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NEW ROUTES TO CONDENSED POLYNUCLEAR COMPOUNDS—IX[1]

SYNTHESIS OF 2,10-, 3,10- AND 4,10-DIAZAPHENANTHRENE[2]

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Abstract—Synthesis of 2,10-, 3,10-, 4,10-diazaphenanthrene and some of their derivatives has been achieved through benzyne cyclisation of anils and reduced anils derived from *o*-chlorobenzaldehyde and the appropriate aminopyridines.

The present work was undertaken to see whether in the newly developed method for constructing polynuclear hetrocycles,[3–5] the benzyne attack on the pyridine ring proceeds satisfactorily. If successful this could serve a two-fold purpose; (i) synthesis of quasisteroidal nitrogen compounds;[6,7] (ii) formation of certain diazaphenanthrenes,[8] the basic systems of which, in spite of their simplicity and pharmacological potential,[9] still remain unsynthesised.

Synthesis of 3,10-diazaphenanthrene (**5**) was investigated first because the involved ring closure could lead only to one product (Fig 1). Condensation[10] of one mole of *o*-chlorobenzaldehyde (**1**) and two moles of 4-aminopyridine (**2**) afforded 4,4'[(2-chlorobenzylidene)diimino] dipyridine (**3**) in excellent yield. Use of equimolar proportions of the reactants gave the same compound as the major product. The desired mono condensation product **4** could be secured only by heating **3** under reduced pressure and distilling off the liberated 4-aminopyridine. The resulting unstable oil on reaction with excess amide ions in liquid ammonia afforded pure 3,10-diazaphenanthrene (**5**) in 20% yield. It was observed that the yields in the benzyne reactions were often better if the anils were reduced prior to cyclisation.[7] Cyclisation of the chlorobenzylamine **6**, secured by sodium borohydride reduction of **3**, proceeded in a good yield (40%). The resulting dihydrodiazaphenanthrene **7** could be dehydrogenated, quantitatively, to **5** by stirring with active manganese dioxide[11] suspended in chloroform.

Direct cyclisation of **3** was then attempted. It was hoped that this material would cyclise through the dianion **8** and subsequently lose a 4-aminopyridine moiety, in a manner parallel to the loss of amide ion postulated for anil cyclisation.[4] The dianion **8** has two equivalent sites for benzyne attack, and the cyclisation should have a better chance against competing reactions like external amination. Indeed, the reaction of **3** with potassium amide gave **5** in excellent yield and 4-aminopyridine could be isolated as a by-product. Thus, not only a more convenient synthesis of 3,10-diazaphenanthrene was achieved but also additional evidence, though indirect, for the proposed mechanism of benzyne cyclisation of anils was adduced. The present work seems to be the first synthesis of the parent 3,10-diazaphenanthrene ring system. Earlier, only a few derivatives of polyhydro-3,10-diazaphenanthrene had been prepared by indirect routes.[12–14] A survey of the literature does not report any synthesis of the later discussed 2,10- and 4,10-diazaphenanthrenes.*

In case of the derivatives (**10** and **11**) from 2-aminopyridine (**9**) attack by benzyne can take place on the ring carbon or nitrogen, both these positions being activated (Fig 2). Treatment of **10** with potassium amide in liquid ammonia, gave a complex mixture from which only 2-aminopyridine could be characterised. A similar reaction of the reduced anil **11**, prepared from **10** by sodium borohydride reduction or by reaction of 2-aminopyridine and *o*-chlorobenzaldehyde in formic acid,[20] also led to a complex tarry mixture. Although, two high melting materials were isolated on extensive work up, none of these could be crystallised or purified for characterisation.

Depending upon the orientation of the cyclisation (Fig 3), the anil (**13**) from 3-aminopyridine (**12**) can lead to a 4,10-(**14**) or a 2,10-diazaphenanthrene (**15**). Condensation of 3-aminopyridine[21] with *o*-chlorobenzaldehyde easily afforded this anil (**13**) which was subjected to benzyne cyclisation in liquid ammonia. The product gave, on chroma-

*In this context an error in *Chemical Abstracts* may be pointed out. The refs[15–18] indexed under benzo(c)(1,7)-, (c)(1,6)- and (c)(1,5)naphthyridine—alternate names for the above systems—actually concern phenanthrolines. A similar mistake in connection with benzo(c)(2,7)naphthyridine (2,9-diazaphenanthrene) has also been pointed out.[19a]

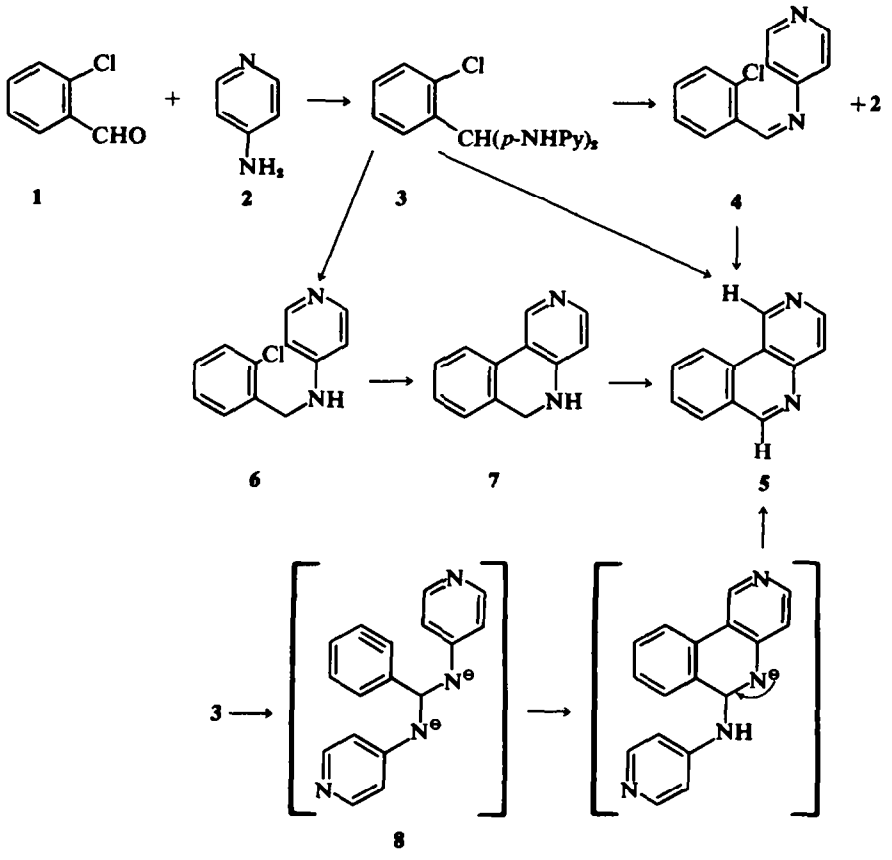

Fig 1.

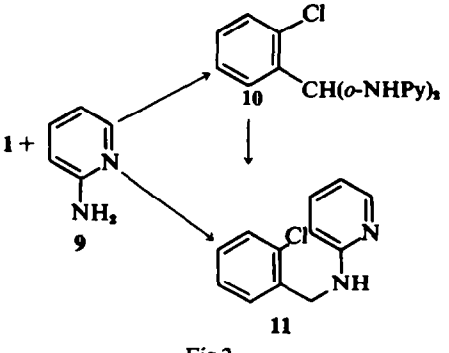

Fig 2.

tography on alumina, two solids: **A**, eluting first (17%), m.p. 99–101°; **B**, eluting second (50%), m.p. 102–104°.

In an alternate approach the reduced anil **16**, obtained either directly from 3-aminopyridine or from the anil **13**, was treated with potassium amide in liquid ammonia. The resulting mixture of bases was crystallised from acetone containing light petroleum to afford a dihydro-diazaphenanthrene (C), m.p. 130–131°, in 66% yield. Chromatography, on alumina, of the residue from the mother liquor gave a second isomer (D), m.p. 89–90°, in 9% yield. The dihydro-diazaphenanthrenes C and D upon manganese dioxide treatment gave **B** and **A** respectively.

Structural assignment of the diazaphenanthrenes (**A** and **B**) has been carried out spectroscopically, by making use of the consistent perturbation effects observed in the NMR spectra of aza-aromatic compounds.[22] Only the lowfield signals (beyond δ8) are considered, since little information can be gleaned from other areas due to extensive overlapping. The NMR spectrum of compound **A** in deuterochloroform shows only one clearly separated signal, a quartet centred around δ8·43 (J = 8 Hz, J = 2 Hz). From its shape and the magnitude of the coupling constants it may be ascribed to a para proton forming an AMX system with protons meta and ortho to a ring nitrogen.[22] Out of all the protons in structures **14** and **15** only H-1 of **14** meets this requirement. Assignment of 4,10-diazaphenanthrene (**14**) structure to compound **A** was confirmed by recording its spectrum in C_6D_6. The spectrum is more clearly resolved and the expected[22] differential solvent effects were observed especially as a

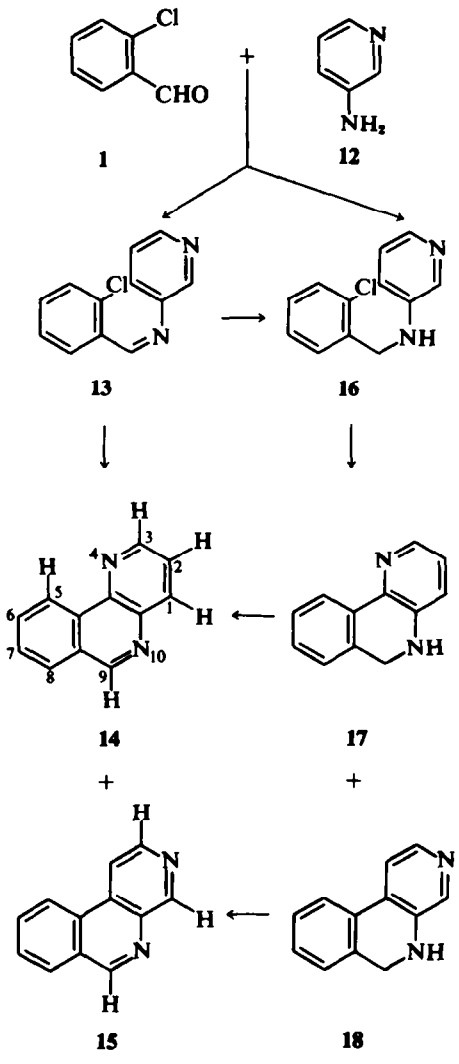

Fig 3.

large downfield shift of the hindered proton (H-5) facing nitrogen. The signal from the H-3 proton could now be seen as an AMX quartet (major J = 4·5 Hz) centred at δ8·82 and the signals from H-5 and H-9 still as an overlap in the δ9·3 area.

The NMR spectrum of compound **B** in $CDCl_3$ was not well resolved but in C_6D_6 the signals corresponding to H-1 (δ9·87, broadened singlet), H-9 (δ9·0, singlet) and H-3 (δ8·7, J = 4·5 Hz, broadened doublet) of structure **15** could be easily delineated. The exceptionally lowfield position of the singlet from H-1 resulted from its being ortho to one nitrogen and peri to another.

The NMR spectrum ($CDCl_3$) of the third isomeric diazaphenanthrene **5**, synthesised in an unambiguous manner, showed besides the δ9·4 signal for H-9, a sharp singlet at δ9·92 arising from H-4. The extreme lowfield position of the H-4 signal is readily understandable in terms of its hindered location and its ortho relationship to the ring nitrogen.[22]

The IR, UV and mass spectral data of **5**, **14** and **15** are also in accord with those expected from diazaphenanthrenes. The mass spectrum of **14** has a molecular ion as the base peak at *m/e* 180 and another prominent peak at *m/e* 90 (M^{2+}). Other notable peaks appear at 153 (M-27), 152 (M-28), 126 (M-54) and 125 (M-55). Genesis[23] of these peaks is ascribed to loss of one or two HCN or H_2CN fragments. The metastable corresponding to loss of HCN from the molecular ion could be observed at 130·5 together with a second metastable at 103·8. The latter indicates a stepwise loss of two HCN fragments. Similar fragmentation patterns were observed in the mass spectra of **5** and **15**.

Comparison of the mass spectra of the three diazaphenanthrenes (**5**, **14** and **15**) showed a greater resemblance between those of **5** and **15** as compared with **14**. If the fragment ions (M-HCN) have a 4-membered ring structure[24] (Fig 4) then both **5** and **15** lead to the same moieties, which might be the reason for this similarity.

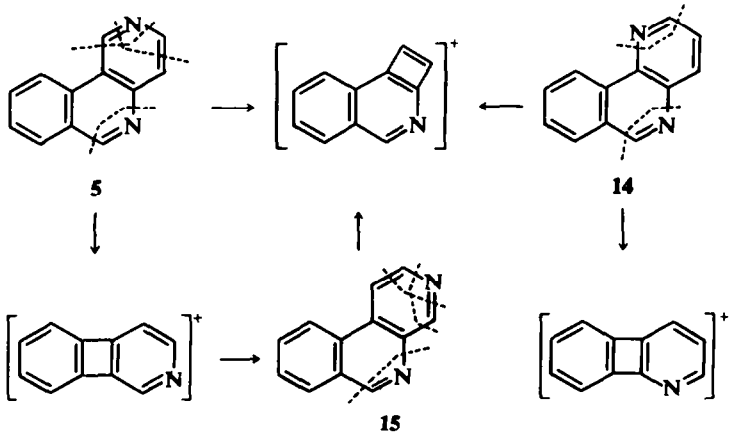

Fig 4.

Reaction of the anil **20**, obtained quantitatively from *o*-chlorobenzaldehyde and 3-amino-2,6-lutidine[25] (**19**), with potassium amide in liquid ammonia was investigated to test the effect of substituents upon benzyne cyclisation and to prepare the analeptic drug 9-amino-1,3-dimethyl-2,10-diazaphenanthrene (**22**), already synthesised[9] by a lengthy route. The cyclisation proceeded efficiently to afford 1,3-dimethyl-2,10-diazaphenanthrene (**21**) which was converted[26] into the desired compound **22**.

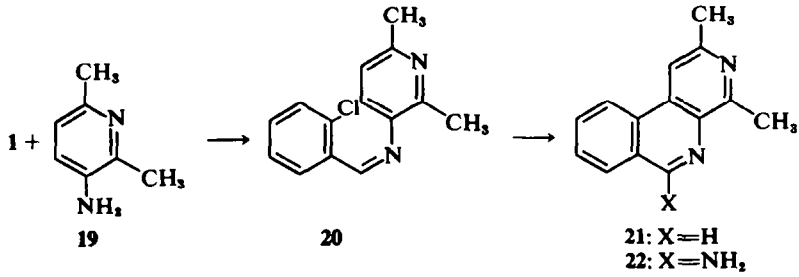

Fig 5.

Preliminary screening[27] of the diazaphenanthrenes **5**, **14**, **15** and **21** showed potent and often specific antimicrobial activity against the different bacterial strains tested.

EXPERIMENTAL

M.ps and b.ps are uncorrected. Microanalyses were performed by Mr. L. K. Khullar, Panjab University, Chandigarh, India. Anhydrous Na_2SO_4 was used as drying agent. IR spectra were determined in nujol mull. The mass spectra were measured on a MS-9 spectrometer at 70 eV. NMR spectra were recorded (TMS reference) on a Varian Associates Model HA-100 instrument. In all cases where the same compound was obtained through different routes, the identity was established through m.m.p. and TLC.

4,4'-[(2-*Chlorobenzylidene*)*diimino*] *dipyridine* (**3**)

A mixture of 4-aminopyridine (3·76 g) and *o*-chlorobenzaldehyde (2·81 g) was heated at 120° for 10 hr to give **3** (5·5 g; 89%), m.p. 183–184° (EtOH); ν_{max} 3230 (N-H) and 1625 cm^{-1}. (Found: C, 65·98; H, 5·13; N, 18·45. $C_{17}H_{15}N_4Cl$ requires: C, 65·67; H, 4·86; N, 18·02%).

4-[(2-*Chlorobenzylidene*)*amino*] *pyridine* (**4**)

Compound **3** (6·21 g) was kept in a molten state under reduced pressure for 1 hr and then the liberated 4-aminopyridine was expelled at 165–170°/11 mm. The residue was distilled at 175°/7 mm to afford an unstable oil (3·7 g; 85%) which was immediately used for the next reaction.

3,10-*Diazaphenanthrene* (**5**)

The anil **4** (1·083 g), suspended in ether (15 ml), was added to well stirred potassium amide (from 2·34 g K) in liquid ammonia (400 ml). After 45 min additional stirring, the mixture was decomposed with NH_4Cl (5 g). Ammonia was allowed to escape and the residue after adding water was extracted with chloroform. The organic layer was washed with water, dried and the solvent removed. The complex residue (TLC) was subjected to sublimation under reduced pressure to afford a solid (0·18 g; 20%), m.p. 114–115°; ν_{max} 1650 and 1635 cm^{-1}; λ_{max}^{EtOH} 246 nm (log ϵ 4·68); NMR ($CDCl_3$) δ9·4 (s,1,H-9), 9·92 (s,1,H-4); mass spectrum *m/e* (rel. intensity): 180(100), 179(49), 154(6), 153(12), 152(7), 129(5), 127(5), 126(5), 125(4), 90(14); metastable peaks at 130·5 and 103·8. (Found: N, 15·30. $C_{12}H_8N_2$ requires: N, 15·55%). Picrate, m.p. 242–244° (dec). (Found: C, 52·71; H, 2·81; N, 17·06. $C_{18}H_{11}O_7N_5$ requires: C, 52·82; H, 2·71; N, 17·11%).

4-[(2-*Chlorobenzyl*)*amino*] *pyridine* (**6**)

A mixture of **3** (1·035 g) in EtOH (31 ml) and $NaBH_4$ (0·345 g) was heated under reflux for 9 hr. Alcohol was evaporated and the residue,* after adding water, was extracted with chloroform. The extract was washed with water, dried and the solvent removed to afford an oil (0·72 g; 99%), m.p. 95–96° (ether-light petroleum); ν_{max} 3230 (N-H) and 1620 cm^{-1}. (Found: N, 13·11. $C_{12}H_{11}N_2Cl$ requires: N, 12·81%).

9,10-*Dihydro*-3,10-*diazaphenanthrene* (**7**)

Reaction of the reduced anil **6** (0·5465 g), dissolved in ether (10 ml), with potassium amide (from 0·585 g K) in liquid ammonia (200 ml) for 1·5 hr gave **7** (0·18 g; 40%), m.p. 146–148° (acetone-light petroleum); ν_{max} 3220 (N-H) and 1620 cm^{-1}. (Found: N, 15·14. $C_{12}H_{10}N_2$ requires: N, 15·38%). Picrate, m.p. 206–207° (dec). (Found: N, 16·61. $C_{18}H_{13}O_7N_5$ requires: N, 17·03%).

3,10-*Diazaphenanthrene* (**5**)

A soln of **7** (0·0364 g) in chloroform (20 ml) was stirred with active MnO_2 (0·364 g) for 10 hr to obtain a solid (0·034 g; 94%), m.p. 114–115° (sublimation), identical with **5**.

3,10-*Diazaphenanthrene* (**5**)

Compound **3** (1·083 g) suspended in ether (10 ml) was reacted with potassium amide (from 1·17 g K) in liquid ammonia (200 ml) for 45 min. The mixture was decomposed with NH_4Cl (3 g). Ammonia was allowed to escape and the well powdered residue (a) was extracted as such with chloroform. The extract was shaken with water and the separated aqueous layer was washed with more chloroform. Addition of picric acid in EtOH to the aqueous layer gave a picrate m.p. 214–215° (EtOH), showing no depression in m.p. on admixture with 4-aminopyridine picrate.[28]

To the remaining part of the residue (a), left after ex-

*Presence of 4-aminopyridine as a by-product of this reaction could be shown by the procedure detailed under preparation of **5** from **3**.

traction with chloroform, water was added and the whole extracted with chloroform. All the above chloroform extracts were combined with this, washed with water, dried and the solvent removed to afford a solid (0·62 g; 98%), m.p. 114–115° (sublimation), identical with **5**.

2,2'-[(2-*Chlorobenzylidene)diimino*] *dipyridine* (**10**)

Reaction of *o*-chlorobenzaldehyde (4·217 g) and 2-aminopyridine (5·64 g) at 120° for 7 hr afforded **10** (9·315 g; quantitative), m.p. 133–135° (dil EtOH); ν_{max} 3250 (N-H) and 1620 cm^{-1}. (Found: N, 17·66. $C_{17}H_{15}N_4Cl$ requires: N, 18·02%).

Attempted cyclisation of 2,2'-[(2-*chlorobenzylidene)diimino*] *dipyridine* (**10**)

Compound **10** (1·083 g) suspended in ether (5 ml) was treated with potassium amide (from 1·17 g K) in liquid ammonia (400 ml) for 45 min. The mixture was decomposed with NH_4Cl (3 g). Ammonia was allowed to escape, the powdered residue, extracted successively with light petroleum, ether and chloroform. All these extracts, were separately washed with water and dried.

The light petroleum extract gave a waxy residue (0·32 g) which did not form a picrate. The ether extract residue (0·07 g) gave a picrate which was crystallised from EtOH, m.p. 235–237° (dec). (Found: N, 17·5%). The chloroform extract left a gummy residue (0·33 g) which was dissolved in EtOH and filtered after adding equal amount of acetone. The filtrate on evaporation yielded a small residue which upon treatment with picric acid gave the same picrate as above. The water washings, from the original chloroform extract, were shaken with chloroform. Picric acid in alcohol was added to the aqueous layer. The picrate obtained m.p. 218–220° (EtOH) was found to be identical with the one obtained from 2-aminopyridine.[28]

2-[(2-*Chlorobenzyl)amino*] *pyridine* (**11**)

(a) 2-Aminopyridine (3 g), *o*-chlorobenzaldehyde (4·68 g) and 98–100% formic acid (6 g) were heated under reflux for 9 hr. The cooled resultant product was poured into a mixture of 50% NaOHaq (10 g) and ice (20 g). The resulting solid was filtered off, washed with water and dried to give the reduced anil **11** (6 g; 86%) which was crystallised from dilute EtOH, m.p. 99–101°; ν_{max} 3240 (N-H) and 1625 cm^{-1}. (Found: N, 13·06. $C_{12}H_{11}N_2Cl$ requires: N, 12·81%).

(b) Reaction of **10** (0·621 g) in EtOH (12 ml), with $NaBH_4$ (0·207 g) for 7 hr gave a solid (0·42 g; 96%), m.p. 99–101° (dil EtOH), identical with **11**.

Attempted cyclisation of 2-[(2-*chlorobenzyl)amino*] *pyridine* (**11**)

A suspension of the reduced anil **11** (1·093 g) in ether (10 ml) was reacted with potassium amide (from 2·34 g K) in liquid ammonia (400 ml) for 3 hr. The mixture was processed as usual. After adding water, it was successively extracted with ether and chloroform. The extracts were separately washed with water, dried and the solvents removed.

The residue (0·4 g) from the ether extract could not be purified. It was heated as such with selenium powder at 300° for 6 hr and then extracted with alcohol and acetone. Removal of the solvent left a tar from which no pure material could be isolated. The residue (0·4 g) from the chloroform extract was treated with EtOAc and the solid formed was collected, m.p. above 320°. The residue obtained on evaporation of the filtrate was passed through alumina using chloroform as eluent and was further purified by precipitation from chloroform with ether. It also did not melt upto 300°. (Found: C, 71·52; H, 5·73; N, 11·1%). The material which was insoluble, after extractions of the original aqueous layer from the cyclisation reaction, was collected and treated with hot EtOH and filtered. The insoluble solid did not melt upto 340°. The filtrate was evaporated, the residue dissolved in EtOH and passed through alumina. The soln obtained gave a solid on addition of ether, m.p. above 320°. (Found: C, 69·56; H, 6·98; N, 8·53%).

3-[(2-*Chlorobenzylidene)amino*] *pyridine* (**13**)

Reaction of *o*-chlorobenzaldehyde (14·057 g) and 3-aminopyridine[21] (9·4 g) at 115° for 5 hr gave **13** (20 g; 92%), b.p. 150°/0·1 mm. (Found: N, 13·17. $C_{12}H_9N_2Cl$ requires: N, 12·90%).

4,10-*Diazaphenanthrene* (**14**) *and* 2,10-*Diazaphenanthrene* (**15**)

Compound **13** (2·166 g) in ether (10 ml) was reacted with potassium amide (from 2·34 g K) in liquid ammonia (400 ml) for 45 min. The mixture was processed as usual and extracted with ether after adding water. The extract was washed with water, dried and the solvent removed. The residue, when chromatographed on alumina (with light petroleum) gave first **14** (0·3 g; 17%) which was sublimed under reduced pressure to afford an analytical sample, m.p. 99–101°; ν_{max} 1650 and 1620 cm^{-1}; λ_{max}^{EtOH} 238 nm (log ϵ 4·70); NMR ($CDCl_3$) δ8·43 (q,1,J = 8·0 Hz, J = 2·0 Hz, H-1); NMR (C_6D_6) δ8·82 (q,1,J = 4·5 Hz, H-3); mass spectrum *m/e* (rel. intensity): 180(100), 179(21), 178(2), 154(4), 153(5), 152(5), 127(4), 126(3), 125(2), 90·5(2), 90(10); metastable peaks at 130·5 and 103·8. (Found: N, 15·57. $C_{12}H_8N_2$ requires: N, 15·55%). Picrate, m.p. 225–226° (EtOH). (Found: C, 52·71; H, 2·83; N, 17·06. $C_{18}H_{11}O_7N_5$ requires: C, 52·82; H, 2·71; N, 17·11%).

From later fractions (eluting with light petroleum and benzene 1:1) 2,10-diazaphenanthrene **15**; (0·9 g; 50%) was obtained. It was also sublimed under reduced pressure to obtain an analytical sample, m.p. 102–104°; ν_{max} 1635 and 1620 cm^{-1}; λ_{max}^{EtOH} 244 nm (log ϵ 4·67); NMR (C_6D_6) δ9·87 (broadened s,1,H-1), 9·0 (s,1,H-9), 8·72 (broadened d,1,J = 4·5 Hz, H-3); mass spectrum *m/e* (rel. intensity): 180(100), 179(47), 154(4), 153(8), 152(12), 127(6), 126(10), 125(5), 90(7); metastable peaks at 130·5 and 103·8. (Found: N, 15·45. $C_{12}H_8N_2$ requires: N, 15·55%). Picrate, m.p. 268–270° (dec). (Found: C, 52·66; H, 3·00; N, 16·69. $C_{18}H_{11}O_7N_5$ requires: C, 52·82; H, 2·71; N, 17·11%).

3-[(2-*Chlorobenzyl)amino*] *pyridine* (**16**)

(a) The usual reaction of 3-aminopyridine (0·5 g), *o*-chlorobenzaldehyde (0·78 g) and 100% formic acid (1 g) gave a material which was crystallised from acetone as **16** (0·41 g; 35%), m.p. 105–107°; ν_{max} 3230 (N-H) and 1600 cm^{-1}. (Found: C, 65·60; H, 5·21; N, 12·74. $C_{12}H_{11}N_2Cl$ requires: C, 65·89; H, 5·07; N, 12·81%).

(b) Reaction of **13** (2·166 g) in EtOH (21 ml), with $NaBH_4$ (0·722 g) for 3 hr afforded a solid (2 g; 92%), m.p. 105–107° (acetone), identical with **16**.

9,10-*Dihydro*-4,10-*diazaphenanthrene* (**17**) and 9,10-*Dihydro*-2,10-*diazaphenanthrene* (**18**)

Reaction of the reduced anil **16** (0·5465 g) dissolved in a mixture of ether (5 ml) and THF (5 ml) with potassium

amide (from 0·585 g K) in liquid ammonia (200 ml) gave a gummy material which upon crystallisation from acetone-light petroleum furnished **18** (0·3 g; 66%), m.p. 130–131°; ν_{max} 3250 (N-H) and 1610 cm^{-1}. (Found: N, 15·81. $C_{12}H_{10}N_2$ requires: N, 15·38%). Picrate, m.p. 201–203° (EtOH). (Found: N, 17·16. $C_{18}H_{13}O_7N_5$ requires: N, 17·03%).

The filtrate from the above crystallisation was evaporated. From the residue, **17** was obtained by chromatography on alumina using light petroleum and benzene (3 : 1) for elution. Crystallisation from light petroleum gave the pure material (0·04 g; 9%), m.p. 89–90°. (Found: N, 14·88. $C_{12}H_{10}N_2$ requires: N, 15·38%).

4,10-*Diazaphenanthrene* (**14**)

Oxidation of **17** (0·0364 g) in chloroform (20 ml) with active MnO_2 (0·364 g) for 17 hr in the usual manner, gave a solid (0·035 g; 95%), m.p. 130–131° (acetone-light petroleum), identical with **14**.

2,10-*Diazaphenanthrene* (**15**)

MnO_2 oxidation of **18**, as above, afforded **15** (92%), m.p. 89–90° (light petroleum).

3-[(2-*Chlorobenzylidene)amino*]-2,6-*lutidine* (**20**)

Condensation of *o*-chlorobenzaldehyde (1·4057 g) with 3-amino-2,6-lutidine[25] (1·22 g) at 120° for 3 hr gave **20** (2·446 g; quantitative), m.p. 81–82° (acetone); ν_{max} 1640 cm^{-1}. (Found: N, 11·32. $C_{14}H_{13}N_2Cl$ requires: N, 11·45%).

1,3-*Dimethyl*-2,10-*diazaphenanthrene* (**21**)

Reaction of **20** (1·223 g), with potassium amide (from 1·17 g K) in the usual manner, gave **21** (1·02 g; 98%), m.p. 158–159° (acetone); ν_{max} 1620 cm^{-1}. (Found: N, 13·93. $C_{14}H_{12}N_2$ requires: N, 13·45%). Its picrate was crystallised from EtOH as yellow needles, m.p. 228–230° (dec). (Found: C, 55·45; H, 3·57; N, 16·50. $C_{20}H_{15}O_7N_5$ requires: C, 54·92; H, 3·46; N, 16·01%).

9-*Amino*-1,3-*dimethyl*-2,10-*diazaphenanthrene* (**22**)

A mixture of **21** (1·04 g) and KNO_3 (0·684 g) was added to the potassium amide (from 0·621 g K) in liquid ammonia (40 ml) and the mixture was kept in a sealed tube at room temp for 17 days. Ammonia was allowed to evaporate and the residue after adding water was extracted with ether. The organic layer was washed with water, dried and the solvent removed to yield **22** (0·2 g; 18%), m.p. 188–190° (light petroleum) (lit,[9] m.p. 187–189°); ν_{max} 3200 (N-H) and 1640 cm^{-1}. (Found: N, 18·46. Calc. for $C_{14}H_{13}N_3$: N, 18·82%). Its picrate was crystallised from EtOH, m.p. 258–260° (dec) [lit,[9] m.p. 260–262° (dec)].

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