5 (1)
C(25)	-0.1919 (3)	0.1695 (1)	-0.0248 (3)	4.8 (1)
C(26)	-0.2760 (3)	0.1709 (1)	0.0428 (3)	4.7 (1)
C(27)	-0.2469 (3)	0.2182 (1)	0.1378 (3)	4.8 (1)
C(28)	-0.1404 (3)	0.2637 (1)	0.1618 (3)	4.2 (1)
C(29)	-0.3919 (4)	0.1219 (2)	0.0155 (4)	6.9 (1)
C(20)	0.5166 (4)	0.1195 (2)	0.6073 (4)	6.5 (1)
C(31)	0.3305 (5)	0.0552 (2)	0.1320 (4)	7.0 (2)

Table 2. *Selected torsion angles* (°)

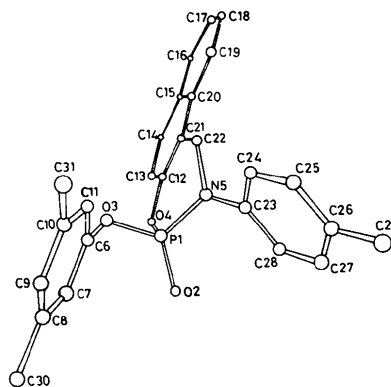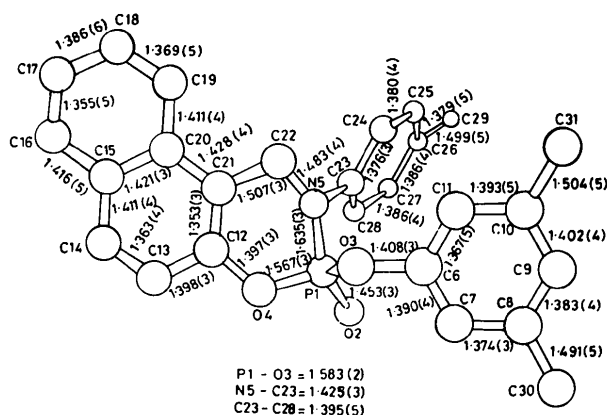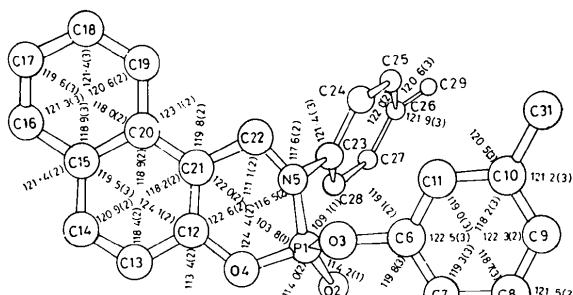|			
O(4)—P(1)—N(5)—C(22)	46.6 (2)	P(1)—N(5)—C(23)—C(28)	61.9 (3)
O(3)—P(1)—N(5)—C(22)	-58.0 (2)	P(1)—N(5)—C(22)—C(21)	-50.2 (3)
O(2)—P(1)—N(5)—C(22)	172.0 (2)	C(22)—N(5)—C(23)—C(24)	38.0 (4)
O(4)—P(1)—N(5)—C(23)	-155.4 (2)	C(22)—N(5)—C(23)—C(28)	-140.4 (3)
O(3)—P(1)—N(5)—C(23)	100.0 (2)	C(23)—N(5)—C(22)—C(21)	150.7 (2)
O(2)—P(1)—N(5)—C(23)	-30.0 (3)	O(3)—C(6)—C(11)—C(10)	177.4 (3)
O(3)—P(1)—O(4)—C(12)	92.2 (3)	O(3)—C(6)—C(7)—C(8)	-177.6 (3)
O(2)—P(1)—O(4)—C(12)	-146.3 (2)	O(4)—C(12)—C(21)—C(22)	1.7 (4)
O(2)—P(1)—O(3)—C(6)	40.9 (3)	O(4)—C(12)—C(21)—C(20)	-179.1 (2)
O(4)—P(1)—O(3)—C(6)	162.3 (2)	O(4)—C(12)—C(13)—C(14)	-179.9 (3)
N(5)—P(1)—O(3)—C(6)	-89.7 (3)	C(13)—C(12)—C(21)—C(22)	-177.4 (3)
N(5)—P(1)—O(4)—C(12)	-20.1 (3)	C(13)—C(12)—C(21)—C(20)	1.8 (4)
P(1)—O(3)—C(6)—C(7)	-78.8 (3)	C(12)—C(21)—C(22)—N(5)	24.6 (3)
P(1)—O(3)—C(6)—C(11)	103.4 (3)	C(20)—C(21)—C(22)—N(5)	-154.6 (2)
P(1)—O(4)—C(12)—C(13)	176.9 (2)	N(5)—C(23)—C(28)—C(27)	179.5 (3)
P(1)—O(4)—C(12)—C(21)	-2.3 (4)	N(5)—C(23)—C(24)—C(25)	-177.2 (3)
P(1)—N(5)—C(23)—C(24)	-119.8 (3)		

Fig. 1. The molecular diagram with the adopted atom-numbering scheme.

P1 - O3 = 1·583 (2)
N5 - C23 = 1·425 (3)
C23 - C28 = 1·395 (5)

(a)

C25 - C26 - C27 = 117·4 (3)
C26 - C27 - C28 = 121·6 (3)
O2 - P1 - N5 = 115·3 (2)
O3 - P1 - O4 = 98·8 (2)
P1 - O3 - C6 = 121·8 (2)
N5 - C23 - C28 = 119·3 (2)
C23 - C24 - C25 = 120·0 (3)
P1 - N5 - C23 = 122·2 (2)
C23 - C28 - C27 = 119·5 (3)

(b)

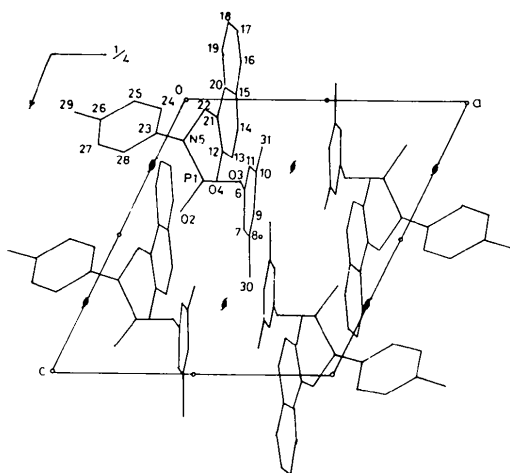Fig. 2. (a) Bond lengths (Å) and (b) bond angles (°).

Fig. 3. The packing of the molecules in the unit cell.

ring is forced to be a sofa; the above rotation occurs so as to avoid interactions with the O(2) atom (Acharya, Tavale, Guru Row, Sahasrabudhe & Tilak, 1984). The dimethylphenyl and methylphenyl rings are *trans* to each other and they occupy axial positions on the phosphorine ring. The P—N bond length and the average P—N—C angle, 1·635 Å and 119°, are in agreement with those in the related structure reported by Acharya, Tavale & Guru Row (1984). The average C—N bond is 1·454 Å, in good agreement with the accepted value. The average C—C distance in the phenoxy group is 1·384 (4) Å and the average angle 120·3 (3)°. Least-squares-planes calculations show that the phenyl rings are planar (maximum deviation 0·01 Å) and that the aromatic rings of the main skeleton are planar within 0·02 Å. The least-squares plane of the dimethylphenoxy ring is included at an angle of 145·2 (1)° with the least-squares plane of the phosphorine ring. The dihedral angle between the two phenyl rings is 72 (1)°. Fig. 3 shows the packing of the molecules in the unit cell. The packing of the molecules in the unit cell essentially involves van der Waals contacts.

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