2·626 (7) Å with the corresponding angle 153 (5)°]. Furthermore, the C(3)—C(4) bond of 1·343 (8) Å has double-bond character and therefore the possible tautomeric keto form of this compound can be ruled out. The benzene ring is planar with the largest deviation from the mean plane 0·014 (7) Å for C(5). The atomic group O(1),C(4),C(3),C(11),O(4) is planar to within 0·023 (7) Å due to the formation of the intramolecular hydrogen bond. The methyl group C(12)H_3 is in the expected *trans* orientation with respect to C(3), the corresponding torsion angle C(3)—C(11)—O(5)—C(12) being −178·0 (5)°. The thiazine ring is in a distorted half-chair conformation with endocyclic torsion angles −49·5 (5) about S(1)—N(2), 39·5 (8) N(2)—C(3), −1·6 (10) C(3)—C(4), −18·8 (10) C(4)—C(10), −0·0 (8) C(9)—C(10), 31·0 (6)° S(1)—C(9).

There are no noticeably short intermolecular contacts in the structure.

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References

Bordner, J., Richards, J. A., Weeks, P. & Whipple, E. B. (1984). *Acta Cryst.* C**40**, 989–990.

Golič, L. & Koželj, M. (1984). *Annu. Yugosl. Cent. Crystallogr.* **19**, 60.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Kojić-Prodić, B. & Ružić-Toroš, Ž. (1982). *Acta Cryst.* B**38**, 2948–2951.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L. Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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Structure of 4-*tert*-Butyl-1-phenoxy-1-λ^5-phosphorinane 1-Oxide

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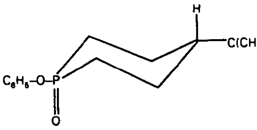
Abstract. $C_{15}H_{23}O_2P$, $M_r = 266{\cdot}32$, monoclinic, $P2_1/a$, $a = 12{\cdot}511$ (2), $b = 8{\cdot}695$ (1), $c = 15{\cdot}035$ (5) Å, $\beta = 111{\cdot}63$ (2)°, $V = 1520{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}16$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 15{\cdot}24$ cm^{-1}, $F(000) = 576$, $T = 298$ K, $R = 0{\cdot}0578$ for 2261 observed reflections. A chair conformation for the heterocyclic ring with slight flattening at the phosphorus end to relieve steric strain was found in this compound. The phenoxy and *tert*-butyl groups are *trans* to each other and in equatorial positions.

Introduction. The stereochemistry of two phosphorinane oxides has been reported by Macdonell, Berlin, Baker, Ealick, van der Helm and Marsi (1978), and by Mazhar-ul-Haque, Ahmed & Horne (1986). The structure of a phosphorinane sulfide has been reported by Quin, McPhail, Lee & Onan (1974). In each case the phosphorinane ring assumes a chair conformation, slightly flattened at the phosphorus end relative to that of the carbocyclic analogue cyclohexane. It is observed that the positioning of the larger substituent on P is important in determining the extent of flattening. We now report the X-ray crystallographic results for a third phosphorinane oxide (I) which supports the above findings.



(I)

Experimental. A crystal *ca* 0·43 × 0·50 × 0·75 mm grown from chloroform, mounted on an Enraf–Nonius CAD-4 diffractometer, take-off angle 5°, graphite-crystal-monochromatized Cu $K\alpha$ radiation used for the determination of unit-cell parameters from 25 accurately centered reflections (27 < θ < 33°) by least-squares refinement. Intensities of 2981 reflections

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($0 < h < 14$, $0 < k < 10$, $-16 < l < 16$) measured, using ω–2θ scan; $\theta_{\max} = 65°$. Intensities of three standard reflections monitored at intervals of 9000 s showed no significant variation; 2582 unique out of which 2261 reflections considered observed having $I > 3\sigma(I)$. Lorentz and polarization corrections applied. Structure solved by direct methods, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealing positions of all non-H atoms. $R_{\rm int} = 0{\cdot}0128$ for merging equivalent reflections. H atoms located from a difference Fourier map. F magnitudes used in full-matrix least-squares refinement of non-H atoms with anisotropic temperature factors, and H atoms isotropically *SHELX*76 (Sheldrick, 1976). No absorption correction was applied, extinction corrections minimized the R value significantly with final extinction parameter $x = 2{\cdot}60 \times 10^{-2}$. Significant improvements were also observed in the standard deviations of some of the bond lengths and angles involving both non-H and H atoms. Weighting scheme of the form $w = 1{\cdot}0/(\sigma^2|F_o| + 0{\cdot}01374F_o^2)$ used to minimize $\sum w\Delta F^2$, final $R = 0{\cdot}0578$, $wR = 0{\cdot}083$, 255 parameters, max. (shift/e.s.d.) = 0·069, max. peak height in final difference Fourier map = 0·30 e Å^{-3}, $S = 1{\cdot}04$. Form factors for non-H atoms from Cromer & Mann (1968); for H atoms from Stewart, Davidson & Simpson (1965). Calculations on IBM 3033 computer of the University of Petroleum and Minerals.

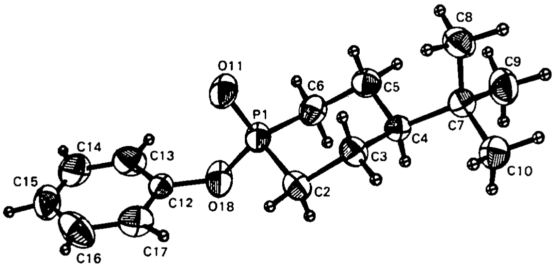

Fig. 1. *ORTEP* diagram of the $C_{15}H_{23}O_2P$ molecule.

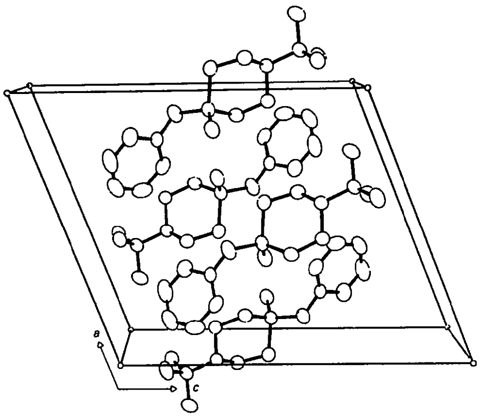

Fig. 2. The unit cell, showing the packing of molecules.

Discussion. Fig. 1 is an *ORTEP* diagram (Johnson, 1976) of the molecule illustrating 30% probability ellipsoids for the non-H atoms and arbitrary spheres of radius 0·1 Å for H atoms. It also shows the conformation and numbering scheme for the title compound. The crystal packing is shown in Fig. 2. The positional parameters together with their U values for non-H atoms are given in Table 1. Selected bond lengths, angles and torsion angles for the title compound are contained in Table 2.*

Table 1. *Fractional atomic positions* (× 10^4) *with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* (Å^2 × 10^3) *for non-hydrogen atoms*

$U_{\rm eq} = \frac{1}{3}$(trace of orthogonalized U_{ij} matrix).

	x	y	z	$U_{\rm eq}$
P(1)	9225 (1)	7452 (1)	5354 (0)	68
C(2)	8944 (2)	6003 (3)	6076 (2)	72
C(3)	9463 (2)	6463 (3)	7139 (2)	69
C(4)	10777 (2)	6650 (3)	7505 (2)	59
C(5)	11094 (2)	7964 (3)	6961 (2)	71
C(6)	10738 (3)	7673 (3)	5883 (2)	74
C(7)	11337 (2)	6820 (3)	8609 (2)	66
C(8)	10966 (3)	8290 (4)	8968 (2)	82
C(9)	12650 (3)	6857 (5)	8923 (2)	94
C(10)	11024 (4)	5456 (4)	9107 (2)	97
O(11)	8552 (2)	8865 (2)	5254 (1)	59
C(12)	8117 (2)	6923 (3)	3521 (2)	92
C(13)	8266 (4)	7795 (4)	2824 (3)	120
C(14)	7397 (6)	8001 (5)	1973 (3)	110
C(15)	6366 (5)	7394 (5)	1795 (3)	118
C(16)	6193 (3)	6489 (6)	2482 (4)	89
C(17)	7094 (3)	6248 (4)	3367 (2)	90
O(18)	9064 (2)	6651 (3)	4341 (1)	97

Table 2. *Selected bond lengths* (Å), *angles* (°), *and torsion angles* (°) *involving non-*H *atoms, with their e.s.d.'s in parentheses*

C(2)–P(1)	1·781 (3)	C(7)–C(4)	1·553 (3)
C(6)–P(1)	1·772 (3)	C(6)–C(5)	1·536 (4)
O(11)–P(1)	1·466 (2)	C(8)–C(7)	1·525 (4)
O(18)–P(1)	1·618 (2)	C(9)–C(7)	1·533 (4)
C(3)–C(2)	1·540 (4)	C(10)–C(7)	1·528 (4)
C(4)–C(3)	1·537 (3)	O(18)–C(12)	1·380 (3)
C(5)–C(4)	1·540 (4)		

C(6)--P(1)–C(2)	102·8 (1)	C(6)–C(5)–C(4)	114·0 (2)
O(11)–P(1)–C(2)	114·8 (1)	C(5)–C(6)–P(1)	109·7 (2)
O(11)–P(1)–C(6)	115·7 (1)	C(8)–C(7)–C(4)	112·5 (2)
O(18)–P(1)–C(2)	106·6 (1)	C(9)–C(7)–C(4)	109·9 (2)
O(18)–P(1)–C(6)	102·2 (1)	C(9)–C(7)–C(8)	107·8 (2)
O(18)–P(1)–O(11)	113·3 (1)	C(10)–C(7)–C(4)	110·7 (2)
C(3)–C(2)–P(1)	110·3 (2)	C(10)–C(7)–C(8)	108·1 (3)
C(4)–C(3)–C(2)	112·7 (2)	C(10)–C(7)–C(9)	107·7 (3)
C(5)–C(4)–C(3)	109·8 (2)	O(18)–C(12)–C(13)	117·9 (3)
C(7)–C(4)–C(3)	113·2 (2)	O(18)–C(12)–C(17)	121·8 (3)
C(7)–C(4)–C(5)	113·2 (2)	C(12)–O(18)–P(1)	122·4 (2)

P(1)–C(2)–C(3)–C(4)	−61·3 (4)	C(4)–C(5)–C(6)–P(1)	60·7 (4)
C(2)–C(3)–C(4)–C(5)	62·8 (4)	C(5)–C(6)–P(1)–C(2)	−52·7 (5)
C(3)–C(4)–C(5)–C(6)	−63·1 (4)	C(6)–P(1)–C(2)–C(3)	53·6 (4)

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* Lists of H-atom coordinates, full lists of bond lengths and angles, together with structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43337 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The heterocyclic ring in the title compound adopts a chair conformation similar to that of cyclohexane, but with slight flattening at the phosphorus end. The *tert*-butyl and phenoxy groups are *trans* to each other and occupy equatorial positions on the phosphorinane ring. The chair conformation in this compound has similar angles to those of *trans*-4-*tert*-butyl-1-phenylphosphorinane 1-oxide (Macdonell *et al.*, 1978) and 1-anilino-4-*tert*-butyl-1-λ^5-phosphorinane 1-oxide (Mazhar-ul-Haque, Ahmed & Horne, 1986). Thus the torsion angle C(6)—P(1)—C(2)—C(3) of 53·6° compares well with the values of 54·7 and 53·2° in the above compounds, and this angle, as expected, is larger than the mean value of *ca* 45° found for cases in which a bulky substituent on phosphorus is axially oriented with respect to the ring (Quin, 1980). The diminution in angle under these circumstances is a direct result of ring flattening induced as a mechanism to relieve steric strain. The P(1)—C(2) and P(1)—C(6) distances of 1·781 (3) and 1·772 (3) Å are significantly shorter than a typical P—C bond of 1·872 Å but are similar to the values of 1·791 (2) and 1·795 (2) Å reported for *trans*-4-*tert*-butyl-1-phenylphosphorinane 1-oxide (Macdonell *et al.*, 1978), and 1·782 (2) and 1·772 (3) Å reported for 1-anilino-4-*tert*-butyl-1-λ^5-phosphorinane 1-oxide (Mazhar-ul-Haque, Ahmed & Horne, 1986). The P(1)—O(11) distance of 1·466 Å is not significantly different from the 1·483 and 1·478 Å found in the above compounds, or the average value of 1·462 Å reported by Corbridge (1974) for a large sample of compounds containing highly electronegative atoms attached to a phosphorus atom. The average C—C distance in the phenoxy group is 1·362 Å and the average angle 120·0 (3)°.

An examination of intermolecular contacts revealed only one distance less than 3·5 Å. This was a distance of 3·24 Å between C(17) and O(18) at $(1{\cdot}5-x,\ y-0{\cdot}5,\ 1-z)$.

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References

Corbridge, D. E. C. (1974). *The Structural Chemistry of Phosphorus*, pp. 215–217, and references cited therein. New York: American Elsevier.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

Macdonell, G. D., Berlin, K. D., Baker, J. R., Ealick, S. E., van der Helm, D. & Marsi, K. L. (1978). *J. Am. Chem. Soc.* **100**, 4535–4540.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Mazhar-ul-Haque, Ahmed, J. & Horne, W. (1986). *Acta Cryst.* C**41**, 975–977.

Quin, L. D. (1980). *The Heterocyclic Chemistry of Phosphorus*, pp. 368–370, and references cited therein. New York: John Wiley.

Quin, L. D., McPhail, A. T., Lee, S. O. & Onan, K. D. (1974). *Tetrahedron Lett.* pp. 3473–3476.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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Structure du Morpholino *o*-Nitrobenzènesélénénamide*

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(*Reçu le* 10 *juin* 1985, *accepté le* 16 *septembre* 1986)

Abstract. $C_{10}H_{12}N_2O_3Se$, $M_r = 286{\cdot}96$, triclinic, $P\bar{1}$, $a = 8{\cdot}039\ (1)$, $b = 8{\cdot}832\ (2)$, $c = 9{\cdot}283\ (2)$ Å, $\alpha = 117{\cdot}29\ (2)$, $\beta = 90{\cdot}24\ (1)$, $\gamma = 99{\cdot}24\ (1)°$, $V = 573{\cdot}7\ (1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}66$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 33{\cdot}8$ cm^{-1}, $F(000) = 144$, 294 K, $R = 0{\cdot}058$ for 1477 reflections. The morpholine ring

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* Séléniure de morpholino et d'*o*-nitrophényle.