BARTCZAK, T. J., WOLF, W. & CAMERON, T. S. (1983). *Acta Cryst.* C**39**. Submitted for publication.
BUKOWSKA-STRZYŻEWSKA, M. & DOBROWOLSKA, W. (1978). *Acta Cryst.* B**34**, 1357–1360.
BUKOWSKA-STRZYŻEWSKA, M., DOBROWOLSKA, W. & GŁOWIAK, T. (1981). *Acta Cryst.* B**37**, 724–727.
BUKOWSKA-STRZYŻEWSKA, M., MICHALSKI, J., MŁOTKOWSKA, B. & SKOWERANDA, J. (1976). *Acta Cryst.* B**32**, 2605–2608.
CORBRIDGE, D. E. C. (1974). *The Structural Chemistry of Phosphorus*, p. 218. Amsterdam: Elsevier.
GAŁDECKI, Z. (1980). *Acta Cryst.* B**36**, 2807–2809.
International Tables for X-ray Crystallography (1974). Vol. IV, Birmingham: Kynoch Press.
MIKOŁAJCZYK, M. (1969*a*). *Angew. Chem.* **81**, 495; *Angew. Chem. Int. Ed. Engl.* **8**, 511.
MIKOŁAJCZYK, M. (1969*b*). *Chem. Commun.* p. 1221.
MIKOŁAJCZYK, M. & ŁUCZAK, J. (1972). *Tetrahedron*, **28**, 5411–5422.
MIKOŁAJCZYK, M. & SCHIEBEL, H. M. (1969). *Angew. Chem.* **81**, 494; *Angew. Chem. Int. Ed. Engl.* **8**, 511.
MILLER, A., WIECZOREK, M. W., KAROLAK-WOJCIECHOWSKA, J., MIKOŁAJCZYK, M. & ZIEMNICKA, B. (1981). *Acta Cryst.* B**37**, 1951–1953.
ROBERTS, P. & SHELDRICK, G. M. (1975). *XANADU*. A program for crystallographic calculations. Univ of Cambridge, England.
SHELDRICK, G. M. (1976). *SHELX76*. A program for crystal structure determination. Univ. of Cambridge, England.
STEWART, J. M., MACHIN, P. A., DICKINSON, C., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY system–version of January 1976, updated May 1976. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Acta Cryst. (1983). C**39**, 1062–1063

4-Phenyl-1,6-dioxaspiro[4.5]decan-4-ol, $C_{14}H_{18}O_3$

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Abstract. $M_r = 234·3$, orthorhombic, *Pbca*, $a = 9·245\,(1)$, $b = 10·907\,(2)$, $c = 24·310\,(2)$ Å, $V = 2451·3\,(9)$ Å^3, $Z = 8$, $D_x = 1·27$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\bar{\alpha}) = 1·5424$ Å, $\mu = 0·72$ mm^{-1}, $F(000) = 1008$, $T = 298$ K. Final $R = 0·045$ for 753 unique observed reflections. The compound was obtained by photochemical cyclization of 1-phenyl-3-[(tetrahydro-2-pyranyl)oxy]-1-propanone. The compound appears as a *Z* isomer concerning the hydroxyl group and the 6-oxa atom with respect to the tetrahydrofuranic ring. The tetrahydropyranic ring remains chair-shaped and the 1-oxa atom is in the axial position.

Introduction. It is known that photochemical irradiation of ketoacetals may often give hydroxyspiroacetal isomers with good yields (Bernasconi, Cottier & Descotes, 1977; Rémy, Cottier & Descotes, 1979,1980,1982; Cottier & Descotes, 1977,1981). So, the photochemical cyclization of 1-phenyl-3-[(tetrahydro-2-pyranyl)oxy]-1-propanone leads to the two isomers of the title compound (Fig. 1) with $Z/E = 0·54$ (Bron, 1982). IR, ^{1}H NMR and ^{13}C NMR spectra have not allowed elucidation of *Z* or *E* isomerization with respect to the tetrahydrofuranic ring because of the influence of the phenyl group. So, the X-ray crystal-structure determination of one of these isomers was performed.

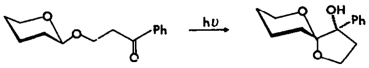

Fig. 1. Reaction scheme.

Experimental. Crystal (petroleum ether) 0·08 × 0·25 × 0·50 mm, Nonius CAD-4 diffractometer, ω–$4/3\theta$ scan, $2 < 2\theta < 146°$. 25 reflections used for refining lattice parameters. 2447 reflections ($0 \leq h \leq 11$, $0 \leq k \leq 13$, $0 \leq l \leq 30$), 753 observed [$I > 1·25\sigma(I)$ and $I > 1·3 \times I_{\mathrm{background}}$]. Lp correction, absorption ignored. Systematic absences: $0kl$ for k odd, $h0l$ for l odd and $hk0$ for h odd. Intensity variation of standard reflection $\bar{1}\bar{1}1$ within 3·4%. Direct-methods program *MULTAN* (Germain, Main & Woolfson, 1970). H from ΔF map, isotropic, with B equal to B_{eq} of bound C or O atom. Final least-squares full-matrix anisotropic F refinement involved all x, y, z, and β_{ij} of C and O atoms. Final $R = 0·045$, $wR = 0·055$, $S = 1·44$; $w = (a + b\,|F_o|)^{-2}$. f_i from *International Tables for X-ray Crystallography* (1974).

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Discussion. The refined atomic coordinates and B_{eq} parameters are listed in Table 1, and the main intramolecular bond distances and angles in Table 2.* The perspective view of the molecule (Fig. 2) was drawn with *ORTEP* (Johnson, 1965).

The tetrahydropyranic ring is in the chair conformation, the calculated least-squares mean plane of which is reported in Table 3. The tetrahydrofuranic ring is not planar; the best least-squares mean plane (Table 3) shows the spirocarbon C(1) out of the plane of the four other atoms in the same way as in some previously studied similar compounds (Rémy, Cottier, Descotes, Faure, Loiseleur & Thomas-David, 1980). The dihedral angle between the two main rings is 70·0°, and between the phenyl ring and the tetrahydrofuranic ring is 83·1°. We may note that O(2) which was, before cyclization, in the equatorial position, is now in the axial position. The molecule of the chosen compound appears as a *Z* isomer concerning the hydroxyl group and 6-oxa atom positions with respect to the tetrahydrofuranic ring. Hence the isomers *Z* and *E* show the same molecular configuration as the compounds in which the phenyl group is replaced by a methyl group (Bernasconi *et al.*, 1977; Cottier & Descotes, 1981).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters for the non-*H *atoms with e.s.d.'s in parentheses*

$$B_{\text{eq}} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)
O(1)	0·8055 (4)	0·2233 (3)	0·0857 (1)	5·3 (2)
O(2)	0·8193 (3)	0·0330 (2)	0·1270 (1)	4·0 (2)
O(3)	0·5690 (4)	0·2814 (3)	0·1388 (2)	5·8 (2)
C(1)	0·7246 (5)	0·1181 (4)	0·1019 (2)	3·7 (2)
C(2)	0·6516 (6)	0·0593 (5)	0·0537 (2)	4·7 (3)
C(3)	0·7565 (7)	0·0338 (6)	0·0072 (2)	6·3 (4)
C(4)	0·8383 (7)	0·1486 (7)	−0·0079 (3)	7·2 (5)
C(5)	0·9093 (7)	0·1977 (5)	0·0427 (3)	6·3 (4)
C(6)	0·6289 (5)	0·1639 (4)	0·1495 (2)	3·9 (3)
C(7)	0·7375 (6)	0·1663 (5)	0·1976 (2)	5·4 (4)
C(8)	0·8596 (6)	0·0808 (6)	0·1791 (2)	5·6 (3)
C(9)	0·5027 (5)	0·0781 (4)	0·1601 (2)	3·9 (3)
C(10)	0·3679 (5)	0·1021 (5)	0·1389 (2)	5·1 (4)
C(11)	0·2537 (7)	0·0188 (7)	0·1468 (2)	6·8 (3)
C(12)	0·2798 (9)	−0·0894 (7)	0·1764 (3)	7·5 (4)
C(13)	0·4113 (8)	−0·1119 (5)	0·1964 (3)	7·4 (4)
C(14)	0·5249 (6)	−0·0304 (4)	0·1888 (2)	5·7 (3)

Table 2. *Main intramolecular bond lengths* (Å) *and bond angles* (°)

C(1)–O(1)	1·425 (4)	C(8)–O(2)	1·420 (6)
C(3)–C(2)	1·515 (7)	C(10)–C(9)	1·374 (7)
C(5)–C(4)	1·493 (9)	C(13)–C(12)	1·333 (9)
C(7)–C(6)	1·542 (7)	C(2)–C(1)	1·496 (6)
C(9)–C(6)	1·518 (6)	C(5)–O(1)	1·446 (7)
C(12)–C(11)	1·403 (9)	C(6)–C(1)	1·540 (6)
C(14)–C(13)	1·388 (8)	C(8)–C(7)	1·532 (8)
C(1)–O(2)	1·415 (5)	C(11)–C(10)	1·407 (8)
C(4)–C(3)	1·508 (9)	C(14)–C(9)	1·389 (6)
C(6)–O(3)	1·420 (5)		
C(1)–O(1)–C(5)	113·1 (3)	C(1)–O(2)–C(8)	107·9 (3)
O(1)–C(1)–O(2)	108·8 (4)	O(1)–C(1)–C(2)	111·5 (4)
O(1)–C(1)–C(6)	104·3 (3)	O(2)–C(1)–C(2)	109·6 (3)
O(2)–C(1)–C(6)	104·1 (3)	C(2)–C(1)–C(6)	118·0 (4)
C(1)–C(2)–C(3)	112·0 (5)	C(2)–C(3)–C(4)	110·5 (5)
C(3)–C(4)–C(5)	108·5 (5)	C(4)–C(5)–O(1)	111·9 (5)
C(1)–C(6)–O(3)	112·3 (4)	C(1)–C(6)–C(7)	101·7 (4)
C(1)–C(6)–C(9)	111·6 (4)	O(3)–C(6)–C(7)	112·2 (4)
O(3)–C(6)–C(9)	106·8 (4)	C(7)–C(6)–C(9)	112·5 (4)
C(6)–C(7)–C(8)	104·3 (4)	O(2)–C(8)–C(7)	107·0 (4)
C(6)–C(9)–C(10)	121·0 (4)	C(6)–C(9)–C(14)	119·8 (4)
C(10)–C(9)–C(14)	119·0 (5)	C(9)–C(10)–C(11)	120·4 (5)
C(10)–C(11)–C(12)	119·0 (6)	C(11)–C(12)–C(13)	119·9 (6)
C(12)–C(13)–C(14)	121·6 (6)	C(13)–C(14)–C(9)	120·0 (6)

Table 3. *Distances* (Å) *to least-squares mean planes*

Plane [O(1), C(1), C(2), C(3), C(4), C(5)]

$$-6{\cdot}052x + 7{\cdot}368y - 8{\cdot}252z + 4{\cdot}157 = 0$$

O(1)	0·220 (3)	C(1)	−0·199 (4)	C(2)	0·207 (5)
C(3)	−0·231 (7)	C(4)	0·244 (7)	C(5)	−0·241 (6)
O(2)	−1·606 (3)	C(6)	0·325 (4)		

Plane [O(2), C(6), C(7), C(8)]

$$4{\cdot}388x + 8{\cdot}321y - 10{\cdot}673z - 2{\cdot}521 = 0$$

O(2)	−0·007 (3)	C(6)	0·007 (4)		
C(7)	−0·010 (6)	C(8)	0·011 (6)		
C(1)	0·553 (4)	O(1)	1·956 (3)	O(3)	0·836 (3)
C(2)	0·258 (5)	C(9)	−1·375 (4)		

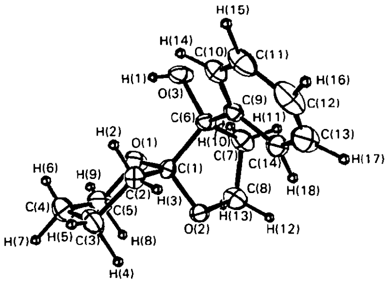

Fig. 2. 25% probability thermal ellipsoids for the C and O atoms. (H atoms are arbitrarily scaled.)

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38496 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bernasconi, C., Cottier, L. & Descotes, G. (1977). *Bull. Soc. Chim. Fr.* pp. 101–106, 107–112.

Bron, P. (1982). Thèse 3ème cycle, n° 1210. Lyon, France.

Cottier, L. & Descotes, G. (1977). *J. Heterocycl. Chem.* **14**, 1271–1273.

Cottier, L. & Descotes, G. (1981). *Tetrahedron*, **37**, 2515–2524.

Germain, G., Main, P. & Woolfson, M. M. (1970). *Acta Cryst.* B**26**, 274–285.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 99. Birmingham: Kynoch Press.

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

Rémy, G., Cottier, L. & Descotes, G. (1979). *Tetrahedron Lett.* **21**, 1847–1850.

Rémy, G., Cottier, L. & Descotes, G. (1980). *Can. J. Chem.* **58**, 2660–2665.

Rémy, G., Cottier, L. & Descotes, G. (1982). *J. Carbohydr. Chem.* **1**, 37–47.

Rémy, G., Cottier, L., Descotes, G., Faure, R., Loiseleur, H. & Thomas-David, G. (1980). *Acta Cryst.* B**36**, 873–877.