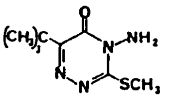
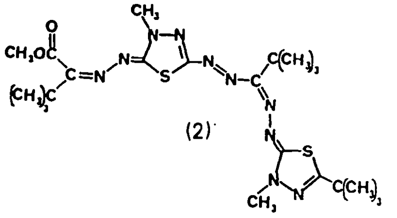
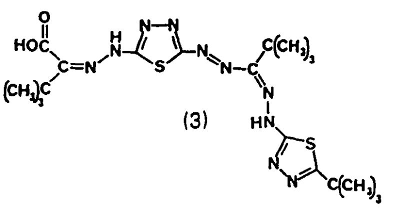
0·32694, 0·74936) as shown in Fig. 2. The maximum deviation from the pseudo-center is 0·213 Å for the pair of C(8) and C(8′), and the mean deviation is 0·060 Å. The dimensions of the rather rare thiadiazole rings found in this study are listed in Table 2 along with those found in the Cambridge Structural Database (Allen *et al.*, 1979). Structure (2), determined by this X-ray analysis, and the NMR and MS data suggest structure (3) for the dye.



(1)



(2)



(3)

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Structure of a New Clerodane Derivative from *Tinospora cordifolia* Miers

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Abstract. (5*R*,10*R*)-4*R*,8*R*-Dihydroxy-2*S*,3*R*:15,16-diepoxycleroda-13(16),14-dieno-17,12*S*:18,1*S*-dilactone, $C_{20}H_{22}O_8$, $M_r = 390·20$, m.p. = 504–506 K, monoclinic, $P2_1$, $a = 7·969$ (3), $b = 12·833$ (5), $c = 8·627$ (2) Å, $\beta = 101·60$ (2)°, $V = 864·11$ Å^3, $Z = 2$, $D_m = 1·490$ (by flotation in $CCl_4 + CHCl_3$), $D_x = 1·498$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 8·77$ cm^{-1}, $F(000) = 412$, $T = 295$ K, $R = 0·0367$, $wR = 0·0445$ for 1654 observed reflections. The structure contains two terpene rings, an epoxy group, two δ-lactones, two tertiary hydroxyl groups, two methyl groups, and a β-substituted furan moiety. The terpene ring *A* is locked into a boat conformation by the C(1)–C(4) lactone bridge. The furan ring is attached equatorially to atom C(12). The structure is stabilized by a network of hydrogen bonds involving the two hydroxyl groups.

Introduction. *Tinospora cordifolia* Miers belongs to the family Menispermaceae. It has been shown to possess several pharmacological activities (Nirmala, Sharadini

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Table 1. *Final atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3) *with e.s.d.'s in parentheses*

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	5074 (3)	2442 (2)	6736 (3)	36 (2)
C(2)	5201 (4)	1289 (2)	7173 (3)	44 (2)
C(3)	5517 (3)	1154 (2)	8864 (3)	42 (2)
C(4)	5531 (3)	2191 (2)	9695 (3)	34 (2)
C(5)	7053 (2)	2867 (2)	9343 (2)	29 (2)
C(6)	6920 (3)	3953 (2)	10069 (3)	36 (2)
C(7)	7761 (3)	4828 (2)	9326 (2)	35 (2)
C(8)	7025 (3)	4891 (2)	7538 (2)	30 (2)
C(9)	7422 (3)	3889 (2)	6701 (2)	29 (2)
C(10)	6851 (2)	2885 (2)	7476 (2)	28 (2)
C(11)	9385 (3)	3831 (2)	6898 (3)	34 (2)
C(12)	10260 (3)	4813 (2)	6448 (3)	38 (2)
C(13)	10600 (3)	4804 (2)	4800 (3)	36 (2)
C(14)	11402 (3)	3975 (3)	4109 (3)	45 (2)
C(15)	11488 (4)	4292 (3)	2656 (3)	54 (3)
C(16)	10254 (4)	5541 (2)	3697 (4)	51 (3)
C(17)	7807 (3)	5844 (2)	6919 (3)	38 (2)
C(18)	3897 (3)	2753 (2)	8933 (3)	38 (2)
C(19)	8719 (3)	2339 (2)	10147 (3)	39 (2)
C(20)	6627 (3)	3935 (2)	4944 (3)	38 (2)
O(1)	10798 (3)	5256†	2368 (2)	61 (2)
O(2)	9367 (3)	5783 (2)	6596 (3)	53 (2)
O(3)	7100 (3)	6675 (2)	6791 (3)	51 (2)
O(4)	5601 (2)	2071 (2)	11324 (2)	43 (2)
O(5)	2854 (2)	3069 (2)	9665 (3)	57 (2)
O(6)	3726 (2)	2917 (2)	7395 (2)	39 (1)
O(7)	3868 (3)	877 (2)	7874 (2)	55 (2)
O(8)	5226 (2)	5016 (1)	7251 (2)	42 (2)

† Fixed to define the origin.

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

C(2)—C(1)	1·524 (4)	C(17)—C(8)	1·517 (4)
C(10)—C(1)	1·540 (3)	O(8)—C(8)	1·413 (3)
O(6)—C(1)	1·447 (3)	C(10)—C(9)	1·561 (3)
C(3)—C(2)	1·440 (4)	C(11)—C(9)	1·541 (3)
O(7)—C(2)	1·426 (4)	C(20)—C(9)	1·522 (3)
C(4)—C(3)	1·511 (4)	C(12)—C(11)	1·528 (4)
O(7)—C(3)	1·460 (3)	C(13)—C(12)	1·500 (3)
C(5)—C(4)	1·569 (3)	O(2)—C(12)	1·453 (3)
C(18)—C(4)	1·518 (3)	C(14)—C(13)	1·430 (4)
O(4)—C(4)	1·404 (3)	C(16)—C(13)	1·331 (4)
C(6)—C(5)	1·540 (3)	C(15)—C(14)	1·332 (4)
C(10)—C(5)	1·586 (3)	O(1)—C(15)	1·357 (4)
C(19)—C(5)	1·527 (3)	O(1)—C(16)	1·355 (4)
C(7)—C(6)	1·515 (4)	O(2)—C(17)	1·329 (4)
C(8)—C(7)	1·538 (3)	O(3)—C(17)	1·202 (3)
C(9)—C(8)	1·538 (3)	O(5)—C(18)	1·211 (3)
		O(6)—C(18)	1·323 (3)

C(10)—C(1)—C(2)	104·3 (2)	C(10)—C(9)—C(8)	112·6 (2)
O(6)—C(1)—C(2)	109·2 (2)	C(11)—C(9)—C(8)	106·8 (2)
O(6)—C(1)—C(10)	112·0 (2)	C(11)—C(9)—C(10)	106·8 (2)
C(3)—C(2)—C(1)	110·9 (2)	C(20)—C(9)—C(8)	110·6 (2)
O(7)—C(2)—C(1)	116·6 (2)	C(20)—C(9)—C(10)	111·1 (2)
O(7)—C(2)—C(3)	61·3 (2)	C(20)—C(9)—C(11)	108·7 (2)
C(4)—C(3)—C(2)	110·9 (2)	C(5)—C(10)—C(1)	108·2 (2)
O(7)—C(3)—C(2)	58·9 (2)	C(9)—C(10)—C(1)	116·8 (2)
O(7)—C(3)—C(4)	114·4 (2)	C(9)—C(10)—C(5)	117·9 (2)
C(5)—C(4)—C(3)	109·1 (2)	C(12)—C(11)—C(9)	115·9 (2)
C(18)—C(4)—C(3)	106·8 (2)	C(13)—C(12)—C(11)	114·4 (2)
C(18)—C(4)—C(5)	106·4 (2)	O(2)—C(12)—C(11)	115·3 (2)
O(4)—C(4)—C(3)	111·9 (2)	O(2)—C(12)—C(13)	106·0 (2)
O(4)—C(4)—C(5)	112·2 (2)	C(14)—C(13)—C(12)	125·6 (2)
O(4)—C(4)—C(18)	110·1 (2)	C(16)—C(13)—C(12)	128·4 (3)
C(6)—C(5)—C(4)	107·6 (2)	C(16)—C(13)—C(14)	106·1 (2)
C(10)—C(5)—C(4)	106·2 (2)	C(15)—C(14)—C(13)	106·1 (3)
C(10)—C(5)—C(6)	113·5 (2)	O(1)—C(15)—C(14)	110·8 (3)
C(19)—C(5)—C(4)	107·6 (2)	O(1)—C(16)—C(13)	110·9 (3)
C(19)—C(5)—C(6)	109·9 (2)	O(2)—C(17)—C(8)	119·7 (2)
C(19)—C(5)—C(10)	111·7 (2)	O(3)—C(17)—C(8)	122·0 (2)
C(7)—C(6)—C(5)	115·2 (2)	O(3)—C(17)—O(2)	118·3 (3)
C(8)—C(7)—C(6)	110·6 (2)	O(5)—C(18)—C(4)	123·5 (2)
C(9)—C(8)—C(7)	110·7 (2)	O(6)—C(18)—C(4)	114·6 (2)
C(17)—C(8)—C(7)	107·1 (2)	O(6)—C(18)—O(5)	121·8 (2)
C(17)—C(8)—C(9)	111·7 (2)	C(16)—O(1)—C(15)	106·1 (2)
O(8)—C(8)—C(7)	110·5 (2)	C(17)—O(2)—C(12)	124·3 (2)
O(8)—C(8)—C(9)	108·0 (2)	C(18)—O(6)—C(1)	113·1 (2)
O(8)—C(8)—C(17)	108·7 (2)	C(3)—O(7)—C(2)	59·9 (2)

C(10)—C(1)—C(2)—C(3)	62·2 (3)	C(6)—C(7)—C(8)—O(8)	56·5 (2)
O(6)—C(1)—C(2)—C(3)	−57·6 (3)	C(17)—C(8)—C(9)—C(11)	55·1 (2)
C(2)—C(1)—C(10)—C(5)	−69·0 (2)	C(7)—C(8)—C(9)—C(20)	177·6 (2)
O(7)—C(2)—C(3)—C(4)	−106·5 (2)	C(9)—C(8)—C(17)—O(2)	−39·9 (3)
C(3)—C(4)—C(5)—C(19)	−67·0 (2)	C(8)—C(9)—C(11)—C(12)	−50·4 (3)
C(5)—C(6)—C(7)—C(8)	56·6 (3)	C(17)—O(2)—C(12)—C(13)	120·1 (3)
C(6)—C(7)—C(8)—C(17)	174·7 (2)	O(2)—C(12)—C(13)—C(14)	−177·6 (3)

& Karandikar, 1984; Quadrat-i-Khuda, Khaleque & Roy, 1964) and several compounds (Ahmed, Khaleque & Wahed, 1978; Hanuman, Bhatt & Sabata, 1986; Hanuman, Mishra & Sabata, 1986; Hanuman, Bhatt & Sabata, 1988; Khaleque, Wahed, Sayeedul Huq & Abdul Bashar, 1970; Quadrat-i-Khuda, Khaleque, Abdul Bashar, Rouf Khan & Roy, 1966) have been isolated from this plant. The structural investigation of the title compound was undertaken as a consequence of the medicinal properties, to determine the stereochemistry.

Experimental. A fine powder made from the dried stems of the above plant was used for extraction with chloroform in a Soxhlet apparatus. The compound was isolated by repeated column chromatography over silica gel with 70% ethyl acetate–petroleum ether (60–80°C) and repeated crystallization from methanol yielded transparent crystals.

A crystal of dimensions 0·45 × 0·40 × 0·22 mm was selected for data collection with an Enraf–Nonius CAD-4 diffractometer using graphite-monochromated Cu $K\alpha$ radiation. Unit-cell parameters obtained by oscillation and Weissenberg photographs were refined by least-squares analysis using 25 reflections, $37 < \theta < 45°$, on the diffractometer. Intensities of two standard reflections recorded for every 5000 s of data collection showed no significant changes. Data were collected in the range $0 < \theta < 70°$ (resolution $d = 0{\cdot}82$ Å, $h_{\max} = 9$, $k_{\max} = 15$, $l_{\min} = -10$, $l_{\max} = 10$). 1860 reflections were collected. Symmetry-equivalent reflections were averaged. Of 1722 unique reflections 32 reflections had $|F_o| < 3\sigma(|F_o|)$. Data were corrected for Lp effects and no absorption correction was made. The structure was solved by *SHELX*86 (Sheldrick, 1986) with TREF = 50. The phase set with $R_E = 0{\cdot}185$ for 477 E values revealed all the 28 non-H atoms. H atoms were located by difference maps. Structure refinement by least squares based on F values, using anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms. Number of parameters refined: 341, including H atoms. In the final calculation, reflections with $|F_o| < 5\sigma(|F_o|)$ and those for which $F_o < F_c$ with $\Delta F/\sigma(|F_o|) > 4{\cdot}0$ were omitted. $(\Delta/\sigma)_{\max} = 0{\cdot}096$ and $\Delta\rho$ in the final difference map $-0{\cdot}1673$ to $0{\cdot}2118$ e Å^{-3}. For 1654 reflections $R = 0{\cdot}0367$ and $wR = 0{\cdot}0445$ (Stout & Jensen, 1968) where $w = k/[\sigma^2(F_o) + g(F_o)^2]$ with $k = 1{\cdot}2639$ and $g = 0{\cdot}002$. *SHELX*76 (Sheldrick, 1976) was used for refinement and a CYBER-180 computer was used for all the calculations. Atomic scattering factors those of *SHELX*76.

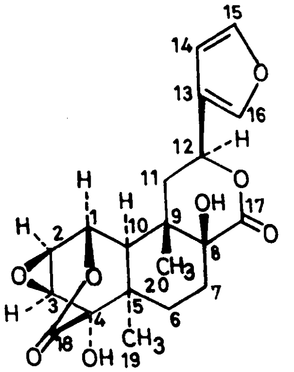

Fig. 1. Chemical structural formula.

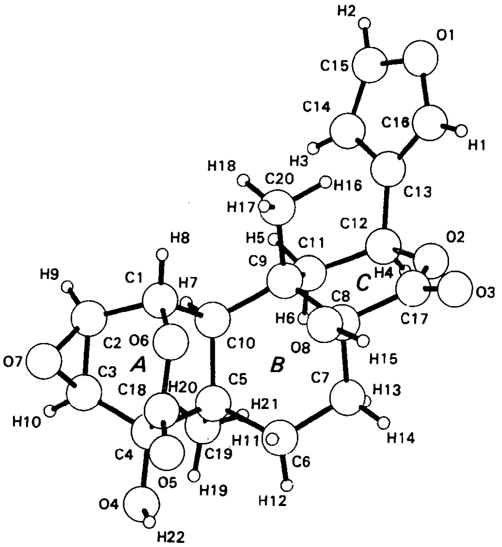

Fig. 2. General view of the molecule.

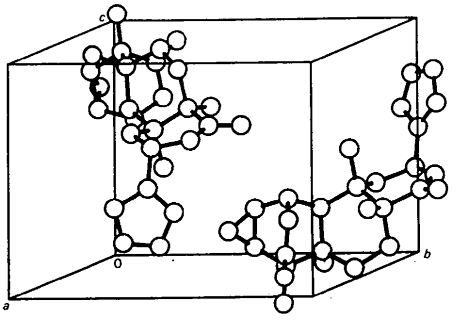

Fig. 3. Unit-cell packing diagram.

Discussion. Final positional parameters with equivalent isotropic temperature factors are given in Table 1.* Important bond lengths, angles and torsion angles can be found in Table 2. The chemical structural formula for the compound is given in Fig. 1. Though the structure of this compound was assigned on the basis of spectroscopic analysis (Bhatt, Hanuman & Sabata, 1988), the stereochemistry about atoms C(8) and C(12) could not be determined. The epoxy oxygen O(7) lies on the same side of the plane of ring *A* as does the lactone bridge (Fig. 2, *PLUTO*, Motherwell & Clegg, 1978). The angles C(10)—C(1)—C(2) = 104·3 (2) and C(5)—C(4)—C(3) = 109·1 (2)° and the torsion angles C(10)—C(1)—C(2)—C(3) = 62·2 (3) and C(2)—C(1)—C(10)—C(5) = −69·0 (2)° indicate that the terpene ring *A* is locked into a boat conformation by the C(1)–C(4) lactone bridge. Atom O(4) is coplanar with the lactone function [deviation from the mean plane of atoms C(1), O(6), C(18), C(4) is 0·0149 (2) Å]. The furan ring is effectively planar and connected equatorially to ring *C* at atom C(12). Atom H(4) is α-oriented [lies below the ring with respect to atom C(12)]. It is interesting to note that atom H(7) is also α-oriented at atom C(10), this indicates a *trans*-diaxial relationship for the lactone at C(1) and H(7) at C(10). The molecular packing is shown in Fig. 3.

Intermolecular hydrogen bonds involve the two hydroxyl groups. Atom O(8) of the hydroxyl at C(8) bonds to O(4) with distance 3·04 (2) Å and O(4) of the hydroxyl at C(4) to O(3) with distance 2·99 (2) Å.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, hydrogen bonds, dihedral angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44921 (13 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 3′-*O*-Acetylthymidine

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Abstract. $C_{12}H_{16}N_2O_6$, $M_r = 284{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}485\ (4)$, $b = 14{\cdot}043\ (7)$, $c = 17{\cdot}145\ (12)$ Å, $U = 1320{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}43$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}08$ cm^{-1}, $F(000) = 600$, $T = 293$ K, $R = 0{\cdot}049$ for 1769 observed reflections. The molecule adopts an *anti* conformation, with a sugar pucker of $^2T_3$ [C(2′)-*endo*/C(3′)-*exo*].

Introduction. The structure determination was performed as part of a series of studies of acetylated nucleosides intended to investigate base-pairing patterns, propeller twisting and acetyl conformations in such compounds. Earlier work has revealed possible biologically interesting acetyl conformations (Wilson & Tollin, 1985) and induced base-pairing in such substituted nucleosides (Wilson, 1985). Since both features of acetyl–base stacking and base-pairing are present in the structure of the closely related compound 3′,5′-di-*O*-acetylthymidine (Wilson, Low, Tollin & Wilson, 1984), it was hoped that the title compound would also show these features.

Experimental. The crystals used were grown from aqueous solution and that used in the structural determination had dimensions 1·2 × 0·34 × 0·19 mm. Cell dimensions and orientation were obtained from the 254, 082, 0,0,10 and symmetry-related reflections. Data were collected on a Nicolet *P*3 four-circle diffractometer in Aberdeen. 2168 reflections were measured using Mo $K\alpha$ radiation. Data collection parameters: $0 < 2\theta < 60°$, $\theta/2\theta$ scans with a 2θ scan range of 2·41–2·8° depending on 2θ value, and a variable scan rate in the range 5·33° min^{-1} ($I < 150$) to 58·6° min^{-1} ($I > 2500$). Range of indices: $h \le 7$, $k \le 19$, $l \le 24$. The reflections 284 and 082 were measured every 50 reflections and showed no significant change in their intensities. No absorption or extinction corrections were applied.

Structure solution and refinement. The Patterson-methods program *PATMET* (Wilson & Tollin, 1986) was used to obtain an oriented model of the planar base group in the structure which was used as *ab initio* structural knowledge (Main, 1976) in the direct-methods package *MITHRIL* (Gilmore, 1984). A direct-methods run led to the appearance of most non-hydrogen atoms in the first calculated *E* map. Fourier recycling completed the definition of the structure, and a series of difference Fourier maps revealed all the H-atom positions. All non-hydrogen atoms were refined anisotropically and the H atoms isotropically. In the final stages of refinement all H parameters were fixed. Refinement was by least squares on *F* using the *BLOCK* program in the *GX* system (Mallinson & Muir, 1985). Final refinement parameters: $R = 0{\cdot}049$ for 1769 observed reflections [$I > 3\sigma(I)$]; $wR = 0{\cdot}053$, $w = 1/[\sigma(F)]^2$; 181 parameters refined; max. shift/e.s.d. = 0·022; max., min. difference peaks = 0·22 and −0·27 e Å^{-3}. Scattering factors taken from *International Tables for X-ray Crystallography* (1974). The *PLUTO* program (Motherwell & Clegg, 1978) was used for structure drawing. All other calculations were carried out using the *GX* package. All computations were performed on

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