

Fig. 1. View of (1) showing the atom-numbering scheme.

M^{II} complexes ($M^{II} = \text{Ni, Cu, Zn}$) have been prepared (Fikar, Bharadwaj, Potenza & Schugar, unpublished results).

As indicated by the torsion angles and bond distances, the molecule shows approximate point symmetry 2 with the pseudo-diad axis normal to the S(2)–S(1) and C(5)–C(6) bond vectors. The C(1)–S(1)–S(2)–C(10) torsion angle in the ten-membered macrocycle ring is substantially larger than that found [52 (1)° (Cheng & Nyburg, 1978)] in the six-membered ring of an oxadithiin derivative and lies within the range observed [78.6 to 101° (Rout, Seshasayee, Subrahmanyam & Aravamudan, 1983)] for several acyclic and presumably unstrained disulfides. Both imino groups are *trans* substituted [C(8)–N(2)–C(9)–C(10), –172.6 (2)°; C(3)–N(1)–C(2)–C(1),

–173.7 (2)°] as is the cyclohexane ring which has the stable chair conformation. The N(1)–C(3)–C(8)–N(2) torsion angle [–63.1 (2)°] is substantially larger than those reported for protonated [–58.5, 58.3° (Morse & Chesick, 1976)] or chelated [53.8, 52.8° (Sato & Saito, 1977)] 1,2-*trans*-diaminocyclohexane, possibly as a result of constraints imposed by the ten-membered ring. Other structural parameters of the cyclohexane ring, including the C–C–C–C torsion angles, are typical.

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1-*p*-Menthene-3,6-diol, a Monoterpene

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Abstract. $\text{C}_{10}\text{H}_{18}\text{O}_2$, $M_r = 170.25$, monoclinic, C_2 , $a = 17.837$ (4), $b = 7.113$ (7), $c = 8.096$ (2) Å, $\beta = 102.17$ (6)°, $V = 1004$ (2) Å³, $Z = 4$, $D_x = 1.13$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 0.7$ cm⁻¹, $F(000) = 376$, $T = 297$ K, $R = 0.036$ for 973 reflections with $I > 2.5\sigma(I)$ (1218 unique). The cyclohexene ring itself is in the half-chair conformation, very close to twofold symmetry [$\Delta C_2 = 1.3$ (2)°] with virtually no torsion [0.5 (3)°] about the double bond. The two

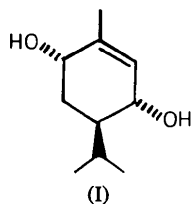
hydroxyl groups are oriented *syn* with respect to one another, *anti* with respect to the isopropyl group; each hydroxyl is in contact with opposite-number hydroxyls on two neighboring molecules [O...O = 2.691 (2), 2.728 (2) Å], resulting in disordered hydrogen bonding.

Experimental. The title compound (I) was isolated from *Melampodium divaricatum* (Rich. in Pers.) DC. (Asteraceae) from Provincia de Alajuela, Costa Rica;

Table 1. *Experimental details*

Crystal	Colorless, 0.6 × 0.4 × 0.2 mm
Instrument	Enraf-Nonius CAD-4 diffractometer
Monochromator	Incident beam, graphite
Unit cell	25 reflections, 22 < 2θ < 24°
Mode	ω-2θ
Standards	10,0,0, 020, 002; ±3.6% variation
R _{int}	0.026
Corrections	Background, Lorentz, polarization
2θ range	2-54°
hkl ranges	h = 0 to 22 k = 0 to 9 l = -10 to 10
Reflections	1291 total 1218 unique 245 unobserved 973 with I > 2.5σ(I)
Solution	Direct methods
Function minimized	Σw(F _o - F _c ²)
Weights	4F _o ² Lp ² /[S ² (C+R ² B)+(0.04F _o ²) ²] S = scan rate, C = integrated count, R = scan time/background time, B = background count
Parameters refined	125
R, wR, R(all)	0.036, 0.048, 0.057
Goodness of fit	1.91
Maximum shift/e.s.d.	0.01
Δρ	0.14, -0.18 e Å ⁻³

collected on 3 July 1981 (C. Hasbun No. 313, voucher at Universidad Nacional Heredia, Costa Rica). A clear, colorless sample was cleaved from a larger prismatic crystal and mounted with epoxy on a glass fiber in random orientation. Details of data collection and structural refinement are given in Table 1.



Structure solved using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); all 12 non-H atoms were located from an *E* map. Refinement by full-matrix least squares with Enraf-Nonius *SDP* (Frenz, 1978); non-H atoms anisotropic; all non-hydroxyl H atoms placed in computed positions with independent isotropic thermal parameters but riding C atoms to which they are attached; disordered half-H atoms located on both hydroxyl groups by ΔF syntheses, then adjusted for reasonable geometry to ride O atoms with fixed thermal parameters. The final cycle of refinement included 125 variable parameters and converged to $R = 0.036$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The absolute configuration was not determined.

Table 2. *Positional and equivalent isotropic thermal parameters and their e.s.d.'s*

	x	y	z	B _{eq} * (Å ²)
C1	0.8942 (1)	0.232	0.6299 (2)	3.44 (4)
C2	0.8875 (1)	0.4152 (4)	0.6476 (3)	3.40 (4)
C3	0.8717 (1)	0.5547 (3)	0.5058 (2)	3.10 (4)
C4	0.8421 (1)	0.4614 (3)	0.3343 (2)	2.94 (4)
C5	0.8915 (1)	0.2887 (3)	0.3227 (2)	3.24 (4)
C6	0.8865 (1)	0.1440 (3)	0.4568 (2)	3.22 (4)
C7	0.9101 (2)	0.0978 (4)	0.7774 (3)	5.04 (6)
C8	0.8354 (1)	0.5993 (3)	0.1847 (2)	3.73 (4)
C9	0.7867 (1)	0.5161 (4)	0.0231 (3)	4.87 (6)
C10	0.9121 (2)	0.6640 (5)	0.1523 (3)	6.11 (6)
O1	0.81554 (8)	0.0403 (2)	0.4117 (2)	4.10 (3)
O2	0.81920 (9)	0.6924 (2)	0.5437 (2)	4.17 (3)

* The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation:

$$1.333(a^2B_{11} + b^2B_{22} + c^2B_{33} + bcB_{23}\cos\alpha + acB_{13}\cos\beta + abB_{12}\cos\gamma).$$

Table 3. *Bond lengths (Å), bond angles (°) and torsion angles (°)*

C1 C2	1.320 (3)	C4 C5	1.526 (2)
C1 C6	1.514 (2)	C4 C8	1.544 (2)
C1 C7	1.508 (3)	C5 C6	1.512 (2)
C2 C3	1.498 (3)	C6 O1	1.444 (2)
C3 C4	1.529 (2)	C8 C9	1.529 (3)
C3 O2	1.432 (2)	C8 C10	1.517 (3)
C2 C1 C6	121.0 (2)	C5 C4 C8	114.4 (1)
C2 C1 C7	123.0 (2)	C4 C5 C6	112.5 (1)
C6 C1 C7	115.9 (2)	C1 C6 C5	112.1 (1)
C1 C2 C3	125.3 (2)	C1 C6 O1	110.3 (1)
C2 C3 C4	112.4 (1)	C5 C6 O1	110.3 (1)
C2 C3 O2	108.4 (1)	C4 C8 C9	111.2 (2)
C4 C3 O2	112.0 (1)	C4 C8 C10	113.9 (2)
C3 C4 C5	108.2 (1)	C9 C8 C10	109.9 (2)
C3 C4 C8	113.1 (1)		
C6 C1 C2 C3	0.5 (3)	O2 C3 C4 C5	-167.2 (2)
C7 C1 C2 C3	180.0 (6)	O2 C3 C4 C8	65.1 (2)
C2 C1 C6 C5	14.6 (3)	C3 C4 C5 C6	61.8 (2)
C2 C1 C6 O1	-108.8 (2)	C8 C4 C5 C6	-171.2 (2)
C7 C1 C6 C5	-165.0 (2)	C3 C4 C8 C9	-165.0 (2)
C7 C1 C6 O1	71.7 (2)	C3 C4 C8 C10	70.2 (2)
C1 C2 C3 C4	15.8 (3)	C5 C4 C8 C9	70.6 (2)
C1 C2 C3 O2	140.2 (2)	C5 C4 C8 C10	-54.2 (2)
C2 C3 C4 C5	-44.8 (2)	C4 C5 C6 C1	-46.2 (2)
C2 C3 C4 C8	-172.6 (2)	C4 C5 C6 O1	77.1 (2)

Final positional and equivalent isotropic thermal parameters for all non-H atoms are given in Table 2, and bond lengths, bond angles and torsion angles are shown in Table 3.* Fig. 1 shows the molecule and the atomic numbering scheme.

Related literature. Isolation from *Eupatorium macrocephalum* and structure by NMR: Gonzales, Bermejo Barrera, Bermejo Barrera & Massanet (1972); crystal

* Lists of structure factors, anisotropic thermal parameters and H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44606 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

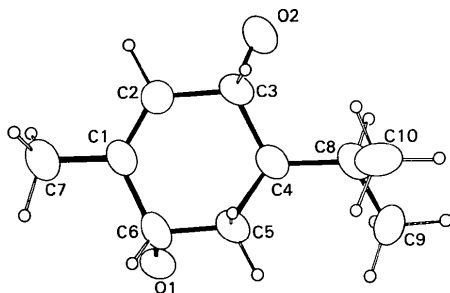


Fig. 1. Molecule of $C_{10}H_{18}O_2$, 50% ellipsoids (Johnson, 1965).

structure of *trans*-1(7)-*p*-menthene-2,8-diol: Scott & Richards (1971); structure of menthyl trimethylammonium iodide: Gabe & Grant (1962); systematics and classification of the genus *Melampodium*: Stuessy (1972).

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Structure of 3'-Azido-3'-deoxythymidine, AZT

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Abstract. $C_{10}H_{13}N_5O_4$, $M_r = 267.2$, monoclinic, $P2_1$, $a = 5.716$ (3), $b = 11.998$ (8), $c = 17.658$ (10) Å, $\beta = 94.26$ (4)°, $V = 1208$ Å³, $Z = 4$, $D_x = 1.47$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 7.5$ cm⁻¹, $F(000) = 560$, $T = 293$ K. $R = 0.060$ for 2138 unique observed [$F > 4\sigma(F)$] reflections. The *N*-glycosidic torsion angles χ have values -125.9 (5) and -172.0 (5)°, in the *anti* range. (Molecule-*A* values are given first throughout.) The sugar puckers are 2_3T (C3'-*exo*/C2'-*endo*), with $P = 171$ (1)° and $\psi_m = 14$ (1)°, and 4_3T (C4'-*endo*/C3'-*exo*), with $P = 213$ (1)° and $\psi_m = 11$ (1)°. The C4–C5 conformations, with $\gamma = 49.7$ (5) and 173.7 (5)°, are *+sc* (*gauche-gauche*) and *ap* (*gauche-trans*). The conformational parameters used follow the guidelines of the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280]. The molecules in the asymmetric unit form a hydrogen-bonded, base-paired dimer. The

bonding is as follows: N3A–0.973 Å–H3A...1.790 Å...O2B, N3A...O2B 2.747 (8) Å, angle at H3A 167° and N3B–0.992 Å–H3B...1.916 Å...O2A, N3B...O2A 2.894 (8) Å, angle at H3B 168°. The propeller twist between the bases is 5° [Wilson & Tollin (1987). *Nucleosides Nucleotides*, **6**, 643–653].

Experimental. Crystals were obtained from aqueous solution. Space group and initial cell dimensions were obtained from Weissenberg photographs. Data were collected on a Nicolet P3 (four-circle) diffractometer in Aberdeen by RAH. The crystal had dimensions 0.6 × 0.3 × 0.2 mm. Cell parameters were measured on the diffractometer using 14 reflections in the 2θ range 15–22°. Range of indices: $0 \leq h \leq 9$; $0 \leq k \leq 18$; $-26 \leq l \leq 26$. Data measured using $\theta/2\theta$ scans in the range $0 < 2\theta < 55^\circ$. Standard reflections, 253 and 106, were measured every 50 reflections. No

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