0·59470 Å^{-1} in h 0/18, k 0/12 and l −14/14, θ–2θ scans, θ-scan width (1·40 + 0·35 tanθ)°, three standard reflections monitored every 100 reflections showed no significant variation in intensity. 2572 unique reflections measured, 2108 intensities observed [$F_o > 3\sigma(F_o)$], no absorption correction. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971). H atoms located on a difference map. Positional parameters of all atoms and anisotropic thermal parameters for P, O, N and C atoms and isotropic thermal parameters for H atoms refined by block-diagonal least squares (Ashida, 1973). $\sum w|\Delta F|^2$ minimized with $w = 1{\cdot}0$ for $0 < F_o < 56$ and $w = [1{\cdot}0 + 0{\cdot}167(F_o - 56)]^{-1}$ for $F_o > 56$. Final $R = 0{\cdot}059$, $wR = 0{\cdot}067$ and $S = 1{\cdot}229$. Maximum positive and maximum negative electron densities in final difference Fourier synthesis are 0·53 and −0·47 e Å^{-3}. Δ/σ in the final cycle = 0·3 (z coordinate of an H atom). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a HITAC 280D at the Tottori University Computing Center and on a FACOM M780/30 at the Data Processing Center of Kyoto University. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond distances and angles are listed in Table 2.* A stereoview of the molecule with atomic numbering drawn by *DCM*-3 (Takenaka, 1977) is shown in Fig. 1.

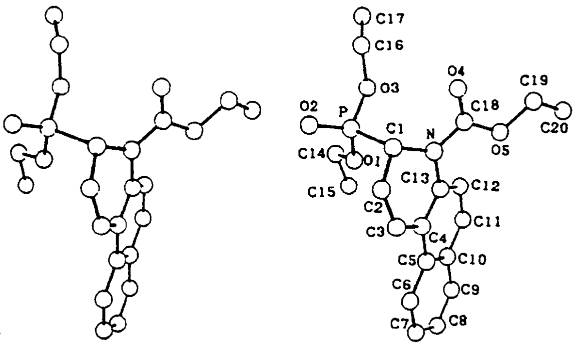

Fig. 1. Stereoview of the title molecule with the atomic numbering system. The absolute configuration of the molecule is arbitrary.

Related literature. The detailed synthetic method and spectral data of the title compound and related compounds are presented by Takeuchi *et al.* (1984).

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* Lists of structure factors, anisotropic temperature factors of the non-H atoms, and isotropic temperature factors and positional parameters of H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52206 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (±)-2-Hydroxymethyl-2,6-dimethylcyclohexan-1-ol

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Abstract. $C_9H_{18}O_2$, $M_r = 158{\cdot}2$, monoclinic, $C2/c$, $a = 21{\cdot}592\,(4)$, $b = 6{\cdot}096\,(1)$, $c = 15{\cdot}166\,(3)$ Å, $\beta = 115{\cdot}00\,(1)°$, $V = 1809{\cdot}2\,(6)$ Å^3, $Z = 8$, $D_m = 1{\cdot}15\,(1)$, $D_x = 1{\cdot}16$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}074$ mm^{-1}, $F(000) = 704$, $T = 298\,(2)$ K, $R = 0{\cdot}044$ for 1274 unique observed reflections. The relative configuration of (±)-2-ethoxycarbonyl-2,6-dimethylcyclohexan-1-one having a 2-methyl substituent chemical shift of δ 1·28 was determined to be (2R*,6R*) by the structure determination of its derivative. The title molecules are linked into infinite chains along **c** by intermolecular hydrogen bonds between the hydroxy groups with O···O distances 2·773 (3) and 2·985 (5) Å.

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Table 1. *Positional parameters* ($\times 10^4$) *and equivalent isotropic temperature factors* (*Hamilton*, 1959)

	x	y	z	B_{eq} (Å^2 × 10)
O(1)	9498 (1)	4350 (2)	5391 (1)	39
O(2)	9536 (1)	−2030 (2)	6530 (1)	44
C(1)	8884 (1)	3066 (3)	4965 (1)	26
C(2)	8893 (1)	1244 (3)	5679 (1)	27
C(3)	8240 (1)	−129 (3)	5194 (1)	38
C(4)	8122 (1)	−1000 (3)	4200 (1)	43
C(5)	8126 (1)	839 (3)	3531 (1)	38
C(6)	8777 (1)	2221 (3)	3964 (1)	28
C(7)	8911 (1)	2282 (3)	6605 (1)	42
C(8)	9532 (1)	−188 (3)	5946 (1)	31
C(9)	8753 (1)	4044 (3)	3264 (1)	41

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

O(1)—C(1)	1·437 (3)	C(2)—C(8)	1·535 (3)
O(2)—C(8)	1·428 (2)	C(3)—C(4)	1·515 (2)
C(1)—C(2)	1·546 (3)	C(4)—C(5)	1·514 (3)
C(1)—C(6)	1·525 (2)	C(5)—C(6)	1·529 (3)
C(2)—C(3)	1·534 (3)	C(6)—C(9)	1·523 (3)
C(2)—C(7)	1·526 (3)		
O(1)···O(1^{i})	2·985 (5)	O(2)···O(2^{ii})	2·773 (3)
O(1)—C(1)—C(2)	109·9 (1)	C(7)—C(2)—C(8)	108·8 (1)
O(1)—C(1)—C(6)	110·8 (2)	C(2)—C(3)—C(4)	113·7 (2)
C(2)—C(1)—C(6)	114·0 (1)	C(3)—C(4)—C(5)	111·2 (1)
C(1)—C(2)—C(3)	108·6 (1)	C(4)—C(5)—C(6)	112·6 (1)
C(1)—C(2)—C(7)	109·6 (1)	C(1)—C(6)—C(5)	109·8 (2)
C(1)—C(2)—C(8)	109·7 (2)	C(1)—C(6)—C(9)	113·2 (1)
C(3)—C(2)—C(7)	109·2 (2)	C(5)—C(6)—C(9)	110·4 (1)
C(3)—C(2)—C(8)	111·0 (2)	O(2)—C(8)—C(2)	112·3 (2)

Symmetry code: (i) $2 - x, 1 - y, 1 - z$; (ii) $2 - x, y, \frac{3}{2} - z$.

Experimental. Colorless prisms grown from a mixed solvent of hexane and ethyl acetate. D_m measured by flotation in an aqueous NaCl solution. Crystal size 0·4 × 0·4 × 0·5 mm, Rigaku AFC-5 four-circle diffractometer, graphite-monchromatized Mo *Kα* radiation, Laue group $2/m$, cell parameters for least-squares refinement of 24 reflections ($20 < 2\theta < 30°$). Intensity measurement performed to $2\theta = 50°$ (h $-25 \rightarrow 25$, k $-7 \rightarrow 0$, l $0 \rightarrow 18$), θ–2θ scan, scan speed 6° min^{-1} in θ. Five standard reflections showed a decay, $0{\cdot}896 < |F_o|/|F_c|_{\rm initial} < 1{\cdot}015$, and a linear correction was applied. Systematic absences (hkl, $h + k$ odd; $h0l$, l odd) indicated the space group to be *Cc* or *C2/c*. 1823 reflections measured, 1322 observed with $|F_o| > 3\sigma(|F_o|)$, 1274 unique after absorption correction ($R_{\rm int} = 0{\cdot}009$; $0{\cdot}968 < A < 0{\cdot}977$). Assuming the centrosymmetric space group *C2/c*, the structure could be successfully solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); positions of all the non-H atoms determined from *E* map and refined by block-diagonal least squares with anisotropic thermal parameters ($R = 0{\cdot}11$) using the *UNICS*III program system (Sakurai & Kobayashi, 1979); all the H atoms were located on a difference synthesis and refined with isotropic thermal parameters. $\sum w||F_o| - |F_c||^2$ minimized, $w^{-1} = \sigma^2(|F_o|) + (0{\cdot}015|F_o|)^2$. Final $R = 0{\cdot}044$, $wR = 0{\cdot}049$, $S = 1{\cdot}95$ for 1274 reflections.* Reflection/parameter ratio 7·0, $\Delta/\sigma < 0{\cdot}20$, $-0{\cdot}16 < \Delta\rho < 0{\cdot}14$ e Å^{-3}. Complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Parameters are given in Table 1, and bond lengths and bond angles in Table 2. The molecular structure is shown in Fig. 1 and the crystal structure is presented in Fig. 2. Each hydrogen bond passes through a center of symmetry or a twofold axis. Therefore, each H atom of the hydroxy groups was considered to have two possible positions, which were located in a difference synthesis. Between the hydroxy groups of two neighbouring molecules there is a 50% probability of an O—H···O′ or O···H′—O′ hydrogen bond existing.

Related literature. In the study of chiral synthesis by utilizing micro-organisms and enzymes, the relative configuration of one of the key intermediates (1) in the synthesis of certain diterpenes had to be determined. The 2-methyl substituent chemical shift δ is 1·28 for (1) and 1·45 for its epimer (Weiss, 1978). Since the compound did not crystallize, the title compound (±)-(2) was prepared by reduction of

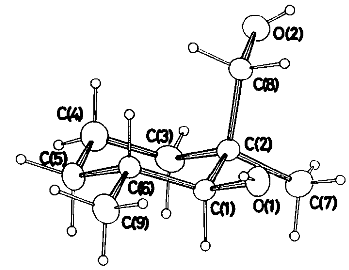

Fig. 1. An *ORTEP* drawing (Johnson, 1965) of the molecule with 25% probability ellipsoids. H atoms are represented by circles of radius 0·08 Å. For the hydroxy H atoms, only one of two possible positions is shown for clarity.

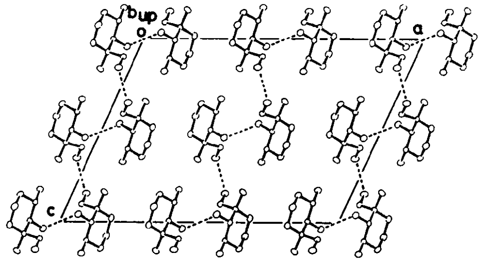

Fig. 2. The crystal structure viewed along **b**. Hydrogen bonds are represented by dashed lines.

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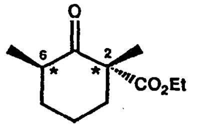
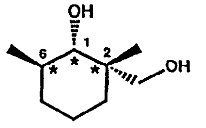
* Lists of structure factors, anisotropic thermal parameters, atomic parameters for H atoms, bond lengths and bond angles involving H atoms, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52223 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(±)-(1) with $LiAlH_4$ (Sugai, Kakeya, Ohta, Morooka & Ohba, 1989). The relative configuration of C(2) and C(6) revealed in the present study is in agreement with the assignment based on the ^1H NMR spectra (Weiss, 1978). The absolute configuration around C(2) in (−)-(1) was determined to be *R* by converting it to (−)-deoxypodocarpic acid methyl ester (Sugai *et al.*, 1989).



(2*R**,6*R**)-(±)-(1)



(1*S**,2*S**,6*R**)-(±)-(2)

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Structure of a Hexahydrodithia[3.3]paracyclophane Involving *trans* Bonds to the Cyclohexane Ring

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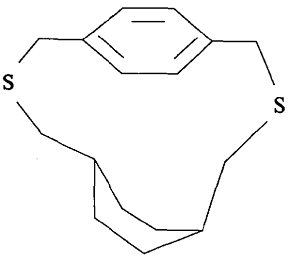
Abstract. 4,5,6,7,8,9-Hexahydro-2,11-dithia[3.3]paracyclophane, $C_{16}H_{22}S_2$, M_r = 278·5, monoclinic, $P2_1/n$, a = 8·851 (1), b = 15·398 (2), c = 11·590 (2) Å, β = 108·86 (1)°, V = 1494·8 (7) Å^3, Z = 4, D_x = 1·24 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 3·2 cm^{-1}, $F(000)$ = 600, T = 293 K, R = 0·036 for 1586 reflections with $F_o^2 > 3\sigma(F_o^2)$. The two —CH_2SCH_2— linkages which bond the phenylene group to the cyclohexyl ring have C—S—C angles of 101·7 (2) and 102·8 (2)°. The cyclohexyl ring is in the twisted boat form with four C—C—C—C torsion angles of 23·7 to 36·1° and two of −58·4 and −63·8°. There are four intramolecular contacts between H atoms and the phenylene ring in the range 2·53 to 2·68 Å which contribute to the 'bent benzene' typical for paracyclophanes: in the aromatic ring the C atoms bonded to the CH_2 groups are displaced 0·057 (2) and 0·065 (3) Å from the plane of the other four C atoms, and the C atoms of these CH_2 groups are 0·303 (3) and 0·358 (3) Å from the same plane.

Experimental. Title compound (I) obtained by the slow addition of *trans*-1,4-bis(mercaptomethyl)cyclohexane and dichloroxylene to basic ethanol using high-dilution techniques. The product was obtained after chromatographic separation on a silica-gel column. The colorless data crystal 0·30 × 0·30 × 0·40 mm was mounted with epoxy on a glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4–16° min^{-1} in θ. Unit cell determined from least-squares analysis of



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