

N(08)...O(17) links equivalent molecules in neighbouring cells ( $x, y \pm 1, z$ ) while the other, N(05)...O(07), spans molecules which belong to chains transformed by a 2<sub>1</sub> axis.

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## Structure and Stereochemistry of 7 $\beta$ -[(+)-Camphorsulfonyl]-9 $\alpha$ -hydroxylongipin-2-en-1-one\*

BY M. SORIANO-GARCÍA,† M. SALMÓN AND R. A. TOSCANO

*Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán 04510, Mexico DF*

AND L. RODRÍGUEZ-SHOMAR AND E. ANGELES

*Facultad de Estudios Superiores – Cuautitlán, UNAM, Campo 1, Depto de Química, Cuautitlán, Izcalli, Estado de Mexico, Mexico DF*

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**Abstract.** 7 $\beta$ -[(+)-7,7-Dimethyl-2-oxobicyclo[2.2.1]-heptane-1-methanesulfonyl]-9 $\alpha$ -hydroxylongipin-2-en-1-one, C<sub>25</sub>H<sub>36</sub>O<sub>6</sub>S,  $M_r = 464.6$ , monoclinic,  $P2_1$ ,  $a = 7.195$  (4),  $b = 11.128$  (4),  $c = 15.003$  (4) Å,  $\beta = 98.45$  (4)°,  $V = 1188$  (1) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.30$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.7107$  Å,  $\mu = 0.166$  mm<sup>-1</sup>,  $F(000) = 500$ ,  $T = 293$  K,  $R = 0.073$  for 1490 observed reflections. The X-ray study confirms that in the solid state the structure and absolute configuration of the title compound are as inferred from chemical and spectroscopic evidence. The cyclohexenone and cyclobutane rings have 1,2-diplanar and normal puckered conformations, respectively. The cycloheptane ring

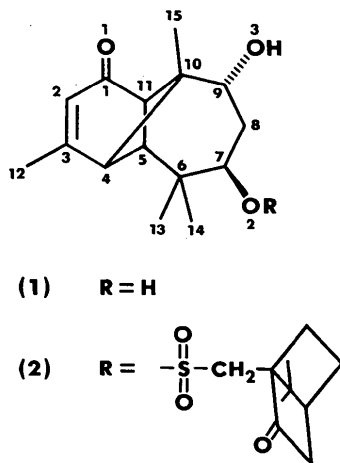
adopts a twist-chair conformation. The geometry and dimensions of the camphor ring system are similar to those in related molecules. The crystal structure is stabilized by an intermolecular hydrogen bond.

**Introduction.** The genus *Stevia* (Compositae) is abundant in Mexico. Some of these plants are used in folk medicine (Altschul, 1975) and as a sweetening agent (Soejarto, Compadre, Medon, Kamath & Kinghorn, 1983). The chemical investigation of the Mexican *Stevia* species showed the occurrence of longipinenes (Román *et al.*, 1985). The longipinene derivative (1) was isolated from the dried and ground roots of *Stevia lucida* Lay, var. *lucida*, collected in the Cubilete mountain, located in the State of Guanajuato, Mexico. Chemical and spectroscopic studies led to the

\* Contribution No. 902 of the Instituto de Química, UNAM.

† To whom correspondence should be addressed.

proposal of the chemical structure (1) (Joseph-Nathan, Cerda, Del Río, Román & Hernández, 1986). In order to determine unambiguously the structure and absolute configuration of (1), a (+)-10-camphorsulfonate derivative was prepared (Salmón & Bidan, 1985). We have undertaken an X-ray study of this compound (2).



**Experimental.** Colourless crystal 0.08 × 0.20 × 0.24 mm. Nicolet R3 four-circle diffractometer, graphite-monochromated MoK $\alpha$  radiation. Lattice parameters from 25 machine-centred reflections with 5.5 < 2 $\theta$  < 27.7°, 2222 reflections with 3 < 2 $\theta$  < 50° for two octants, 1490 independent with  $I > 2.5\sigma(I)$ , index range  $h \pm 8$ ,  $k 0 \rightarrow 12$ ,  $l 0 \rightarrow 16$ ,  $\omega$ -scan mode, variable scan speed, scan width 1.0° ( $\theta$ ), two standard reflections (20 $\bar{4}$ ,  $\bar{3}1\bar{1}$ ) monitored every 50 measurements, Lp correction applied but absorption ignored. Structure solved by combination of direct methods and partial structure expansion by an iterative *E*-Fourier procedure using *SHELXTL* (Sheldrick, 1981). Least-squares refinement of all non-H atoms anisotropic; H atoms of CH, CH<sub>2</sub> and CH<sub>3</sub> were allowed to ride on bonded C. The hydroxyl H atom was located on a difference Fourier map at an advanced stage of anisotropic refinement and its coordinates refined; all H atoms assigned fixed isotropic temperature factor,  $U = 0.06 \text{ \AA}^2$ .  $\sum w(\Delta F)^2$  minimized,  $w = [\sigma^2(F_o) +$

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )

$$U_{eq} = (U_{11} \times U_{22} \times U_{33})^{1/3}$$

	x	y	z	$U_{eq}$
S(1)	-24 (3)	9923	4235 (1)	35 (1)
O(1)	-1704 (10)	8607 (6)	-679 (4)	62 (3)
O(2)	-1945 (7)	10147 (6)	3605 (3)	41 (2)
O(3)	325 (8)	11603 (6)	1511 (4)	43 (2)
O(4)	1016 (8)	9059 (7)	3822 (4)	50 (2)
O(5)	852 (9)	11038 (7)	4484 (5)	63 (3)
O(6)	1130 (10)	10374 (10)	6752 (5)	101 (4)
C(1)	-2751 (13)	9252 (8)	-312 (6)	39 (3)
C(2)	-4674 (12)	9584 (8)	-723 (5)	41 (3)
C(3)	-5639 (10)	10301 (7)	-253 (5)	31 (3)
C(4)	-4696 (9)	10726 (8)	660 (5)	30 (3)
C(5)	-3885 (11)	9539 (7)	1148 (5)	29 (3)
C(6)	-3501 (11)	9410 (9)	2184 (5)	38 (3)
C(7)	-1990 (11)	10243 (7)	2614 (5)	31 (3)
C(8)	-2270 (12)	11542 (8)	2351 (5)	33 (3)
C(9)	-1668 (11)	11806 (8)	1441 (5)	35 (3)
C(10)	-2647 (10)	11144 (7)	596 (5)	24 (2)
C(11)	-2198 (10)	9762 (7)	624 (5)	27 (3)
C(12)	-7601 (12)	10703 (11)	-588 (7)	56 (4)
C(13)	-2890 (15)	8104 (9)	2370 (6)	49 (4)
C(14)	-5409 (12)	9610 (10)	2531 (6)	49 (4)
C(15)	-2284 (12)	11872 (9)	-225 (5)	40 (3)
C(16)	-1835 (10)	10032 (9)	5782 (5)	35 (3)
C(17)	-542 (12)	10450 (12)	6624 (7)	66 (4)
C(18)	-1778 (14)	10835 (13)	7283 (7)	72 (5)
C(19)	-3777 (14)	10566 (10)	6800 (6)	48 (3)
C(20)	-4238 (15)	11490 (9)	6082 (6)	56 (4)
C(21)	-2832 (16)	11196 (9)	5408 (6)	58 (4)
C(22)	-3413 (11)	9417 (8)	6240 (6)	34 (3)
C(23)	-5125 (15)	9050 (14)	5611 (9)	88 (5)
C(24)	-2717 (17)	8331 (10)	6820 (8)	72 (5)
C(25)	-900 (12)	9228 (8)	5158 (5)	35 (3)

$0.002(F_o)^2]^{-1}$ , where  $\sigma$  is standard deviation of observed amplitudes based on counting statistics; isotropic extinction parameter  $X = 0.0009$ . In the last cycle  $(\Delta/\sigma)_{max} = 0.035$ ;  $\Delta\rho$  within  $\pm 0.34 e \text{ \AA}^{-3}$ ,  $S = 1.09$ , final  $R = 0.073$ ,  $wR = 0.077$ ; scattering factors from *International Tables for X-ray Crystallography* (1974). All computations were performed on a Nova 4S computer and plots drawn on a Tektronix plotter with the *SHELXTL* system of programs.

**Discussion.** A perspective view of the molecule and the atomic numbering are shown in Fig. 1. Final atomic coordinates are given in Table 1\* and bond distances and angles in Table 2.

The carbon skeleton of the molecule comprises a system of fused cycloheptane and cyclohexenone rings bridged between C(4) and C(10) to form a cyclobutane ring, and a long side chain at C(7).

All bond lengths and angles are close to corresponding values in rastevione acetate (Román, Del Río, Hernández, Joseph-Nathan, Zabel & Watson, 1981). The cyclohexenone and cyclobutane rings adopt 1,2-diplanar and normal puckered conformations,

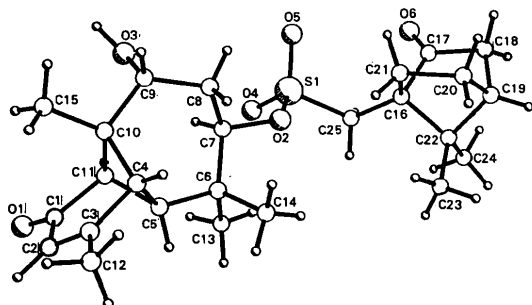


Fig. 1. View of molecule (2), showing the atom labelling.

\* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates, bond angles and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44974 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

respectively. The cycloheptane ring occurs in a twist-chair conformation. This type of conformation was also observed in rastevione acetate (Román *et al.*, 1981).

Table 2. Bond lengths (Å) and angles (°); *e.s.d.*'s are given in parentheses

S(1)–O(2)	1.576 (5)	S(1)–O(4)	1.415 (7)
S(1)–O(5)	1.417 (7)	S(1)–C(25)	1.779 (9)
O(1)–C(11)	1.228 (12)	O(2)–C(7)	1.486 (9)
O(3)–C(9)	1.440 (10)	O(6)–C(17)	1.193 (11)
C(1)–C(2)	1.477 (12)	C(1)–C(11)	1.513 (11)
C(2)–C(3)	1.326 (12)	C(3)–C(4)	1.513 (11)
C(3)–C(12)	1.496 (11)	C(4)–C(5)	1.579 (11)
C(4)–C(10)	1.562 (10)	C(5)–C(6)	1.545 (11)
C(5)–C(11)	1.559 (11)	C(6)–C(7)	1.500 (12)
C(6)–C(13)	1.531 (14)	C(6)–C(14)	1.554 (12)
C(7)–C(8)	1.504 (12)	C(8)–C(9)	1.520 (12)
C(9)–C(10)	1.544 (11)	C(10)–C(11)	1.571 (12)
C(10)–C(15)	1.528 (12)	C(16)–C(17)	1.527 (12)
C(16)–C(21)	1.546 (13)	C(16)–C(22)	1.568 (12)
C(16)–C(25)	1.522 (12)	C(17)–C(18)	1.487 (15)
C(18)–C(19)	1.542 (14)	C(19)–C(20)	1.491 (14)
C(19)–C(22)	1.573 (14)	C(20)–C(21)	1.566 (16)
C(22)–C(23)	1.494 (13)	C(22)–C(24)	1.529 (14)
O(2)–S(1)–O(4)	108.5 (3)	O(2)–S(1)–O(5)	109.7 (4)
O(4)–S(1)–O(5)	117.8 (4)	O(2)–S(1)–C(25)	99.0 (3)
O(4)–S(1)–C(25)	108.5 (4)	O(5)–S(1)–C(25)	111.7 (4)
S(1)–O(2)–C(7)	120.0 (5)	O(1)–C(1)–C(2)	123.9 (8)
O(1)–C(1)–C(11)	122.8 (8)	C(2)–C(1)–C(11)	113.3 (8)
C(1)–C(2)–C(3)	117.4 (7)	C(2)–C(3)–C(4)	118.0 (7)
C(2)–C(3)–C(12)	122.9 (8)	C(4)–C(3)–C(12)	119.1 (8)
C(3)–C(4)–C(5)	104.1 (7)	C(3)–C(4)–C(10)	109.8 (6)
C(5)–C(4)–C(10)	89.4 (5)	C(4)–C(5)–C(6)	122.9 (7)
C(4)–C(5)–C(11)	84.0 (6)	C(6)–C(5)–C(11)	119.0 (6)
C(5)–C(6)–C(7)	112.7 (7)	C(5)–C(6)–C(13)	106.0 (7)
C(7)–C(6)–C(13)	109.7 (7)	C(5)–C(6)–C(14)	106.7 (6)
C(7)–C(6)–C(14)	112.8 (7)	C(13)–C(6)–C(14)	108.6 (8)
O(2)–C(7)–C(6)	107.1 (6)	O(2)–C(7)–C(8)	108.3 (6)
C(6)–C(7)–C(8)	114.8 (7)	C(7)–C(8)–C(9)	112.1 (7)
O(3)–C(9)–C(8)	108.5 (6)	O(3)–C(9)–C(10)	108.3 (7)
C(8)–C(9)–C(10)	119.3 (7)	C(4)–C(10)–C(9)	114.4 (6)
C(4)–C(10)–C(11)	84.2 (6)	C(9)–C(10)–C(11)	112.2 (6)
C(4)–C(10)–C(15)	118.8 (6)	C(9)–C(10)–C(15)	107.3 (7)
C(11)–C(10)–C(15)	118.8 (7)	C(1)–C(11)–C(5)	106.9 (6)
C(1)–C(11)–C(10)	108.5 (6)	C(5)–C(11)–C(10)	89.8 (6)
C(17)–C(16)–C(21)	103.8 (8)	C(17)–C(16)–C(22)	99.4 (6)
C(21)–C(16)–C(22)	101.4 (7)	C(17)–C(16)–C(25)	114.7 (7)
C(21)–C(16)–C(25)	119.5 (7)	C(22)–C(16)–C(25)	115.3 (8)
O(6)–C(17)–C(16)	126.1 (10)	O(6)–C(17)–C(18)	126.9 (9)
C(16)–C(17)–C(18)	106.7 (7)	C(17)–C(18)–C(19)	103.8 (8)
C(18)–C(19)–C(20)	107.4 (9)	C(18)–C(19)–C(22)	101.1 (8)
C(20)–C(19)–C(22)	102.2 (7)	C(19)–C(20)–C(21)	103.1 (8)
C(16)–C(21)–C(20)	104.4 (7)	C(16)–C(21)–C(19)	93.9 (7)
C(16)–C(21)–C(23)	115.1 (8)	C(19)–C(22)–C(23)	111.9 (8)
C(16)–C(22)–C(24)	113.3 (8)	C(19)–C(22)–C(24)	113.9 (8)
C(23)–C(22)–C(24)	108.3 (9)	S(1)–C(25)–C(16)	117.6 (7)

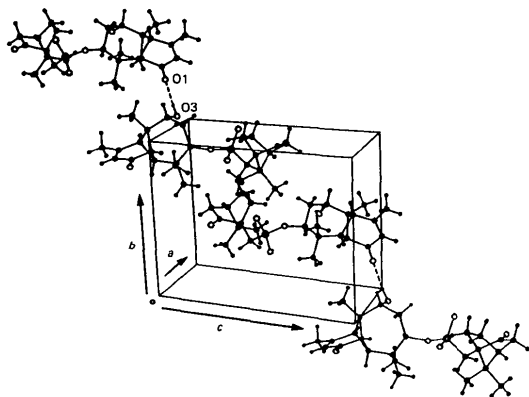


Fig. 2. A perspective drawing of the packing arrangement. The dashed lines indicate the intermolecular hydrogen bonds.

The norbornane skeleton of the (+)-10-camphorsulfonyl group is similar to that in (+)-3-bromocamphor (Allen & Rogers, 1971) and (+)-8-bromocamphor (Bear & Trotter, 1975). The absolute configuration of the longipinene system (1) may thus be inferred by internal comparison with the known absolute stereochemistry of (+)-camphor (Allen & Rogers, 1971). The six-membered ring, C(16)–C(21), shows a boat conformation. The values of the Cremer & Pople (1975) ring-puckering parameters are  $\theta = 55$  (1),  $\varphi = 237$  (1)° and  $Q = 0.85$  (1) Å. The bridging atom C(22) is a member of two five-membered rings: C(10)–C(21)–C(20)–C(19)–C(22) and C(16)–C(17)–C(18)–C(19)–C(22) which adopt  $\alpha$ - and  $\beta$ -envelope conformations, respectively.  $\Delta$  and  $\varphi_m$  (Altona, Geise & Romers, 1968) values for the  $\alpha$ - and  $\beta$ -envelope conformations are 24.2 (9); 58.1 (9) and –31.7 (10); 56.7 (10)°, respectively. The angle C(16)–C(22)–C(19) of 93.9 (7)° is typical for compounds with bicyclo[2.2.1]heptane skeletons (Bear & Trotter, 1975; Poloński & Dauter, 1986). The dihedral angles made by the bridge three-atom plane C(16)–C(22)–C(19) to the four-atom planes C(16)–C(17)–C(18)–C(19) and C(16)–C(21)–C(20)–C(19) are 124.2 (8) and 54.9 (8)°, respectively. The S=O and S–O bond lengths are 1.416 (7) (mean) and 1.576 (5)°, respectively.

Unit-cell contents are shown in Fig. 2. There is only one intermolecular hydrogen bond between the O(3)–H hydroxyl group and the O(1) carbonyl group of an adjacent molecule at  $(-x, 0.5 + y, -z)$ . The O(3)···O(1) and H(3)···O(1) distances are 2.807 (10) and 1.91 (10) Å and the O(3)–H···O(1) angle is 165 (9)°. An intermolecular C–H···O contact  $< 3.42$  Å is present: C(24)···O(3)( $-x, -0.5 + y, 1 - z$ ) with a value of 3.415 (10) Å.

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## Bis(2,4-dichlorophenyl) Ether\*

BY KARI RISSANEN, JUSSI VALKONEN AND LIISA VIRKKI

*Department of Chemistry, University of Jyväskylä, SF-40100 Jyväskylä, Finland*

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**Abstract.** C<sub>12</sub>H<sub>6</sub>Cl<sub>4</sub>O, *M<sub>r</sub>* = 308.0, orthorhombic, *Fdd2*, *a* = 21.782 (3), *b* = 11.309 (3), *c* = 10.137 (3) Å, *V* = 2497.1 Å<sup>3</sup>, *Z* = 8 (one half of the molecule in the asymmetric unit), *D<sub>x</sub>* = 1.64 Mg m<sup>-3</sup>, λ(Mo *Kα*) = 0.71073 Å, μ = 0.93 mm<sup>-1</sup>, *F*(000) = 1232, *T* = 296 K, final *R* = 0.025 for 278 unique observed reflections. The oxygen atom is situated on the twofold axis and the angle (C–O–C) between the symmetry-related phenyl rings is 120.6 (6)°. The molecule has a twist conformation, with a twist angle of 68.4 (8)° between the phenyl rings. The chlorine substituents do not deviate markedly from the least-squares plane formed by the oxygen and the benzene carbon atoms [deviations 0.030 (2) and 0.092 (2) Å].

**Introduction.** Polychlorinated diphenyl ethers occur, together with related chlorinated aromatics, as impurities in various commercial chlorophenol preparations (Gará, Andersson, Nilsson & Norström, 1981) and are now turning up in environmental samples (Paasivirta, Tarhanen & Soikkeli, 1986). Under photochemical exposure, polychlorinated diphenyl ethers give rise by ring closure to polychlorinated dibenzofurans (Norström, Andersson & Rappe, 1976). Some of these, especially 2,3,7,8-tetrachlorodibenzofuran, are considered acutely toxic. The problem of the preferred conformations of diphenyl ethers and the mechanism of the interconversion between stable conformers have been studied extensively for several years mainly by NMR techniques (Benjamins, Dar & Chandler, 1974; Edlund & Norström, 1977). A major reason for this interest is the structural relationships between diphenyl ethers and thyroid hormones (Lehmann, 1972). Four possible forms have been suggested to be involved in the conformational equilibrium of diphenyl ethers (Edlund & Norström, 1977; Fig. 1). The non-toxicity of the

polychlorinated diphenyl ethers compared to polychlorinated biphenyls has been explained by the non-planarity of the diphenyl ethers. Thus they do not fit into the rectangular (3 × 10 Å) dioxin receptor as do the toxic biphenyls (Singh & McKinney, 1980). The crystal structures of halogenated diphenyl ethers have not been extensively studied; only the crystal structure of bis(3,4-dichlorophenyl) ether has been published (Singh & McKinney, 1980). In this series we will be reporting the crystal and molecular structures of various polychlorinated diphenyl ethers, beginning here with the crystal structure of bis(2,4-dichlorophenyl) ether.

**Experimental.** Colourless crystals synthesized by known method (Nilsson, Norström, Hansson & Andersson, 1977), 0.20 × 0.20 × 0.20 mm, mounted on a glass fibre, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo *Kα*, ω–2θ method, lattice parameters from 25 reflections with 3 < θ < 12°, two standard reflections measured every hour, no loss of intensity, 629 reflections (*h*: 0→25, *k*: 0→13, *l*: 0→12) with θ < 25°, 629 independent, 278 with *I* > 3σ(*I*), *L<sub>p</sub>* correction, empirical absorption correction (Walker & Stuart, 1983); correction factors: max. = 1.352 and min. = 0.572, direct methods, refinement by full-matrix

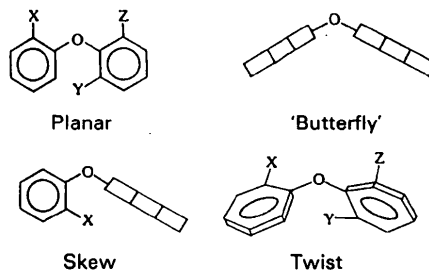


Fig. 1. Possible conformers of diphenyl ether according to Edlund & Norström (1977).

\* Structures of Chlorinated Diphenyl Ethers. I.