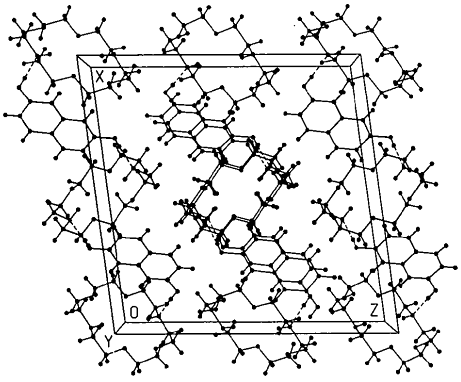

Fig. 3. Packing diagram for the 1:1 complex. Hydrogen bonds are shown as dashed lines.

refine, the two crown molecules are not related *via* a mirror plane containing C(4a)—C(8a), nor is an alternate space group apparent. The low residual electron density does not indicate alternate positions; however, solvent loss may be possible. Except for the C(2)—O(2) and C(7)—O(7) distances, the two dihydroxynaphthalene molecules are statistically equivalent, and both molecules are essentially planar (r.m.s.d. = 0·011 Å). Packing diagrams of the 1:1 and 1:2 complexes are shown in Figs. 3 and 4.

We thank the Welch Foundation (P-074) and the TCU Research Fund for their financial support.

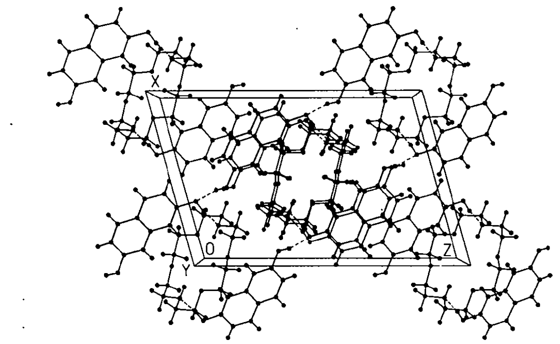

Fig. 4. Packing diagram for the 1:2 complex. Hydrogen bonds are shown as dashed lines.

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*Acta Cryst.* (1989). C**45**, 306–308

# The Absolute Configuration of (+)-Isoconcinndiol

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(*Received* 7 *June* 1988; *accepted* 19 *September* 1988)

**Abstract.** 6-Bromo-α-ethenyldecahydro-2-hydroxy-α,2,5,5,8a-pentamethyl-1-naphthalenepropanol, $C_{20}H_{35}BrO_2$, $M_r = 387{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}830\,(1)$, $b = 12{\cdot}736\,(2)$, $c = 23{\cdot}289\,(2)$ Å, $V = 2026\,(5)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}27$ Mg m$^{-3}$, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 2{\cdot}82$ mm$^{-1}$, $F(000) = 824$, $T = 293$ K, final $R = 0{\cdot}051$ for 682 observed reflections. (+)-Isoconcinndiol was isolated from the mollusc *Aplysia dactylomela*. Its structure and absolute configuration are those of (3*S*,8*R*,13*S*)-labd-14-ene-8,13-diol. Hydrogen bonds O(2)H···O(1) [2·68 (1) Å, 165·4 (6)°] and O(1)H···O(2)($x-\frac{1}{2}$, $-y+\frac{1}{2}$, $-z+1$) [2·65 (1) Å, 166·9 (6)°] link the crystal structure.

**Introduction.** The brominated diterpene isoconcinndiol was first isolated from the marine alga *Laurencia snyderae*, var. *guadalupensis* (Dawson) (Howard & Fenical, 1980). The structure of the compound was proposed as shown in (1), mainly on the basis of its spectral data. A recent report (Murai, Abiko & Masamune, 1984) concerning the synthesis of the C(13) diastereoisomers of racemic (1), agrees that

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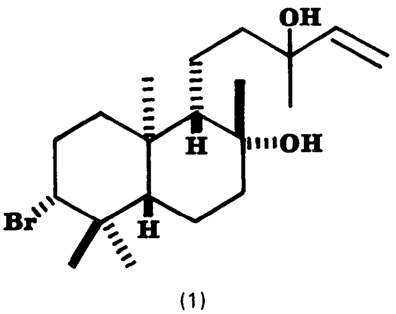
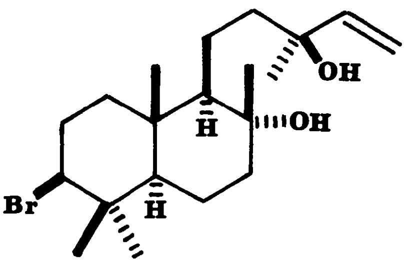
neither of the diastereoisomers is natural isoconcinndiol, and the proposed structure should be revised.



(1)



(2)

**Experimental.** Colourless crystal, 0·2 × 0·4 × 0·9 mm. Diffraction maxima were collected on a computer-controlled four-circle Siemens AED diffractometer, using graphite-monochromated Cu $K\alpha$ radiation and $\omega$:$\theta$ scan mode. Two standard reflections monitored every hour showed no significant intensity decay. Cell parameters were established by least-squares adjustment of 22 reflections in the range $20° < 2\theta < 30°$. Of 684 measured independent reflections in the range $3° < 2\theta < 100°$, 682 with $I > 3\sigma(I)$ were judged as observed and corrected for Lorentz and polarization factors, index range $h$ 0,4; $k$ 0,12; $l$ 0,23; no absorption correction was performed. The structure was solved by direct methods (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Atomic coordinates and displacement parameters, anisotropic for Br and isotropic for the other non-hydrogen atoms, were refined by least squares using unit weights (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Most of the H atoms were located on a difference electron-density map and the remainder in calculated positions (Fayos & Martinez-Ripoll, 1980). An appropriate weighting scheme to normalize $\langle w\Delta^2F\rangle$ *vs* $\langle F_o\rangle$ and $\langle(\sin\theta)/\lambda\rangle$ was carried out (Martinez-Ripoll & Cano, 1980). A final weighted anisotropic full-matrix refinement (fixed isotropic contribution for H atoms) gave for the correct enantiomer the discrepancy indices $R = 0{\cdot}051$ and $wR = 0{\cdot}063$.

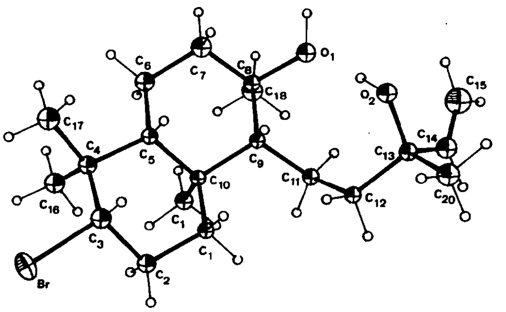


Fig. 1. Perspective view of the title compound.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and* $U_{eq}$ *values* (Å$^2$)

$U_{\text{eq}} = \frac{1}{3}\sum_i\sum_j[U_{ij}a_i^*a_j^*a_ia_j\cos(\mathbf{a}_i,\mathbf{a}_j)] \times 10^3$.

| | $x$ | $y$ | $z$ | $U_{\text{eq}}$ |
|---|---|---|---|---|
| Br | 4439 (3) | 1845 (1) | 2853 (1) | 72 (1) |
| O(1) | −933 (15) | 6690 (5) | 4646 (2) | 45 (2) |
| O(2) | 2077 (16) | 7966 (5) | 4392 (2) | 44 (2) |
| C(1) | 2105 (22) | 4923 (8) | 3052 (4) | 32 (3) |
| C(2) | 2740 (22) | 3891 (8) | 2765 (4) | 39 (3) |
| C(3) | 3305 (24) | 3099 (8) | 3240 (4) | 46 (3) |
| C(4) | 1658 (23) | 2822 (7) | 3653 (4) | 39 (3) |
| C(5) | 916 (23) | 3906 (7) | 3900 (3) | 32 (3) |
| C(6) | −802 (30) | 3794 (8) | 4355 (4) | 45 (3) |
| C(7) | −890 (26) | 4810 (8) | 4699 (4) | 51 (3) |
| C(8) | −1353 (24) | 5748 (7) | 4321 (4) | 33 (3) |
| C(9) | 212 (24) | 5835 (6) | 3825 (3) | 30 (2) |
| C(10) | 372 (25) | 4780 (6) | 3459 (4) | 29 (2) |
| C(11) | −89 (23) | 6815 (7) | 3448 (3) | 34 (2) |
| C(12) | 1671 (23) | 7491 (7) | 3386 (3) | 30 (2) |
| C(13) | 1952 (25) | 8385 (8) | 3825 (4) | 37 (3) |
| C(14) | 249 (28) | 9168 (8) | 3765 (4) | 52 (3) |
| C(15) | −976 (35) | 9432 (11) | 4168 (6) | 84 (5) |
| C(16) | 101 (27) | 2173 (8) | 3371 (4) | 50 (3) |
| C(17) | 2493 (27) | 2170 (9) | 4143 (5) | 58 (3) |
| C(18) | −1491 (24) | 4545 (8) | 3099 (4) | 41 (3) |
| C(19) | −3447 (29) | 5803 (9) | 4142 (5) | 62 (4) |
| C(20) | 3859 (26) | 8931 (9) | 3725 (5) | 55 (3) |

The absolute configuration was determined by comparison of 40 Bijvoet pairs with $F_o > 10\sigma(F_o)$, which are in the range $5 < F_c < 50$ and $0{\cdot}08 < (\sin\theta)/\lambda < 0{\cdot}45$ Å$^{-1}$ (Martinez-Ripoll & Fayos, 1980). The averaged Bijvoet differences are 0·77 for the correct enantiomer *vs* 3·66 for the wrong one.

Scattering factors from *International Tables for X-ray Crystallography* (1974), maximum electron density in final difference map 0·26 e Å$^{-3}$, maximum $\Delta/\sigma$ 0·16 (for non-H atoms).*

**Discussion.** Since the spectroscopic data of our natural sample, isolated from *Aplysia dactylomela*, are identical to those given by Howard & Fenical (1980) for isoconcinndiol and since the synthetic C(13) diastereoisomeric diols (1) are both different from the natural

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, absolute configuration data and complete geometrical data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51384 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| Br–C(3) | 1·991 (11) | C(6)–C(7) | 1·522 (14) |
| C(1)–C(8) | 1·447 (11) | C(7)–C(8) | 1·518 (14) |
| O(2)–C(13) | 1·428 (11) | C(8)–C(9) | 1·577 (18) |
| C(1)–C(2) | 1·537 (14) | C(8)–C(19) | 1·491 (25) |
| C(1)–C(10) | 1·526 (19) | C(9)–C(10) | 1·595 (12) |
| C(2)–C(3) | 1·548 (14) | C(9)–C(11) | 1·540 (12) |
| C(3)–C(4) | 1·520 (19) | C(10)–C(18) | 1·552 (21) |
| C(4)–C(5) | 1·579 (14) | C(11)–C(12) | 1·485 (19) |
| C(4)–C(16) | 1·499 (20) | C(12)–C(13) | 1·543 (13) |
| C(4)–C(17) | 1·522 (16) | C(13)–C(14) | 1·538 (22) |
| C(5)–C(6) | 1·587 (21) | C(13)–C(20) | 1·494 (23) |
| C(5)–C(10) | 1·560 (13) | C(14)–C(15) | 1·301 (24) |
| | | | |
| C(2)–C(1)–C(10) | 112·8 (8) | C(1)–C(2)–C(3) | 108·4 (8) |
| Br–C(3)–C(2) | 107·2 (6) | C(2)–C(3)–C(4) | 114·8 (10) |
| Br–C(3)–C(4) | 112·9 (7) | C(3)–C(4)–C(17) | 108·9 (12) |
| C(3)–C(4)–C(16) | 112·0 (8) | C(3)–C(4)–C(5) | 105·4 (8) |
| C(16)–C(4)–C(17) | 107·1 (9) | C(5)–C(4)–C(17) | 108·9 (8) |
| C(5)–C(4)–C(16) | 114·4 (11) | C(4)–C(5)–C(10) | 117·4 (7) |
| C(4)–C(5)–C(6) | 113·7 (8) | C(6)–C(5)–C(10) | 109·1 (9) |
| C(5)–C(6)–C(7) | 107·7 (10) | C(6)–C(7)–C(8) | 111·8 (8) |
| O(1)–C(8)–C(7) | 107·9 (7) | C(7)–C(8)–C(19) | 113·5 (10) |
| C(7)–C(8)–C(19) | 113·5 (10) | C(7)–C(8)–C(9) | 109·8 (9) |
| O(1)–C(8)–C(19) | 107·3 (9) | O(1)–C(8)–C(9) | 101·0 (8) |
| C(9)–C(8)–C(19) | 116·2 (8) | C(8)–C(9)–C(11) | 112·6 (9) |
| C(8)–C(9)–C(10) | 112·3 (8) | C(10)–C(9)–C(11) | 112·8 (7) |
| C(5)–C(10)–C(9) | 105·4 (7) | C(1)–C(10)–C(9) | 106·5 (9) |
| C(1)–C(10)–C(5) | 108·0 (10) | C(9)–C(10)–C(18) | 113·2 (9) |
| C(5)–C(10)–C(18) | 114·3 (8) | C(1)–C(10)–C(18) | 108·9 (8) |
| C(9)–C(11)–C(12) | 114·7 (9) | C(11)–C(12)–C(13) | 117·6 (9) |
| O(2)–C(13)–C(12) | 110·1 (7) | C(12)–C(13)–C(20) | 110·4 (9) |
| C(12)–C(13)–C(14) | 108·9 (9) | O(2)–C(13)–C(20) | 108·9 (9) |
| O(2)–C(13)–C(14) | 111·8 (8) | C(14)–C(13)–C(20) | 110·1 (10) |
| C(13)–C(14)–C(15) | 126·1 (11) | | |

sample, the structure of isoconcinndiol might differ from (1) in the stereochemistry at C(8) and should be represented by (2) or its C(13) diastereoisomer. The X-ray single-crystal analysis described in this paper confirmed the structure and absolute configuration of (+)-isoconcinndiol as (3*S*,8*R*,13*S*)-labd-14-ene-8,13-diol, depicted in (2).

Fig. 1 is a computer-generated perspective molecular drawing, showing the absolute configuration. Atomic parameters are given in Table 1. Bond distances and angles are listed in Table 2.

The six-membered rings in isoconcinndiol are *trans* fused and are both in a chair conformation. The OH group on C(8) and the side chain on C(9) are equatorial.

The crystal structure is built by hydrogen bonds, comprising an intramolecular O(2)H···O(1), with distance 2·68 (1) Å and angle 165·4 (6)°, and another intermolecular O(1)H···O(2), through symmetry operation $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$, with distance 2·65 (1) Å and angle 166·9 (6)°.

This work was supported by grants from the Spanish CAICYT to JDM and MLR.

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*Acta Cryst.* (1989). C**45**, 308–311

# Structure of Bis(4,4,5,5-tetramethyl-2-thioxo-1,3,2$\lambda^5$-dioxaphospholan-2-yl) Disulfide, $[\overline{\text{OC(Me)}_2\text{C(Me)}_2\text{OP}}\text{(S)S}]_2$

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(*Received* 20 *May* 1988; *accepted* 18 *August* 1988)

**Abstract.** $C_{12}H_{24}O_4P_2S_4$, $M_r = 422·5$, monoclinic, space group $C2$, $a = 14·913(9)$, $b = 6·847(6)$, $c = 11·427(6)$ Å, $\beta = 120·18(6)°$, $V = 1008·7$ Å$^3$, $Z = 2$, $D_x = 1·39$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 6·195$ cm$^{-1}$, $T = 295$ K, $F(000) = 444$, $R = 0·0656$ for 957 unique reflections. The molecule has a twofold axis of symmetry and contains a planar zigzag array of S=P–S–S–P=S linkages with two dioxaphospholane

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0108-2701/89/020308-04$03.00

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