Synthesis and *X*-Ray Crystal Structures of 1-(*NN*-Dimethylaminomethylidene)-3-phenylisoindolenine and 1-Formyl-3-phenylisoindole

Raymond Bonnett,* Michael B. Hursthouse, Stephanie A. North, and Jill Trotter
Chemistry Department, Queen Mary College, Mile End Road, London E1 4NS

1-Phenylisoindole reacts with the Vilsmeier reagent to give 1-(*NN*-dimethylaminomethylidene)-3-phenylisoindole (4), which on basic hydrolysis furnishes 1-formyl-3-phenylisoindole (5). *X*-Ray structure analyses have been carried out on (4) and (5). The latter structure is the first available for an isoindole with a simple substitution pattern, and provides information bearing on the aromaticity of the isoindole nucleus.

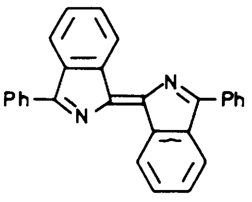
Although the isoindole unit, as part of the phthalocyanine macrocycle,[1] featured in the pioneering work on the determination of complex organic structures by *X*-ray crystallography, very little has been done on the crystal structures of the isoindoles themselves. In view of the marked interest in the properties, especially the aromaticity, of the isoindole system, this must be largely attributable to the reactivity (polymerisation, autoxidation) of simple isoindoles.[2] Certain stabilised skeleta, such as phthalimide [3] and phthalimidine [4] derivatives, have been studied, and structures are also available for dimeric species such as 3,3′-diphenyl-1,1′-bi-(isoindolylidene) (1).[5] Structures have been described for the unusual isoindolenine ylide (2) [6] and, since the completion of the present work, for the highly encumbered 1-alkylthio-derivative (3).[7]

We provide here a full account of the synthesis and *X*-ray structures of 1-(*NN*-dimethylaminomethylidene)-3-phenylisoindolenine (4) and its hydrolysis product 1-formyl-3-phenylisoindole (5). The structure of the latter compound has been reported in a preliminary way: [2] this structure and that of compound (3), which emerged independently but contemporaneously, are the first examples of crystal structures of simple (stabilised) isoindole derivatives.

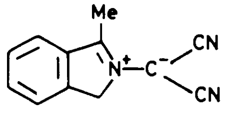
1-(NN-*Dimethylaminomethylidene*)-3-*phenylisoindolenine*.—Electrophilic substitution occurs readily in the isoindole system at the 1,3-positions.[2] When 1-phenylisoindole was treated with the Vilsmeier reagent, followed by rapid work-up with sodium hydrogencarbonate solution, the intermediate iminium salt was obtained as the free base in the form of the dimethylaminomethylidene derivative (4). An analogous reaction is known with indole.[8] Compound (4) formed greenish yellow needles from ethyl acetate and petroleum and had $\lambda_{max.}$ (EtOH) 396 nm (ε 38 400). The n.m.r. spectrum (Figure 1) was temperature dependent: at 55 °C the two methyl groups appeared as a sharp singlet (δ 3.56), while at -27 °C this signal was replaced by two sharp singlets (δ 3.89 and 3.33). This behaviour is attributed to restricted rotation about the C–N bond of the aminomethylidene group, which, as part of an extended vinylogous amidine system, is expected to have some double bond character, as represented in canonical structure (6). The observed coalescence temperature (14 °C) corresponds to a $\Delta G^{\ddagger}$ value of *ca.* 50.3 kJ mol^{-1} for the rotational process.

The structure with the *E*-configuration [rather than the *Z*-configuration shown in (4)] at the exocyclic double bond was considered, although it was regarded as much less likely because of steric interaction between the C(7) proton and the *N*-methyl group. In the event, the *X*-ray work confirmed that the exocyclic double bond of the compound isolated has the *Z*-configuration.

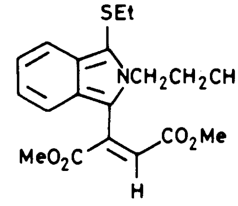
Crystals of (4) suitable for *X*-ray diffraction were obtained from ethanol–pentane using the isothermal distillation method.



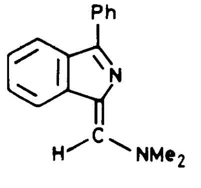
(1)



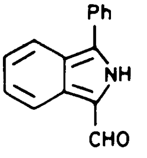
(2)



(3)



(4)



(5)

The molecular structure resulting from *X*-ray analysis and the crystallographic numbering scheme are shown in Figure 2. Two distinct conformers of (4) were present in the crystallographic asymmetric unit, and are designated a and b [(4a) is actually shown in Figure 2]. The bond lengths and angles of conformers a and b are given in Table 1, and data on least-squares planes is collected in Table 2. The crystal packing and unit cell are illustrated in Figure 3.

Molecules (4a and b) exhibit a difference in conformation about the phenyl–isoindolenine bond. Taking a view along this bond in the direction C(3)–C(10), then in conformer (4a) the phenyl group is rotated in a clockwise sense out of the plane of the heterocyclic system, while in conformer (4b) it is rotated anticlockwise. The magnitude of the torsion angles C(9)–C(3)–C(10)–C(11) are almost identical in both molecules (29.2 and $-28.9°$). Conformers (4a and b) are equally favoured in the crystal structure. The twisting of the phenyl group out of plane presumably arises so as to avoid the non-bonded interaction which would otherwise exist between H(4)

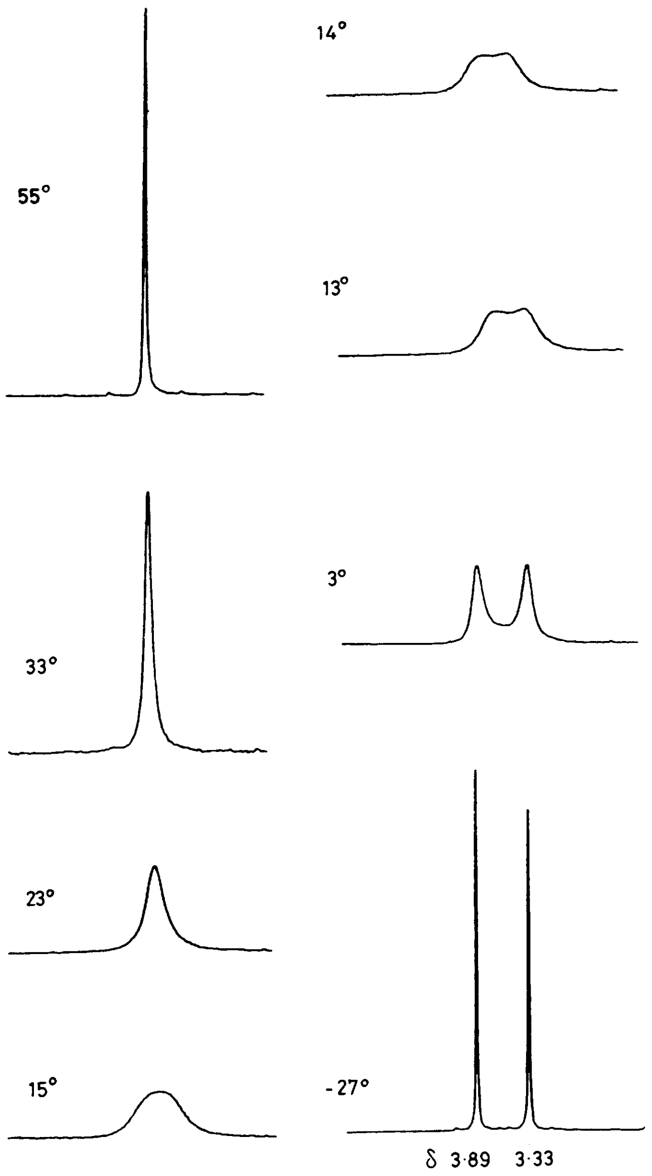


Figure 1. Evidence for rotamers of compound (4) in $CDCl_3$: *N*-methyl region of the n.m.r. spectrum (80 MHz) at various temperatures (°C)

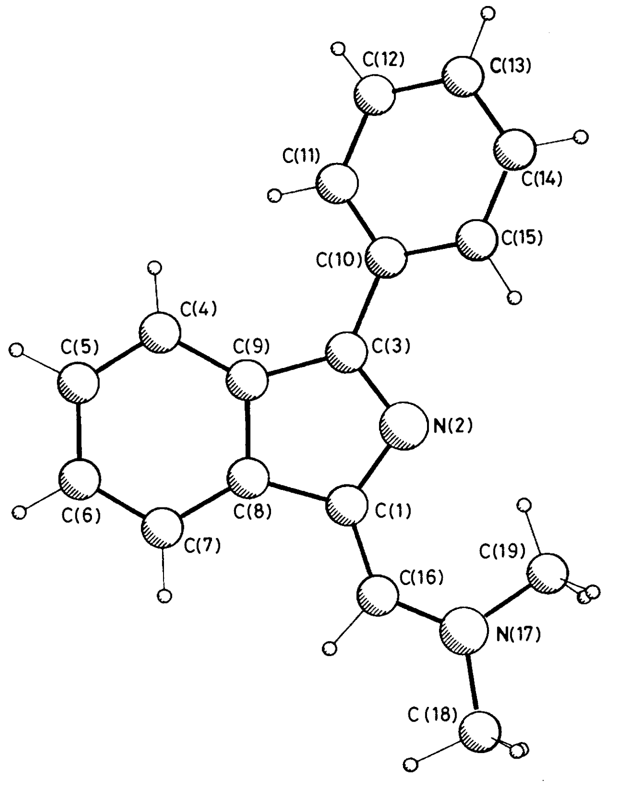


Figure 2. Molecular structure and crystallographic numbering scheme for 1-(*NN*-dimethylaminomethylidene)-3-phenylisoindole (4) (conformer a)

and H(11). Models indicate that in a planar arrangement the distance H(4)–H(11) would be *ca.* 1.6 Å, which is considerably less than twice the van der Waals radius of hydrogen (2 × 1.0 Å). Rotation about C(3)–C(10) increases the distance between these hydrogen atoms to a more comfortable value [2.12 Å and 2.21 Å in (4a and b), respectively]. Apart from minor differences about the N(17)–C(19) bonds there are no further significant differences between conformers (4a and b).

Carbon–nitrogen bond lengths in the heterocyclic ring accord with the double bond distribution shown in structure (4). In the carbocyclic ring of this isoindolenine system there exists a limited degree of bond alternation: the average bond lengths for C(5)–C(6), C(4)–C(5), and C(4)–C(9) are 1.39, 1.37, and 1.40 Å, respectively. In valence bond terms this may be attributed to a contribution from structure (6), already postulated to account for n.m.r. behaviour. In accord with this interpretation, C(1)–C(16) (1.38 Å) evidently has much single bond character, while, conversely, C(16)—N(17) (1.32 Å) possesses double bond character.*

1-Formyl-3-phenylisoindole.—Hydrolysis of the aminomethylidene derivative (4) with hot alkali gave 1-formyl-3-phenylisoindole (5) as bright yellow needles, $\lambda_{max.}$ (EtOH) 384 nm (ε 29 800). The carbonyl absorption in the infrared occurred at remarkably low frequency (Nujol; 1 630 cm^{-1}): this, and the failure of this aldehyde readily to form common carbonyl derivatives, is attributed to the importance of the polarised canonical structure (7). An analogous situation is found in pyrrole chemistry.[10] The effect of the electron-withdrawing substituent on the chemical properties of the isoindole system was evidenced in other ways. Compared with isoindole [11] or simple alkylisoindoles the compound was robust and easily manipulated; it underwent *N*-methylation with diazomethane; and it failed to undergo a Diels–Alder reaction with *N*-methylmaleimide in tetrahydrofuran.

The formylisoindole (5) was obtained from ethyl acetate–light petroleum as yellow monoclinic prisms suitable for *X*-ray diffraction studies. The resulting molecular structure is shown in Figure 4, together with the crystallographic numbering system. Bond distances and bond angles are given in Table 1 in such a way as to facilitate comparison with the corresponding data for compound (4): data on planarity are provided in the Supplementary Publication (see later). The unit cell and crystal packing are illustrated in Figure 5. The phenyl and isoindole units are individually planar, but are not coplanar. The interplanar angle (29.2°) is essentially the same as that observed with compound (4), but in the present case only one

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* Assuming 1.42 Å for a C–N bond involving trigonally hybridised atoms, and 1.27 Å for the C=N double bond.[9]

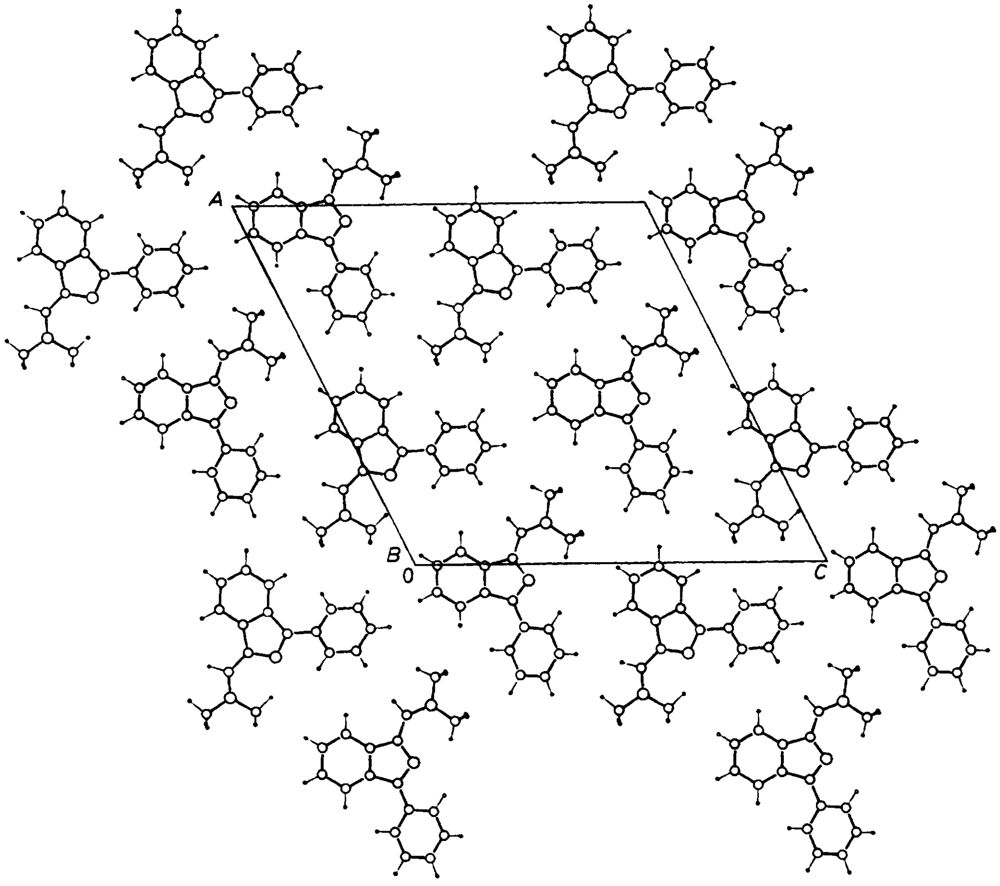
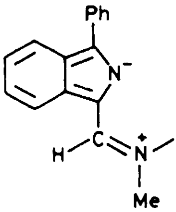
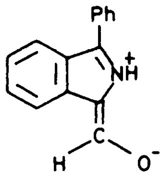


Figure 3. Packing diagram for (4)



(6)



(7)

conformer is observed: all molecules of (5) have the conformation earlier designated a in respect of compound (4). This conformation is illustrated in Figure 6, where the plane of the phenyl substituent is set perpendicular to the page to emphasise this point. The formyl group of (5) is coplanar with the isoindole skeleton. This is a second difference with respect to compound (4) since there the CH–NMe$_2$ group, although itself planar, is slightly tilted (*ca.* 8°) with respect to the plane of the fused rings.

In the five-membered ring the C(1)–N(2)–C(3) angle (112.2°) is somewhat larger than it is in 2-ethoxycarbonylpyrrole derivatives (*ca.* 109°).[12] The N(2)–C(3) bond (1.348 Å) is markedly shorter than is the C(1)–N(2) bond (1.376 Å). This accords with a significant contribution of the canonical structure (7).

In the carbocyclic ring of the fused system a marked degree of bond alternation is found. The bond lengths for C(5)–C(6), C(4)–C(5), and C(4)–C(9) are 1.409, 1.367, and 1.414 Å, respectively. This recalls the geometry of naphthalene

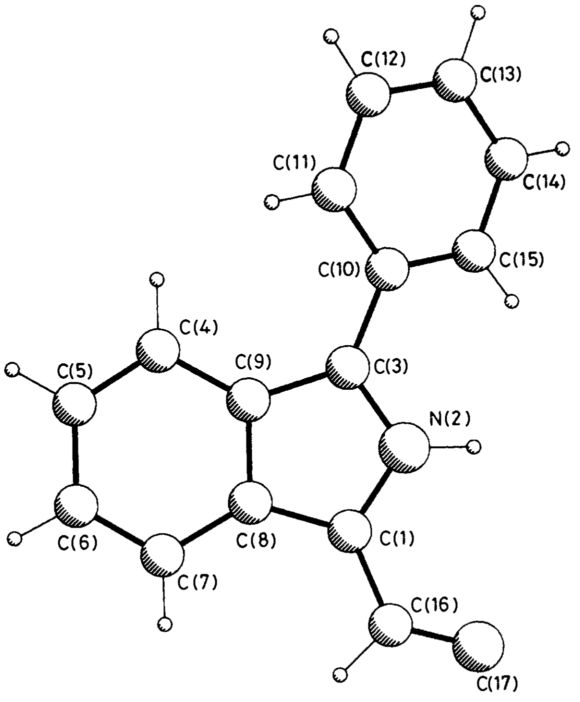


Figure 4. Molecular structure and crystallographic numbering scheme for 1-formyl-3-phenylisoindole (5)

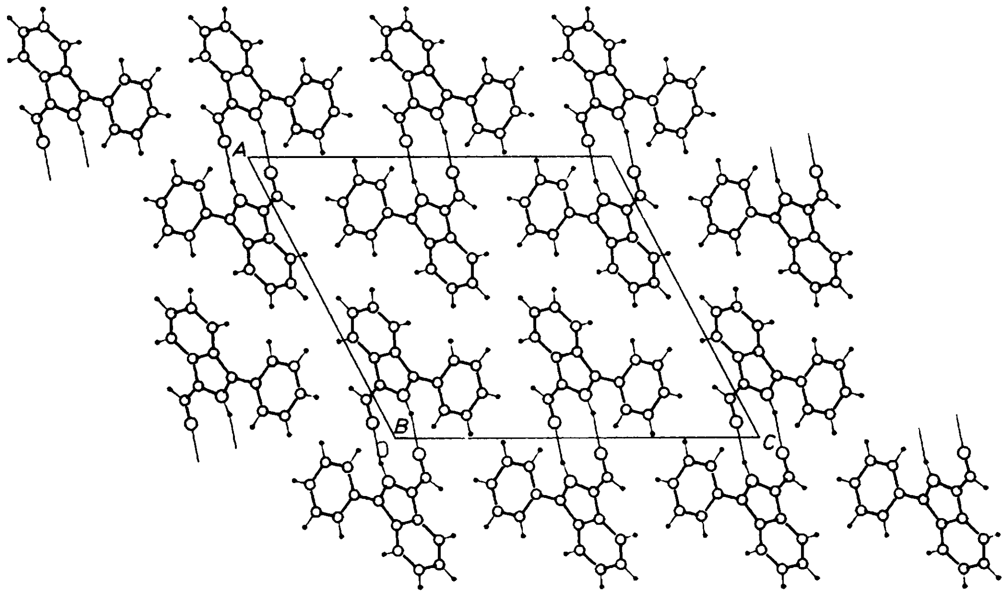


Figure 5. Packing diagram for (5)

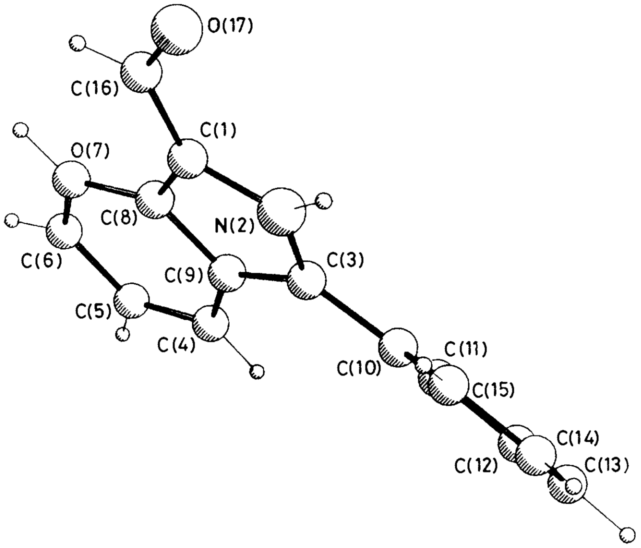


Figure 6. View of 1-formyl-3-phenylisoindole (5) with the phenyl substituent perpendicular to the paper

(corresponding bond lengths 1.411, 1.377, and 1.424 Å)[13] and provides good structural evidence for the aromaticity of the isoindole moiety as a unit.[2] As would be anticipated, the carbocyclic ring of the isoindolenine system in compound (2) does not show this alternation.[6] Thus we now have structural data on a simple isoindole which can be related to the aromaticity of the system: it remains of considerable interest to determine the geometry of a system devoid of a strongly polarising and/or stabilising group, since, in the present example, the formyl group (especially) clearly distorts the structure.

Intermolecular Interactions.—In compound (4) there are no short intermolecular contacts less than the normal van der Waals contact distances (Figure 3). On the other hand, in the formyl derivative (5) pairs of centrosymmetrically related molecules are linked by $N(2)^1-H^1 \cdots O(17)^2$ hydrogen bonds to form dimers (Figure 5) with the following geometry:

$N(2)^1 \cdots O(17)^2$	2.88 Å
$H(2)^1 \cdots O(17)^2$	1.95 Å
$N(2)^1-H(2)^1 \cdots O(17)^2$	167.2°

Symmetry code $1 = x, y, z$; $2 = 2 - x, 1 - y, 1 - z$

The higher density of crystals of the formyl compound (5) with respect to crystals of the aminomethylidene derivative (4) (1.34 and 1.22 g cm^{-3}, respectively) indicates that the molecules of (5) are arranged more compactly, and this is further substantiated by the occurrence of a number of intermolecular contacts of <3.5 Å in addition to the hydrogen bonding interaction (Table 2). In compound (4) only such one contact occurs [$C(3b)^1 \cdots C(18a)^2$ 3.474 Å. $1 = x,y,z$; $2 = \frac{1}{2} - x, +y, \frac{1}{2} - z$]. It seems to us that a likely explanation for the occurrence of only one conformation in the crystals of the formyl compound (5) is that the presence of two conformations [as found in (4)] would prevent such efficient molecular packing.

Experimental

Petroleum means that fraction of light petroleum with b.p. 40—60°.

1-(NN-*Dimethylaminomethylidene*)-3-*phenylisoindolenine*. [1-(NN-*Dimethylaminomethylidene*)-3-*phenyl*-1H-*isoindole*] (4).—Phosphoryl chloride (2.5 g) was added to dimethylformamide (20 ml) with stirring (−5 to −10°; argon). 1-Phenylisoindole[14] (1.5 g) in dimethylformamide (20 ml) was added dropwise, and the mixture was stirred at *ca.* −5° for 1 h, then at room temperature for 5 h. The mixture was poured into saturated sodium hydrogencarbonate (500 ml) with stirring, and the resulting brownish yellow solid was removed, washed with water, and dried (1.84 g). Column chromatography (silica gel, 35 g; eluted with petroleum–acetone 2 : 1) and crystallisation from ethyl acetate–light petroleum gave brilliant yellow green needles (1.31 g, 68%) of 1-(NN-*dimethylaminomethylidene*)-3-*phenylisoindolenine*, m.p. 152—153° (Found: C, 81.95; H, 6.5; N, 11.15%; M^+, 248.131. $C_{17}H_{16}N_2$

Table 1. Molecular geometry of 1-(*NN*-dimethylaminomethylidene)-3-phenylisoindole [two conformers (4a and b)] and of 1-formyl-3-phenylisoindole (5)

(a) Bond lengths (Å)

Bond	(4a)	(4b)	(5) *
C(1)–N(2)	1.386(3)	1.392(3)	1.376
N(2)–C(3)	1.329(2)	1.327(2)	1.348
C(3)–C(9)	1.450(4)	1.451(4)	1.428
C(4)–C(9)	1.405(3)	1.401(3)	1.414
C(8)–C(9)	1.403(3)	1.409(3)	1.420
C(4)–C(5)	1.378(5)	1.369(5)	1.367
C(5)–C(6)	1.394(4)	1.383(4)	1.409
C(6)–C(7)	1.377(3)	1.367(4)	1.365
C(7)–C(8)	1.402(4)	1.404(4)	1.414
C(1)–C(8)	1.442(3)	1.441(3)	1.412
C(3)–C(10)	1.475(3)	1.469(3)	1.462
C(10)–C(11)	1.396(2)	1.394(3)	1.399
C(11)–C(12)	1.390(4)	1.383(4)	1.380
C(12)–C(13)	1.383(5)	1.391(5)	1.381
C(13)–C(14)	1.374(3)	1.372(3)	1.383
C(14)–C(15)	1.382(4)	1.375(4)	1.381
C(10)–C(15)	1.390(4)	1.399(4)	1.395
C(1)–C(16)	1.386(3)	1.376(3)	1.412
C(16)–N(17)	1.323(3)	1.322(3)	
C(16)–O(17)			1.228
N(17)–C(18)	1.452(3)	1.452(3)	
N(17)–C(19)	1.453(4)	1.464(5)	

* All e.s.d.s are 0.002 Å.

(b) Bond angles (°)

Angle	(4a) †	(4b) †	(5) *
N(2)–C(1)–C(8)	109.8	109.5	106.6
N(2)–C(1)–C(16)	126.4	126.7	124.2
C(8)–C(1)–C(16)	123.7	123.8	129.2
C(1)–N(2)–C(3)	107.3	107.6	112.2
N(2)–C(3)–C(9)	111.4	111.4	106.9
N(2)–C(3)–C(10)	120.4	120.6	123.1
C(9)–C(3)–C(10)	128.2	127.9	129.9
C(5)–C(4)–C(9)	118.3	119.1	118.5
C(4)–C(5)–C(6)	121.0	121.2	121.6
C(5)–C(6)–C(7)	121.7(3)	121.6(3)	121.3
C(6)–C(7)–C(8)	118.2	118.4	118.6
C(7)–C(8)–C(9)	120.4	120.4	120.1
C(7)–C(8)–C(1)	133.7	133.6	132.4
C(1)–C(8)–C(9)	105.9	106.0	107.4
C(3)–C(9)–C(4)	133.8	135.0	133.1
C(3)–C(9)–C(8)	105.6	105.5	107.0
C(4)–C(9)–C(8)	120.5	119.4	119.9
C(3)–C(10)–C(11)	121.9	122.5	120.0
C(3)–C(10)–C(15)	120.1	119.6	121.8
C(11)–C(10)–C(15)	118.0	117.8	118.2
C(10)–C(11)–C(12)	120.7	120.9	120.7
C(11)–C(12)–C(13)	120.3	120.1	120.5
C(12)–C(13)–C(14)	119.3(3)	119.4(3)	119.4
C(13)–C(14)–C(15)	120.8(3)	120.8(3)	120.6
C(14)–C(15)–C(10)	120.9	120.9	120.6
C(1)–C(16)–N(17)	130.5(3)	131.0(3)	
C(1)–C(16)–O(17)			126.7
C(16)–N(17)–C(18)	120.5	120.9	
C(16)–N(17)–C(19)	123.7	122.9	
C(18)–N(17)–C(19)	115.7	116.1	

* All e.s.d.s 0.1°. † E.s.d.s 0.2° except where indicated otherwise.

Table 2. Intermolecular contacts <3.5 Å in (5) (excluding contacts involving hydrogen)

C(16)^1 · · · C(3)^2	3.31 Å
O(17)^1 · · · C(3)^2	3.41 Å
C(16)^1 · · · C(9)^2	3.48 Å
O(17)^1 · · · C(14)^3	3.30 Å
O(17)^1 · · · C(15)^4	3.37 Å
O(17)^1 · · · O(17)^4	3.29 Å
O(17)^1 · · · O(17)^4	3.29 Å

Symmetry code: $1 = x, y, z$; $2 = x, 1 + y, z$; $3 = 2 - x, -y, 1 - z$; $4 = 2 - x, 1 - y, 1 - z$.

requires C, 82.2; H, 6.5; N, 11.3%; *M*, 248.131), $\lambda_{max.}$ (EtOH) 241(i), 251 (31 400), 276 (10 500), 302(i), and 396 nm (38 400), $\lambda_{max.}$ (EtOH + HCl) 247(i), 253.5 (20 900), 276 (18 400), and 413 nm (39 000), $\nu_{max.}$ (KBr) 3 015, 1 615, 1 590, 1 550, 1 395, 1 360, 1 320, 1 135, 1 040, 920, 780, 755, 750, 695, and 680 cm^{-1}, δ(CDCl$_3$; 33°; see also Figure 1) 8.2—7.0 (m, 10 H aryl and olefinic H) and 3.57br (s, 6 H, NMe$_2$), *m/e* (79°) 248 (100%, *M*), 233 (36, *M*-Me), 219 (40), 207 (16), 206 (67), 193 (3), 165 (6), 128 (10), 124 (12), 116 (28), and 77 (10).

1-Formyl-3-phenylisoindole (5).—1-(*NN*-Dimethylaminomethylidene)-3-phenylisoindolenine (0.218 g), ethanol (20 ml), and sodium hydroxide (1.3 ml of a 4% aqueous solution) were refluxed under argon for 3.25 h. The solvent was removed under reduced pressure, the residue being dissolved in dichloromethane (30 ml) and washed with water (2 × 15 ml), the washings being back extracted with dichloromethane. The combined organic solutions were dried (minimum Na$_2$SO$_4$), and evaporated. The residue was triturated with ether (2 ml), the ether was removed (Pasteur pipette), and the resulting yellow solid was recrystallised twice from ethyl acetate to give bright yellow needles (93 mg, 48%) of *1-formyl-3-phenylisoindole*, m.p. 183—184° (Found: C, 81.25; H, 5.0; N, 6.25%; M^+, 221.084. $C_{15}H_{11}NO$ requires C, 81.45; H, 5.0; N, 6.35%; *M*, 221.084), $\lambda_{max.}$ (EtOH) 242 (15 500), 264 (23 200), and 384 nm (29 800) $\nu_{max.}$ (Nujol) 3 210, 1 630, 1 615, 1 290, 1 275, 1 230, and 745 cm^{-1}, δ([^{2}H$_6$]DMSO) 13.34br (s, NH, exchangeable), 9.96 (s, CHO), and 8.25—7.17 (m, aryl H), *m/e* (104°) 221 (100%, *M*), 192 (10), 165 (30, *M* − CHO − HCN), and 110(10).

1-Formyl-2-methyl-3-phenylisoindole.—1-Formyl-3-phenylisoindole (44 mg) in tetrahydrofuran (5 ml; N$_2$ flush) was treated with an excess of ethereal diazomethane for 6 h. The reaction was worked up in the usual way to give a greenish yellow oil, which was purified by t.l.c. (silica; petroleum–acetone 3 : 1). The lower yellow band proved to be starting material (9 mg). The upper yellow band was recovered to afford *1-formyl-2-methyl-3-phenylisoindole* (28 mg, 59%) as a greenish yellow oil (Found: C, 82.1; H, 6.35; N, 6.0%; M^+, 235.100. $C_{16}H_{13}NO$ requires C, 81.7; H, 5.6; N, 5.95%; *M*, 235.100), $\lambda_{max.}$ (EtOH) 243 (13 600), 265 (20 500), and 376 nm (27 200), $\nu_{max.}$ (liquid film) 3 060, 1 680 (br, imp?), 1 635, 1 615, 1 600, 1 455, 1 390, 1 340, and 835 cm^{-1}, δ(CDCl$_3$) 10.03 (s, CHO), 7.63—6.98 (m, aryl H), and 4.18 (s, NMe).

X-Ray Crystallography.—Crystallographic data and details of intensity data collection are given in Table 3. Fractional atomic co-ordinates are given in Table 4. For both substances the crystal system, space group, and preliminary unit cell dimensions were determined photographically. Accurate cell parameters were obtained by least squares refinement from the diffractometer setting angles of 25 high angle reflexions. *X*-Ray intensity data were collected on a Nonius CAD4 diffractometer using Ni-filtered Cu-K_α radiation and an ω–2θ scan technique. Details of the data collection procedure have been given previously.[15] Structure solution and refinement were carried out using the SHELX 76 program,[16] and the diagrams were drawn using the program PLUTO.[17] Tables of

Table 3. Crystal data

	$C_{17}H_{16}N_2$ (4)	$C_{15}H_{11}NO$ (5)
a (Å)	20.387(2)	14.956(1)
b (Å)	7.161(1)	4.878(1)
c (Å)	20.642(2)	16.951(1)
β (°)	116.07(1)	117.3(1)
U (Å^3)	2 707.1	1 098.5
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$ [a]	$P2_1/c$ [b]
M	248.3	221.2
Z	8	4
D_X (g cm^{-3})	1.22	1.34
$F(000)$	1 056	464
μ (Cu-K_a) (cm^{-1})	4.8	5.8
Scan parameters a, b (°)	0.80, 0.15	0.80, 0.15
Aperture parameters A, B (mm)	4.0, 0.0	4.0, 0.0
Reflexions measured	$hk \pm l$	$hk \pm l$
θ range	3.0—65.0°	3.0—70.0°
Approximate crystal dimensions (mm)	0.50 × 0.25 × 0.09	0.15 × 0.15 × 0.38
No. of reflexions measured	5 274	2 475
No. of unique reflexions with $F \geqslant 3\sigma(F)$	3 241	1 784
R ($\Sigma\Delta/\Delta F_0$)	0.051	0.034
R_W ($= \Sigma w\Delta/\Sigma wF_0$)	0.049	0.041
$w = [\sigma^2(F_0) + gF_0^2]^{-1}$ g	0.000 55	0.000 34
No. of least-squares parameters	471	198

[a] Determined unambiguously from reflexion conditions $h0l$ $h + l = 2n$, $0k0$ $k = 2n$. [b] Determined unambiguously from reflexion conditions $h0l$ $l = 2n$, $0k0$ $k = 2n$.

Table 4. Atom co-ordinates (× 10^4)

	x	*y*	*z*
(4)			
C(1a)	167(1)	2 660(3)	2 440(1)
N(2a)	−442(1)	2 980(2)	2 558(1)
C(3a)	−1 003(1)	3 280(3)	1 919(1)
C(4a)	−1 140(1)	3 505(3)	595(1)
C(5a)	−744(2)	3 351(4)	202(1)
C(6a)	−4(2)	2 898(4)	536(1)
C(7a)	363(1)	2 621(3)	1 270(1)
C(8a)	−31(1)	2 781(3)	1 679(1)
C(9a)	−780(1)	3 191(3)	1 342(1)
C(10a)	−1 731(1)	3 720(3)	1 867(1)
C(11a)	−2 371(1)	3 318(4)	1 249(1)
C(12a)	−3 048(1)	3 806(4)	1 206(1)
C(13a)	−3 098(1)	4 688(4)	1 778(2)
C(14a)	−2 472(1)	5 047(4)	2 395(2)
C(15a)	−1 795(1)	4 571(3)	2 442(1)
C(16a)	874(1)	2 377(3)	2 963(1)
N(17a)	1 141(1)	2 294(3)	3 674(1)
C(18a)	1 923(1)	2 173(4)	4 121(2)
C(19a)	693(2)	2 398(5)	4 059(1)
C(1b)	2 634(1)	1 983(3)	−88(1)
N(2b)	2 508(1)	2 179(2)	518(1)
C(3b)	3 151(1)	2 353(3)	1 087(1)
C(4b)	4 506(1)	2 449(3)	1 246(1)
C(5b)	4 908(1)	2 349(4)	862(2)
C(6b)	4 575(2)	2 107(4)	122(2)
C(7b)	3 834(1)	1 943(4)	−255(1)
C(8b)	3 409(1)	2 041(3)	126(1)
C(9b)	3 745(1)	2 278(3)	880(1)
C(10b)	3 196(1)	2 705(3)	1 806(1)
C(11b)	3 809(1)	2 232(3)	2 439(1)
C(12b)	3 842(2)	2 640(4)	3 107(1)
C(13b)	3 264(2)	3 556(4)	3 157(1)
C(14b)	2 653(2)	3 995(4)	2 537(1)
C(15b)	2 615(1)	3 581(3)	1 871(1)
C(16b)	2 115(1)	1 820(3)	−792(1)
N(17b)	1 397(1)	1 678(3)	−1 070(1)
C(18b)	952(2)	1 693(4)	−1 846(1)
C(19b)	999(2)	1 606(5)	−628(2)
(5)			
C(1)	8 095(1)	3 969(3)	4 657(1)
N(2)	8 463(1)	2 559(2)	4 165(1)
C(3)	7 798(1)	709(3)	3 618(1)
C(4)	6 023(1)	−655(3)	3 425(1)
C(5)	5 341(1)	−39(3)	3 725(1)
C(6)	5 524(1)	2 039(3)	4 358(1)
C(7)	6 400(1)	3 504(3)	4 708(1)
C(8)	7 125(1)	2 911(3)	4 415(1)
C(9)	6 930(1)	856(3)	3 763(1)
C(10)	8 014(1)	−1 113(3)	3 043(1)
C(11)	7 227(1)	−2 155(3)	2 266(1)
C(12)	7 414(1)	−4 003(4)	1 743(1)
C(13)	8 386(1)	−4 815(3)	1 969(1)
C(14)	9 175(1)	−3 734(3)	2 721(1)
C(15)	8 995(1)	−1 906(3)	3 254(1)
C(16)	8 607(1)	6 140(3)	5 242(1)
O(17)	9 443(1)	7 039(2)	5 410(1)

anisotropic thermal parameters, hydrogen atom co-ordinates, and observed and calculated structure factors for both structures have been deposited as Supplementary Publication No. SUP 23858 (28pp).*

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* For details, see Notice to Authors No. 7, *J. Chem. Soc., Perkin Trans. 2*, 1984. Issue 1.

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