

- DOYLE, P. A. & TURNER, P. S. (1968). *Acta Cryst.* **A24**, 390–397.
- GALDECKI, Z. & GŁÓWKA, M. L. (1977a). *Acta Cryst.* **B33**, 2650–2653.
- GALDECKI, Z. & GŁÓWKA, M. L. (1977b). *Acta Cryst.* **B33**, 3908–3911.
- GALDECKI, Z. & GŁÓWKA, M. L. (1978). *Acta Cryst.* **B34**, 2938–2940.
- GŁÓWKA, M. L. (1978). *Acta Cryst.* **B34**, 3089–3091.
- HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
- International Tables for X-ray Crystallography* (1962). Vol. III, pp. 202–203. Birmingham: Kynoch Press.
- LADELL, J., McDONALD, T. R. R. & SCHMIDT, G. M. J. (1956). *Acta Cryst.* **9**, 195.
- LEISEROWITZ, L. (1976). *Acta Cryst.* **B32**, 775–802.
- STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1981). **B37**, 1783–1784

Structure of 1-[Benzyl(phenyl)phosphinoyl]-1-phenylethanol

BY M. L. GŁÓWKA AND Z. GALDECKI

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

(Received 26 February 1981; accepted 30 March 1981)

Abstract. $C_{21}H_{21}O_2P$, $M_r = 336.37$, monoclinic, $P2_1/c$, $Z = 4$, $a = 8.765$ (2), $b = 20.224$ (4), $c = 10.182$ (2) Å, $\beta = 97.21$ (2)°, $V = 1790.6$ Å³, $D_o = 1.23$ (floatation), $D_c = 1.247$ Mg m⁻³, $F(000) = 712$. The structure was solved with *MULTAN* and refined by least squares to a final R of 0.047 ($R_w = 0.036$) for all 1658 intensities collected on a diffractometer with Cu radiation. The molecules form chains by means of hydrogen bonds with an O...O distance of 2.693 Å. The chains run along glide planes. The hydroxyl group is in an antiperiplanar position with respect to the phosphinoyl oxygen.

Introduction. The present investigation is part of our study on the structures, conformations and hydrogen bonding in phosphine oxide derivatives containing a hydroxyl group (Galdecki & Głowska, 1980*a,b,c*, 1981). Colourless crystals of the compound were kindly furnished by Dr W. Waszkuć from the Institute of Organic Chemistry, Technical University of Łódź. Intensities from a spherical crystal (about 0.3 mm in diameter) were recorded on a CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation. All the 1658 independent intensities were used in the refinement. Absorption was neglected. The calculations were performed by means of the XRAY 76 system (Stewart, 1976) on a Riad 22 computer, and *MULTAN* (Germain, Main & Woolfson, 1971) on an Odra 1305 machine. The weighting scheme applied was $w^{-2} = \sigma(F_o)$.

The structure was solved with *MULTAN* based on 200 $E > 1.553$. Three cycles of refinement by full-matrix least squares with isotropic temperature factors followed by two anisotropic cycles (block-

diagonal) enabled us to locate all the H atoms on a difference Fourier map. The H atoms were refined in two cycles by full-matrix least squares, and finally three cycles of block-diagonal refinement of the parameters of all the atoms (H isotropic, remaining atoms anisotropic) were carried out. The final R and R_w

Table 1. *Positional parameters* ($\times 10^4$) *and temperature factors* (Å² $\times 10^3$) *for the non-hydrogen atoms*

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
P	10904 (3)	6873 (1)	2313 (3)	36 (2)
O(1)	10849 (7)	7023 (3)	862 (6)	40 (4)
O(2)	9377 (7)	7003 (3)	4410 (7)	44 (4)
C(1)	7795 (10)	6677 (4)	2414 (10)	34 (6)
C(2)	7353 (11)	6674 (6)	1059 (11)	51 (8)
C(3)	6127 (12)	6281 (6)	517 (13)	69 (9)
C(4)	5353 (13)	5898 (6)	1333 (13)	69 (9)
C(5)	5746 (12)	5901 (5)	2671 (13)	64 (8)
C(6)	6994 (11)	6290 (5)	3238 (12)	51 (7)
C(7)	9113 (10)	7117 (4)	3028 (10)	32 (6)
C(8)	8748 (11)	7848 (5)	2710 (11)	47 (7)
C(10)	12544 (10)	7262 (5)	3333 (11)	42 (6)
C(11)	13108 (10)	7889 (5)	2763 (10)	39 (6)
C(12)	12659 (11)	8503 (5)	3182 (12)	54 (7)
C(13)	13223 (13)	9080 (5)	2676 (14)	74 (9)
C(14)	14246 (13)	9038 (6)	1756 (14)	77 (9)
C(15)	14708 (12)	8432 (7)	1337 (13)	70 (9)
C(16)	14135 (11)	7855 (6)	1836 (12)	55 (7)
C(21)	11134 (10)	5995 (4)	2646 (10)	39 (6)
C(22)	11087 (13)	5576 (5)	1568 (13)	61 (8)
C(23)	11317 (17)	4898 (6)	1797 (15)	88 (10)
C(24)	11567 (15)	4660 (5)	3068 (15)	87 (10)
C(25)	11626 (14)	5080 (5)	4146 (14)	74 (9)
C(26)	11412 (12)	5757 (5)	3938 (12)	52 (8)

factors were 0.047 and 0.036, respectively.* The positional parameters and isotropic thermal parameters for the non-hydrogen atoms are in Table 1.

Discussion. A view of the molecule on the *xy* plane and the atom-numbering system are shown in Fig. 1. The bond lengths and angles listed in Table 2 are close to those commonly observed in similar structures. Relative configurations of both chiral atoms and the conformation along the P—C(7) bond are shown in Fig. 2. The antiperiplanar conformation is adopted and hydrogen bonding is intermolecular, although an intramolecular connection was proposed for such compounds (Musierowicz & Waszkuć, 1977). The molecules transformed by glide plane *c* are connected into chains by means of hydrogen bonds with O...O = 2.693 (9), H...O = 1.76 (20) Å and O...H—O = 173 (20)°, as compared with the values found in 2-hydroxy-2-methyl-1-phenylphospholane 1-oxide (Gałdecki & Głowska, 1980c) (2.702, 1.67 Å, 174°), in 2-hydroxy-1-phenylphospholane 1-oxide (Gałdecki &

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36090 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

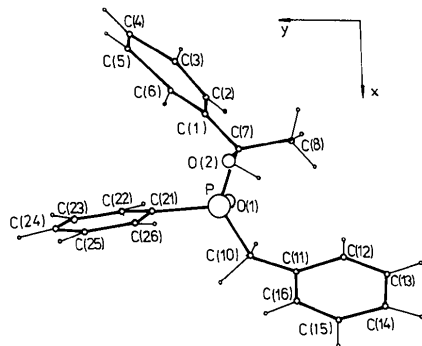


Fig. 1. View of the molecule along [001] showing the atom numbering.

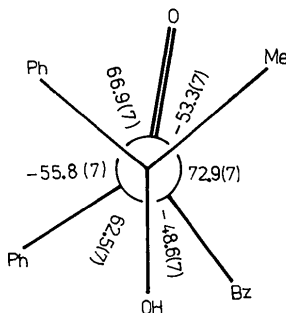


Fig. 2. Newman projection of the molecule along P—C(7). Torsion angles are in degrees.

Table 2. Bond lengths (Å) and bond angles (°)

P—O(1)	1.503 (7)	C(10)—C(11)	1.503 (14)
P—C(7)	1.878 (10)	C(11)—C(12)	1.387 (14)
P—C(10)	1.841 (9)	C(12)—C(13)	1.391 (16)
P—C(21)	1.815 (9)	C(13)—C(14)	1.379 (20)
C(1)—C(2)	1.386 (15)	C(14)—C(15)	1.374 (18)
C(1)—C(7)	1.529 (12)	C(15)—C(16)	1.392 (17)
C(2)—C(3)	1.394 (15)	C(16)—C(11)	1.386 (15)
C(3)—C(4)	1.376 (18)	C(21)—C(22)	1.383 (16)
C(4)—C(5)	1.363 (19)	C(22)—C(23)	1.402 (16)
C(5)—C(6)	1.411 (14)	C(23)—C(24)	1.373 (21)
C(6)—C(1)	1.399 (15)	C(24)—C(25)	1.383 (19)
C(7)—O(2)	1.416 (12)	C(25)—C(26)	1.396 (15)
C(7)—C(8)	1.538 (13)	C(26)—C(21)	1.393 (15)
O(1)—P—C(7)	113.8 (4)	C(8)—C(7)—O(2)	111.7 (8)
O(1)—P—C(10)	113.3 (4)	P—C(10)—C(11)	114.5 (7)
O(1)—P—C(21)	111.8 (4)	C(10)—C(11)—C(12)	121.1 (8)
C(7)—P—C(10)	107.5 (4)	C(10)—C(11)—C(16)	119.7 (9)
C(7)—P—C(21)	105.3 (4)	C(12)—C(11)—C(16)	119.1 (9)
C(10)—P—C(21)	104.6 (4)	C(11)—C(12)—C(13)	120.7 (11)
C(2)—C(1)—C(6)	119.8 (9)	C(12)—C(13)—C(14)	119.4 (11)
C(2)—C(1)—C(7)	120.8 (9)	C(13)—C(14)—C(15)	120.6 (11)
C(6)—C(1)—C(7)	119.4 (9)	C(14)—C(15)—C(16)	120.0 (11)
C(1)—C(2)—C(3)	120.1 (11)	C(15)—C(16)—C(11)	120.2 (10)
C(2)—C(3)—C(4)	119.8 (11)	P—C(21)—C(22)	117.3 (8)
C(3)—C(4)—C(5)	121.3 (11)	P—C(21)—C(26)	121.1 (8)
C(4)—C(5)—C(6)	119.8 (11)	C(22)—C(21)—C(26)	121.6 (9)
C(5)—C(6)—C(1)	119.3 (11)	C(21)—C(22)—C(23)	118.5 (11)
P—C(7)—O(2)	107.6 (6)	C(22)—C(23)—C(24)	120.2 (12)
P—C(7)—C(1)	108.3 (6)	C(23)—C(24)—C(25)	121.3 (11)
P—C(7)—C(8)	109.4 (7)	C(24)—C(25)—C(26)	119.4 (12)
C(1)—C(7)—O(2)	109.4 (8)	C(25)—C(26)—C(21)	119.1 (11)
C(1)—C(7)—C(8)	110.2 (7)		

Głowska, 1981) (2.616, 1.49 Å, 170°), and in 2-hydroxy-5-hydroxymethylene-1-phenylphospholane 1-oxide (Gałdecki & Głowska, 1980d) (2.744, 2.743; 1.85, 1.79 Å; 171, 161°).

The authors are grateful to Dr W. Waszkuć for suggesting the problem and supplying the crystals. They are also grateful to the staff of the Regional Laboratory of Physicochemical Analysis and Structural Research, Kraków, for collecting the intensities. This research was supported by project MR.I.9 from the Polish Academy of Sciences.

References

- GALDECKI, Z. & GŁOWKA, M. L. (1980a). *Acta Cryst.* **B36**, 1495–1497.
- GALDECKI, Z. & GŁOWKA, M. L. (1980b). *Acta Cryst.* **B36**, 2188–2190.
- GALDECKI, Z. & GŁOWKA, M. L. (1980c). *Acta Cryst.* **B36**, 2191–2193.
- GALDECKI, Z. & GŁOWKA, M. L. (1980d). *Acta Cryst.* **B36**, 2809–2812.
- GALDECKI, Z. & GŁOWKA, M. L. (1981). *Acta Cryst.* **B37**, 459–461.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
- MUSIEROWICZ, S. & WASZKUĆ, W. T. (1977). *Phosphorus Sulfur Relat. Elem.* **3**, 345–350.
- STEWART, J. M. (1976). The XRAY system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.