molecules in compact clusters stabilized in the packing by six hydrogen bonds.

Fig. 3 displays the trimeric asymmetric unit (*PLUTO*; Motherwell & Clegg, 1978) viewed along the *c* axis with the connecting hydrogen bonds. The molecules are associated using all the possible polar atoms, including both donor and acceptor hydrogen bonds for the O(5) hydroxyl function in the three molecules. As a consequence, there are no polar contacts closer than 3·5 Å between the different trimers of the packing.

In conclusion, this X-ray analysis revealed that the expected ring expansion in (1) was correct but that the reaction proceeded one step ahead of the predicted result, *i.e.* compound (3) and not (2). Further chemical syntheses are in progress to take advantage of this extra step towards a new route to the taxol backbone.

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Structure of Methyl 3-Hydroxy-1-(1-isoquinolyl)-3-phenyl-3,3a,8,8a-tetrahydroindeno[1,2-*c*]pyrrol-8a-ylcarboxylate

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Abstract. $C_{28}H_{22}N_2O_3$, $M_r = 434{\cdot}5$, triclinic, $P\bar{1}$, $a = 11{\cdot}081$ (1), $b = 10{\cdot}247$ (1), $c = 15{\cdot}074$ (2) Å, $\alpha = 93{\cdot}584$ (2), $\beta = 130{\cdot}11$ (2), $\gamma = 113{\cdot}09$ (2)°, $V = 1076{\cdot}7$ (9) Å^3, $Z = 2$, $D_x = 1{\cdot}340$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}067$ mm^{-1}, $F(000) = 456$, $T = 294$ K, final $R = 0{\cdot}041$ for 2742 unique reflections. The regiochemistry of the condensation product of 2-methoxycarbonylindene with 2-benzoyl-1,2-dihydroisoquinaldonitrile fluoroborate was established in order to show that it was a derivative of indeno[1,2-*c*]pyrrole. The two pentagonal rings have an envelope shape. Molecules are linked by a hydrogen bond, O(31)—H(31)···N(2^{i}) [2·936 (2) Å, 174 (2)°] [(i) $1-x, -y, 1-z$].

Introduction. The reaction of 2-benzoyl-1,2-dihydroisoquinaldonitrile fluoroborate (1) with indene (2) led to a condensation product (see scheme below), the formula of which could not be found completely from ^{1}H NMR data. So the condensation of (1) with

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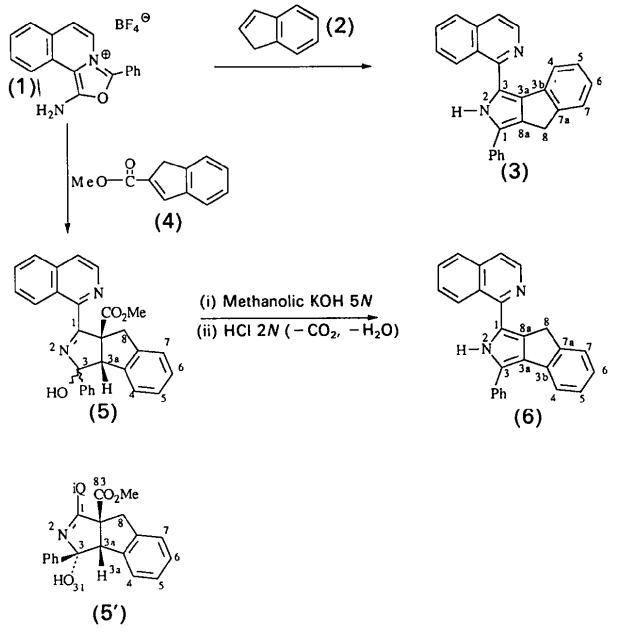
2-methoxycarbonylindene (4) was attempted and was successful. Crystals sufficiently large were obtained for an X-ray crystallographic study as detailed below. Their formula proved to be (5).



Experimental. Crystal of hexagonal platelet shape, 0·13 × 0·10 × 0·08 mm. Cell parameters determined from 25 reflections having $7{\cdot}72 \le \theta \le 14{\cdot}51°$. Enraf–Nonius CAD-4 diffractometer. θ–2θ scans; scan range $(0{\cdot}80 + 0{\cdot}35\tan\theta)°$. $0{\cdot}023 \le \sin\theta/\lambda \le 0{\cdot}572$ Å^{-1}, $11 \le h \le 12$, $0 \le k \le 11$, $-16 \le l \le 16$. Intensity control reflections 502, 566, 354. Intensity variation during measurements was not significant. No absorption correction. 3183 unique reflections; 441 unobserved reflections $[I < 2\sigma(I)]$. Direct methods, program *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refinement based on F's. To refine the structure properly it was necessary to introduce two positions for the methoxycarbonyl group substituted on C(8a) with occupancy factors close to 0·5: C(83), O(84), O(85) and C(86) on one hand, C(83), O(84*B*), O(85*B*) and C(86*B*) on the other. The two positions are rotated by nearly 180° around C(8a)—C(83).

H-atom locations were calculated, when possible, before refinement. It was not possible to locate the H-atom positions for the methyl group C(86) [or C(86*B*)] from the difference Fourier map; apparently there are two favoured positions for each group (occupancy factor 0·25 for each H atom). Atomic diffusion factors from *International Tables for X-ray Crystallography*, (1974, Vol. IV, pp. 99, 149). Full-matrix refinements. Refined parameters were x, y, z for all atoms and β_{ij} for C, N and O atoms. For each H atom, B was chosen equal to (1 Å^2 + B_{eq} of the neighbouring heavy atom). $wR = 0{\cdot}048$, $w = 1/\sigma^2(F)$, $S = 2{\cdot}14$. Maximum shift to e.s.d. ratio $(\Delta/\sigma)_{max} < 0{\cdot}01$. $\Delta\rho_{max} = 0{\cdot}23$ (5), $\Delta\rho_{min} = -0{\cdot}21$ (5) e Å^{-3}.

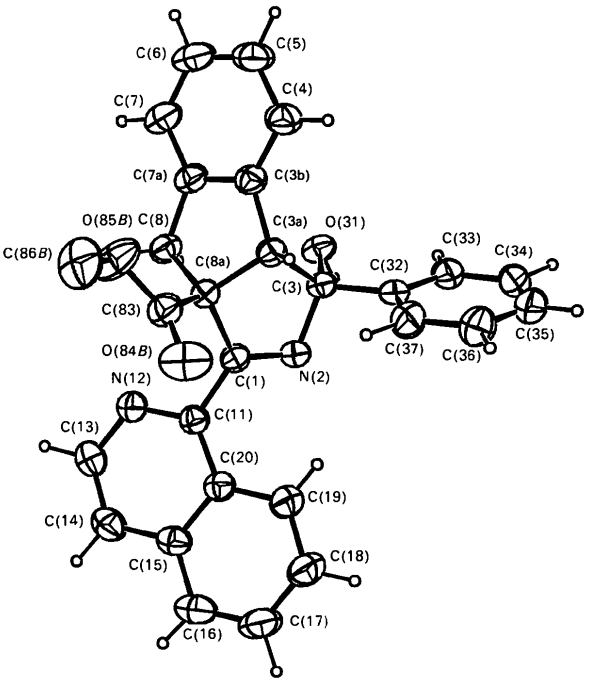


Fig. 1. *ORTEP* view of the molecule.

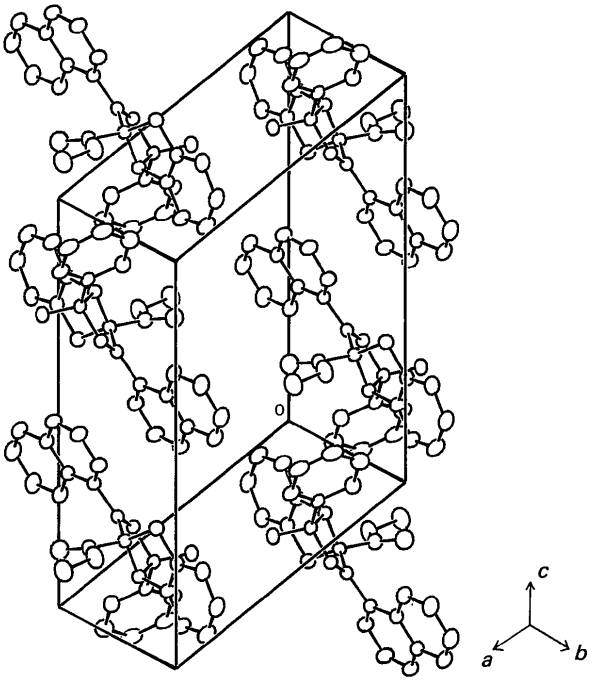


Fig. 2. *ORTEP* view of the packing.

Table 1. *Positional and thermal parameters and their e.s.d.'s*

Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as $B_{eq} = (4/3) \times (\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab\cos\gamma + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\alpha)$.

	x	y	z	B_{eq}(Å^2)
C(1)	0·8800 (1)	0·2673 (2)	0·2069 (1)	2·49 (4)
N(2)	0·7464 (1)	0·1382 (1)	0·10119 (9)	2·61 (3)
C(3)	0·6761 (2)	0·1761 (2)	−0·0107 (1)	2·55 (4)
C(3a)	0·8308 (2)	0·3562 (2)	0·0517 (1)	2·64 (4)
C(3b)	0·7608 (2)	0·4575 (2)	−0·0005 (1)	2·88 (4)
C(4)	0·7102 (2)	0·4818 (2)	−0·1060 (1)	3·89 (5)
C(5)	0·6575 (2)	0·5875 (2)	−0·1336 (1)	4·53 (6)
C(6)	0·6518 (2)	0·6641 (2)	−0·0589 (2)	4·48 (6)
C(7)	0·6989 (2)	0·6377 (2)	0·0457 (1)	3·76 (5)
C(7a)	0·7572 (2)	0·5353 (2)	0·0757 (1)	3·03 (4)
C(8)	0·8257 (2)	0·4935 (2)	0·1877 (1)	3·04 (4)
C(8a)	0·9262 (2)	0·4193 (2)	0·1921 (1)	2·49 (4)
C(11)	0·9844 (2)	0·2719 (2)	0·3371 (1)	2·66 (4)
N(12)	1·0220 (1)	0·3854 (1)	0·4158 (1)	3·36 (4)
C(13)	1·1150 (2)	0·3994 (2)	0·5373 (1)	3·88 (5)
C(14)	1·1778 (2)	0·3068 (2)	0·5831 (1)	3·72 (5)
C(15)	1·1476 (2)	0·1897 (2)	0·5031 (1)	3·20 (4)
C(16)	1·2176 (2)	0·0946 (2)	0·5476 (1)	4·26 (5)
C(17)	1·1906 (2)	−0·0128 (2)	0·4684 (2)	4·80 (6)
C(18)	1·0945 (2)	−0·0311 (2)	0·3424 (2)	4·47 (6)
C(19)	1·0224 (2)	0·0568 (2)	0·2954 (1)	3·71 (5)
C(20)	1·0464 (2)	0·1693 (2)	0·3748 (1)	2·80 (4)
O(31)	0·5018 (1)	0·1560 (1)	−0·07191 (8)	3·12 (3)
C(32)	0·6545 (2)	0·0685 (2)	−0·1013 (1)	2·74 (4)
C(33)	0·4757 (2)	−0·0530 (2)	−0·2219 (1)	3·48 (5)
C(34)	0·4588 (2)	−0·1514 (2)	−0·3029 (1)	4·43 (6)
C(35)	0·6199 (2)	−0·1267 (2)	−0·2649 (1)	4·86 (5)
C(37)	0·8157 (2)	0·0888 (2)	−0·0630 (1)	3·96 (5)
C(83)	1·1411 (2)	0·5383 (2)	0·2906 (1)	3·33 (4)
O(84)	1·2303 (3)	0·6790 (3)	0·3610 (2)	5·8 (1)
O(85)	1·2331 (2)	0·4782 (3)	0·3124 (2)	3·74 (6)
C(86)	1·4390 (4)	0·5601 (5)	0·4255 (3)	5·9 (1)
O(84*B*)	1·2432 (3)	0·4866 (3)	0·3243 (2)	5·79 (9)
O(85*B*)	1·1893 (3)	0·6846 (3)	0·3109 (2)	4·48 (8)
C(86*B*)	1·3887 (4)	0·8051 (5)	0·3884 (4)	5·6 (1)

Computer programs of the *SDP* system (B. A. Frenz & Associates, Inc., 1982). *ORTEP* (Johnson, 1976) was used to represent the molecule (Fig. 1) and the packing (Fig. 2). Atomic coordinates are listed in Table 1 and bond lengths and angles in Table 2.*

Discussion. Bond lengths for the central part of the compound are consistent with formula (5) above, and more precisely with (5′), where iQ = 1-isoquinolinyl.

Mean planes of the different rings. Planes were defined as follows: Π(1) C(1), N(2), C(3) and C(8a); Π(2) C(3a), C(3b), C(7a) and C(8); Π(3) C(3b) C(4), C(5), C(6), C(7) and C(7a); Π(4) C(11), N(12), C(13), C(14), C(15) and C(20); Π(5) C(15)–C(20); Π(6) Π(4) and Π(5) together; Π(7) C(32)–C(37); Π(8) C(8a), C(83), O(84), O(84) and C(86); Π(8*B*) C(8a), C(83), O(84*B*), O(85*B*) and C(86*B*).

The planarity of the aromatic rings [Π(3), Π(4), Π(5), Π(7)] and the isoquinolinyl group [Π(6)] is good.

* Lists of structure factors, coordinates of the H atoms, anisotropic thermal parameters for the non-H atoms, bond distances involving H atoms, average ring planes and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54076 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and angles* (°) *with their e.s.d.'s*

C(1)—N(2)	1·281 (1)	C(13)—C(14)	1·358 (3)
C(1)—C(8a)	1·518 (2)	C(14)—C(15)	1·410 (3)
C(1)—C(11)	1·487 (2)	C(15)—C(16)	1·417 (3)
N(2)—C(3)	1·492 (2)	C(15)—C(20)	1·418 (2)
C(3)—C(3a)	1·567 (2)	C(16)—C(17)	1·358 (4)
C(3)—O(31)	1·411 (2)	C(17)—C(18)	1·403 (3)
C(3)—C(32)	1·526 (3)	C(18)—C(19)	1·366 (3)
C(3a)—C(3b)	1·498 (2)	C(19)—C(20)	1·418 (3)
C(3a)—C(8a)	1·557 (2)	C(32)—C(33)	1·386 (1)
C(3b)—C(4)	1·385 (3)	C(32)—C(37)	1·390 (3)
C(3b)—C(7a)	1·387 (3)	C(33)—C(34)	1·395 (3)
C(4)—C(5)	1·388 (3)	C(34)—C(35)	1·374 (4)
C(5)—C(6)	1·381 (4)	C(35)—C(36)	1·380 (1)
C(6)—C(7)	1·392 (3)	C(36)—C(37)	1·386 (3)
C(7)—C(7a)	1·392 (3)	C(83)—O(84)	1·234 (3)
C(7a)—C(8)	1·518 (2)	C(83)—O(85)	1·270 (4)
C(8)—C(8a)	1·549 (3)	C(83)—O(84*B*)	1·257 (4)
C(8a)—C(83)	1·518 (2)	C(83)—O(85*B*)	1·315 (3)
C(11)—N(12)	1·321 (2)	O(85)—C(86)	1·457 (3)
C(11)—C(20)	1·428 (3)	O(85*B*)—C(86*B*)	1·458 (4)
N(12)—C(13)	1·362 (2)		

N(2)—C(1)—C(8a)	115·6 (1)	C(11)—N(12)—C(13)	118·3 (2)
N(2)—C(1)—C(11)	123·4 (1)	N(12)—C(13)—C(14)	123·3 (2)
C(8a)—C(1)—C(11)	121·0 (1)	C(13)—C(14)—C(15)	119·5 (2)
C(1)—N(2)—C(3)	109·6 (1)	C(14)—C(15)—C(16)	122·1 (2)
N(2)—C(3)—C(3a)	104·99 (8)	C(14)—C(15)—C(20)	118·5 (2)
N(2)—C(3)—O(31)	108·1 (1)	C(16)—C(15)—C(20)	119·4 (2)
N(2)—C(3)—C(32)	108·8 (1)	C(15)—C(16)—C(17)	119·9 (2)
C(3a)—C(3)—O(31)	108·9 (1)	C(16)—C(17)—C(18)	121·0 (2)
C(3a)—C(3)—C(32)	113·1 (2)	C(17)—C(18)—C(19)	120·7 (2)
O(31)—C(3)—C(32)	112·65 (8)	C(18)—C(19)—C(20)	120·0 (2)
C(3)—C(3a)—C(3b)	117·3 (1)	C(11)—C(20)—C(15)	116·7 (2)
C(3)—C(3a)—C(8*a*)	102·6 (1)	C(11)—C(20)—C(19)	124·2 (2)
C(3b)—C(3a)—C(8a)	103·9 (1)	C(15)—C(20)—C(19)	119·0 (2)
C(3a)—C(3b)—C(4)	128·3 (2)	C(3)—C(32)—C(33)	121·3 (2)
C(3a)—C(3b)—C(7a)	110·6 (2)	C(3)—C(32)—C(37)	120·2 (1)
C(4)—C(3b)—C(7a)	121·1 (2)	C(33)—C(32)—C(37)	118·5 (2)
C(3b)—C(4)—C(5)	118·6 (2)	C(32)—C(33)—C(34)	120·5 (2)
C(4)—C(5)—C(6)	120·6 (2)	C(33)—C(34)—C(35)	120·2 (1)
C(5)—C(6)—C(7)	121·0 (2)	C(34)—C(35)—C(36)	119·9 (2)
C(6)—C(7)—C(7a)	118·4 (2)	C(35)—C(36)—C(37)	120·0 (2)
C(3b)—C(7a)—C(7)	120·3 (2)	C(32)—C(37)—C(36)	120·9 (1)
C(3b)—C(7a)—C(8)	110·9 (2)	C(8a)—C(83)—O(84)	125·2 (2)
C(7)—C(7a)—C(8)	128·7 (2)	C(8a)—C(83)—O(85)	114·7 (1)
C(7a)—C(8)—C(8a)	103·1 (2)	C(8a)—C(83)—O(84*B*)	118·3 (2)
C(1)—C(8a)—C(3a)	100·8 (1)	C(8a)—C(83)—O(85*B*)	112·2 (2)
C(1)—C(8a)—C(8)	116·1 (2)	O(84)—C(83)—O(85)	119·3 (2)
C(1)—C(8a)—C(83)	110·7 (1)	O(84)—C(83)—O(84*B*)	114·7 (2)
C(3a)—C(8a)—C(8)	104·6 (1)	O(84*B*)—C(83)—O(85*B*)	128·3 (2)
C(3a)—C(8a)—C(83)	111·3 (2)	C(83)—O(85)—C(86)	123·8 (3)
C(8)—C(8a)—C(83)	112·5 (1)	C(83)—O(85*B*)—C(86*B*)	116·6 (3)
C(1)—C(11)—N(12)	113·1 (2)		
C(1)—C(11)—C(20)	123·2 (2)		
N(12)—C(11)—C(20)	123·6 (1)		

The ring C(1), N(2), C(3), C(3a) and C(8a) exhibits an envelope shape; this is the same shape for the ring C(3a), C(3b), C(7a), C(8) and C(8a). As a consequence, according to the concept proposed by Duax & Norton (1975), the predominant symmetry is close to a plane passing through C(3a) and the middle of C(1)—N(2) in one cycle, and through C(8a) and the middle of C(3b)—C(7a) in the other. Discrepancies from ideal symmetry are represented as $\Delta C_s^{3a} = 1{\cdot}4°$ and $\Delta C_s^{8a} = 0{\cdot}3°$.

The angle Π(2) makes with Π(3) is 2·3 (1·5)° and Π(1)–Π(2) = 51·5 (1)°. Π(2), Π(3) and Π(6) are approximately parallel. Π(8) and Π(9), the two positions for the methoxycarbonyl group, are approximately twisted by half a turn [180–18·0 (3)°]; they are approximately perpendicular to Π(1) and Π(2).

Torsion angles. The torsion angles H(3a)—C(3a)—C(8a)—C(83) = 24·4 (1·4) and H(3a)—C(3a)—C(3)—C(31) = −149·7 (1·1)° show that H(3a) and

C(83) are in *cis* positions, whereas H(3a) and O(31) are in *trans* positions.

Packing. The crystal exhibits a hydrogen bond, O(31)—H(31)···N(2^{i}) [2·936 (2) Å, 174 (2)°] [(i) = 1 − x, −y, 1 − z].

When subjected to saponification, (5) was transformed into (6), another indeno[1,2-*c*]pyrrole derivative and a regioisomer of (3). Finally, the crystal structure determination of (5) enabled us to prove not only the formula of (6) but also that of (3).

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N-2-(2-Bromo-4,5-dimethoxyphenyl)-1-(2,3,4-trimethoxyphenyl)ethylformamide

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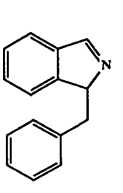
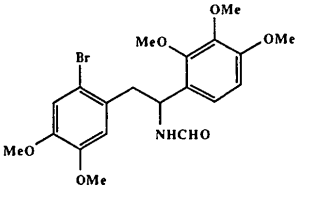
Abstract. $C_{20}H_{24}BrNO_6$, M_r = 454·3, monoclinic, *Cc*, a = 27·479 (4), b = 5·128 (1), c = 21·811 (3) Å, β = 139·35 (1)°, V = 2002 (1) Å^3, Z = 4, D_x = 1·51 Mg m^{-3}, Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 2·07 mm^{-1}, F(000) = 936, T = 130 K, R = 0·030 for 4151 observed reflections. The X-ray study confirms that in the solid state the structure of the title compound is similar to that inferred from chemical and spectroscopic evidence. Steric hindrance from different chemical groups is minimized by the adoption of a staggered conformation at the central C(8)—C(9) bond, with aryl groups in an *anti* disposition. There is a delocalized orbital along the N—C—O fragment of the *N*-formylamino group.

Introduction. The title compound was prepared (Dominguez, Lete, Villa & Iriondo, 1984) as part of a program directed towards the synthesis of new heterocyclic compounds. We planned a new approach for the preparation of the 1-benzylisoindole (1) ring system, which consisted of adapting the Bischler–Napieralski reaction (Dominguez & Lete, 1983) to the synthesis of five-membered rings. However, it was found that the required substrates, 2′-bromo-1,2-diarylethylamides, always failed to undergo cyclization (Villa, Lete & Dominguez, 1986). In as much as the projected cyclization is formally a 5-*endo*-dig process and is favoured according to Baldwin's rules (Baldwin, 1976), we decided to determine the crystal structure of (2) to evaluate the structural factors inhibiting the desired cyclization.



(1)



(2)

Experimental. *NMR*. The ^{1}H and ^{13}C NMR spectra were run on a Bruker AC250 spectrometer at 298 K operating in the pulse-Fourier transform mode and provided with an ASPECT 3000 computer. 5 mm

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