Isoindole

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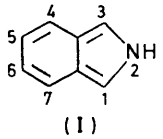
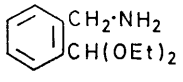
Isoindole has been isolated for the first time. It is obtained as a white solid at *ca.* −196° following pyrolysis of 2-(methoxycarbonyloxy)isoindoline in a silica tube at 500° and 0·01 mmHg. Chemical reactions and spectroscopic data are reported.

ALTHOUGH substituted, and especially *N*-substituted, isoindoles have become well known in recent years,[1] the parent compound (I) has remained elusive. Indole, in sharp contrast, has been known[2] since 1866. The earliest attempt to prepare isoindole † was described in 1928 by Fenton and Ingold,[4] who treated 2-tosylisoindoline with concentrated aqueous base: no isoindole was isolated, but a small amount of the hydrolysis product, isoindoline, was obtained. Bornstein and his colleagues[5] prepared the amine-acetal (II), but attempts to promote an acid-catalysed cyclisation gave red resins.



(I)



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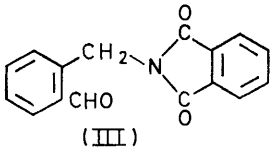
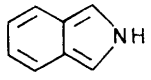


(III)

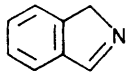
Polymers were also obtained by Veber and Lwowski[6] when they attempted to make isoindole by the hydrazinolysis of α-phthalimido-*o*-tolualdehyde (III). Isoindole has possibly been detected as an intermediate in the electrochemical reduction of 1,2-dihydrophthalazine.[7]

The only (partial) success in this long period of experimentation was recorded in a communication[8] by Kreher and Seubert, who trapped isoindole (generated in solution by base-promoted elimination from 2-benzyloxyisoindoline) as its Diels–Alder adducts with maleic anhydride and *N*-phenylmaleimide.

The non-appearance of isoindole stimulated theoretical calculations on this system. The numerical values predicted vary, but such studies[6,9,10] have been in general agreement that isoindole, although expected to have a delocalisation energy less than that of indole, should be aromatic. Moreover the isoindole tautomer (Ia) was predicted[6,10] to have a delocalisation energy greater (by *ca.* 30 kJ mol^{-1}) than that of the 1*H*-form (Ib).



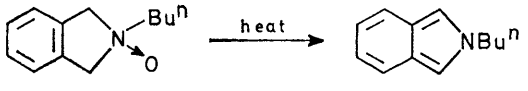
(Ia)



(Ib)

In preliminary experiments[11] the elimination of toluenesulphinate ion from 2-tosylisoindoline was re-examined using a strong base (KOBu^t) in an aprotic solvent (dimethyl sulphoxide).[8] Steam distillation in the dark under nitrogen gave a low yield of a yellowish solid which was recognised (mass spectrum; Ehrlich reaction) as crude isoindole, but which rapidly resinified and blackened. Although such wet methods are probably now capable of further development, it was thought desirable to adopt an approach which avoided solvents and slow purification stages. A pyrolytic method was therefore developed.

Our starting compound was 2-hydroxyisoindoline (VII). The preparation of this compound had been described by Thesing and his colleagues,[12] although the relevant experimental section is confused by the incorrect designation of the starting material for the preparation of (VII). We have confirmed that pyrolysis of 2-n-butylisoindoline 2-oxide (IV) gives the corresponding isoindole (V) in good yield, whereas pyrolysis of



(IV) (V)

2-(β-phenylethyl)isoindoline 2-oxide (VI) at 105° and 0·02 mmHg gives 2-hydroxyisoindoline (VII). The latter was contaminated with 2-(β-phenylethyl)iso-

† As in earlier papers[3] the use of the name isoindole in a general context does not imply the absence of the tautomeric 1*H*-isoindole.

[1] For a review, see J. D. White and M. E. Mann, *Adv. Heterocyclic Chem.*, 1969, **10**, 113.
[2] A. Baeyer, *Annalen*, 1866, **140**, 295.
[3] C. O. Bender, R. Bonnett, and R. G. Smith, *J.C.S. Perkin I*, 1972, 771 and references therein.
[4] G. W. Fenton and C. K. Ingold, *J. Chem. Soc.*, 1928, 3295.
[5] J. Bornstein, S. F. Bedell, P. E. Drummond, and C. L. Kosloski, *J. Amer. Chem. Soc.*, 1956, **78**, 83.
[6] D. F. Veber and W. Lwowski, *J. Amer. Chem. Soc.*, 1964, **86**, 4152.
[7] H. Lund and E. T. Jensen, *Acta Chem. Scand.*, 1970, **24**, 1867.
[8] R. Kreher and J. Seubert, *Z. Naturforsch.*, 1965, **20b**, 75.
[9] M. J. S. Dewar, *Trans. Faraday Soc.*, 1946, **42**, 764; H. C. Longuet-Higgins and C. A. Coulson, *ibid.*, 1947, **43**, 87; J. S. Dewar, A. J. Harget, N. Trinajstić, and S. D. Worley, *Tetrahedron*, 1970, **26**, 4505; B. A. Hess, L. J. Schaad, and C. W. Holyoke, *ibid.*, 1972, **28**, 3657.
[10] J. Kopecký, J. E. Shields, and J. Bornstein, *Tetrahedron Letters*, 1967, 3669.
[11] C. O. Bender, Ph.D. Thesis, London, 1967; R. G. Smith, Ph.D. Thesis, London, 1970.
[12] J. Thesing, W. Schäfer, and D. Melchior, *Annalen*, 1964, **671**, 119.

indoline, which was removed by partitioning the product between petroleum and water.

Treatment of (VII) with methyl *p*-nitrophenylcarbonate gave 2-methoxycarbonyloxyisoindoline (VIII). This was expected to decompose thermally as shown [(VIII) ⟶ (I), arrows] in the Scheme with the formation of isoindole and the volatile innocuous by-products carbon dioxide and methanol. To test this proposal a trapping reaction with *N*-phenylmaleimide was carried out in boiling xylene. This gave the *exo*-adduct (X), which had been reported earlier by Kreher and Seubert.[8] The latter authors did not assign its stereochemistry, but it is now assigned the *exo*-configuration since no coupling is observed between the bridgehead protons and the vicinal (CH·CO) protons [structure (X), τ values].[13] No *endo*-isomer was detected in this preparation.

In order to obtain isoindole in substance the carbonate (VIII) was pyrolysed in an unpacked silica tube at *ca.* 500° and 0·01 mmHg (Figure).[14] Isoindole collected as a white deposit, in some cases obviously crystalline (needles), in a shallow depression at −196°. The properties of this material which support the conclusion that it is isoindole are as follows.

(i) The solid readily decomposed at room temperature in air becoming first yellow and then darkening and resinifying. Solutions appeared to be more stable, especially under nitrogen.

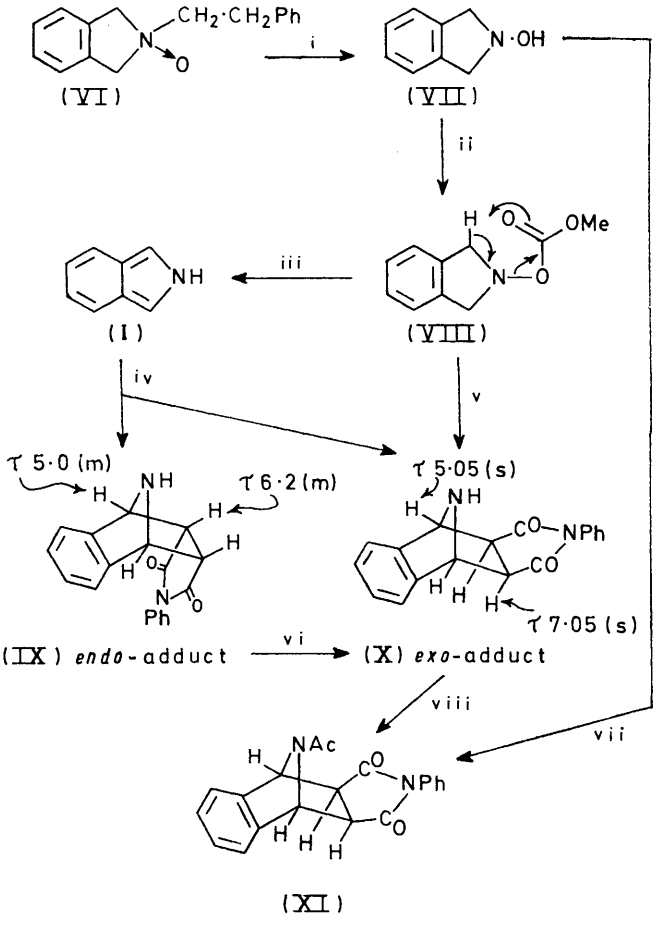
(ii) It gave a positive reaction with Ehrlich's reagent (a red-purple colour, slowly turning blue; 2-methylisoindole is reported [15] to behave similarly), and an immediate deep red colour with ethanolic 1,3,5-trinitrobenzene.

(iii) The i.r. spectrum (solid film, collected on NaCl target at −196° but measured at room temperature; some decomposition expected) showed no carbonyl absorption (*i.e.* none of the precursor had survived). The strongest band, at 740 cm^{-1}, is ascribed to aromatic C–H out-of-plane deformation modes [*cf.* (V), 758 cm^{-1}].

(iv) The u.v. spectrum (hexane, *in vacuo*) showed strong end absorption (250—230 nm) and, in the 320 nm region, an envelope showing clear fine structure [16] (vibrational spacing *ca.* 700 cm^{-1}, *cf.* the strong i.r. band in this region). The most prominent maxima occurred at 306·5, 312·5, 320, 326·5, and 335 nm. This spectrum strikingly resembles those of 2-n-butylisoindole (V) and isobenzofuran.[17] Recent theoretical studies [18] predict that isoindole should show absorption at 273 and 315 nm, in fair agreement with experiment. A strong band probably lies a little below 230 nm, however.

(v) The hexane solution from (iv) showed a greenish blue fluorescence in u.v. light (medium-pressure mercury lamp).

(vi) Although it indicated the presence of isoindole (and the expected by-product, methanol) the n.m.r. spectrum in chloroform (60 MHz; −46°) was broad and not sufficiently helpful for structural work. However, in [$^2H_6$]acetone (100 MHz; −40°) the spectrum was well resolved, and, in the aromatic region, was broadly similar to that of 2-n-butylisoindole (V). The multiplets centred at τ 3·2 and 2·5 are interpreted as an AA′BB′ system, and are assigned as shown on the basis of the calculated π-electron densities in the benzenoid ring.[18] The singlet at τ 2·72 is assigned to the 1(3)-protons: this singlet is rather broad ($W_{\frac{1}{2}}$ 3·5 Hz) and this is ascribed



SCHEME Preparation and reactions of isoindole

i, 105°, vac.; ii, $MeO{\cdot}CO_2{\cdot}C_6H_4{\cdot}NO_2$-*p*; iii, 500°, vac.; iv, *N*-phenylmaleimide, CH_2Cl_2, <20°; v, *N*-phenylmaleimide, xylene, heat; vi, xylene, heat; vii, Ac_2O, *N*-phenylmaleimide, CH_2Cl_2; viii, AcCl–K_2CO_3,CH_2Cl_2

to coupling with the 2-proton (which is not otherwise detected) since the signal sharpens considerably ($W_{\frac{1}{2}}$ 2·0 Hz) [16] on deuterium exchange at C-2.

(vii) The accurate mass of the molecular ion accorded with the formula C_8H_7N. This ion was the base peak, but important fragments were observed at *m/e* 90 $(M - HCN)^+$, 89 $(M - HCN - H)^+$, and 63 $(C_5H_3)^+$.

[13] P. Laszlo and P. von R. Schleyer, *J. Amer. Chem. Soc.*, 1964, **86**, 1171; R. Kreher and J. Seubert, *Angew. Chem. Internat. Edn.*, 1966, **5**, 967.

[14] Preliminary communication, R. Bonnett and R. F. C. Brown, *J.C.S. Chem. Comm.*, 1972, 393.

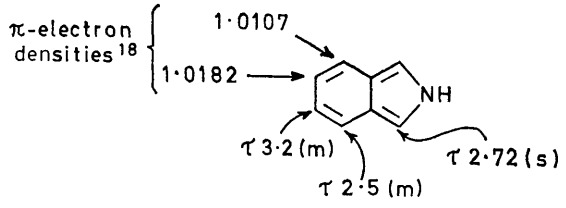
[15] G. Wittig, H. Tenhaeff, W. Schoch, and G. Koenig, *Annalen*, 1951, **572**, 1.

[16] For displayed spectrum see ref. 14.

[17] R. N. Warrener, *J. Amer. Chem. Soc.*, 1971, **93**, 2346; D. Wege, *Tetrahedron Letters*, 1971, 2337.

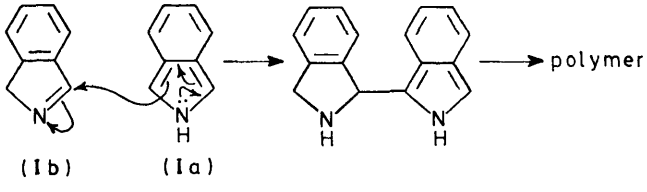
[18] L. Klasinc, E. Pop, N. Trinajstić, and J. V. Knop, *Tetrahedron*, 1972, **28**, 3465.

(viii) Treatment of isoindole with *N*-phenylmaleimide in the cold (see Experimental section) gave a 2 : 3 mixture of the adducts (IX) and (X) in good yield. The *exo*-adduct (X) could be isolated by recrystallisation of the

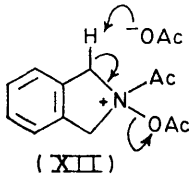


mixture from ethanol. The *endo*-adduct (IX) was not isolated, but the n.m.r. spectrum of the mixture clearly indicated its presence and its stereochemistry, since the bridgehead and CH·CO protons were now coupled, and the latter protons were no longer shielded by the benzenoid ring of the isoindoline [structures (IX) and (X), τ values]. Moreover, when the mixture of (IX) and (X) was refluxed in xylene, the n.m.r. spectrum of the total product showed that the expected transformation [(IX) ⟶ (X)] into the thermodynamically more stable isomer had occurred.

The above evidence is regarded as convincing proof that isoindole has at last been made.[19] We are now examining its chemistry further. That it is aromatic, as predicted,[9] is indicated by the chemical shift values and supported by the strong molecular ion in the mass spectrum. The available evidence (u.v., n.m.r.) indicates that the isoindole form (Ia) is important, as expected:[6,10] but the presence of a proportion of 1*H*-tautomer (Ib) cannot be excluded. An n.m.r. study with solvent variation[20] should provide an answer here. Isoindole readily autoxidises; but even under nitrogen at room temperature it resinifies, and this may be a consequence of interaction between tautomeric forms as illustrated. Such a process is known with pyrrole[21] but



requires an acidic medium to generate the (protonated) 2*H*-pyrrole system formally equivalent to (Ib). Various attempts were made to seek other routes to isoindole from the hydroxylamine (VII). In the mass spectrometer loss of water to give the isoindole molecular ion (accurate mass measurement) was an important fragmentation (metastable peak observed). Pyrolysis of (VII) at 600° and 0·05 mmHg on the apparatus already described was not a satisfactory preparative method, however. The discoloured deposit which formed contained isoindole [isolated as the adduct (X)] together with other components including the starting material. However, treatment of (VII) with acetic anhydride, followed by *N*-phenylmaleimide, gave the adduct (XI), which was also formed on acetylation of (X). We envisage that the formation of 2-acetylisoindole from (VII) proceeds *via* an intermediate species such as (XII).



EXPERIMENTAL

Spectroscopic measurements were made as previously indicated.[20] Values of ν (cm⁻¹) and λ (nm; ε in parentheses; i = inflection) refer to maxima unless otherwise stated. Petroleum means light petroleum of b.p. 60—80°.

2-*n-Butylisoindole* (V).—2-n-Butylisoindoline 2-oxide[12] (0·175 g) was pyrolysed in a short-path distillation apparatus at 117° and 0·2 mmHg. The colourless oil (0·143 g) which collected in the cooled bulb darkened on exposure to light and air. It was shown to be 98% 2-n-butylisoindole by g.l.c. (5 ft × 1/8 in 15% polypropylene glycol LB-550-X on Chromosorb W; Perkin-Elmer F11 at 170°; retention time 22·4 min). Apparent λ (EtOH) 224 (72,600), 260i (2200), 266 (2100), 271 (2000), 277 (2100), 281·5i (1400), 313i (3750), 326·5 (4700), and 340i (3600); ν (film) 3120w, 3060, 3030, 2960, 2935, 2875, 1660w, 1550w, 1366, 1330, 1158, 1140, and 758s; τ ($CDCl_3$; observed by Dr. P. F. V. Williams) multiplets centred at 2·52 and 3·14 (AA'BB', C_6H_4), 3·07 (s, C-1 and C-3 H), 6·00 (t, *J* 7 Hz, N·CH_2), and 7·90—9·14 (m, CH_2·CH_2·CH_3), M^+ 173·120 (Calc. for $C_{12}H_{15}N$: *M*, 173·120), *m/e* 174 (10%), 173 (70), 172 (11), 132 (11), 131 (100), 130 (21), and 117 (22).

2-*Hydroxyisoindoline* (VII).—2-(β-Phenylethyl)isoindoline 2-oxide[12] (4 × 0·50 g) was heated (10 h) at 105° and 0·02 mmHg, the products being collected in a horizontal water-cooled condenser. Recrystallisation of the sublimate from petroleum gave a grey crystalline solid, m.p. 70—84°, which contained (t.l.c.) 2-(β-phenylethyl)isoindoline. Distribution of the product between petroleum (50 ml) and water (200 ml) and evaporation of the aqueous layer gave 2-*hydroxyisoindoline*, which was crystallised from petroleum–ether to give prisms (0·70 g, 62%), m.p. 99° (lit.,[12] 74—76°) (Found: C, 70·9; H, 6·85; N, 10·3. C_8H_9NO requires C, 71·1; H, 6·7; N, 10·35%), ν (Nujol) 3200; τ ($CDCl_3$) 2·70br (exchangeable, OH), 2·76 (s, ArH), and 5·66 (s, CH_2), *m/e* 135 (93%), 134 (100), 117 (16), 116 (34), 90 (34), 89 (22), 88 (24), and 63 (16), *m** 101·5 (135 ⟶ 117).

2-*Methoxycarbonyloxyisoindoline* (VIII).—A solution of methyl *p*-nitrophenyl carbonate[22] (0·197 g) and 2-hydroxyisoindoline (0·137 g) in ether (7 ml) was kept (room temp., N_2, dark) for 48 h. Petroleum (b.p. 40—60°; 40

[19] Since our preliminary communication two confirmatory reports on the preparation of isoindole have appeared: J. Bornstein, D. E. Remy, and J. E. Shields, *J.C.S. Chem. Comm.*, 1972, 1149; G. M. Priestley and R. N. Warrener, *Tetrahedron Letters*, 1972, 4295.

[20] C. O. Bender, R. Bonnett, and R. G. Smith, *J. Chem. Soc.* (*C*), 1970, 1251.

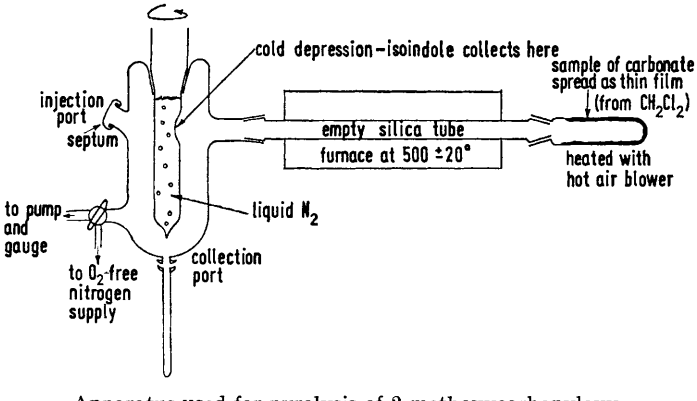
[21] H. A. Potts and G. F. Smith, *J. Chem. Soc.*, 1957, 4018.

[22] J. M. A. Hoeflake, *Rec. Trav. chim.*, 1916, **36**, 24.

ml) was added and the solution was washed repeatedly (8 × 3 ml) with a solution of potassium carbonate (5 g) in saturated aqueous sodium hydrogen carbonate (25 ml) to remove *p*-nitrophenol. The pink organic layer was dried (Na_2SO_4) and filtered through silica gel (0·03 g). Evaporation gave a light brown solid (0·179 g) which was crystallised from petroleum (b.p. 40—60°) to give light brown leaflets (0·143 g, 73%), m.p. 70—72°. Sublimation (50° at 0·01 mmHg) gave crystals of *2-methoxycarbonyloxyisoindoline*, m.p. 74—75° (Found: C, 62·3; H, 5·85; N, 7·25. $C_{10}H_{11}NO_3$ requires C, 62·15; H, 5·75; N, 7·25%); ν (Nujol) 1770; τ ($CDCl_3$) 2·77 (s, ArH), 5·52 (s, C-1 and C-3 H_2), and 6·20 (s, OMe); *m/e* (no molecular ion) 117 (100%), 90 (48), 89 (26), 44 (59), 32 (17), and 31 (22).

Thermal Decomposition of 2-Methoxycarbonyloxyisoindoline in Xylene. Trapping of Isoindole as a Diels–Alder Adduct.—2-Methoxycarbonyloxyisoindoline (0·045 g) and *N*-phenylmaleimide (0·2 g) were refluxed in xylene (3 ml; 45 min; N_2). The cold solution was diluted with ether (20 ml) and the basic product was extracted with N-sulphuric acid (3 × 5 ml). The acid extract was basified (NH_3) and the crystalline precipitate was extracted with dichloromethane. Removal of solvent gave the crude adduct (0·053 g, 78%), which was recrystallised from ethanol to give the *exo*-adduct (X) (0·046 g, 68%) as prisms, m.p. 208—209° (decomp.) [lit.,[8] 208—209° (decomp.)] (Found: C, 74·35; H, 4·7; N, 9·8. Calc. for $C_{18}H_{14}N_2O_2$: C, 74·45; H, 4·85; N, 9·65%); ν (Nujol) 3290—3300, 1773, and 1705; τ ($CDCl_3$) 2·4—3·0 (m, ArH), 5·05 (s, bridgehead H), 7·05 (s, CH·CO), and 7·55br (NH, exchangeable). The n.m.r. spectra of the crude and purified adducts were indistinguishable.

Isoindole (I).—(*a*) 2-Methoxycarbonyloxyisoindoline was pyrolysed, after volatilisation (50—70° and *ca.* 0·01 mmHg) from a Pyrex B24 test tube, in an empty silica tube (i.d. 20 mm) heated over 30 cm to 500 ± 20° (electric furnace). The products were collected in a depression on a cold finger (liquid nitrogen) 10 cm from the end of the heated zone.



Apparatus used for pyrolysis of 2-methoxycarbonyloxyisoindoline (VIII)

Pressures of 0·005—0·01 mmHg were observed at the pump (Figure). To remove the isoindole the apparatus was back-flushed with oxygen-free nitrogen and a solvent was injected through the septum into the depression, where it solidified. As the liquid nitrogen evaporated the solvent melted, carrying the isoindole through a collection port at the bottom of the trap and into a receiver (*e.g.* an n.m.r. tube) cooled with liquid nitrogen.

(*b*) 2-Methoxycarbonyloxyisoindoline (44 mg) was pyrolysed. Colourless needles formed in the cold depression over 30 min, and were transferred to an n.m.r. tube, with [$^2H_6$]acetone (0·6 ml) as described in (*a*); τ (100 MHz; −40°) 2·5 and 3·2 [m, m, protons at C-5(6) and C-4(7), respectively] and 2·72 (s, =C*H*·NH, $W_{\frac{1}{2}}$ 3·5 Hz). The spectrum did not change significantly on warming the solution to 0°; addition of a trace of D_2O–Et_3N caused sharpening of the singlet at τ 2·72 ($W_{\frac{1}{2}}$ 2 Hz).

(*c*) The pyrolysate from (VIII) (1·5 mg) was washed with hexane (5 ml; spectroscopic) through a syringe needle into a modified Thunberg silica cell (1 cm) provided with a septum. Further additions of hexane and withdrawals of solution were similarly made through the septum; λ <230vs, 263·5, 268·5, 275, 286·5, 294i, 300, 306·5, 312·5, 320, 326·5, and 335.[16] On exposure to air the colourless hexane solution slowly became pink and then blue (λ 540—570) and the fine structure in the u.v. disappeared.

(*d*) The pyrolysate from (VIII) (6 mg) was collected directly on a sodium chloride plate (15 × 15 mm) held with metal foil against a flat surface of the cold finger. The colourless film became yellow at room temperature; ν (sublimed film, within *ca.* 4 min of exposure to air) 3350, 3110, 3070, 3030, 2900, 2850, 1460, 1360, 1230, 1110, 1065, 1020, 985, and 740.

(*e*) The mass spectrum of a sample of isoindole was taken with as little delay as possible: M^+ 117·0576 (C_8H_7N requires *M*, 117·0578), *m/e* 118 (26%), 117 (100), 90 (74), 89 (56), 63 (38), 44 (100, CO_2), and 32 (100, MeOH).

(*f*) The pyrolysate from (VIII) (22 mg) was collected in the depression which had already been coated at room temperature with *N*-phenylmaleimide (100 mg). Dichloromethane (3 ml) was injected under nitrogen onto the cold target area, which was then allowed to warm to room temperature. The reaction mixture was removed at the collection port and worked up as before to give a yellow crystalline basic fraction (25 mg, *ca.* 75%). T.l.c. [EtOAc–petroleum (1 : 1); silica gel] revealed, besides a trace of *N*-phenylmaleimide, the *exo*-adduct (X), together with another component of slightly lower R_F. This basic mixture of *exo*- and *endo*-isomers [(X) + (IX)] was examined as follows.

(i) Recrystallisation of a part from ethanol gave the *exo*-adduct (X), m.p. 204—205° (decomp.), identical (i.r.) with the sample prepared before.

(ii) The n.m.r. spectrum ($CDCl_3$) showed signals due to the *exo*-adduct, and a second set of signals assigned to the *endo*-isomer (*ca.* 3 : 2, respectively). The *endo*-isomer (IX) showed τ 3·55 (m, 2ArH shielded by C=O), 5·00 (m, bridgehead H), and 6·20 (m, CHCO).

(iii) The adduct mixture was heated in refluxing xylene (N_2; 45 min). The total basic product was examined by n.m.r., which now revealed only the *exo*-adduct.

Diels–Alder Adduct (XI) *of 2-Acetylisoindole and N-Phenylmaleimide.*—(a) *From 2-hydroxyisoindoline.* Acetic anhydride (0·5 g) was added in one portion to a solution of 2-hydroxyisoindoline (0·203 g) in dichloromethane (1·5 ml; −10°; N_2). After 3 min *N*-phenylmaleimide (0·5 g) in dichloromethane (*ca.* 1 ml) was added (hypodermic needle, septum). After 2 h at <0° ethanol (1 ml) was added, and the solvent was evaporated off under reduced pressure. The residue in dichloromethane (5 ml) was washed (aq. $NaHCO_3$) and the dried solution was chromatographed [silica gel (12 g)]. Elution with dichloromethane (150 ml) removed *N*-phenylmaleimide (0·276 g). Subsequent elution

with dichloromethane–ethanol (9 : 1) gave a red gum which was rechromatographed on silica gel (35 g). The fraction eluted with ethyl acetate–petroleum (3 : 2; 400 ml) gave a crystalline product, which was recrystallised from ethanol to give needles (0·136 g, 27%) of the *exo*-Diels–Alder adduct (XI) of 2-acetylisoindole and *N*-phenylmaleimide, m.p. 230° (lit.,[8] 223—224°), M^+ 332. (Calc. for $C_{20}H_{16}N_2O_3$: *M*, 332); ν (CH_2Cl_2) 1780, 1710, and 1655; τ ($CDCl_3$; 35°) 2·5—2·9 (m, ArH), 4·0—4·6vbr (non-equivalent bridgehead H), 6·92 (s, CH·CO), and 8·05 (s, NAc), τ ($CD_3{\cdot}NO_2$; 35°) similar; τ ($CD_3{\cdot}NO_2$; −20°) 2·3—2·9 (m, ArH) 4·06, 4·23 (both s, bridgehead H), 6·73 (ABq, *J* 6·8 Hz, CH·CO), and 7·99 (s, NAc).

(b) *From the Adduct* (X). Acetylation of (X) with acetyl chloride–potassium carbonate in dichloromethane gave the *N*-acetyl derivative, m.p. 228—229°, identical (mixed m.p.) with the sample prepared before.

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