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

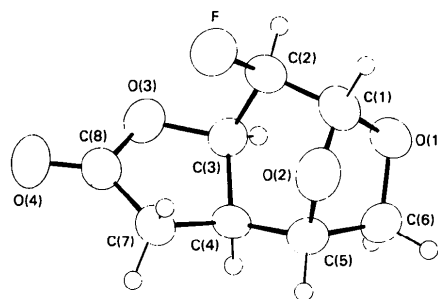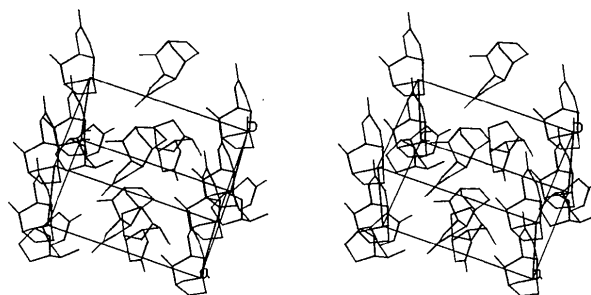|			
F—C(2)	1·396 (3)	O(1)—C(1)	1·419 (4)
O(1)—C(6)	1·435 (3)	O(2)—C(1)	1·411 (4)
O(2)—C(5)	1·438 (3)	O(3)—C(3)	1·439 (3)
O(3)—C(8)	1·355 (3)	O(4)—C(8)	1·198 (3)
C(1)—C(2)	1·503 (4)	C(2)—C(3)	1·514 (4)
C(3)—C(4)	1·527 (4)	C(4)—C(5)	1·515 (4)
C(4)—C(7)	1·521 (4)	C(5)—C(6)	1·522 (4)
C(7)—C(8)	1·498 (4)		
C(1)—O(1)—C(6)	107·1 (2)	C(1)—O(2)—C(5)	101·9 (2)
C(3)—O(3)—C(8)	110·5 (2)	O(1)—C(1)—O(2)	106·8 (3)
O(1)—C(1)—C(2)	108·6 (3)	O(2)—C(1)—C(2)	109·4 (2)
F—C(2)—C(1)	107·4 (2)	F—C(2)—C(3)	109·2 (2)
C(1)—C(2)—C(3)	109·6 (2)	O(3)—C(3)—C(2)	110·0 (2)
O(3)—C(3)—C(4)	104·8 (2)	C(2)—C(3)—C(4)	114·1 (2)
C(3)—C(4)—C(5)	111·8 (2)	C(3)—C(4)—C(7)	101·9 (2)
C(5)—C(4)—C(7)	116·5 (2)	O(2)—C(5)—C(4)	109·3 (2)
O(2)—C(5)—C(6)	100·8 (2)	C(4)—C(5)—C(6)	112·1 (2)
O(1)—C(6)—C(5)	102·8 (2)	C(4)—C(7)—C(8)	103·5 (2)
O(3)—C(8)—O(4)	119·9 (3)	O(3)—C(8)—C(7)	109·7 (2)
O(4)—C(8)—C(7)	130·4 (3)		

Fig. 1. The atomic arrangement.

are listed in Table 1,* bond lengths and valency angles in Table 2. The atomic arrangement and crystal packing are shown in Figs. 1 and 2.

Related literature. Tricyclic lactones have been used as synthetic intermediates en route to thromboxanes (Kelly & Roberts, 1980). A detailed NMR study of the title compound (I) has shown interesting long-range couplings between F and H(7A), H(7B) and between F and C(4), C(7) (Roberts, 1986). We undertook the X-ray study to obtain accurate geometrical details of the molecule with particular reference to the F environment.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, selected mean planes and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44586 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 2. Crystal packing diagram.

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Structure of a Novel, Macrocyclic Schiff Base

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Abstract. (*E,E*)-1,2,3,4,4aα,12aβ-Hexahydro-7,7,10,-10-tetramethyl-7*H*,10*H*-8,9-dithia-5,12-diazabenzo-cyclodecene, $C_{14}H_{24}N_2S_2$, $M_r = 284·49$, monoclinic, $P2_1/n$, $a = 8·962$ (1), $b = 19·356$ (3), $c = 10·178$ (1) Å, $\beta = 114·49$ (1)°, $V = 1606·7$ (7) Å^3, $Z = 4$, $D_m = 1·21$ (1), $D_x = 1·176$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0·71073$ Å) = 0·31 mm^{-1}, $F(000) = 616$, $T = 296$ (1) K, $R_F = 0·031$ for 2066 reflections. The mole-

Table 1. *Fractional coordinates and isotropic thermal parameters for non-hydrogen atoms in* (1)

$$B_{eq} = 4\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j/3.$$

	x	y	z	B or $B_{eq}(\text{Å}^2)$
S(1)	0.48733 (6)	0.10347 (3)	0.87346 (5)	4.36 (1)
S(2)	0.41989 (6)	0.10116 (3)	0.65761 (5)	4.34 (1)
N(1)	0.3589 (2)	0.28795 (7)	0.8315 (1)	3.36 (3)
N(2)	0.5620 (2)	0.28233 (7)	0.6824 (1)	3.41 (3)
C(1)	0.3660 (2)	0.17050 (9)	0.9183 (2)	3.51 (4)
C(2)	0.4389 (2)	0.23890 (9)	0.9094 (2)	3.33 (4)
C(3)	0.4521 (2)	0.34841 (9)	0.8238 (2)	3.31 (4)
C(4)	0.3675 (2)	0.4149 (1)	0.8345 (2)	4.30 (4)
C(5)	0.4548 (3)	0.4779 (1)	0.8127 (2)	4.69 (5)
C(6)	0.4658 (3)	0.4746 (1)	0.6677 (2)	4.69 (5)
C(7)	0.5515 (2)	0.4089 (1)	0.6557 (2)	4.31 (4)
C(8)	0.4678 (2)	0.34472 (9)	0.6787 (2)	3.40 (4)
C(9)	0.4827 (2)	0.23230 (9)	0.6077 (2)	3.32 (4)
C(10)	0.5524 (2)	0.16170 (9)	0.6102 (2)	3.57 (4)
C(11)	0.4055 (3)	0.1545 (1)	1.0764 (2)	4.82 (5)
C(12)	0.1853 (2)	0.1659 (1)	0.8234 (2)	4.33 (5)
C(13)	0.7311 (2)	0.1552 (1)	0.7113 (2)	4.76 (5)
C(14)	0.5190 (3)	0.1401 (1)	0.4552 (2)	5.20 (5)

Table 2. *Selected bond distances* (Å) *and angles* (°) *for* (1)

S(1)–S(2)	2.0229 (6)	C(3)–C(4)	1.521 (2)	
S(1)–C(1)	1.868 (2)	C(3)–C(8)	1.542 (2)	
S(2)–C(10)	1.869 (2)	C(4)–C(5)	1.514 (3)	
N(1)–C(2)	1.254 (2)	C(5)–C(6)	1.521 (3)	
N(1)–C(3)	1.459 (2)	C(6)–C(7)	1.517 (3)	
N(2)–C(8)	1.465 (2)	C(7)–C(8)	1.518 (2)	
N(2)–C(9)	1.254 (2)	C(9)–C(10)	1.499 (2)	
C(1)–C(11)	1.496 (2)	C(10)–C(13)	1.508 (2)	
C(1)–C(11)	1.530 (2)	C(10)–C(14)	1.537 (2)	
C(1)–C(12)	1.505 (2)			

S(2)–S(1)–C(1)	109.79 (5)	C(3)–C(4)–C(5)	111.6 (1)
S(1)–S(2)–C(10)	109.33 (5)	C(4)–C(5)–C(6)	110.7 (2)
C(2)–N(1)–C(3)	116.8 (1)	C(5)–C(6)–C(7)	110.7 (2)
C(8)–N(2)–C(9)	116.5 (1)	C(6)–C(7)–C(8)	112.0 (2)
S(1)–C(1)–C(2)	106.7 (2)	N(2)–C(8)–C(3)	107.1 (1)
S(1)–C(1)–C(11)	102.2 (1)	N(2)–C(8)–C(7)	111.2 (1)
S(1)–C(1)–C(12)	112.5 (1)	C(3)–C(8)–C(7)	110.9 (1)
C(2)–C(1)–C(11)	109.1 (1)	N(2)–C(9)–C(10)	124.5 (2)
C(2)–C(1)–C(12)	113.2 (1)	S(2)–C(10)–C(9)	105.7 (2)
C(11)–C(1)–C(12)	112.4 (1)	S(2)–C(10)–C(13)	112.3 (1)
N(1)–C(2)–C(1)	124.0 (2)	S(2)–C(10)–C(14)	102.7 (1)
N(1)–C(3)–C(4)	111.2 (2)	C(9)–C(10)–C(13)	113.9 (1)
N(1)–C(3)–C(8)	107.2 (1)	C(9)–C(10)–C(14)	109.2 (2)
C(4)–C(3)–C(8)	110.7 (1)	C(13)–C(10)–C(14)	112.2 (2)

C(1)–S(1)–S(2)–C(10)	90.8 (1)	C(8)–C(3)–C(4)–C(5)	−55.7 (2)
S(2)–S(1)–C(1)–C(2)	−77.0 (1)	N(1)–C(3)–C(8)–N(2)	−63.1 (2)
S(1)–S(2)–C(10)–C(9)	−80.1 (1)	N(1)–C(3)–C(8)–C(7)	175.4 (2)
C(3)–N(1)–C(2)–C(1)	−173.7 (2)	C(4)–C(3)–C(8)–N(2)	175.4 (2)
C(2)–N(1)–C(3)–C(4)	−136.6 (2)	C(4)–C(3)–C(8)–C(7)	53.9 (2)
C(2)–N(1)–C(3)–C(8)	102.3 (2)	C(3)–C(4)–C(5)–C(6)	57.3 (2)
C(9)–N(2)–C(8)–C(3)	105.7 (2)	C(4)–C(5)–C(6)–C(7)	−56.8 (2)
C(9)–N(2)–C(8)–C(7)	−133.0 (2)	C(5)–C(6)–C(7)–C(8)	56.2 (2)
C(8)–N(2)–C(9)–C(10)	−172.6 (2)	C(6)–C(7)–C(8)–N(2)	−173.8 (1)
S(1)–C(1)–C(2)–N(1)	124.5 (2)	C(6)–C(7)–C(8)–C(3)	−54.8 (2)
N(1)–C(3)–C(4)–C(5)	−174.7 (2)	N(2)–C(9)–C(10)–S(2)	123.3 (2)

cule has approximate point symmetry 2 and consists of a 1,2-*trans*-disubstituted cyclohexane (chair conformation) fused to a ten-membered ring that contains one disulfide and two, *trans*-substituted, imino groups. Structural parameters associated with the disulfide group, including the C—S—S—C torsion angle [90.8 (1)°], are similar to those reported for several acyclic disulfides. The N—C—C—N torsion angle [−63.1 (2)°] associated with disubstitution of the cyclohexane ring is substantially larger than those found for protonated or chelated 1,2-*trans*-diaminocyclohexane.

Experimental. The title compound (1) was prepared by a modification of the procedure of D'Amico & Dahl (1975). *trans*-1,2-Diaminocyclohexane was condensed with α,α'-dithiodiisobutyraldehyde which was prepared by the reaction of sulfur monochloride with isobutyraldehyde. Colorless, diffraction-quality plates were obtained by evaporation from acetonitrile/ethanol (3/1, v/v).

Structure solution: D_m by flotation; crystal 0.37 × 0.33 × 0.27 mm; Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Mo *K*α radiation; θ–2θ scan. Reciprocal-lattice symmetry and systematic absences consistent with space group $P2_1/n$. Cell constants from setting angles of 25 reflections with $16.00 \le \theta \le 20.21°$; data corrected for Lorentz, polarization and absorption (empirical) effects. Variation in intensity of 3 standard reflections ±1.4%; 2820 unique reflections measured with $2 \le 2\theta \le 50°$; 2066 with $I \ge 3\sigma(I)$ used in refinement. Data collected: h, k, ±l.

Structure solved by direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and difference Fourier techniques. Full-matrix least-squares refinement on F; non-H atoms

anisotropic; H atoms isotropic; H-atom temperature factors not refined; secondary-extinction parameter refined [final $g = 5.9$ (6) × 10^{-7}]; $w = 4F_o^2/$ $[\sigma^2(F_o^2)+0.0016F_o^4]$. Final $R_F = 0.031$, $wR_F = 0.044$, $S = 1.50$, $(\Delta/\sigma)_{max} = 0.35$, $\Delta\rho_{max} = 0.18$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs from Enraf–Nonius (1983) *SDP*.*

Atomic parameters are listed in Table 1, bond lengths and angles in Table 2. Fig. 1 shows the molecular structure and labeling scheme.

Related literature. The title compound was prepared as part of a project to study novel metal complexes containing tetradentate N_2S_2 ligands. Such complexes, particularly those with aliphatic thiolate ligation, may serve as useful models in helping to understand the active sites of several metal-containing proteins (Bharadwaj, Potenza & Schugar, 1986). Reduction of (1) with LiAlH₄ yields the saturated dithiol from which
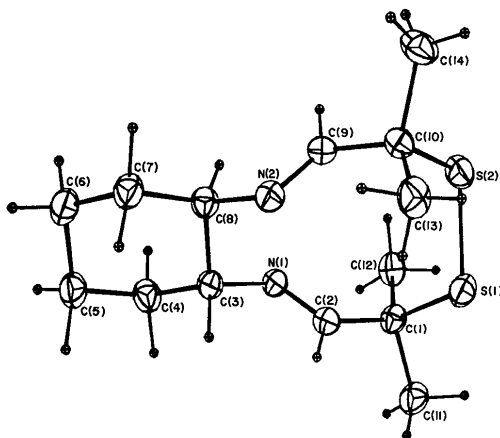
Fig. 1. View of (1) showing the atom-numbering scheme.

M^{II} complexes (M^{II} = 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, Subrahmanyan & 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. $C_{10}H_{18}O_2$, $M_r = 170·25$, monoclinic, $C2$, $a = 17·837$ (4), $b = 7·113$ (7), $c = 8·096$ (2) Å, $\beta = 102·17$ (6)°, $V = 1004$ (2) Å^3, $Z = 4$, $D_x = 1·13$ 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;