

pourront être utilisées pour l'étude ultérieure de relations entre la structure spatiale et l'activité pharmacologique et pour simuler les interactions avec d'éventuels récepteurs biologiques.

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2-Hydroxy-1-phenylphospholane 1-Oxide

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Abstract. $C_{10}H_{13}O_2P$, $M_r = 196.2$, monoclinic, $P2_1/c$, $Z = 4$, $a = 6.331$ (2), $b = 8.600$ (2), $c = 18.970$ (3) Å, $\beta = 103.26$ (2)°, $V = 1005.4$ Å³, $D_x = 1.30$, $D_m = 1.29$ Mg m⁻³, $F(000) = 416$, $\mu = 2.13$ mm⁻¹. The structure was solved by direct methods and refined to a final R of 0.043 for 1333 reflections. The molecules form dimers with an O...O distance of 2.616 (4) Å. The phospholane ring has a C(3) sofa conformation with $\Delta C_3^1 = 1.8$ (3), $\Delta C_2^1 = 22.7$ (2) and $\phi = 28.6$ (2)°.

Introduction. This study was undertaken as a continuation of our investigations on the structures and ring conformations of phospholane derivatives [Galdecki & Główka (hereinafter GG), 1980*a,b*]. Prismatic crystals were crystallized by cooling a saturated solution in acetone. Intensities from an irregular crystal were collected on a Syntex $P2_1$ diffractometer with graphite-monochromated Cu $K\alpha$ radiation. From 1333 ($\theta_{max} = 72.4^\circ$) recorded reflections, 183 with $F \leq 2\sigma$ were considered as 'less-thans'. The structure was solved by means of *MULTAN* (Germain, Main & Woolfson, 1971). The E map based on 200 $E \geq 1.45$ gave the positions of five atoms. The remaining atoms were easily found from a Fourier map. Full-matrix least-squares refinement resulted in an

R of 0.075 after five cycles of calculations with isotropic and three with anisotropic temperature factors. Five successive cycles of refinement including H atoms located from a difference Fourier map (with isotropic temperature factors) reduced R to 0.043. The weights were $w = \sigma^{-2}$. Neutral-atom scattering factors for P, O and C were taken from Doyle & Turner (1968) and for H from *International Tables for X-ray*

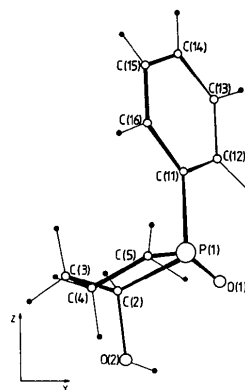


Fig. 1. Projection of the molecule along [100] showing the numbering of atoms.

Table 1. Atomic parameters (positional $\times 10^3$ for hydrogens and $\times 10^4$ for others)
$$B_{\text{iso}} = \frac{1}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j \text{ for P, O and C.}$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{iso} (Å ²)
P(1)	8142 (1)	9145 (1)	983 (1)	4.0 (1)
O(1)	7536 (4)	10635 (3)	642 (1)	5.4 (2)
O(2)	6045 (5)	7711 (3)	-226 (1)	6.9 (2)
C(2)	6624 (7)	7468 (5)	534 (2)	5.0 (2)
C(3)	8269 (8)	6165 (5)	696 (2)	5.9 (3)
C(4)	10400 (8)	6831 (6)	566 (2)	6.5 (3)
C(5)	10802 (7)	8417 (6)	937 (2)	5.7 (3)
C(11)	7819 (5)	9078 (4)	1904 (2)	4.0 (2)
C(12)	6121 (6)	9964 (5)	2051 (2)	5.2 (2)
C(13)	5606 (8)	9845 (6)	2725 (2)	6.5 (3)
C(14)	6770 (9)	8884 (6)	3242 (2)	6.7 (3)
C(15)	8484 (10)	8047 (5)	3115 (2)	6.8 (3)
C(16)	9030 (7)	8137 (5)	2440 (2)	5.5 (2)
H(2)	459 (8)	851 (6)	-38 (3)	17.6 (16)
H(21)	536 (5)	719 (3)	67 (1)	3.7 (7)
H(31)	852 (5)	588 (4)	121 (2)	4.4 (8)
H(32)	774 (5)	523 (4)	40 (2)	4.8 (8)
H(41)	1025 (6)	700 (4)	4 (2)	5.6 (9)
H(42)	1161 (6)	603 (4)	71 (2)	5.8 (9)
H(51)	1165 (5)	827 (3)	139 (2)	3.6 (7)
H(52)	1159 (5)	920 (4)	71 (2)	5.4 (9)
H(12)	534 (5)	1077 (4)	168 (2)	4.1 (8)
H(13)	436 (6)	1055 (4)	281 (2)	6.0 (9)
H(14)	649 (6)	880 (4)	373 (2)	7.5 (11)
H(15)	950 (5)	741 (4)	345 (2)	4.6 (8)
H(16)	1032 (5)	742 (3)	235 (2)	4.1 (8)

Crystallography (1962). The calculations were made using the XRAY 70 system of Stewart, Kundell & Baldwin (1970) on a RIAD 32 computer and *MULTAN* for which an Odra 1305 computer was used.

Table 1 lists the atomic parameters.* A view of the molecule with the atom numbering is shown in Fig. 1.

Discussion. Bond lengths and valency angles (Tables 2 and 3) are similar to those reported earlier for 2-hydroxy-2-methyl-1-phenylphospholane 1-oxide (II) (GG, 1980*a*) and 2-hydroxy-5-hydroxymethyl-1-phenylphospholane 1-oxide (III) (GG, 1980*b*), if differences in the chemical formula of (III) are taken into account. The conformations of the phospholane rings in these structures are shown in Fig. 2. The substituents at positions 2 and 5 appear to affect the ring conformation. In structures (I) and (II), these interactions cause an increase in the torsion angle C(5)–P(1)–C(2)–C(3) with a simultaneous decrease of C(4)–C(5)–P(1)–C(2). As a result of these changes the phospholane ring assumes a C(3) sofa conformation. The situation is different in the case of (III),

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35657 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å)

P(1)–O(1)	1.486 (3)	O(1)···H(2)	1.50 (5)
P(1)–C(2)	1.832 (4)	O(2)–H(2)	1.13 (5)
P(1)–C(5)	1.818 (5)	C(2)–H(21)	0.93 (3)
P(1)–C(11)	1.806 (3)	C(3)–H(31)	0.98 (3)
O(2)–C(2)	1.417 (4)	C(3)–H(32)	1.00 (3)
C(2)–C(3)	1.513 (6)	C(4)–H(41)	1.00 (4)
C(3)–C(4)	1.537 (7)	C(4)–H(42)	1.02 (3)
C(4)–C(5)	1.529 (7)	C(5)–H(51)	0.92 (3)
C(11)–C(12)	1.397 (6)	C(5)–H(52)	1.00 (4)
C(12)–C(13)	1.394 (6)	C(12)–H(12)	1.03 (3)
C(13)–C(14)	1.362 (6)	C(13)–H(13)	1.04 (4)
C(14)–C(15)	1.374 (8)	C(14)–H(14)	0.98 (4)
C(15)–C(16)	1.400 (6)	C(15)–H(15)	0.97 (3)
C(16)–C(11)	1.384 (5)	C(16)–H(16)	1.07 (3)
O(1)···O(2)	2.616 (4)		

Table 3. Valency angles (°)

O(1)–P(1)–C(2)	116.4 (1)	C(2)–O(2)–H(2)	111 (3)
O(1)–P(1)–C(5)	115.8 (2)	C(2)–C(3)–H(31)	109 (2)
O(1)–P(1)–C(11)	112.0 (2)	C(2)–C(3)–H(32)	111 (2)
C(2)–P(1)–C(5)	95.7 (2)	C(3)–C(2)–H(21)	110 (2)
C(2)–P(1)–C(11)	105.2 (2)	C(3)–C(2)–H(21)	109 (2)
C(5)–P(1)–C(11)	110.3 (2)	C(3)–C(4)–H(41)	110 (2)
P(1)–C(2)–O(2)	110.1 (3)	C(3)–C(4)–H(42)	110 (2)
P(1)–C(2)–C(3)	103.0 (3)	C(4)–C(3)–H(31)	108 (2)
P(1)–C(5)–C(4)	105.7 (3)	C(4)–C(3)–H(32)	114 (2)
P(1)–C(11)–C(12)	116.0 (2)	C(4)–C(3)–H(51)	108 (2)
P(1)–C(11)–C(16)	124.2 (3)	C(4)–C(5)–H(52)	117 (2)
O(2)–C(2)–C(3)	108.8 (3)	C(5)–C(4)–H(41)	108 (2)
C(2)–C(3)–C(4)	106.2 (4)	C(5)–C(4)–H(42)	117 (2)
C(3)–C(4)–C(5)	108.6 (4)	C(11)–C(12)–H(12)	120 (2)
C(11)–C(12)–C(13)	119.7 (4)	C(11)–C(16)–H(16)	122 (2)
C(12)–C(13)–C(14)	120.2 (5)	C(12)–C(13)–H(13)	117 (2)
C(13)–C(14)–C(15)	120.8 (4)	C(13)–C(12)–H(12)	120 (2)
C(14)–C(15)–C(16)	120.0 (4)	C(13)–C(14)–H(14)	122 (2)
C(15)–C(16)–C(11)	119.6 (4)	C(14)–C(13)–H(13)	123 (2)
C(16)–C(11)–C(12)	119.7 (3)	C(14)–C(15)–H(15)	128 (2)
P(1)–C(2)–H(2)	118 (2)	C(15)–C(14)–H(14)	117 (2)
P(1)–C(5)–H(51)	111 (2)	C(15)–C(16)–H(16)	118 (2)
P(1)–C(5)–H(52)	111 (2)	C(16)–C(15)–H(15)	112 (2)
O(2)–C(2)–H(2)	106 (2)	H(31)–C(3)–H(32)	109 (3)
O(2)–H(2)···O(1)	170 (4)	H(41)–C(4)–H(42)	105 (3)
P(1)–O(1)···H(2)	133 (2)	H(51)–C(5)–H(52)	105 (3)

where repulsive interactions of the hydroxyl group at C(2) and the hydroxymethyl group at C(5) with the phosphoryl O atom equalize and as a consequence the torsion angles mentioned above assume almost identical values, while the ring adopts a P(1) half-chair conformation.

In the crystals the molecules of (I) form *RS* dimers by means of hydrogen bonds $\geq\text{P}=\text{O}\cdots\text{HO}-$, analogous to compound (II), (GG, 1980*a*).

The distances O···O, O–H and H···O and angle O–H···O amount to 2.616 (4), 1.13 (6), 1.50 (6) Å and 170 (4)°, respectively. The angle between the least-squares planes of the benzene and phospholane rings is about 83° in comparison with the 87 and 89° in (II) and (III) (GG, 1980*a,b*), respectively.

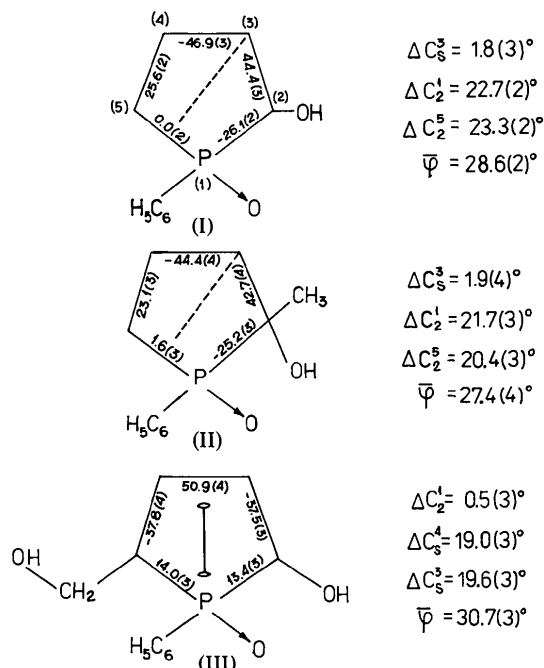


Fig. 2. Conformations and asymmetry parameters (Duax & Norton, 1975) of the phospholane rings in 2-hydroxy-1-phenylphospholane 1-oxide derivatives.

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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