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

Cl(1*a*)–C(19*a*)	1·748 (7)	Cl(1*b*)–C(19*b*)	1·756 (5)
O(1*a*)–C(1*a*)	1·254 (5)	O(1*b*)–C(1*b*)	1·242 (9)
C(1*a*)–C(2*a*)	1·486 (6)	C(1*b*)–C(2*b*)	1·494 (7)
C(1*a*)–C(12*a*)	1·458 (7)	C(1*b*)–C(12*b*)	1·435 (7)
C(2*a*)–C(3*a*)	1·517 (7)	C((2*b*)–C(3*b*)	1·528 (12)
C(3*a*)–C(4*a*)	1·529 (6)	C(3*b*)–C(4*b*)	1·522 (7)
C(3*a*)–C(16*a*)	1·513 (7)	C(3*b*)–C(16*b*)	1·517 (8)
C(3*a*)–C(17*a*)	1·529 (8)	C(3*b*)–C(17*b*)	1·526 (6)
C(4*a*)–C(13*a*)	1·497 (6)	C(4*b*)–C(13*b*)	1·497 (6)
N(5*a*)–C(13*a*)	1·374 (6)	N(5*b*)–C(13*b*)	1·367 (6)
N(5*a*)–C(14*a*)	1·403 (6)	N(5*b*)–C(14*b*)	1·411 (5)
C(6*a*)–C(7*a*)	1·361 (7)	C(6*b*)–C(7*b*)	1·392 (6)
C(6*a*)–C(14*a*)	1·392 (8)	C(6*b*)–C(14*b*)	1·382 (8)
C(7*a*)–C(8*a*)	1·373 (7)	C(7*b*)–C(8*b*)	1·358 (10)
C(8*a*)–C(9*a*)	1·388 (9)	C(8*b*)–C(9*b*)	1·380 (9)
C(9*a*)–C(15*a*)	1·399 (7)	C(9*b*)–C(15*b*)	1·370 (5)
N(10*a*)–C(11*a*)	1·478 (5)	N(10*b*)–C(11*b*)	1·484 (4)
N(10*a*)–C(15*a*)	1·395 (8)	N(10*b*)–C(15*b*)	1·420 (7)
C(11*a*)–C(12*a*)	1·486 (6)	C(11*b*)–C(12*b*)	1·506 (6)
C(11*a*)–C(18*a*)	1·531 (7)	C(11*b*)–C(18*b*)	1·538 (5)
C(12*a*)–C(13*a*)	1·369 (5)	C(12*b*)–C(13*b*)	1·376 (8)
C(14*a*)–C(15*a*)	1·390 (6)	C(14*b*)–C(15*b*)	1·398 (7)
C(18*a*)–C(19*a*)	1·389 (7)	C(18*b*)–C(19*b*)	1·366 (7)
C(18*a*)–C(23*a*)	1·376 (8)	C(18*b*)–C(23*b*)	1·406 (7)
C(19*a*)–C(20*a*)	1·357 (10)	C(19*b*)–C(20*b*)	1·361 (6)
C(20*a*)–C(21*a*)	1·372 (11)	C(20*b*)–C(21*b*)	1·383 (9)
C(21*a*)–C(22*a*)	1·357 (9)	C(21*b*)–C(22*b*)	1·365 (9)
C(22*a*)–C(23*a*)	1·386 (8)	C(22*b*)–C(23*b*)	1·383 (6)
O(1)–C(1)	1·340 (10)	C(1)–C(2′)	1·204 (33)
C(1)–C(2″)	1·246 (30)		

3·236 (4), 2·825 (8) Å and the H···*A* distances 2·03 (6), 2·39 (5), 2·79 (5), 1·95 (6) Å, respectively. The corresponding *D*—H···*A* angles are 161 (3), 161 (3), 145 (3), 149 (3)°.

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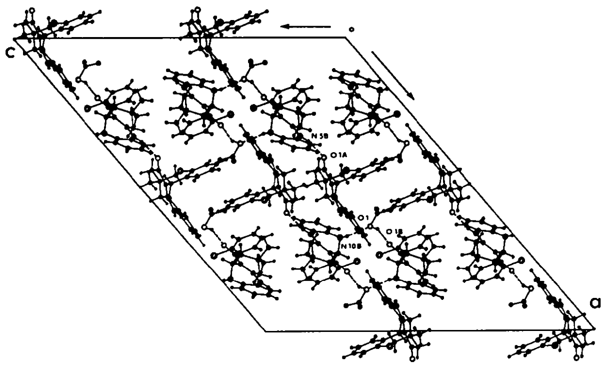

Fig. 2. The packing arrangement of the title compound as viewed along **b**. The dashed lines indicate hydrogen bonds.

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Structure of (3β,25*R*)-Spirost-5-ene-3,17-diol Hemihydrate (Pennogenin)*

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Abstract. $C_{27}H_{42}O_4.\frac{1}{2}H_2O$, $M_r = 439{\cdot}6$, monoclinic, $P2_1$, $a = 18{\cdot}880$ (8), $b = 7{\cdot}446$ (2), $c = 19{\cdot}911$ (7) Å, $\beta = 117{\cdot}78$ (4)°, $V = 2476$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}18$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}073$ mm^{-1}, $F(000) = 964$, $T = 293$ K. Final $R = 0{\cdot}047$ for 2575 observed reflections. The molecular structure is determined from the X-ray data and confirms the structure previously assigned on the basis of chemical and spectroscopic evidence. In the two independent molecules rings *A*, *C* and *F* have chair conformations and ring *B* adopts a half-chair conformation. The methyl substituents at the *A*/*B* and *C*/*D* ring junctions are *axial* and the ring *A* hydroxyl group and the ring *F*

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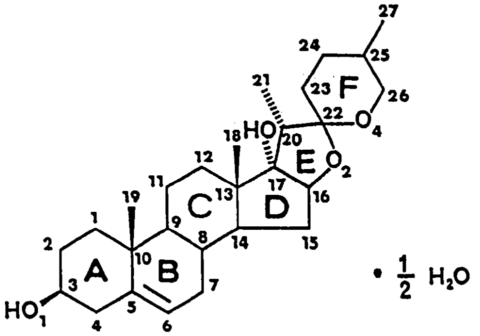
methyl group are *equatorial.* The structure consists of sheets of molecules along the [011] direction, linked by hydrogen bonds between hydroxyl groups of neighbouring molecules and *via* a molecule of water.

Introduction. The steroidal constituents of *Dioscorea mexicana,* a member of the Compositae family widely distributed in Mexico, were investigated and the steroidal sapogenin, pennogenin, was isolated by chromatography. Chemical and spectroscopic studies led to the proposal of structure (1) for pennogenin (Marker, Wagner, Goldsmith, Ulshafer & Ruof, 1943; Marker, Wagner, Ulshafer, Wittbecker, Goldsmith & Ruof, 1947). It was of interest to determine the crystal structure of (1) in order to ascertain its conformation and molecular geometry.



(1)

Experimental. Colourless crystal $0.10 \times 0.36 \times 0.40$ mm. Nicolet $R3$ four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters from 25 machine-centred reflections with $4.9 < 2\theta < 18.1°$. 4192 unique reflections with $3 < 2\theta < 55°$, 2575 with $I > 3.0\sigma(I)$, index range $h \pm 21$, $k\,0 \rightarrow 9$, $l\,0 \rightarrow 25$, ω-scan mode, variable scan speed, scan width $1.0°$ (θ), two standard reflections ($2\bar{1}0$, $2\bar{1}1$) monitored every 50 measurements, no intensity variation, Lp correction, absorption ignored; structure solved by combination of direct methods and partial structure expansion by an iterative E–Fourier procedure using *SHELXTL* (Sheldrick, 1981); least-squares anisotropic refinement of all non-H atoms; H atoms riding on bonded C, coordinates of H atoms bonded to O refined with fixed isotropic temperature factor $U = 0.06$ Å^2, function minimized $\sum w(\Delta F)^2$, $w = [\sigma^2(F_o) + 0.0005(F_o)^2]^{-1}$, where σ is standard deviation of observed amplitudes, based on counting statistics; isotropic extinction parameter $X = 0.00198$. In the last cycle $(\Delta/\sigma)_{\max} = 0.39$; $\Delta\rho$ from -0.16 to 0.17 e Å^{-3}; final $R = 0.047$, $wR = 0.046$; scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates are in Table 1.* The atomic numbering scheme for (1) is shown in Fig. 1. The bond lengths for non-H atoms are listed in Table 2. The average e.s.d. in the bond lengths is 0.007 Å and in angles 0.4°.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2 $\times 10^3$)

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	U_{eq}
O(1*A*)	10259 (2)	8867	5910 (2)	54 (2)
O(2*A*)	8092 (2)	8980 (4)	10397 (2)	45 (1)
O(3*A*)	6955 (2)	6937 (5)	8656 (2)	54 (2)
O(4*A*)	6939 (2)	7807 (4)	10389 (2)	45 (1)
C(1*A*)	9399 (3)	6443 (7)	7012 (3)	60 (3)
C(2*A*)	9899 (3)	6715 (7)	6594 (3)	53 (3)
C(3*A*)	9812 (3)	8641 (7)	6327 (3)	45 (2)
C(4*A*)	10090 (3)	9908 (7)	6993 (3)	43 (2)
C(5*A*)	9657 (3)	9610 (6)	7458 (3)	35 (2)
C(6*A*)	9342 (3)	10943 (7)	7649 (3)	44 (3)
C(7*A*)	8919 (3)	10832 (6)	8136 (3)	42 (2)
C(8*A*)	9029 (3)	9018 (6)	8515 (2)	31 (2)
C(9*A*)	8980 (3)	7496 (6)	7973 (3)	36 (2)
C(10*A*)	9630 (3)	7684 (6)	7703 (2)	35 (2)
C(11*A*)	8947 (4)	5652 (7)	8281 (3)	56 (3)
C(12*A*)	8313 (4)	5450 (7)	8549 (3)	55 (3)
C(13*A*)	8423 (3)	6881 (6)	9137 (3)	34 (2)
C(14*A*)	8383 (3)	8714 (6)	8776 (2)	32 (2)
C(15*A*)	8332 (3)	10029 (7)	9347 (3)	48 (3)
C(16*A*)	7769 (3)	9060 (7)	9585 (2)	40 (2)
C(17*A*)	7709 (3)	7060 (6)	9331 (2)	37 (2)
C(18*A*)	9212 (3)	6613 (8)	9869 (3)	59 (3)
C(19*A*)	10459 (3)	7202 (9)	8344 (3)	69 (3)
C(20*A*)	7735 (3)	5986 (7)	10008 (3)	44 (2)
C(21*A*)	7092 (4)	4549 (7)	9809 (3)	66 (3)
C(22*A*)	7743 (3)	7425 (7)	10557 (2)	39 (2)
C(23*A*)	8240 (3)	6980 (8)	11389 (2)	54 (3)
C(24*A*)	8166 (3)	8429 (8)	11904 (3)	56 (3)
C(25*A*)	7305 (3)	8834 (8)	11673 (3)	52 (3)
C(26*A*)	6872 (3)	9220 (8)	10840 (3)	52 (3)
C(27*A*)	7202 (4)	10370 (11)	12122 (3)	90 (4)
O	10521 (2)	12454 (6)	5649 (2)	81 (2)
O(1*B*)	8668 (2)	990 (5)	3974 (2)	64 (2)
O(2*B*)	5038 (2)	−1 (5)	7272 (2)	45 (2)
O(3*B*)	6505 (2)	3120 (5)	7867 (2)	54 (2)
O(4*B*)	5389 (2)	1750 (5)	8357 (2)	51 (2)
C(1*B*)	7908 (3)	3389 (7)	5191 (3)	55 (3)
C(2*B*)	8179 (3)	3218 (8)	4584 (3)	58 (3)
C(3*B*)	8407 (3)	1297 (7)	4534 (3)	47 (2)
C(4*B*)	7703 (3)	67 (8)	4355 (3)	59 (3)
C(5*B*)	7378 (3)	281 (7)	4919 (3)	43 (2)
C(6*B*)	7253 (3)	−1122 (8)	5254 (3)	61 (3)
C(7*B*)	6905 (3)	−1079 (7)	5791 (3)	51 (3)
C(8*B*)	6525 (3)	718 (6)	5788 (2)	35 (2)
C(9*B*)	7068 (3)	2246 (6)	5774 (3)	37 (2)
C(10*B*)	7187 (3)	2180 (7)	5059 (2)	38 (2)
C(11*B*)	6827 (4)	4111 (7)	5945 (3)	68 (3)
C(12*B*)	6614 (4)	4157 (7)	6603 (4)	67 (3)
C(13*B*)	5991 (3)	2720 (6)	6498 (3)	40 (2)
C(14*B*)	6356 (3)	924 (6)	6462 (3)	37 (2)
C(15*B*)	5838 (3)	−461 (6)	6586 (3)	45 (3)
C(16*B*)	5778 (3)	363 (7)	7260 (3)	43 (2)
C(17*B*)	5835 (3)	2449 (7)	7202 (2)	35 (2)
C(18*B*)	5211 (3)	3091 (9)	5788 (3)	72 (3)
C(19*B*)	6442 (3)	2879 (9)	4362 (3)	70 (3)
C(20*B*)	5038 (3)	3095 (7)	7170 (3)	44 (2)
C(21*B*)	5042 (4)	4977 (8)	7476 (4)	68 (3)
C(22*B*)	4849 (3)	1562 (7)	7570 (2)	38 (2)
C(23*B*)	3999 (3)	1363 (8)	7433 (3)	56 (3)
C(24*B*)	3911 (3)	−99 (8)	7919 (3)	59 (3)
C(25*B*)	4506 (3)	151 (9)	8746 (3)	63 (3)
C(26*B*)	5347 (3)	315 (9)	8814 (3)	68 (3)
C(27*B*)	4487 (4)	−1355 (12)	9250 (4)	118 (5)

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* Lists of structure factors, anisotropic thermal parameters, bond angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43739 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The title compound crystallizes with two independent molecules per asymmetric unit and one water molecule. The two independent molecules (1*A*) and (1*B*) are closely similar as regards their bond lengths and angles. However, some of the individual distances are somewhat different from their expected values. The C(2)—C(3) and C(3)—C(4) bond distances are significantly shorter than the expected value of 1·533 Å for a C—C bond length in *n*-hydrocarbons (Bartell, 1959) but are in agreement with the values determined in hecogenin (Soriano-García, Toscano, López, Hernández & Enríquez, 1984).

The stereochemistry of both (1*A*) and (1*B*) is as follows: C(8)—βH is *trans* to both C(9)—αH and C(14)—αH; C(9)—αH is *trans* to C(10)—βCH_3; C(17)—αOH is *cis* to C(16)—αH and C(20)—αCH_3 and *trans* to C(13)—βCH_3.

In both molecules, the *A*/*B*, *B*/*C* and *C*/*D* ring junctions are *trans*; the *D*/*E* ring junction is *cis*. Rings *A*, *C* and *F* all exist in chair conformations with average values for the endocyclic torsion angles 53·4 (5), 54·4 (5) and 54·3 (6)° in molecule (1*A*) and 53·6 (5), 52·7 (5) and 54·7 (7) in molecule (1*B*), in agreement with those in hecogenin (Soriano-García *et al.*, 1984) and in cyclohexane (Kahn, Fourme, André & Renaud, 1973). The substituents at C(3) and C(25) are equatorial. The conformation of the *B* ring is half-chair in both molecules.

In (2*A*) the five-membered *D* and *E* rings have Δ and φ_m values (Altona, Geise & Romers, 1968) of −3·0 (4), 48·5 (4)° and 23·8 (5), 41·1 (5)°, indicating half-chair and β-envelope conformations, respectively, whereas, in (1*B*), the Δ and φ_m values are 26·8 (4), −47·2 (4)° and −35·2 (4), 45·1 (4)°, indicating β-envelope and α-envelope conformations.

The molecular arrangement, consisting of layers of molecules along the [011] direction, is shown in Fig. 2. The molecules (1*A*) and (1*B*) are linked by an extensive three-dimensional hydrogen-bond network which stabilizes the crystal structure. Each molecule is linked to a neighbour *via* O(1*B*)—H(1*B*)···O(1*A*) (2−*x*, −0·5 + *y*, 1−*z*). The water molecule is involved in three hydrogen

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

O(1*A*)—C(3*A*)	1·443 (8)	O(1*B*)—C(3*B*)	1·432 (8)
O(2*A*)—C(16*A*)	1·437 (5)	O(2*B*)—C(16*B*)	1·433 (7)
O(2*A*)—C(22*A*)	1·440 (6)	O(2*B*)—C(22*B*)	1·426 (6)
O(3*A*)—C(17*A*)	1·434 (4)	O(3*B*)—C(17*B*)	1·429 (5)
O(4*A*)—C(22*A*)	1·422 (6)	O(4*B*)—C(22*B*)	1·422 (5)
O(4*A*)—C(26*A*)	1·428 (7)	O(4*B*)—C(26*B*)	1·429 (8)
C(1*A*)—C(2*A*)	1·536 (10)	C(1*B*)—C(2*B*)	1·521 (10)
C(1*A*)—C(10*A*)	1·541 (7)	C(1*B*)—C(10*B*)	1·549 (8)
C(2*A*)—C(3*A*)	1·511 (7)	C(2*B*)—C(3*B*)	1·511 (8)
C(3*A*)—C(4*A*)	1·508 (7)	C(3*B*)—C(4*B*)	1·513 (8)
C(4*A*)—C(5*A*)	1·511 (9)	C(4*B*)—C(5*B*)	1·518 (10)
C(5*A*)—C(6*A*)	1·301 (8)	C(5*B*)—C(6*B*)	1·319 (9)
C(5*A*)—C(10*A*)	1·523 (7)	C(5*B*)—C(10*B*)	1·518 (7)
C(6*A*)—C(7*A*)	1·520 (9)	C(6*B*)—C(7*B*)	1·494 (10)
C(7*A*)—C(8*A*)	1·514 (7)	C(7*B*)—C(8*B*)	1·517 (7)
C(8*A*)—C(9*A*)	1·539 (7)	C(8*B*)—C(9*B*)	1·540 (7)
C(8*A*)—C(14*A*)	1·549 (8)	C(8*B*)—C(14*B*)	1·524 (8)
C(9*A*)—C(10*A*)	1·558 (8)	C(9*B*)—C(10*B*)	1·541 (8)
C(9*A*)—C(11*A*)	1·517 (7)	C(9*B*)—C(11*B*)	1·548 (8)
C(10*A*)—C(19*A*)	1·531 (6)	C(10*B*)—C(19*B*)	1·535 (6)
C(11*A*)—C(12*A*)	1·527 (11)	C(11*B*)—C(12*B*)	1·538 (11)
C(12*A*)—C(13*A*)	1·524 (7)	C(12*B*)—C(13*B*)	1·532 (8)
C(13*A*)—C(14*A*)	1·527 (6)	C(13*B*)—C(14*B*)	1·522 (7)
C(13*A*)—C(17*A*)	1·572 (8)	C(13*B*)—C(17*B*)	1·572 (8)
C(13*A*)—C(18*A*)	1·536 (5)	C(13*B*)—C(18*B*)	1·517 (6)
C(14*A*)—C(15*A*)	1·537 (8)	C(14*B*)—C(15*B*)	1·518 (8)
C(15*A*)—C(16*A*)	1·531 (9)	C(15*B*)—C(16*B*)	1·527 (9)
C(16*A*)—C(17*A*)	1·560 (7)	C(16*B*)—C(17*B*)	1·566 (7)
C(17*A*)—C(20*A*)	1·549 (8)	C(17*B*)—C(20*B*)	1·553 (8)
C(20*A*)—C(21*A*)	1·525 (8)	C(20*B*)—C(21*B*)	1·527 (8)
C(20*A*)—C(22*A*)	1·525 (7)	C(20*B*)—C(22*B*)	1·526 (8)
C(22*A*)—C(23*A*)	1·512 (6)	C(22*B*)—C(23*B*)	1·504 (8)
C(23*A*)—C(24*A*)	1·540 (8)	C(23*B*)—C(24*B*)	1·516 (9)
C(24*A*)—C(25*A*)	1·499 (8)	C(24*B*)—C(25*B*)	1·512 (7)
C(25*A*)—C(26*A*)	1·496 (6)	C(25*B*)—C(26*B*)	1·535 (9)
C(25*A*)—C(27*A*)	1·520 (10)	C(25*B*)—C(27*B*)	1·516 (11)

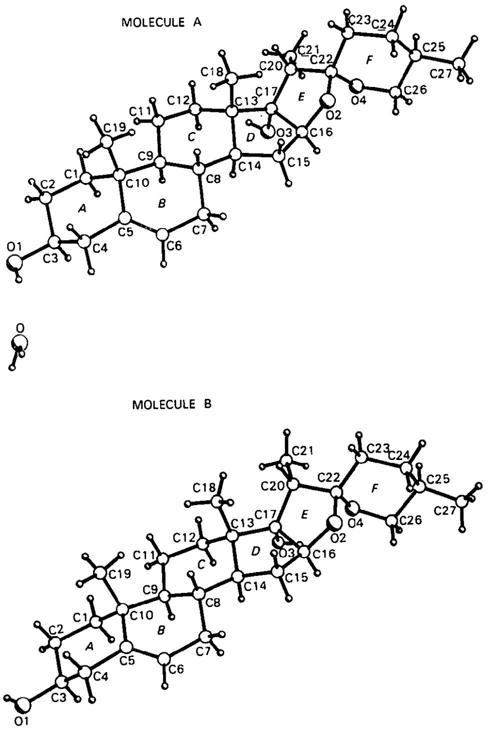

Fig. 1. The molecular structures of (1*A*) and (1*B*) showing the atom labelling.

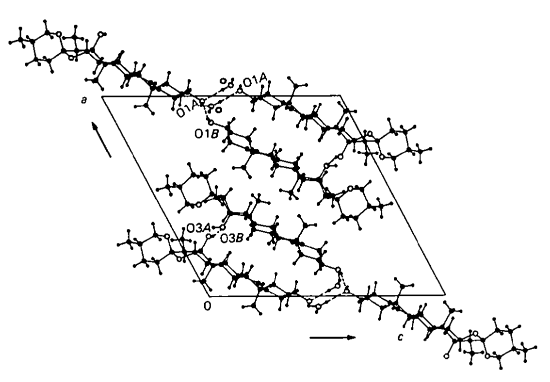

Fig. 2. The packing arrangement of the molecules as viewed along **b**. The dashed lines indicate hydrogen bonds.

bonds [O(1*A*)—H(1*A*)···O (*x*, *y*, *z*); O—H(1)···O(1*A*) (2−*x*, 0·5 + *y*, 1−*z*) and O—H(2)···O(1*B*) (2−*x*, 1·5 + *y*, 1−*z*)]. Finally, a hydrogen-bond interaction between the hydroxyl groups at C(17) of neighbouring molecules constitutes the major intermolecular attraction between 'ends' of the molecules [O(3*A*)—H(3*A*)···O(3*B*) (*x*, *y*, *z*)]. The *D*···*A* lengths are, respectively 2·882 (5), 2·809 (5), 2·941 (5), 2·961 (6) and 3·166 (5) Å, H···*A* 2·09 (5), 2·02 (4), 2·08 (4), 2·23 (5) and 2·42 (5) Å and the corresponding *D*—H···*A* angles are 159 (4), 148 (4), 178 (4), 161 (4) and 163 (5)°.

In addition, there are nine intramolecular C—H···O approaches with C···O < 3·3 Å, C(12*A*)—H(12*A*)···O(3*A*) 2·89 (11); C(14*A*)—H(14*A*)···O(3*A*) 2·91 (1); C(18*A*)—H(18*C*)···O(2*A*) 3·28 (1); C(21*A*)—H(21*B*)···O(3*A*) 2·82 (1); C(21*A*)—H(21*B*)···O(4*A*) 2·76 (1); C(24*A*)—H(24*A*)···O(2*A*) 2·97 (1); C(12*B*)—H(12*C*)···O(3*B*) 2·73 (1); C(21*B*)—H(21*E*)···O(4*B*) 2·87 (1) and C(24*B*)—H(24*C*)···O(2*B*) 2·96 (1) Å, which stabilize the molecular conformation.

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Structure of 1,5-Bis[(4-ethylphenyl)amino]-9,10-anthracenedione

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Abstract. $C_{30}H_{26}N_2O_2$, $M_r = 446{\cdot}6$, monoclinic, $P2_1/c$, $a = 4{\cdot}687\ (1)$, $b = 12{\cdot}119\ (3)$, $c = 20{\cdot}558\ (4)$ Å, $\beta = 93{\cdot}783\ (3)°$, $V = 1165{\cdot}2$ Å^3, $Z = 2$, $D_x = 1{\cdot}273$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 5{\cdot}50$ cm^{-1}, $F(000) = 472$, $T = 297$ K, final $R = 0{\cdot}0621$ for 1529 unique observed reflections. The phenyl ring is twisted by 50·3 (5)° out of the anthraquinone plane. Intramolecular hydrogen bonds are formed between the amino groups of the substituents and the carbonyl oxygens [H···O = 1·84 (2) Å].

Introduction. The title compound (D-35, according to BDH nomenclature) belongs to a series of pleochroic dyes that were developed for application in guest–host liquid-crystal displays (Pellatt, Roe & Constant, 1980). As part of our investigations of pleochroic dyes, the molecular and crystal structures of D-35 were determined. The results are discussed in relation to the known structures of the similar dyes 1-{[4-(dimethylamino)phenyl]amino}-4-hydroxy-9,10-anthracenedione (D-27) (Foitzik, Paulus & Haase, 1986*a*), 1-{[4-(dimethylamino)phenyl]amino}-9,10-anthracenedione (D-52M) (Foitzik & Paulus, 1986), and 1,5-bis{[4-(pentyloxy)phenyl]amino}-9,10-anthracenedione (D-43) (Foitzik, Paulus & Haase, 1986*b*).

Experimental. Substance from BDH Chemicals Ltd, UK, no further purification; black–violet needles by slow evaporation of a solution in $CHCl_3$/cyclohexane; crystal dimensions 0·1 × 0·1 × 4 mm; cell constants and e.s.d.'s from least-squares refinement of 48 reflections, $29{\cdot}7 < 2\theta < 58{\cdot}3°$; Stoe–Stadi 4 diffractometer; 2528 intensities measured, $-1 < h < 6$, $-14 < k < 6$, $-24 < l < 24$, max. $(\sin\theta)/\lambda = 0{\cdot}5617$ Å^{-1}; three standard reflections ($11\bar{4}$, 122, $1\bar{2}2$), intensity variation <4%, 1641 unique reflections, $R_{\mathrm{int}} = 0{\cdot}0160$, 112 reflections unobserved with $F_o < 2\sigma(F_o)$; no absorption correction; structure solved by direct methods with *SHELX*76 (Sheldrick, 1976); most of the H atoms from difference Fourier synthesis; for comparison with the other structures of this series all C-bound H atoms placed at calculated positions (C—H = 1·08 Å), H(1N) refined; all H atoms with isotropic thermal parameters fixed at 1·1 times the average value of the diagonal

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