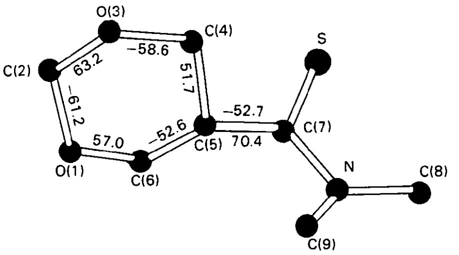

Fig. 2. Torsion angles (°) in the 1,3-dioxane ring. The e.s.d.'s are all in the range 0·3–0·4°.

The torsion angles in the dioxane ring of the title compound are shown in Fig. 2. The dioxane ring, as expected, is puckered in the O—C—O region, the mean of the torsional angles for O—C—O—C being 62·3 (3)°. The torsion angles for C(12)—C(10)—C(2)—O(1) and C(11)—C(10)—C(2)—O(1) are 61·4 (3) and −178·3 (3)°, respectively. The torsion angle for S—C(7)—C(5)—C(6) is 70·4 (3)°, in contrast to 28(1)° found in a *trans*-substituted-5-carbothioamide (Shoja & Kaloustian, 1983). The dimethylcarbothioamide group is at a dihedral angle of 98·7 (2)° with respect to the plane of the dioxane ring. The equation of this plane calculated with respect to orthogonal coordinates is $(0\cdot8055)X + (0\cdot5376)Y + (-0\cdot244)Z - (1\cdot7954) = 0$.

There are no intermolecular distances less than 3·0 Å.

References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1973). NRC Crystallographic Programs for the IBM/360 system. Accession Nos. 133–147, *J. Appl. Cryst.* **6**, 324–325.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

International Tables for X-ray Crystallography (1968). Vol III, 2nd ed., pp. 202–203. Birmingham: Kynoch Press.

Shoja, M. & Kaloustian, M. K. (1983). In preparation.

Shoja, M. & White, J. G. (1979). *Acta Cryst.* **35**, 206–208.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Willis, B. T. M. & Pryor, A. W. (1975). *Thermal Vibrations in Crystallography*, pp. 101–102. Cambridge Univ. Press.

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Structure of a Racemic Compound: The Dicyclohexylammonium Salt of (2*R*,4*R*,2*S*,4*S*)-*cis*-4-Methyl-2-oxido-1,3,2-dioxaphosphorinane 2-Sulphide, $C_{12}H_{24}N^+.C_4H_8O_3PS^-$

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Abstract. $M_r = 349\cdot5$, monoclinic, $P2_1/c$, $a = 20\cdot621$ (6), $b = 11\cdot592$ (2), $c = 18\cdot182$ (5) Å, $\beta = 115\cdot63$ (2)°, $V = 3918\cdot5$ Å^3, $Z = 8$, $D_m = 1\cdot171$ (10), $D_x = 1\cdot184$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot22$ mm^{-1}, $F(000) = 1520$, $T = 293$ K, $R = 0\cdot0712$, $R_w = 0\cdot0797$ for 2928 observed reflections $[F_o \geq 3\sigma(F_o)]$. The salt consists of discrete cations $[C_{12}H_{24}N]^+$ and anions $[C_4H_8O_3PS]^-$ bonded by strong hydrogen bonds to form tetramers. Both the 1,3,2-dioxaphosphorinane and the cyclohexane rings exist in a more or less distorted chair conformation.

Introduction. During a systematic study of the stereochemistry of organophosphorus cyclic compounds, Mikołajczyk & Łuczak (1972) investigated the geometrical isomerism in cyclic five- and six-membered phosphorus monothioacid derivatives which occurs when the molecule contains at least one asymmetric centre at a ring carbon atom. The dicyclohexylammonium (hereafter DCHA) salts of *cis*- and *trans*-4-methyl-2-oxido-1,3,2-dioxaphosphorinane 2-sulphide (the dioxaphosphorinane ring hereafter DOPN) were obtained as diastereoisomerically pure compounds using several different synthetic routes, starting from 2-hydroxy-4-methyl-1,3,2-dioxaphosphorinane 2-sulphide or its sodium salt (Mikołajczyk, 1969 *a,b*; Mikołajczyk & Schiebel, 1969; Mikołajczyk & Łuczak, 1972). Here we present the crystal structure of the *cis* isomer (m.p. 481–484 K) which was studied to investigate the conformation of the DOPN ring when present in the crystal as an anion linked to a bulky positively charged DCHA group. This is a continuation of earlier work on the crystal structure of 4-methyl-1,3,2-dioxaphosphorinanes (Bartczak, Christensen, Kinas & Stec, 1975, 1976). The crystal structure of a similar compound, the ammonium salt of *cis*-4-methyl-2-oxido-1,3,2-DOPN 2-sulphide, has also been solved (Bartczak, Wolf & Cameron, 1983). These are probably the first two examples of crystal structures containing ionic forms of 4-methyl-1,3,2-dioxaphosphorinane rings. The convention is used throughout this paper, following Mikołajczyk &

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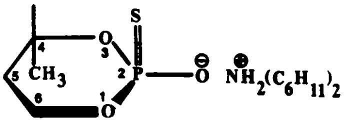
Łuczak (1972), that *cis* refers to the relation between the C(4) methyl group and the exocyclic oxygen atom (Fig. 1); C(4) and C(24) in the notation used in this paper.



Experimental. Spherical crystal, approx. 0·3 mm diameter, D_m by pycnometry, Syntex $P2_1$ automated four-circle diffractometer, graphite-monochromatized Mo $K\alpha$, no absorption correction, scanning mode ω–2θ, max. *hkl* 19,11,17, max. $\sin\theta/\lambda$ 0·481 Å^{-1}; centrosymmetric direct methods, 3199 reflections measured, $R_{\text{int}} = 0{\cdot}0426$, structure refined on F by standard least-squares methods and difference electron density syntheses; isotropic least-squares refinement gave $R = 0{\cdot}138$, anisotropic refinement required blocked full-matrix technique; all H atoms located in a weighted difference Fourier synthesis based on reflections with $\sin\theta/\lambda \le 0{\cdot}3$ Å^{-1}; positional and isotropic thermal parameters of H atoms, taken as for their parent C atoms × 1·5, included in final calculations of structure factors but not refined except for positional parameters of H atoms bonded to amine nitrogen atoms, *i.e.* H(1), H(2), H(6) and H(7); 413 parameters refined, number of reflections: number of parameters = 7:1, $R_w = 0{\cdot}0797$, $w = 1{\cdot}7522/[\sigma^2(F) + 0{\cdot}0015\,F_o^2]$, max. LS shift/error = 0·5 for z coordinate of P(1), max. height in final difference Fourier *ca* 0·3 e Å^{-3}, scattering factors and anomalous dispersion parameters from *International Tables for X-ray Crystallography* (1974). All calculations performed with *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *BONDLA* of XRAY76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).*

Discussion. Final atomic coordinates are given in Table 1, bond lengths and angles in Table 2. The numbering scheme is shown in Fig. 1.

1,3,2-DOPN ring systems have been extensively investigated during the last decade, but comparatively less information is available on 4-methyl derivatives than on 5,5-dimethyl species (Bukowska-Strzyżewska & Dobrowolska, 1978). In the title salt the C(4) methyl group is in *cis* relation to the exocyclic O atom as postulated by Mikołajczyk & Łuczak (1972). Methyl and phosphoryl groups are equatorial to the ring and the thiophosphoryl group is axial (Fig. 1). Bond lengths and angles found are within the observed ranges for similar compounds and will not be discussed in detail (Bukowska-Strzyżewska, Dobrowolska & Głowiak, 1981; Corbridge, 1974). The flattening of the 1,3,2-DOPN rings is as usual more marked at the phosphorus end of the ring than at the carbon end. The appropriate values are: 44·4 (5), 47·1 (5) and 51·0 (5), 57·3 (5)° and also lie within the range observed for the structures solved so far (Bukowska-Strzyżewska, Michalski, Młotkowska & Skoweranda, 1976; Miller, Wieczorek, Karolak-Wojciechowska, Mikołajczyk & Ziemnicka, 1981; Bartczak *et al.*, 1975, 1976; Gałdecki, 1980). The cyclohexane rings are in the conformation of a slightly distorted chair.

Table 1. *Positional parameters* (× 10^4) *and equivalent U values* (Å^2 × 10^3) *for the non-hydrogen atoms with e.s.d.'s in parentheses* (*amine hydrogen atoms are included*)

$$\bar{U}_{\text{eq}} = \frac{U_{11} + U_{22}\sin^2\beta + U_{33} + 2U_{13}\cos\beta}{3(1 - \cos^2\beta)}.$$

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
P(1)	9614 (1)	4737 (1)	6543 (1)	54 (2)
S(1)	8700 (1)	5403 (2)	6340 (1)	106 (2)
O(2)	9855 (2)	4886 (4)	5895 (2)	85 (3)
O(1)	9615 (3)	3371 (3)	6704 (3)	98 (3)
O(3)	10246 (2)	5139 (3)	7370 (2)	68 (3)
C(1)	197 (4)	4823 (5)	8133 (3)	71 (4)
C(2)	206 (4)	3555 (6)	8176 (4)	89 (4)
C(3)	9551 (5)	3042 (7)	7440 (4)	121 (4)
C(4)	862 (6)	5322 (8)	8817 (5)	151 (4)
N(1)	8871 (2)	5093 (4)	4232 (3)	51 (3)
H(1)	9330 (19)	5124 (22)	4139 (19)	73*
H(2)	8968 (19)	5161 (22)	4703 (20)	73*
C(5)	8475 (3)	6223 (4)	3947 (3)	53 (4)
C(6)	8958 (4)	7179 (5)	4433 (4)	75 (4)
C(7)	8619 (4)	8344 (5)	4146 (4)	91 (4)
C(8)	8383 (4)	8511 (6)	3234 (4)	86 (4)
C(9)	7908 (4)	7550 (6)	2743 (4)	86 (4)
C(10)	8260 (3)	6366 (5)	3038 (3)	64 (4)
C(11)	8465 (3)	3990 (5)	3892 (3)	52 (4)
C(12)	7831 (3)	3853 (5)	4103 (4)	67 (4)
C(13)	7455 (3)	2710 (5)	3761 (4)	77 (4)
C(14)	7971 (4)	1684 (5)	4105 (4)	77 (4)
C(15)	8606 (5)	3911 (4)	1836 (5)	83 (4)
C(16)	8992 (3)	3007 (5)	4233 (4)	77 (4)
P(2)	5377 (1)	4880 (1)	1920 (1)	53 (2)
S(2)	6221 (1)	5782 (2)	2568 (1)	85 (2)
O(22)	5176 (2)	4831 (3)	1040 (2)	72 (3)
O(21)	5456 (2)	3583 (3)	2228 (3)	93 (3)
O(23)	4680 (2)	5289 (3)	2014 (2)	71 (3)
C(21)	4663 (4)	5113 (5)	2792 (4)	94 (4)
C(22)	4779 (4)	3833 (6)	3020 (4)	102 (4)
C(23)	5458 (4)	3386 (6)	3018 (5)	119 (4)
C(24)	3938 (5)	5543 (8)	2702 (6)	179 (4)
N(2)	3894 (2)	4814 (4)	9671 (3)	50 (3)
H(6)	4132 (18)	4812 (22)	9271 (19)	69*
H(7)	4277 (18)	4789 (22)	10180 (20)	69*
C(25)	3452 (3)	5907 (5)	9586 (3)	54 (4)
C(26)	2820 (3)	5969 (5)	8763 (4)	62 (4)
C(27)	2413 (3)	7084 (6)	8650 (4)	78 (4)
C(28)	2902 (4)	8125 (6)	8797 (4)	88 (4)
C(29)	3526 (4)	8058 (5)	9627 (4)	85 (4)
C(30)	3944 (3)	6920 (5)	9739 (4)	69 (4)
C(31)	3518 (3)	3672 (5)	9572 (3)	52 (4)
C(32)	4032 (3)	2734 (5)	9600 (4)	74 (4)
C(33)	3686 (4)	1551 (5)	9504 (4)	80 (4)
C(34)	3398 (4)	1349 (6)	10137 (4)	93 (4)
C(35)	2910 (4)	2309 (6)	10126 (5)	105 (4)
C(36)	3247 (3)	3479 (5)	10212 (4)	81 (4)

* U_{iso} (not refined).

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* Lists of structure factors, anisotropic thermal parameters, positional parameters and isotropic U values of hydrogen atoms, hydrogen-bonding contacts, planes and distances of atoms therefrom, torsion angles and asymmetry parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38490 (31 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) *and angles* (°)

E.s.d.'s of angles involving H atoms are 1·5–3·3°.

S(1)–P(1)	1·919 (3)	S(2)–P(2)	1·933 (2)
O(2)–P(1)	1·473 (5)	O(22)–P(2)	1·470 (4)
O(1)–P(1)	1·611 (4)	O(21)–P(2)	1·588 (4)
P(1)–O(3)	1·576 (3)	P(1)–O(23)	1·592 (5)
C(1)–O(3)	1·479 (8)	C(21)–O(23)	1·445 (10)
C(3)–O(1)	1·451 (11)	C(23)–O(21)	1·452 (11)
C(2)–C(3)	1·551 (9)	C(22)–C(23)	1·494 (13)
C(1)–C(2)	1·471 (9)	C(21)–C(22)	1·531 (9)
C(4)–C(1)	1·513 (10)	C(24)–C(21)	1·516 (14)
N(1)–H(1)	1·033 (44)	N(2)–H(2)	1·040 (44)
N(1)–H(2)	0·794 (37)	N(2)–H(7)	0·921 (28)
C(5)–N(1)	1·511 (7)	C(25)–N(2)	1·529 (7)
C(11)–N(1)	1·507 (7)	C(31)–N(2)	1·504 (7)
C(6)–C(5)	1·497 (8)	C(26)–C(25)	1·503 (7)
C(10)–C(5)	1·523 (9)	C(30)–C(25)	1·498 (8)
C(6)–C(7)	1·506 (9)	C(26)–C(27)	1·506 (9)
C(7)–C(8)	1·525 (11)	C(27)–C(28)	1·520 (10)
C(8)–C(9)	1·497 (9)	C(28)–C(29)	1·500 (10)
C(9)–C(10)	1·537 (9)	C(29)–C(30)	1·541 (9)
C(11)–C(12)	1·520 (10)	C(31)–C(32)	1·504 (9)
C(11)–C(16)	1·511 (8)	C(31)–C(36)	1·509 (11)
C(12)–C(13)	1·525 (8)	C(32)–C(33)	1·520 (9)
C(13)–C(14)	1·536 (9)	C(33)–C(34)	1·526 (13)
C(14)–C(15)	1·506 (12)	C(34)–C(35)	1·494 (11)
C(15)–C(16)	1·554 (8)	C(35)–C(36)	1·501 (9)
O(2)–P(1)–S(1)	116·3 (2)	O(22)–P(2)–S(2)	116·0 (2)
O(1)–P(1)–S(1)	110·8 (2)	O(21)–P(2)–S(2)	117·7 (2)
O(1)–P(1)–O(2)	106·7 (3)	O(21)–P(2)–O(22)	106·6 (3)
O(1)–P(1)–O(3)	100·4 (2)	O(21)–P(2)–O(23)	101·8 (3)
O(3)–P(1)–S(1)	113·7 (2)	O(23)–P(2)–S(2)	113·0 (2)
O(3)–P(1)–O(2)	107·6 (3)	O(23)–P(2)–O(22)	106·6 (2)
C(1)–O(3)–P(1)	117·3 (4)	C(21)–O(23)–P(2)	117·9 (4)
C(3)–O(1)–P(1)	115·7 (4)	C(23)–O(21)–P(2)	117·0 (4)
C(2)–C(3)–O(1)	107·6 (7)	C(22)–C(23)–O(21)	110·3 (5)
C(1)–C(2)–C(3)	110·6 (5)	C(21)–C(22)–C(23)	111·9 (7)
C(2)–C(1)–O(3)	107·1 (6)	C(22)–C(21)–O(23)	109·6 (6)
C(4)–C(1)–O(3)	105·7 (7)	C(24)–C(21)–O(23)	106·7 (6)
C(4)–C(1)–C(2)	110·7 (6)	C(24)–C(21)–C(22)	112·3 (7)
H(2)–N(1)–H(1)	110·6	H(7)–N(2)–H(6)	104·1
C(5)–N(1)–H(1)	108·1	C(25)–N(2)–H(6)	111·9
C(5)–N(1)–H(2)	97·6	C(25)–N(2)–H(7)	110·0
C(11)–N(1)–H(1)	110·4	C(31)–N(2)–H(6)	106·9
C(11)–N(1)–H(2)	111·2	C(31)–N(2)–H(7)	105·4
C(11)–N(1)–C(5)	118·2 (4)	C(31)–N(2)–C(25)	117·6 (4)
C(6)–C(5)–N(1)	108·5 (4)	C(26)–C(25)–N(2)	111·3 (4)
C(10)–C(5)–N(1)	109·1 (5)	C(30)–C(25)–N(2)	107·7 (5)
C(10)–C(5)–C(6)	111·5 (5)	C(30)–C(25)–C(26)	111·9 (5)
C(7)–C(6)–C(5)	112·0 (5)	C(27)–C(26)–C(25)	111·7 (5)
C(6)–C(7)–C(8)	111·9 (6)	C(26)–C(27)–C(28)	111·6 (6)
C(7)–C(8)–C(9)	111·9 (8)	C(27)–C(28)–C(29)	110·6 (6)
C(8)–C(9)–C(10)	111·5 (5)	C(28)–C(29)–C(30)	111·4 (5)
C(9)–C(10)–C(5)	110·3 (5)	C(29)–C(30)–C(25)	110·5 (5)
C(12)–C(11)–N(1)	111·7 (5)	C(32)–C(31)–N(2)	108·2 (5)
C(16)–C(11)–N(1)	107·3 (4)	C(36)–C(31)–N(2)	112·5 (5)
C(16)–C(11)–C(12)	111·2 (5)	C(36)–C(31)–C(32)	110·9 (5)
C(13)–C(12)–C(11)	109·2 (6)	C(33)–C(32)–C(31)	111·1 (6)
C(14)–C(13)–C(12)	111·5 (4)	C(34)–C(33)–C(32)	111·6 (6)
C(15)–C(14)–C(13)	109·5 (6)	C(35)–C(34)–C(33)	110·8 (6)
C(16)–C(15)–C(14)	111·7 (6)	C(36)–C(35)–C(34)	113·2 (7)
C(15)–C(16)–C(11)	110·0 (5)	C(35)–C(36)–C(31)	111·2 (6)

The crystal structure comprises discrete cations and anions hydrogen bonded together in tetramers built of two such complexes arranged around the centres of symmetry (Fig. 2). The system of strong hydrogen bonds accounts for the observed hardness of the crystals and their high melting point (481–484 K). The 1,3,2-DOPN rings lie on the twofold screw axes and are more (complex II) or less (complex I) parallel to the *ac* plane. The 1,3,2-DOPN rings lie in the unit cell approximately at $y = 0$, $\frac{1}{4}$, $\frac{1}{2}$, and 1. Both crystallographically independent DCHA ions are arranged in the structure in such a way that the base planes of cyclohexyl rings are almost parallel to the *b* axis.

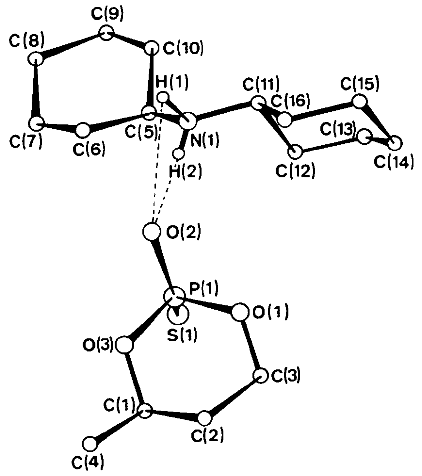

Fig. 1. 4-Methyl-2-oxido-1,3,2-DOPN 2-sulphide ion (I) as projected onto the least-squares plane defined by O(1), O(3), C(1) and C(3). The ion is bonded to the DCHA ion by the hydrogen bond N(1)–H(1)···O(2). The O(2)···H(1) distance is 1·71 (7) and N(1)···O(1) is 2·74 (7) Å. In complex (II) the distances O(22)···H(6) and O(22)···H(7) are both 1·84 (7) Å.

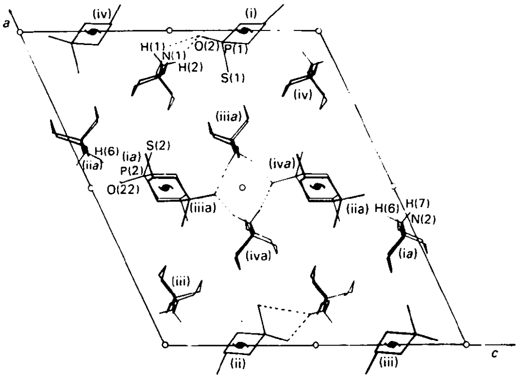

Fig. 2. The crystal structure, hydrogen atoms omitted except for those belonging to the amine nitrogen atoms, projected down the *b* axis. Roman numerals correspond to the following equivalent positions relative to the *x*, *y*, *z* coordinates in Table 1: (ii), (ii*a*) $1-x$, $1-y$, $1-z$, (iii), (iii*a*) $1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$, (iv, iv*a*) x, $\frac{1}{2}-y$, $\frac{1}{2}+z$.

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References

Bartczak, T. J., Christensen, A. T., Kinas, R. & Stec, W. J. (1975). *Cryst. Struct. Commun.* **4**, 701–704.

Bartczak, T. J., Christensen, A. T., Kinas, R. & Stec, W. J. (1976). *Cryst. Struct. Commun.* **5**, 21–24.

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.
Galdecki, 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.

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

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Abstract. $M_r = 234{\cdot}3$, orthorhombic, *Pbca*, $a = 9{\cdot}245\,(1)$, $b = 10{\cdot}907\,(2)$, $c = 24{\cdot}310\,(2)$ Å, $V = 2451{\cdot}3\,(9)$ Å^3, $Z = 8$, $D_x = 1{\cdot}27$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\bar{\alpha}) = 1{\cdot}5424$ Å, $\mu = 0{\cdot}72$ mm^{-1}, $F(000) = 1008$, $T = 298$ K. Final $R = 0{\cdot}045$ 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 $Z/E = 0{\cdot}54$ (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.

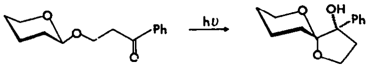

Fig. 1. Reaction scheme.

Experimental. Crystal (petroleum ether) $0{\cdot}08 \times 0{\cdot}25 \times 0{\cdot}50$ mm, Nonius CAD-4 diffractometer, ω–$4/3\theta$ scan, $2 < 2\theta < 146°$. 25 reflections used for refining lattice parameters. 2447 reflections ($0 \le h \le 11$, $0 \le k \le 13$, $0 \le l \le 30$), 753 observed [$I > 1{\cdot}25\sigma(I)$ and $I > 1{\cdot}3 \times I_{\mathrm{background}}$]. Lp correction, absorption ignored. Systematic absences: $0kl$ for k odd, $h0l$ for l odd and $hk0$ for h odd. Intensity variation of standard reflection $\overline{1}\overline{1}1$ 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 $R = 0{\cdot}045$, $wR = 0{\cdot}055$, $S = 1{\cdot}44$; $w = (a + b\,|F_o|)^{-2}$. f_i from *International Tables for X-ray Crystallography* (1974).

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