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Stereochemical Studies of an Optically Active Bornane Derivative

By Biing-Jiun Uang, Hung-Hsin Liu and Sue-Lein Wang*

Department of Chemistry, National Tsing Hua University, Hsinchu, Taiwan 30043

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Abstract. (−)-(1*R*,2*S*,4′*S*)-(1,7,7-Trimethylbicyclo-[2.2.1]heptane)-2-spiro-2′-(4′-phenylmethyl-1′,3′-oxathiolan-5′-one), $C_{19}H_{24}O_2S$, $[\alpha]_D^{27^\circ C} = -18{\cdot}5^\circ$[CHCl₃, 2 g dm^{-3}] $M_r = 316{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}491\ (2)$, $b = 11{\cdot}078\ (3)$, $c = 20{\cdot}279\ (6)$ Å, $V = 1682{\cdot}8\ (8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}249$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}88$ cm^{-1}, $F(000) = 680$, $T = 295$ K, $R = 3{\cdot}10\%$, $wR = 3{\cdot}20\%$, for 1481 independent reflections [$I \geq 2{\cdot}5\sigma(I)$]. The S atom on the lactone ring is oriented at the *endo* position of the bornane moiety and the phenylmethyl substituent at C(4′) is *anti* to the C(1) atom of the bornane moiety. The absolute configuration is 1*R*,2*S*,4′*S*.

Experimental. The title compound was prepared from the major product of an asymmetric acetalization of (+)-(1*R*)-camphor and thioglycolic acid followed by a diastereoselective benzylation. After recrystallization from *n*-hexane, the title compound was obtained as transparent and rod-like crystals. Nicolet *R*3*m*/*V* diffractometer, graphite-monochromated Mo $K\alpha$ radiation; θ–2θ scan technique. Cell parameters on crystal 0·35 × 0·45 × 0·7 mm from least-squares procedure on 22 reflections ($6{\cdot}0 < 2\theta < 28^\circ$). Systematic absences: $h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; $00l$, $l = 2n + 1$. Total of 1743 reflections measured with $(\sin\theta/\lambda)_{\max} = 0{\cdot}595$ Å^{-1} in the ranges $0 \leq h \leq 8$, $0 \leq k \leq 13$, $0 \leq l \leq 24$. No significant variation in intensities of three standards monitored every 50 reflections. Scan width 1·2° plus $K\alpha$ separation, scan speed 2·93–14·95° min^{-1}, and a scan to background ratio of 0·25. 1481 unique structure amplitudes with $I \geq 2{\cdot}5\sigma(I)$. The structure was solved by direct methods. Refinement on *F*. The correct positions for all non-H atoms were deduced from an *E* map and were refined with anisotropic temperature factors. H atoms were located from difference electron-density maps and were refined

Table 1. *Atomic coordinates* (× 10⁴) *and equivalent isotropic displacement parameters* (Å² × 10³)

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	$U_{\rm eq}$
S(3′)	1706 (1)	1432 (1)	3604 (1)	48 (1)
O(1′)	1567 (3)	−863 (2)	3913 (1)	49 (1)
O(13′)	−662 (3)	−1565 (2)	3318 (1)	70 (1)
C(1)	3172 (4)	339 (2)	4735 (1)	39 (1)
C(2)	2813 (3)	122 (2)	4000 (1)	36 (1)
C(3)	4650 (4)	−241 (3)	3718 (2)	46 (1)
C(4)	5880 (4)	−171 (3)	4322 (2)	49 (1)
C(5)	6142 (5)	1165 (3)	4490 (2)	60 (1)
C(6)	4298 (5)	1528 (3)	4755 (2)	52 (1)
C(7)	4662 (4)	−606 (3)	4893 (2)	45 (1)
C(8)	1547 (5)	363 (4)	5170 (2)	54 (1)
C(9)	4109 (6)	−1934 (3)	4837 (2)	60 (1)
C(10)	5497 (7)	−446 (4)	5575 (2)	66 (1)
C(4′)	160 (4)	501 (3)	3151 (2)	46 (1)
C(5′)	295 (4)	−735 (3)	3462 (2)	47 (1)
C(6′)	413 (5)	466 (4)	2400 (2)	52 (1)
C(7′)	2259 (4)	84 (3)	2182 (1)	45 (1)
C(8′)	3530 (5)	918 (3)	2011 (2)	51 (1)
C(9′)	5241 (5)	561 (4)	1831 (2)	60 (1)
C(10′)	5682 (6)	−637 (4)	1814 (2)	61 (1)
C(11′)	4437 (6)	−1475 (4)	1982 (2)	60 (1)
C(12′)	2716 (5)	−1133 (3)	2160 (2)	53 (1)

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

S—C(2)	1·854 (3)	S—C(12)	1.804 (3)
O(1)—C(2)	1·446 (3)	O(1)—C(11)	1·328 (4)
O(2)—C(11)	1·202 (4)	C(1)—C(2)	1·535 (4)
C(1)—C(6)	1·565 (4)	C(1)—C(7)	1·563 (4)
C(1)—C(8)	1·503 (5)	C(2)—C(3)	1·543 (4)
C(3)—C(4)	1·535 (4)	C(4)—C(5)	1·530 (5)
C(4)—C(7)	1·552 (4)	C(5)—C(6)	1·535 (5)
C(7)—C(9)	1·533 (5)	C(7)—C(10)	1·527 (5)
C(11)—C(12)	1·511 (4)	C(12)—C(13)	1·535 (5)
C(13)—C(14)	1·512 (5)	C(14)—C(15)	1·392 (5)
C(14)—C(19)	1·372 (5)	C(15)—C(16)	1·391 (5)
C(16)—C(17)	1·359 (6)	C(17)—C(18)	1·369 (6)
C(18)—C(19)	1·390 (5)		
C(2)—S—C(12)	93·4 (1)	C(2)—O(1)—C(11)	117·8 (2)
C(2)—C(1)—C(6)	104·5 (2)	C(2)—C(1)—C(7)	102·7 (2)
C(6)—C(1)—C(7)	100·0 (2)	C(2)—C(1)—C(8)	115·5 (3)
C(6)—C(1)—C(8)	114·0 (3)	C(7)—C(1)—C(8)	118·1 (2)
S—C(2)—O(1)	104·4 (2)	S—C(2)—C(1)	112·1 (2)
O(1)—C(2)—C(1)	110·4 (2)	S—C(2)—C(3)	116·3 (2)
O(1)—C(2)—C(3)	109·5 (2)	C(1)—C(2)—C(3)	104·1 (2)
C(2)—C(3)—C(4)	103·1 (2)	C(3)—C(4)—C(5)	107·6 (3)
C(3)—C(4)—C(7)	103·1 (2)	C(5)—C(4)—C(7)	102·1 (3)
C(4)—C(5)—C(6)	102·5 (3)	C(1)—C(6)—C(5)	104·8 (3)
C(1)—C(7)—C(4)	93·4 (2)	C(1)—C(7)—C(9)	115·7 (3)
C(4)—C(7)—C(9)	113·7 (3)	C(1)—C(7)—C(10)	113·6 (3)
C(4)—C(7)—C(10)	113·5 (3)	C(9)—C(7)—C(10)	106·9 (3)
O(1)—C(11)—O(2)	120·9 (3)	O(1)—C(11)—C(12)	115·6 (3)
O(2)—C(11)—C(12)	123·5 (3)	S—C(12)—C(11)	105·2 (2)
S—C(12)—C(13)	116·1 (2)	C(11)—C(12)—C(13)	112·6 (3)
C(12)—C(13)—C(14)	114·2 (3)	C(13)—C(14)—C(15)	120·4 (3)
C(13)—C(14)—C(19)	121·3 (3)	C(15)—C(14)—C(19)	118·3 (3)
C(14)—C(15)—C(16)	120·0 (3)	C(15)—C(16)—C(17)	121·0 (4)
C(16)—C(17)—C(18)	119·4 (4)	C(17)—C(18)—C(19)	120·3 (4)
C(14)—C(19)—C(18)	121·0 (3)		

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* To whom correspondence should be addressed.

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with isotropic temperature factors. The absolute structure was determined with final refinements of the structure with Rogers' η value (Rogers, 1981) which gave $\eta = 1{\cdot}76$ (1) for the final positions that appear in Table 1.* At convergence $R = 3{\cdot}10\%$, $wR = 3{\cdot}20\%$, $w = [\sigma^2(F) + 0{\cdot}00025F^2]^{-1}$, $\sigma^2(F)$ based on counting statistics, $(\Delta/\sigma)_{\max} = 0{\cdot}033$. GOF = 1·53, $(\Delta\rho)_{\max} = 0{\cdot}15$, $(\Delta\rho)_{\min} = -0{\cdot}14$ e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a MicroVAX II computer system using the *SHELXTL-PLUS* programs.

Atomic positions and thermal parameters are listed in Table 1, bond lengths and angles in Table 2. A stereoscopic view of the molecular structure of $C_{19}H_{24}O_2S$ is depicted in Fig. 1.

Related literature. The observed configuration of the 1′,3′-oxathiolan-5′-one ring (the lactone ring with the S atom at the tip of the envelope form) is in agreement with what had been suggested from NMR studies by Pihlaja, Nikkila, Neuvonen & Keskinen (1976).

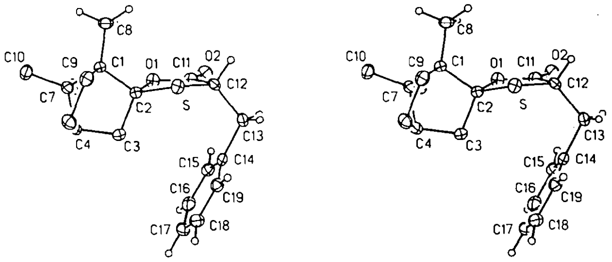

Fig. 1. A stereoscopic view of the molecular structure of $C_{19}H_{24}O_2S$.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52568 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,3,5-Triallyl-4,6-diphenyl-1,3,5-triazacyclohexan-2-one

By J.-P. Declercq

Laboratoire de chimie physique et de cristallographie, Université Catholique de Louvain,
1 place Louis Pasteur, 1348 Louvain la Neuve, Belgium

and I. Marek

Laboratoire de chimie organique de synthèse, Université Catholique de Louvain, 1 place Louis Pasteur,
1348 Louvain la Neuve, Belgium

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Abstract. $C_{24}H_{27}N_3O$, $M_r = 373{\cdot}5$, monoclinic, $P2_1/n$, $a = 9{\cdot}315$ (3), $b = 14{\cdot}674$ (6), $c = 15{\cdot}855$ (6) Å, $\beta = 98{\cdot}44$ (3)°, $V = 2144$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}16$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 5{\cdot}7$ cm^{-1}, $F(000) = 800$, $T = 291$ K, $R = 0{\cdot}062$ for 2900 observed reflections. X-ray analysis was undertaken to establish the exact nature of cycloaddition reaction product and its unambiguous stereochemical configuration. The presence of an exocyclic double bond at C2 forces the triazacyclohexane ring to adopt an envelope conformation, with N5 on the flap and a mirror plane through C2 and N5. The symmetry of the central ring is not retained by the phenyl substituents: C14—C19 in equatorial position and C23—C28 in axial position. Two of the N atoms (N1 and N3) are slightly pyramidal, as shown by the distances from the planes defined by the three covalently bonded C atoms: 0·05 and 0·19 Å respectively. The pyramidal character of N5 is well established, with a corresponding distance of 0·40 Å.

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