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Structure and Absolute Configuration of (8*R*)-8-Benzyloxy-6-(1,3-dioxan-2-yl)-1,4-dithiaspiro[4.4]non-6-ene, $C_{18}H_{22}O_3S_2$

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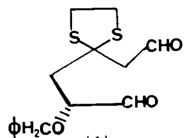
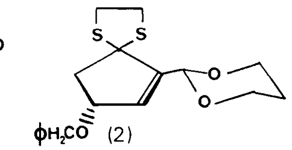
Abstract. $M_r = 350{\cdot}50$, monoclinic, $P2_1$, $a = 15{\cdot}399$ (4), $b = 7{\cdot}785$ (5), $c = 16{\cdot}128$ (4) Å, $\beta = 113{\cdot}01$ (2)°, $Z = 4$ (two independent molecules), $D_x = 1{\cdot}309$ Mg m^{-3}, $V = 1780$ (2) Å^3, μ(Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å) $= 1{\cdot}7$ mm^{-1}, $F(000) = 744$, $R = 0{\cdot}067$ for 1951 observed reflections. The two molecules of the unit cell are compared. Only van der Waals contacts are involved in the crystal packing.

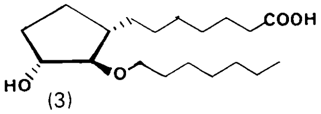
Introduction. Synthetic approaches to enantiomerically pure substituted cyclopentanes are of great interest because they are decisive intermediates in the total synthesis of natural cyclopentane derivatives such as prostaglandins (Bindra & Bindra, 1977). Since our manifold interest in these compounds lies especially in their pharmacological activity, we needed cyclopentanes which are substituted in one proper natural configuration. Recently we have reported (Barrière, Chiaroni, Cléophax, Géro, Riche & Vuilhorgne, 1981) a short, flexible and stereocontrolled synthesis of cyclopentenes leading to 11α-hydroxy-13-oxaprostanoic acid (3), from (−)-quinic acid. The sequence implies generation of intermediates such as (2) (the title compound), obtained from an acyclic precursor (1) by an intramolecular aldolization dehydration reaction. The stereochemistry of the crystalline compound (2) was secured by the present X-ray analysis.



(1)



(2)



(3)

Experimental. Crystal 0·3 × 0·4 × 0·4 mm. Graphite-monochromated Cu $K\bar{\alpha}$ radiation, four-circle Philips PW 1100 diffractometer, θ–2θ scan-technique. Unit-cell dimensions refined from setting angles of 23 reflections. Three standard reflections measured every 2 h: no fluctuation in intensity. Two sets of equivalent reflections collected up to $2\theta = 135°$ ($-17 \le h \le 16$, $0 \le k \le 9$, $0 \le l \le 19$). 3670 *hkl* reflections, 3505 unique, 1951 considered as observed with $I > 2{\cdot}50\sigma(I)$; $\sigma(I)$ derived from counting statistics; $R_{\rm int} = 0{\cdot}076$ (from 103 multiple reflections). 3510 $\overline{hkl}$ unique reflections also recorded (of which 1948 'observed') for the absolute-configuration determination. Lp corrections, absorption ignored. Direct methods (*MULTAN*77; Main, Lessinger, Woolfson, Germain & Declercq, 1977). S atoms revealed on the best *E* map, other atoms from successive Fourier syntheses. Refinement by full-matrix least squares with anisotropic temperature factors (*ORION*; André, Fourme & Renaud, 1971); $\sum w|F_o - |F_c||^2$ minimized, $w = 1/\sigma^2(F_o)$. Not all H atoms on difference maps; therefore they were placed at idealized positions (C—H = 1·0 Å) and assigned the equivalent isotropic thermal factor of the bonded C atom. Absolute configuration determined by examination of several Bijvoet pairs. Final $R = 0{\cdot}067$, $R_w = 0{\cdot}076$ (for the 1951 *hkl* reflections), $S = 0{\cdot}72$. $(\Delta/\sigma)_{\max} = 0{\cdot}8$. Final difference-map excursions $\le 0{\cdot}33$ e Å^{-3}. Atomic scattering factors and anomalous-dispersion terms for S from *International Tables for X-ray Crystallography* (1974). No correction for secondary extinction.

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Discussion. Final atomic parameters are in Table 1,* bond distances and angles in Table 2, and torsion angles in Table 3. Molecule (I) is shown in Fig. 1.

Both molecules are almost identical and exhibit the same chirality: C(4) is *R*.† This is proved by the torsion angles C(2)–C(3)–C(4)–O(11′) and C(3)–C(4)–O(11′)–C(12′) which have the same signs and almost equal values (130·6, 121·7° and 72·0, 70·0°).

The coordinates of molecules (I) and (II) are related by a pseudo-glide plane defined as $x_1 - x_2 \simeq 0$, $y_1 + y_2 \simeq 0{\cdot}80$ and $z_1 - z_2 = 0{\cdot}50$. This explains the opposite signs observed for the endocyclic torsion angles (Table 3), except those cited above.

The differences affect the ring conformations: in (I) the cyclopentene ring has an envelope shape with C(5) as the flap [out of the plane C(1)–C(2)–C(3)–C(4) by 0·42 Å], while in (II) this ring is perfectly planar.

In both molecules the five-membered rings containing the S atoms are in the envelope conformation, but in (I) C(9′) is displaced 0·38 Å from the plane of the other four atoms, while in (II) C(9′) is displaced 0·78 Å.

Table 1. *Atomic coordinates* (×10⁴) *with e.s.d.'s in parentheses and equivalent isotropic thermal parameters*

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

	x	y	z	$B_{\rm eq}$(Å^2)
Molecule (I)				
C(1)	8380 (6)	3108 (13)	4418 (6)	3·3
C(2)	8374 (5)	3110 (12)	5325 (5)	2·4
C(3)	7807 (7)	1910 (14)	5402 (6)	3·7
C(4)	7344 (7)	889 (13)	4554 (7)	3·7
C(5)	7948 (7)	1335 (16)	4046 (6)	4·2
C(1′)	8879 (6)	4430 (13)	6002 (5)	3·2
O(2′)	9675 (5)	3666 (9)	6644 (4)	4·7
C(3′)	10208 (9)	4880 (16)	7311 (8)	6·5
C(4′)	9583 (10)	5657 (19)	7786 (7)	6·3
C(5′)	8705 (9)	6314 (18)	7083 (9)	6·6
O(6′)	8261 (4)	5000 (11)	6441 (5)	5·5
S(7′)	7655 (2)	4911 (4)	3758 (2)	5·5
C(8′)	8550 (12)	6025 (22)	3520 (11)	10·8
C(9′)	9299 (10)	5023 (20)	3578 (8)	6·6
S(10′)	9556 (2)	3428 (4)	4421 (2)	4·8
O(11′)	7407 (5)	−954 (9)	4683 (5)	4·4
C(12′)	6827 (8)	−1585 (15)	5114 (8)	5·5
C(13′)	5779 (7)	−1594 (13)	4544 (6)	3·4
C(14′)	5435 (6)	−2437 (13)	3713 (6)	3·5
C(15′)	4465 (7)	−2503 (13)	3226 (6)	4·2
C(16′)	3832 (7)	−1773 (17)	3525 (7)	4·6
C(17′)	4171 (7)	−912 (15)	4332 (8)	4·9
C(18′)	5115 (9)	−836 (15)	4817 (7)	4·8
Molecule (II)				
C(1)	3481 (6)	4906 (15)	9358 (6)	3·8
C(2)	3508 (6)	5138 (16)	10333 (6)	3·6
C(3)	3140 (6)	6573 (13)	10451 (6)	3·3
C(4)	2796 (6)	7633 (14)	9599 (7)	3·9
C(5)	2998 (8)	6572 (17)	8892 (7)	6·3
C(1′)	3919 (6)	3731 (13)	11038 (6)	3·5
O(2′)	4667 (5)	4395 (10)	11745 (4)	5·4
C(3′)	5132 (9)	3107 (18)	12408 (7)	6·1
C(4′)	4444 (12)	2343 (16)	12713 (8)	7·7
C(5′)	3601 (8)	1697 (20)	11924 (9)	7·1
O(6′)	3244 (4)	3057 (13)	11257 (5)	6·5
S(7′)	2773 (2)	2973 (5)	8831 (2)	6·3
C(8′)	3598 (12)	1700 (21)	8610 (9)	8·6
C(9′)	4268 (10)	3084 (23)	8427 (8)	8·6
S(10′)	4626 (1)	4559 (4)	9371 (1)	4·2
O(11′)	1828 (4)	8020 (9)	9281 (4)	3·6
C(12′)	1573 (6)	9224 (15)	9829 (7)	4·2
C(13′)	549 (6)	9557 (12)	9349 (6)	3·1
C(14′)	180 (7)	10523 (14)	8573 (6)	3·8
C(15′)	−745 (8)	10790 (16)	8120 (7)	4·7
C(16′)	−1360 (8)	10137 (18)	8470 (8)	5·7
C(17′)	−1036 (7)	9208 (16)	9269 (8)	5·4
C(18′)	−47 (8)	8886 (15)	9736 (6)	4·6

Table 2. *Bond lengths* (Å) *and angles* (°) *for molecules* (I) *and* (II).

Mean e.s.d. for angles: 1°.

	(I)	(II)		(I)	(II)
C(1)–C(2)	1·47 (1)	1·57 (1)	C(4′)–C(5′)	1·48 (2)	1·51 (2)
C(1)–C(5)	1·55 (2)	1·54 (2)	C(5′)–O(6′)	1·43 (2)	1·46 (2)
C(1)–S(7′)	1·85 (1)	1·86 (1)	S(7′)–C(8′)	1·79 (2)	1·75 (2)
C(1)–S(10′)	1·82 (1)	1·78 (1)	C(8′)–C(9′)	1·36 (3)	1·60 (3)
C(2)–C(3)	1·32 (2)	1·30 (2)	C(9′)–S(10′)	1·77 (1)	1·81 (2)
C(2)–C(1′)	1·48 (1)	1·53 (2)	O(11′)–C(12′)	1·42 (2)	1·44 (1)
C(3)–C(4)	1·50 (1)	1·51 (1)	C(12′)–C(13′)	1·51 (2)	1·48 (1)
C(4)–C(5)	1·50 (2)	1·54 (2)	C(13′)–C(14′)	1·40 (1)	1·38 (1)
C(4)–O(11′)	1·45 (1)	1·41 (1)	C(13′)–C(18′)	1·39 (2)	1·40 (2)
C(1′)–O(2′)	1·39 (1)	1·37 (1)	C(14′)–C(15′)	1·39 (2)	1·34 (2)
C(1′)–O(6′)	1·46 (1)	1·33 (1)	C(15′)–C(16′)	1·37 (2)	1·38 (2)
O(2′)–C(3′)	1·43 (2)	1·44 (1)	C(16′)–C(17′)	1·37 (2)	1·39 (2)
C(3′)–C(4′)	1·57 (2)	1·46 (2)	C(17′)–C(18′)	1·36 (2)	1·43 (2)
C(2)–C(1)–C(5)	103	102	O(2′)–C(3′)–C(4′)	110	109
C(2)–C(1)–S(7′)	110	109	C(3′)–C(4′)–C(5′)	108	111
C(2)–C(1)–S(10′)	113	112	C(4′)–C(5′)–O(6′)	110	109
C(5)–C(1)–S(7′)	112	112	C(1′)–O(6′)–C(5′)	111	112
C(5)–C(1)–S(10′)	113	115	C(1)–S(7′)–C(8′)	99	102
S(7′)–C(1)–S(10′)	106	107	S(7′)–C(8′)–C(9′)	114	103
C(1)–C(2)–C(3)	111	114	C(8′)–C(9′)–S(10′)	113	106
C(1)–C(2)–C(1′)	122	120	C(1)–S(10′)–C(9′)	110	96
C(3)–C(2)–C(1′)	127	126	C(4)–O(11′)–C(12′)	113	115
C(2)–C(3)–C(4)	113	111	O(11′)–C(12′)–C(13′)	116	106
C(3)–C(4)–C(5)	102	106	C(12′)–C(13′)–C(14′)	120	123
C(3)–C(4)–O(11′)	114	113	C(12′)–C(13′)–C(18′)	123	117
C(5)–C(4)–O(11′)	107	110	C(14′)–C(13′)–C(18′)	117	120
C(1)–C(5)–C(4)	105	107	C(13′)–C(14′)–C(15′)	119	124
C(2)–C(1′)–O(2′)	108	109	C(14′)–C(15′)–C(16′)	123	118
C(2)–C(1′)–O(6′)	108	110	C(15′)–C(16′)–C(17′)	119	121
O(2′)–C(1′)–O(6′)	109	115	C(16′)–C(17′)–C(18′)	120	120
C(1′)–O(2′)–C(3′)	111	112	C(13′)–C(18′)–C(17′)	124	117

Table 3. *Torsion angles for molecules* (I) *and* (II) (°)

	(I)	(II)
C(5)–C(1)–C(2)–C(3)	−16·6 (1·0)	−0·0 (0·8)
C(1)–C(2)–C(3)–C(4)	0·5 (0·8)	−0·7 (0·8)
C(2)–C(3)–C(4)–C(5)	16·3 (1·1)	1·1 (0·9)
C(3)–C(4)–C(5)–C(1)	−25·1 (0·8)	−1·1 (0·9)
C(4)–C(5)–C(1)–C(2)	25·9 (0·8)	0·7 (0·9)
C(2)–C(1)–S(7′)–C(8′)	120·2 (0·7)	−120·1 (0·7)
S(10′)–C(1)–S(7′)–C(8′)	−1·9 (0·7)	0·8 (0·6)
C(1)–S(7′)–C(8′)–C(9′)	21·8 (1·1)	−31·1 (0·9)
S(7′)–C(8′)–C(9′)–S(10′)	−33·7 (1·2)	51·2 (1·0)
C(8′)–C(9′)–S(10′)–C(1)	28·8 (1·1)	−48·2 (1·0)
C(9′)–S(10′)–C(1)–S(7′)	−12·6 (0·6)	25·4 (0·6)
C(9′)–S(10′)–C(1)–C(5)	111·2 (0·8)	−99·3 (0·8)
C(2)–C(3)–C(4)–O(11′)	130·6 (0·9)	121·7 (1·0)
C(3)–C(4)–O(11′)–C(12′)	72·0 (1·1)	70·0 (0·8)
C(4)–O(11′)–C(12′)–C(13′)	72·6 (1·1)	175·9 (0·7)
C(1)–C(2)–C(1′)–O(2′)	−106·7 (0·7)	119·6 (0·8)
C(2)–C(1′)–O(2′)–C(3′)	178·8 (1·0)	−175·9 (0·8)
C(1′)–O(2′)–C(3′)–C(4′)	58·9 (1·1)	−57·2 (0·9)
O(2′)–C(3′)–C(4′)–C(5′)	−52·7 (1·3)	53·9 (1·3)
C(3′)–C(4′)–C(5′)–O(6′)	53·7 (1·3)	−51·5 (1·2)
C(4′)–C(5′)–O(6′)–C(1′)	−60·7 (1·1)	52·1 (1·2)
C(5′)–O(6′)–C(1′)–O(2′)	64·7 (0·9)	−57·7 (0·9)
O(6′)–C(1′)–O(2′)–C(3′)	−63·9 (0·8)	60·6 (1·0)
O(6′)–C(1′)–C(2)–C(3)	−37·8 (0·7)	64·3 (1·0)
O(11′)–C(12′)–C(13′)–C(14′)	55·1 (1·0)	−72·5 (1·1)

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* 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 38902 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† Note that the crystallographic numbering scheme differs from that used to name the compound.

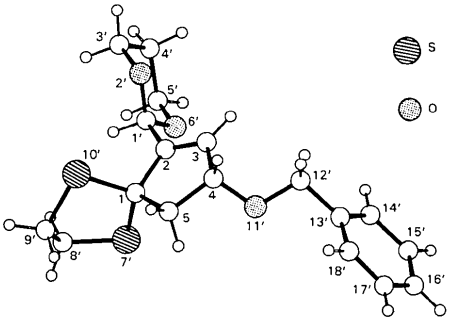

Fig. 1. Perspective view of molecule (I) with atomic labeling.

The phenyl rings are in quite different orientations with respect to the rest of the molecule, as shown by the torsion angles along the O(11')—C(12') and C(12')—C(13') bonds. This is probably a consequence of the packing in the crystal, where only normal van der Waals contacts are observed.

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A 1:1 Complex of 2,9-Dimethyl-1,10-phenanthroline and Resorcinol, $C_{14}H_{12}N_2.C_6H_6O_2$

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Abstract. $M_r = 318{\cdot}38$, triclinic, $P\bar{1}$, $a = 7{\cdot}461\,(1)$, $b = 11{\cdot}942\,(2)$, $c = 14{\cdot}392\,(3)$ Å, $\alpha = 139{\cdot}25\,(1)$, $\beta = 91{\cdot}16\,(2)$, $\gamma = 94{\cdot}87\,(1)^\circ$, $V = 826{\cdot}9\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}28$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 6{\cdot}79$ cm^{-1}, $F(000) = 336$, room temperature, $R = 0{\cdot}078$ for 1755 unique reflections with $I > 3\sigma(I)$. Parallel sheets of 2,9-dimethyl-1,10-phenanthroline molecules are almost perpendicular, 81·7 (4)°, to parallel sheets of resorcinol molecules. There is extensive hydrogen bonding with each phenanthroline bound from above and below by resorcinol molecules.

Introduction. Interactions between neutral organic molecules are of biological interest as well as of importance in nonaqueous laboratory systems. We have investigated a series of crown-ether complexes with neutral organic molecules (Galloy, Watson, Vögtle & Müller, 1982; Grossie, Watson, Vögtle & Müller, 1982; Watson, Galloy, Grossie, Vögtle & Müller, 1983) and bipyridyl complexes with barium and calcium (Watson, Grossie, Vögtle & Müller, 1983). The title complex represents a class of molecular complex which is different from three reported in the earlier papers.

Experimental. Complex prepared by mixing equimolar quantities of the reactants in alcoholic solvents Recrystallization from acetone–methanol (3:1) yielded faceted prismatic crystals. Although the crystals were of poor quality, a useable single crystal was obtained; 0·5 × 0·5 × 0·5 mm. Syntex $P2_1$ diffractometer, θ:2θ scan, $2\theta_{\max} = 116^\circ$, graphite-monochromated Cu $K\alpha$. Lattice parameters from least-squares refinement of 15 medium-angle reflections; angles measured by centering routine associated with the diffractometer system. No systematic absences. Monitored reflection showed no significant change in intensity. 1878 independent reflections measured ($0 \le h \le 8$, $-9 \le k \le 9$, $-11 \le l \le 11$), 1755 with $I > 3\sigma(I)$. Lorentz and polarization corrections; no absorption correction. Direct methods (*MULTAN*78: Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Several heavy atoms and all H atoms found in difference Fourier map.

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