

Fig. 2. Conformations and asymmetry parameters (Duax & Norton, 1975) of the phospholane rings in 2-hydroxy-1-phenylphospholane 1-oxide derivatives.

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3-Methoxy-1,2-diphenyl-2-phospholene 1-Oxide

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Abstract. $C_{17}H_{17}O_2P$, $M_r = 284.3$, triclinic, $P\bar{1}$, $Z = 2$, $a = 9.55(5)$, $b = 6.19(4)$, $c = 14.07(5)$ Å, $\alpha = 91.3(3)$, $\beta = 95.8(5)$, $\gamma = 116.5(5)^\circ$, $V = 738.5$ Å³, $D_o = 1.274$ (by flotation), $D_c = 1.278$ Mg m⁻³, $F(000) = 300$. The structure was solved with *SHELX* and refined by full-matrix least squares to a final R of 0.100 for 1070 visually estimated intensities collected on a Weissenberg camera. The phospholene ring is nearly planar with asymmetry parameters $\Delta C_2^4 = 0.4(8)$, $\Delta C_2^1 = 0.9(8)$, $\bar{\varphi} = 0.7(8)^\circ$. The phenyl group attached to C(2) is not coplanar with the phospholene ring but is tilted away from the methoxy group as indicated by the torsional angle $P(1)-C(2)-C(21)-C(26) = 14.4^\circ$, whereas the phenyl ring attached to P is nearly perpendicular to the phospholene ring with an angle between the normals to these planes of 90.9° .

Introduction. The title compound was a model for studying the mechanism of 2-phospholene ring for-

mation. In addition, this study is a continuation of X-ray investigations of the structure and conformation of the 2-phenyl-2-phospholene 1-oxide system (Galdecki, 1979; Galdecki & Głowska, 1980a,b).

Colourless crystals of the compound were kindly supplied by Dr K. M. Pietrusiewicz. Unit-cell parameters were calculated from high-order reflections on Weissenberg photographs. The total of 1070 observed reflections were collected on the photographs (equi-inclination Weissenberg camera and multiple-film method) by means of Ni-filtered Cu radiation from the unshaped crystal of approximate dimensions $0.3 \times 0.5 \times 1.0$ mm. The intensities of the reflections were estimated visually. The data were not corrected for absorption.

The structure of the crystal has been solved by direct methods. All calculations have been performed by means of the *SHELX* program (Sheldrick, 1976). Full-matrix least-squares refinement of the model

Table 1. Atomic positions and isotropic temperature factors for P, O and C atoms

	x	y	z	B_{iso} (\AA^2)
P(1)	1.0796 (4)	0.6282 (6)	0.1986 (2)	2.6 (1)
O(1)	1.1376 (10)	0.8812 (15)	0.1737 (5)	3.9 (2)
O(2)	0.6965 (10)	0.0887 (14)	0.0778 (5)	3.7 (2)
C(1)	0.6668 (16)	-0.1374 (24)	0.0175 (9)	4.4 (3)
C(2)	0.8646 (12)	0.4332 (18)	0.1744 (7)	1.9 (2)
C(3)	0.8447 (14)	0.2500 (20)	0.1123 (7)	2.8 (2)
C(4)	0.9792 (14)	0.2262 (22)	0.0772 (8)	3.4 (3)
C(5)	1.1300 (15)	0.4417 (22)	0.1235 (8)	3.6 (3)
C(11)	1.1395 (10)	0.6138 (11)	0.3215 (5)	1.8 (2)
C(12)	1.2249 (10)	0.8303 (11)	0.3786 (5)	3.9 (3)
C(13)	1.2771 (10)	0.8262 (11)	0.4745 (5)	5.0 (3)
C(14)	1.2439 (10)	0.6058 (11)	0.5134 (5)	5.3 (3)
C(15)	1.1585 (10)	0.3893 (11)	0.4563 (5)	5.0 (3)
C(16)	1.1063 (10)	0.3933 (11)	0.3604 (5)	4.0 (3)
C(21)	0.7568 (9)	0.5029 (10)	0.2152 (5)	2.3 (2)
C(22)	0.6003 (9)	0.3311 (10)	0.2154 (5)	2.9 (3)
C(23)	0.4947 (9)	0.3946 (10)	0.2554 (5)	3.7 (3)
C(24)	0.5456 (9)	0.6299 (10)	0.2952 (5)	4.4 (3)
C(25)	0.7020 (9)	0.8017 (10)	0.2950 (5)	4.4 (3)
C(26)	0.8076 (9)	0.7382 (10)	0.2550 (5)	3.0 (2)

Table 2. Bond lengths (\AA) and angles ($^\circ$)

Both phenyl rings were refined as rigid groups with bond lengths of 1.395 \AA and valency angles of 120.0 $^\circ$.

P(1)—O(1)	1.474 (9)	P(1)—C(11)—C(16)	121.6 (2)
P(1)—C(2)	1.848 (11)	O(1)—P(1)—C(2)	116.8 (5)
P(1)—C(5)	1.795 (12)	O(1)—P(1)—C(5)	115.0 (5)
P(1)—C(11)	1.783 (8)	O(1)—P(1)—C(11)	111.1 (4)
C(2)—C(3)	1.347 (14)	C(1)—O(2)—C(3)	120.3 (10)
C(3)—C(4)	1.487 (16)	C(2)—C(3)—O(2)	118.2 (11)
C(4)—C(5)	1.528 (16)	C(2)—C(21)—C(22)	119.2 (5)
C(2)—C(21)	1.443 (13)	C(2)—C(21)—C(26)	120.8 (5)
C(3)—O(2)	1.346 (13)	C(3)—C(2)—C(21)	135.4 (10)
C(1)—O(2)	1.517 (15)	C(4)—C(3)—O(2)	119.0 (10)
		C(2)—P(1)—C(5)	94.8 (5)
P(1)—C(2)—C(3)	106.2 (8)	C(2)—C(3)—C(4)	122.6 (11)
P(1)—C(2)—C(21)	120.2 (6)	C(3)—C(4)—C(5)	107.1 (10)
P(1)—C(11)—C(12)	118.4 (2)	C(4)—C(5)—P(1)	109.3 (9)

structure resulted in an R of 0.153 after three cycles with isotropic thermal parameters. The phenyl groups were treated as rigid groups with fixed bond lengths and valency angles (Table 2). The final R factor was 0.100 after three additional cycles of refinement with rigid phenyl rings. The H atoms were located geometrically and were not included in the refinement. The calculations were performed on an ODR-1305 computer.* The atomic parameters are in Table 1 and a view of the molecule is shown in Fig. 1.

* Lists of structure factors and H-atom positional parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35685 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

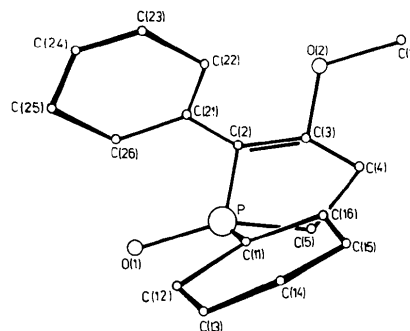


Fig. 1. A view of a molecule of the title compound.

Discussion. On comparing the bond lengths and valency angles in this structure (IV) with those found in similar compounds (Fig. 2), good agreement is observed when experimental errors are taken into account. The most interesting result is the planarity of the phospholene ring, the asymmetry parameters (Duax & Norton, 1975) being: $\Delta C_2^4 = 0.3$ (8), $\Delta C_5^1 = 0.9$ (8), $\bar{\varphi} = 0.7$ (8) $^\circ$. The torsional angle $\varphi_{2,3}$ is 1.3 $^\circ$, which indicates a weak repulsion between the C(2)-phenyl and the C(3)-methoxy groups. Very similar values were found in other phospholene rings examined by us: 2.0 $^\circ$ in (II) (7-benzyl-4-*tert*-butyl-9-hydroxy-8-phenyl-7-phospha-*cis*-bicyclo[4.3.0]non-8-ene *anti*-7-oxide) (Gałdecki & Głowska, 1980b); 2.8 $^\circ$ in (III) (7-benzyl-9-methoxy-8-phenyl-7-phospha-*cis*-bicyclo[4.3.0]non-8-ene *anti*-7-oxide) (Gałdecki, 1979); and 1.1 $^\circ$ in (V) (4-hydroxy-1-phenyl-2-phospholene 1-oxide) (Gałdecki & Głowska, 1980a). Unexpectedly, van der Helm, Washecheck, Burks & Ealick (1976) reported a value of 11.4 $^\circ$ in 1-benzyl-3-hydroxy-4,5-dimethyl-2-phenyl-2-phospholene 1-oxide (I) although the structure is very close to (II) and (III); moreover, this torsional angle, connected with a double bond, is the highest value of the ring torsional angles in (I). Such a large deviation from planarity of the double-bond system in (I) (van der Helm *et al.*, 1976) can thus be assumed to result from intermolecular rather than intramolecular interactions.

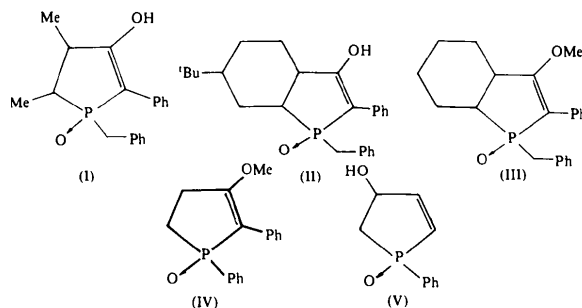


Fig. 2. The title compound (IV) and related structures.

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5,5'-Dichloro-3,3'-dinitro-2,2'-biphenyldiol*

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Abstract. $C_{12}H_6Cl_2N_2O_6$, monoclinic, $C2/c$, $a = 17.114$ (2), $b = 7.343$ (1), $c = 11.676$ (1) Å, $\beta = 118.11$ (1)°, $U = 1294.2$ (5) Å³, $D_m = 1.77$ (1), $D_c = 1.77$ Mg m⁻³, $Z = 4$. $\mu(Cu K\alpha) = 4.67$ mm⁻¹; $R = 0.047$ for 969 observed reflections. The molecule has exact 2 symmetry and the dihedral angle between the perpendiculars to the phenyl rings is 43.6 (5)°.

Introduction. Niclofolan, the title compound, also known as Bilevon, Dertil, Distolan and Menichlophan (Bard, 1972), is an anthelmintic (worm-killing) drug used against *Fasciola hepatica* (liver-fluke) infections in sheep and cattle (Australian Bureau of Animal Health, 1977). It is recognized that biphenol and bisphenol flukicides act by inhibiting ATP production in the flukes, and the active site is thought to be the succinate dehydrogenase complex (Metzger & Düwel, 1973). The molecular conformation of the biphenol reported here provides a comparison with those of several bisphenol flukicide structures reported earlier (Hay & Mackay, 1979, 1980; Hay, DeMunk & Mackay, 1980). The latter, which contain a bridging C atom, have the same ring substituents as niclofolan in which the phenyl rings are directly linked.

Weissenberg photographs showed that the blade-like crystals grown from ethanol were monoclinic, and systematic absences indicated either the space group $C2$ or $C2/c$ (subsequently confirmed as $C2/c$). Intensities were measured with Cu $K\alpha$ radiation (graphite-crystal monochromator, $\lambda = 1.5418$ Å) from a crystal $0.40 \times 0.14 \times 0.04$ mm mounted on a Rigaku-AFC

four-circle diffractometer. Of the 1287 non-equivalent terms ($2\theta_{\max} = 130^\circ$) recorded by an ω - 2θ scan of 2° min⁻¹ and with 10s stationary background counts, the 969 for which $|F_o| > 2\sigma|F_o|$ were used for the structure refinement. The intensities were corrected for Lorentz and polarization effects and for absorption; no corrections for extinction were made. The scattering factors for Cl, O, N and C were from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were made for the non-hydrogen atoms with the values of Cromer & Liberman (1970).

The structure was solved by direct methods with *SHELX* 76 (Sheldrick, 1976). Full-matrix least-squares refinement with individual isotropic temperature factors for the non-hydrogen atoms yielded $R = 0.15$. After refinement of these atoms with anisotropic temperature factors, the H atoms were located on a difference map. Final refinement, in which the positional parameters and isotropic temperature factors of the H atoms were allowed to vary, converged at $R = 0.047$ and $R_w = (\sum w|F_o| - |F_c|^2 / \sum w|F_o|^2)^{1/2} = 0.063$. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with the terms weighted according to $(\sigma^2|F_o| + 0.003|F_o|^2)^{-1}$. The final difference map contained no peaks >0.3 e Å⁻³; the largest peaks were near the Cl atom. Final atomic coordinates are given in Table 1.† Figs. 1 and 2 have been prepared with *ORTEP* (Johnson, 1965).

* Bisphenol Flukicides. III. Part I: Hay & Mackay (1979, 1980). Part II: Hay, DeMunk & Mackay (1980).

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35664 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.