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The Structure of Racemic 7-Benzyl-9-methoxy-8-phenyl-7-phospha-*cis*-bicyclo[4.3.0]non-8-ene *anti*-7-Oxide*†

BY Z. GAŁDECKI

Institute of General Chemistry, Technical University of Łódź, 36 Żwirki, 90-924 Łódź, Poland

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Abstract. $C_{22}H_{25}O_2P$, $M_r = 352{\cdot}42$, orthorhombic, *Pbca*, $a = 16{\cdot}728$ (4), $b = 22{\cdot}557$ (5), $c = 10{\cdot}001$ (3) Å, $Z = 8$, D_m (flotation) $= 1{\cdot}294$, $D_c = 1{\cdot}240$ Mg m^{-3}. The structure was solved by the *MULTAN* program and refined by full-matrix least-squares calculations to a final R of 0·098 for 1757 reflections. The phospholene ring has a conformation intermediate between sofa and half-chair whereas the cyclohexane ring is in a slightly distorted chair conformation. The two rings have a *cis* junction.

Introduction. The present study is the first in a series of X-ray investigations of the structures of phosphaindole derivatives, especially of the conformations of the fused phospholane (or phospholene) and cyclohexane rings. So far, only four similar structures, 1-benzyl-3-hydroxy-4,5-dimethyl-2-phenylphosphol-2-ene (Washecheck, Helm, Purdum & Berlin, 1974), 1-hydroxy-1-oxophospholane (Alver & Kjøge, 1969), 1-benzylphosphole (Coggon, Engel, McPhail & Quin, 1970) and 1-hydroxydibenzo-1*H*-phosphole 1-oxide (Boer & Bright, 1974), have been examined by the X-ray method. Better known are structures containing a 1,3-dioxaphospholane ring.

Intensities from a crystal of irregular shape were collected on a Syntex $P2_1$ four-circle diffractometer with graphite-monochromated Cu $K\alpha$ radiation, up to $2\theta = 96°$. From the 1757 recorded reflections 1275 were contributed to the last cycles of refinement [$F \geq 3\sigma(F)$]. The structure was solved by a multisolution technique (*MULTAN*; Germain, Main & Woolfson, 1971). The E map based on 340 $E \geq 1{\cdot}3$ revealed all the heavy atoms. The structure was refined by least-squares calculations, $\sum w(\Delta F)^2$ being minimized. After four cycles with isotropic ($R = 0{\cdot}135$, $R_w = 0{\cdot}090$) and a further four cycles of full-matrix refinement with anisotropic temperature factors, the agreement factor was equal to 0·122 ($R_w = 0{\cdot}073$). During the anisotropic refinement the molecule was divided into two parts. One part was refined while the other was kept fixed because there were too many variables for the program. A difference synthesis revealed the positions of all H atoms, which were not refined (the C—H bond lengths differ considerably from the 'standard' value especially after test refinement). Finally three cycles of

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† Note: The atom numbering used in the text, Fig. 1 and tables of this paper is arbitrary and does not correspond to that used in naming the compound.

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refinement of the non-hydrogen atoms were performed without weighting. The final R and R_w indices were 0·098 and 0·095. Neutral-atom scattering factors (Doyle & Turner, 1968) were employed. The calculations were performed using the XRAY 70 system (Stewart, Kundell & Baldwin, 1970) on an IBM 370/145 computer.

Discussion. Positional parameters are given in Table 1.* The molecule and atom-numbering scheme are shown in Fig. 1. The bond distances and angles and their estimated standard deviations, calculated by *BONDLA* of XRAY 70, are given in Table 2; torsion angles are shown in Fig. 2. Details of the best planes are given in Tables 3 and 4. The molecules are packed in the crystal nearly perpendicularly to the z axis (Fig. 3) and are well separated. The shortest intermolecular C···C and C···O distances are 3·416 and 3·425 Å, respectively (Table 5). As usual P–C(sp^2) (1·788 Å) is shorter than the P–C(sp^3) lengths (1·814 and 1·823 Å) [*cf.* values of Washecheck, Helm, Purdum & Berlin (1974)]. The angle inside the ring at the P atom is 94·1°. The visible shortening of C(3)–O(2) and C(2)–C(21) bond lengths is probably due to the presence of the conjugated system O(2)–C(3)–C(2)–C(21).

The five-membered ring is in a conformation intermediate between C(8) sofa and C(2) half-chair. The asymmetry parameters (Duax & Norton, 1975) are: $\Delta C_s^8 = 6{\cdot}3$, $\Delta C_2^2 = 9{\cdot}2$ and $\Delta C_s^9 = 18{\cdot}3°$. The average torsion angle is 19·9°. The four atoms P, C(2), C(3) and C(9) lie nearly in a plane ($\sigma_{mean} = 0{\cdot}013$ Å) while C(8) deviates from that plane by 0·525 Å. The cyclo-

* Lists of structure factors, anisotropic thermal parameters, H atom positional parameters and H–C bond lengths have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34198 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *for non-hydrogen atoms with e.s.d.'s in parentheses*

	x	y	z
P	8001 (2)	742 (1)	6502 (3)
O(1)	8314 (4)	975 (3)	5208 (7)
O(2)	6456 (5)	−528 (3)	6460 (9)
C(1)	5721 (7)	−611 (5)	7193 (13)
C(2)	7677 (6)	−13 (5)	6512 (10)
C(3)	6887 (7)	−14 (4)	6629 (10)
C(4)	6090 (7)	683 (5)	5218 (12)
C(5)	5819 (7)	1324 (5)	5072 (12)
C(6)	6542 (8)	1743 (5)	5327 (13)
C(7)	6830 (7)	1677 (5)	6784 (13)
C(8)	7060 (7)	1026 (4)	7128 (10)
C(9)	6440 (7)	577 (5)	6659 (12)
C(10)	8715 (6)	842 (4)	7859 (11)
C(11)	8800 (7)	1498 (5)	8172 (11)
C(12)	9231 (7)	1880 (5)	7339 (11)
C(13)	9222 (8)	2491 (6)	7630 (15)
C(14)	8800 (10)	2707 (7)	8721 (17)
C(15)	8385 (9)	2337 (6)	9542 (13)
C(16)	8383 (7)	1724 (6)	9259 (12)
C(21)	8199 (6)	−537 (4)	6289 (10)
C(22)	8034 (8)	−1095 (5)	6856 (10)
C(23)	8522 (9)	−1571 (5)	6609 (14)
C(24)	9209 (9)	−1499 (6)	5787 (15)
C(25)	9402 (7)	−955 (6)	5289 (12)
C(26)	8888 (8)	−470 (5)	5542 (11)

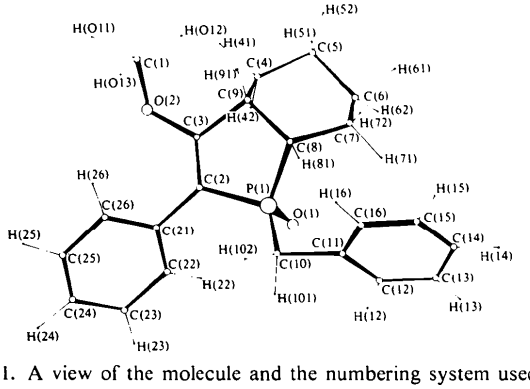

Fig. 1. A view of the molecule and the numbering system used in this paper.

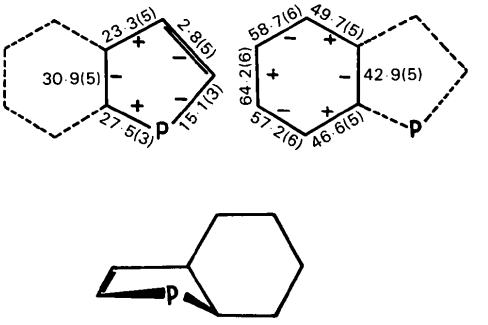

Fig. 2. Torsional angles and the conformation of the two fused rings.

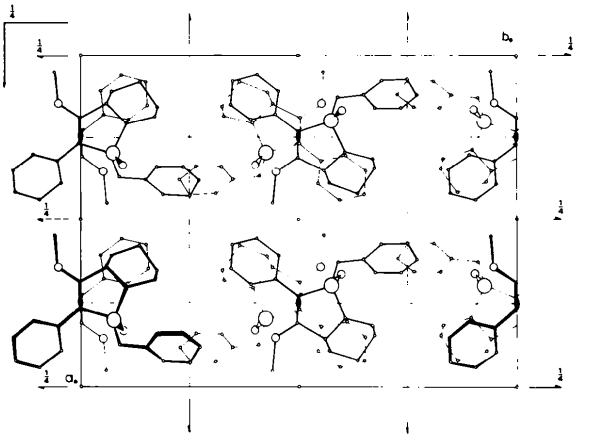

Fig. 3. Packing of molecules in the unit cell.

Table 2. *Bond lengths* (Å) *and angles* (°) *for non-hydrogen atoms with e.s.d.'s in parentheses*

P–O(1)	1·492 (8)	C(8)–C(9)	1·526 (16)
P–C(2)	1·788 (11)	C(10)–C(11)	1·522 (15)
P–C(8)	1·814 (12)	C(11)–C(12)	1·400 (16)
P–C(10)	1·823 (11)	C(12)–C(13)	1·410 (18)
O(2)–C(1)	1·446 (15)	C(13)–C(14)	1·389 (22)
O(2)–C(3)	1·376 (12)	C(14)–C(15)	1·362 (21)
C(2)–C(3)	1·330 (15)	C(15)–C(16)	1·411 (19)
C(2)–C(21)	1·487 (14)	C(16)–C(11)	1·389 (17)
C(3)–C(9)	1·530 (15)	C(21)–C(22)	1·408 (14)
C(4)–C(5)	1·522 (16)	C(22)–C(23)	1·374 (18)
C(4)–C(9)	1·575 (17)	C(23)–C(24)	1·424 (21)
C(5)–C(6)	1·558 (17)	C(24)–C(25)	1·364 (19)
C(6)–C(7)	1·542 (18)	C(25)–C(26)	1·416 (18)
C(7)–C(8)	1·558 (15)	C(26)–C(21)	1·383 (16)

O(1)–P–C(2)	116·5 (4)	C(6)–C(7)–C(8)	112·1 (10)
O(1)–P–C(8)	118·7 (4)	C(5)–C(6)–C(7)	109·9 (10)
O(1)–P–C(10)	111·9 (5)	C(4)–C(5)–C(6)	109·2 (9)
C(2)–P–C(8)	94·1 (5)	C(5)–C(4)–C(9)	110·1 (9)
C(2)–P–C(10)	108·2 (5)	C(10)–C(11)–C(12)	121·7 (10)
C(8)–P–C(10)	105·6 (5)	C(10)–C(11)–C(16)	118·2 (10)
C(1)–O(2)–C(3)	119·6 (9)	C(11)–C(12)–C(13)	118·3(11)
P–C(2)–C(3)	107·8 (8)	C(12)–C(11)–C(16)	120·00 (11)
P–C(2)–C(21)	125·4 (7)	C(12)–C(13)–C(14)	120·8 (13)
P–C(8)–C(9)	104·6 (7)	C(13)–C(14)–C(15)	121·2 (14)
P–C(8)–C(7)	118·2 (8)	C(14)–C(15)–C(16)	118·8 (13)
P–C(10)–C(11)	109·5 (7)	C(15)–C(16)–C(11)	121·0 (12)
O(2)–C(3)–C(2)	121·0 (9)	C(21)–C(22)–C(23)	120·7 (11)
O(2)–C(3)–C(9)	118·7 (10)	C(22)–C(23)–C(24)	119·7 (11)
C(2)–C(3)–C(9)	119·1 (9)	C(22)–C(21)–C(2)	122·4 (10)
C(3)–C(2)–C(21)	126·6 (10)	C(23)–C(24)–C(25)	120·4 (12)
C(3)–C(9)–C(8)	104·6 (9)	C(24)–C(25)–C(26)	119·1 (12)
C(3)–C(9)–C(4)	107·3 (9)	C(25)–C(26)–C(21)	121·3 (10)
C(7)–C(8)–C(9)	113·0 (9)	C(26)–C(21)–C(22)	118·7 (9)
C(4)–C(9)–C(8)	115·7 (9)	C(26)–C(21)–C(2)	118·9 (9)

Table 3. *Details of five-membered-ring geometry*

The equations of the least-squares planes are given in the form $PX + QY + RZ = S$ in direct space. P, Q, R and S are: 1·592, −3·532, 9·839, 7·563 (I); 1·105, −0·080, 9·986, 7·365 (II); and 1·035, −3·630, 9·858, 7·235 (III). Distances from the planes are in Å.

Plane	Atoms in the plane		Other atoms	
(I)	P	−0·154 (4)	O(2)	0·007 (10)
	C(2)	0·071 (10)	C(1)	0·641 (9)
	C(3)	0·061 (10)	C(21)	0·120 (10)
	C(9)	−0·189 (12)		
	C(8)	0·212 (10)		
(II)	P	0·006 (4)	C(8)	0·525 (10)
	C(2)	−0·014 (10)	O(2)	−0·197 (9)
	C(3)	0·016 (10)	C(1)	0·455 (13)
	C(9)	−0·008 (12)	C(21)	−0·175 (10)
(III)	O(2)	−0·008 (9)	P	0·267 (4)
	C(2)	−0·017 (10)	C(1)	0·669 (13)
	C(3)	0·017 (10)	C(9)	−0·214 (12)
	C(21)	0·075 (10)		

Table 4. *Least-squares planes of benzene rings*

Benzyl group: A (13·570X − 2·620Y + 5·753Z = 16·252); phenyl group: B (8·859X + 5·024Y + 8·197Z = 12·171). The mean distances of the atoms from the plane are: 0·003 (A) and 0·018 (B) Å.

	Distances of the atoms from the planes (Å)	
	A	*B*
C(11) or C(21)	−0·002 (12)	−0·023 (10)
C(12) or C(22)	0·003 (12)	0·016 (11)
C(13) or C(23)	−0·001 (14)	0·007 (15)
C(14) or C(24)	−0·003 (17)	−0·022 (15)
C(15) or C(25)	0·004 (14)	0·014 (12)
C(16) or C(26)	−0·001 (12)	0·009 (12)

Table 5. *Intermolecular distances for non-hydrogen atoms (less than* 3·5 Å)

(I)	(II)	Distance (Å)	Equivalent position of atom (II)
O(1)···C(14)		3·425 (1)	$x, \frac{1}{2}-y, z-\frac{1}{2}$
O(2)···C(16)		3·495 (3)	$1\frac{1}{2}-x, -y, z-\frac{1}{2}$
C(1)···C(25)		3·440 (2)	$x-\frac{1}{2}, y, 1\frac{1}{2}-z$
C(13)···C(15)		3·416 (1)	$x, \frac{1}{2}-y, z-\frac{1}{2}$

hexane ring is in a distinctly twisted chair conformation: $\Delta C_2^{8,9} = 2{\cdot}5$, $\Delta C_s^8 = 5{\cdot}8$, $\Delta C_s^9 = 14{\cdot}7$ and $\bar{\varphi} = 53{\cdot}2°$. The rings are *cis*-fused. Similar conformation, bond lengths and angles have been found in the 9-hydroxy-4-*tert*-butyl analogue (Gałdecki & Główka, 1979).

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