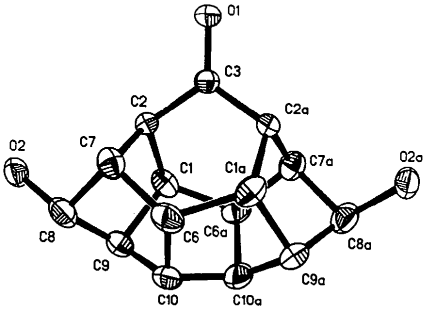

Fig. 2. A view of (1) showing the twofold axis. The benzoyl groups, not shown, are also related by this twofold axis. The thermal ellipsoids are shown at the 20% probability level.

in the norbornadiene moiety five are significantly larger than normal tetrahedral angles [ave. = 115·3 (4)°] and 13 are significantly smaller than the expected value of 109·5° [ave. = 101·8 (4)°]. The internal angle at the bridge atom, C(8), is extremely small [C(7)—C(8)—C(9) = 95·4 (4)°]. Molecular-mechanics calculations were carried out with the 1980 version of program *MM*2 (Allinger, 1976) to see if the strain found in this system could be mathematically predicted. In fact, all the distances along the C(8)—C(9)—C(10)—C(10a)—C(9a)—C(8a) chain are extraordinarily short [ave. = 1·466 (7) Å] yet are predicted to be 1·52 or 1·53 Å by *MM*2. An analysis of the thermal ellipsoids for these atoms indicated corrections of only 0·007 Å for these bonds, indicating that the shortening is not due to thermal motion. These bonds involve atoms separated from double-bonding systems by at least two single bonds. Therefore, while these calculations did predict the angular strain, no meaningful correlations were noted between the bond lengths found in the X-ray study and those calculated by the molecular-mechanics program (Table 2).

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Structure of an Antileukaemic Agent, 5,5′,10,10′-Dihydro-5,5′-dioxo-1,1′,3,3′-tetraphenyl-10,10′-bi(benz[*g*])isoquinoline*

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Abstract. $C_{50}H_{32}N_2O_2$, $M_r = 692·78$, triclinic, $P\bar{1}$, $a = 10·4411$ (9), $b = 11·746$ (1), $c = 15·929$ (1) Å, $\alpha = 86·21$ (1), $\beta = 82·63$ (1), $\gamma = 64·57$ (1)°, $V = 1750·3$ Å^3, $Z = 2$, $D_m = 1·322$ (2), $D_x = 1·315$ g cm^{-3}, Cu $K\alpha$ ($\lambda = 1·5418$ Å), $\mu = 5·44$ cm^{-1}, $F(000) = 724$, $T = 293$ K, $R = 0·041$ for 3636 unique reflections with $F > 5·0\sigma(F)$. The molecule consists of two similar fragments linked through a weak C(sp^3)—C(sp^3) bond of length 1·617 (6) Å. The nitrogens of the pyridine rings are not protonated. The rotations of the phenyl rings from the plane of the pyridine ring prevent the conjugation of the unsaturated systems. The molecular packing in the unit cell is stabilized by van der Waals forces.

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‡ Contribution No. 709.

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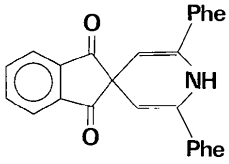
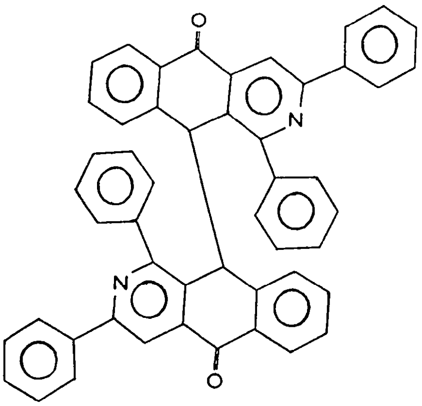
Introduction. The compound was synthesized from indan-1,3-dione as starting material (Padmanabhan, Jagannadha Rao, Ramana & Ramadas, 1981) and was tested for its antileukaemic activity. The structure suggested was spiro(2,6-diphenyl-1*H*-pyridine-4,2′-indan-1′,3′-dione), (I).



(I)

A crystal structure analysis was undertaken to determine the three-dimensional structure, which produced the structure (II).



(II)

Experimental. Yellow rectangular crystals from glacial acetic acid, 0·15 × 0·25 × 0·30 mm, D_m by flotation, three-dimensional intensity data using four-circle automated Picker diffractometer, $\theta/2\theta$ scan, Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å), 38 reflections with $2\theta \leq 100°$ for lattice-parameter measurement and refinement, $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}5615$ Å^{-1}, 5186 unique reflections with $2\theta \leq 120°$, $-10 \leq h \leq 11$; $0 \leq k \leq 12$; $-17 \leq l \leq 17$; two reflections were measured after every 100 reflections and there was no significant variation in intensity, $R_{\rm int} = 0{\cdot}012$, 1550 unobserved reflections with $F < 5{\cdot}0\sigma(F)$. Structure solution using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); after repeated trials, the assumption that a fragment was correctly oriented but randomly positioned (*ORIENT*) gave the complete molecule; block-diagonal least-squares refinement on $F_{\rm obs}$ (Shiono, 1968), H atoms geometrically fixed and checked against $\Delta\rho$ synthesis, non-hydrogens refined [minimizing $\sum w(\Delta F)^2$] anisotropically and hydrogens isotropically, with Cruickshank's weighting scheme (Cruickshank, Bujosa, Lovell & Truter, 1961); $\sigma = \{[A + B(F_{\rm obs}) + C(F_{\rm obs})^2]^{1/2}$, $A = 5{\cdot}0$, $B = 1{\cdot}0$, $C = 0{\cdot}025\}$, $R = 0{\cdot}041$, $wR = 0{\cdot}064$, goodness of fit $S = 0{\cdot}263$ for 614 parameters, $(\Delta/\sigma)_{\rm ave} = 0{\cdot}08$, $(\Delta/\sigma)_{\max} = 0{\cdot}34$, $|\Delta\rho|_{\max} = \pm 0{\cdot}28$ e Å^{-3}. Lp correction, no absorption correction. Atomic scattering factors for non-hydrogen atoms from *International Tables for X-ray Crystallography* (1968), and for H from Stewart, Davidson & Simpson (1965).

Discussion. Fractional atomic parameters and temperature coefficients for non-hydrogen atoms are given in Table 1.* Atom numbering is given in Fig. 1. Some of the bond lengths and bond angles involving the non-hydrogen atoms are given in Table 2. The average e.s.d. on bond lengths is 0·007 Å and on bond angles is 0·4°. N(1) through C(27) are denoted as fragment I and N(28) through C(54) as fragment II. The bond lengths and angles in the two fragments are in good agreement with each other and also with standard values. The angles around C(12) and C(39) deviate from the normal tetrahedral values, obviously owing to strain caused by dimer formation. In similar molecules with planar outer rings, the internal angles at the central sp^3 C atoms are usually greater than the tetrahedral value, as observed in 10-bromoanthrone [117·0 (7)°] (Destro, D'Alfonso & Simonetta, 1973). N(1) and N(28) are not protonated and the average C—N bond length is 1·341 (6) Å; C—N—C is 120·0 (4)°. The angles N(1)—C(2)—C(16) 115·7 (4), N(28)—C(29)—C(43) 117·1 (4), N(1)—C(14)—C(22) 114·7 (4) and N(28)—C(41)—C(49) 112·7 (4)° are much less than the standard value at a C(sp^2) atom. This is observed in other 1,6-substituted unsaturated hetero ring systems, *e.g.* 3-acetyl-4-hydroxy-6-phenyl-2-pyrone (Thailambal & Vasantha Pattabhi, 1985); 4-hydroxy-3-(3-hydroxy-1-oxo-3-phenyl-2-propenyl)-6-methyl-2-pyrone (Thailambal, Vasantha Pattabhi & Gabe, 1986). The bond C(12)—C(39), 1·617 (6) Å, linking the two fragments is rather long, which may have been necessary for avoiding steric hindrance during dimer formation. A similarly high value, 1·612 (7) Å, is reported in 1,1′,8,8′-tetrahydroxy-10,10′-bi-9(10*H*)-anthrone (Ahmed & Neville, 1982).

The torsion angles within the pyridine ring vary from −3·9 (7) to 6·6 (7)° in fragment I and −8·5 (7) to 8·1 (6)° in fragment II, showing that they are not strictly planar. The central ring in the tricyclic group of I is between a sofa and boat conformation with Duax asymmetry values (Duax, Weeks, & Rohrer, 1976) of

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* Lists of structure factors, anisotropic thermal parameters, bond distances and angles, torsion angles, least-squares-planes equations and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44266 (41 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

$\Delta C_{s1} = 8{\cdot}3\ (7)$ and $\Delta C_{s2} = 13{\cdot}0\ (7)$. The corresponding ring in fragment II is in a distorted sofa conformation with $\Delta C_s = 15{\cdot}0\ (7)$. The torsion angles around C(12)—C(39) show that the substitutions are in staggered conformation.

Table 1. *Positional parameters* (× 10^4) *and equivalent isotropic temperature factors* (Å^2) *for non-hydrogen atoms with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
N(1)	5047 (4)	2396 (3)	4220 (2)	3·5 (1)
C(2)	6280 (5)	1562 (4)	4500 (3)	3·5 (1)
C(3)	6769 (5)	284 (4)	4366 (3)	3·6 (1)
C(4)	6053 (4)	−138 (4)	3884 (3)	3·3 (1)
C(5)	6618 (5)	−1519 (4)	3717 (3)	4·1 (2)
C(6)	5657 (5)	−1935 (4)	3357 (3)	3·8 (1)
C(7)	5952 (6)	−3209 (5)	3395 (4)	4·8 (2)
C(8)	5031 (7)	−3621 (5)	3113 (4)	5·3 (2)
C(9)	3795 (7)	−2761 (5)	2808 (3)	5·0 (2)
C(10)	3485 (5)	−1490 (5)	2762 (3)	4·0 (2)
C(11)	4410 (5)	−1053 (4)	3033 (3)	3·4 (1)
C(12)	4106 (4)	317 (4)	2943 (3)	3·0 (1)
C(13)	4819 (4)	724 (4)	3555 (3)	3·0 (1)
C(14)	4321 (4)	1989 (4)	3781 (3)	3·1 (1)
O(15)	7792 (4)	−2247 (3)	3905 (3)	6·7 (2)
C(16)	7089 (5)	2115 (5)	4903 (3)	3·9 (2)
C(17)	6696 (6)	3401 (5)	4859 (3)	4·8 (2)
C(18)	7479 (6)	3927 (6)	5187 (4)	5·7 (2)
C(19)	8691 (6)	3172 (7)	5559 (4)	6·3 (2)
C(20)	9093 (6)	1895 (7)	5619 (4)	6·4 (2)
C(21)	8305 (5)	1352 (6)	5298 (3)	5·1 (2)
C(22)	2951 (5)	2995 (4)	3526 (3)	3·2 (1)
C(23)	1652 (5)	2982 (5)	3830 (3)	4·1 (2)
C(24)	395 (5)	3932 (5)	3593 (3)	4·7 (2)
C(25)	425 (5)	4888 (5)	3051 (3)	4·5 (2)
C(26)	1706 (6)	4914 (5)	2761 (3)	4·4 (2)
C(27)	2967 (5)	3969 (4)	2998 (3)	3·7 (1)
N(28)	8567 (4)	−1010 (3)	1580 (2)	3·3 (1)
C(29)	8727 (5)	−2091 (4)	1252 (3)	3·1 (1)
C(30)	7554 (5)	−2232 (4)	1044 (3)	3·3 (1)
C(31)	6204 (5)	−1285 (4)	1229 (3)	3·1 (1)
C(32)	4983 (5)	−1377 (4)	879 (3)	3·6 (1)
C(33)	3606 (5)	−256 (4)	952 (3)	3·3 (1)
C(34)	2493 (5)	−225 (5)	526 (3)	3·8 (2)
C(35)	1195 (5)	799 (5)	604 (3)	4·2 (2)
C(36)	987 (5)	1819 (5)	1087 (3)	4·3 (2)
C(37)	2075 (5)	1790 (5)	1513 (3)	3·7 (1)
C(38)	3397 (5)	756 (4)	1454 (3)	3·2 (1)
C(39)	4524 (4)	690 (4)	1986 (3)	3·0 (1)
C(40)	6005 (4)	−202 (4)	1651 (3)	2·9 (1)
C(41)	7259 (5)	−82 (4)	1762 (3)	3·0 (1)
O(42)	5173 (4)	−2313 (3)	501 (3)	5·7 (1)
C(43)	10206 (5)	−3049 (4)	1034 (3)	3·3 (1)
C(44)	10499 (5)	−4280 (4)	827 (3)	4·1 (2)
C(45)	11888 (6)	−5134 (5)	584 (3)	4·6 (2)
C(46)	12997 (5)	−4794 (5)	543 (4)	4·8 (2)
C(47)	12733 (5)	−3585 (5)	757 (4)	4·8 (2)
C(48)	11349 (5)	−2732 (4)	1009 (3)	3·9 (2)
C(49)	7295 (5)	1108 (4)	2015 (3)	3·4 (1)
C(50)	8243 (5)	1047 (5)	2581 (3)	4·1 (2)
C(51)	8385 (6)	2119 (6)	2763 (4)	5·1 (2)
C(52)	7608 (6)	3254 (5)	2379 (4)	5·7 (2)
C(53)	6687 (6)	3329 (5)	1801 (4)	5·3 (2)
C(54)	6523 (5)	2255 (4)	1624 (3)	4·1 (2)

Table 2. *Some relevant bond lengths* (Å) *and bond angles* (°) *involving the non-hydrogen atoms*

N(1)—C(2)	1·349 (6)	C(2)—N(1)—C(14)	119·6 (4)
N(1)—C(14)	1·333 (6)	C(29)—N(28)—C(41)	120·3 (4)
C(2)—C(16)	1·486 (7)	C(11)—C(12)—C(13)	113·1 (4)
C(12)—C(39)	1·617 (6)	C(11)—C(12)—C(39)	112·1 (4)
C(14)—C(22)	1·496 (7)	C(13)—C(12)—C(39)	109·9 (4)
C(29)—C(43)	1·483 (7)	C(12)—C(39)—C(38)	107·9 (4)
C(41)—C(49)	1·497 (6)	C(12)—C(39)—C(40)	111·5 (4)
N(28)—C(29)	1·340 (6)	C(38)—C(39)—C(40)	113·4 (4)
N(28)—C(41)	1·343 (6)		

All the phenyl rings are planar with a maximum $\sum\Delta^2/\langle\sigma^2\rangle = 13{\cdot}0$. The phenyl rings substituted at the pyridine rings make angles of 14·6 (6) and 63·3 (6)° with the pyridine ring in I and 16·1 (6) and 47·6 (6)° in II. These rotations prevent conjugation between the unsaturated systems and also may be due to steric forces. The tricyclic groups take a butterfly shape which is common in anthrone ring systems and helps to avoid the crowding of the atoms (Destro, D'Alfonso & Simonetta, 1973). The dihedral angles between the outer rings in the tricyclic group are 17·5 (7) and 18·9 (7)° in the two fragments. The mean planes through the tricyclic groups make an angle of 27·8 (6)° with each other. Thermal ellipsoids for the non-hydrogen atoms drawn at the 50% probability level (Johnson, 1976) and a stereoview of the molecule

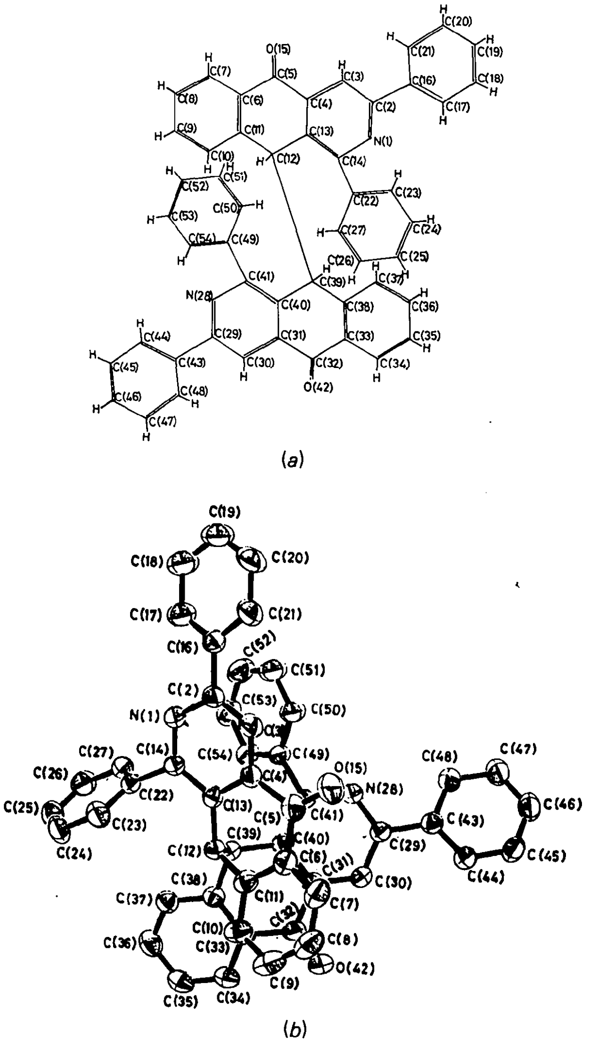

Fig. 1. (*a*) Schematic diagram with atom numbering. (*b*) Thermal ellipsoids drawn at 50% probability level for the non-hydrogen atoms (Johnson, 1976).

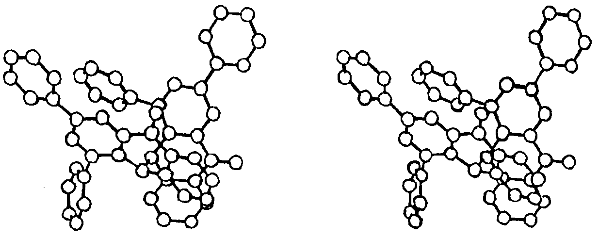

Fig. 2. Stereoview of the single molecule (Motherwell & Clegg, 1978).

(Motherwell & Clegg, 1978) are given in Figs. 1(*b*) and 2, respectively.

The C(47)···O(42) (1 + *x*, *y*, *z*) contact distance is 3·438 (8) Å with C—H 1·00 (7), H···O 2·46 (7) Å, C—H···O 165 (4)°, which is rather long for a C—H···O hydrogen bond. The packing of the molecules in the unit is stabilized by van der Waals forces.

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Structure of 2,3-Dihydro-9-hydroxy-2-(1-hydroxy-1-methylethyl)-7*H*-furo[3,2-*g*][1]benzopyran-7-one Monohydrate*

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Abstract. $C_{14}H_{14}O_5.H_2O$, M_r = 280·3, monoclinic, *a* = 24·629 (2), *b* = 8·244 (1), *c* = 6·739 (1) Å, *β* = 104·21 (1)°, *V* = 1326·43 Å^3, *Z* = 4, D_x = 1·403 (1), D_m = 1·400 (2) g cm^{-3}, Cu *Kα* (*λ* = 1·5418 Å), *μ* = 8·9 cm^{-1}, *T* = 293 K, *F*(000) = 592, *R*(*F*) = 0·0441 for 1038 reflections with $I > 2{\cdot}5\sigma(I)$. The central ring (*B*) makes angles of 2·4 (4) and 3·4 (4)° with the mean planes through the pyran ring (*A*) and the furan ring (*C*) respectively. The molecules are nearly perpendicular to the *c* axis [88·0 (3)°] and the *B* rings of the twofold-related molecules are stacked along the *c* axis with a stacking distance of about 3·4 Å. The protons available from the water molecule and the two hydroxyl groups take part in hydrogen bonding.

Introduction. $C_{14}H_{14}O_5.H_2O$ was extracted from *Atalantia racemosa,* a herb used in Indian medicine. The structure solution was undertaken to determine the three-dimensional structure. Structure (I) has been

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‡ Contribution No. 708.

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