Polyhalogenoheterocyclic Compounds.   Part XXIX.[1]   Perchloro-acridine, -phenanthridine, and -benzo[h]quinoline

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Perchloro-acridine, -phenanthridine, and -benzo[h]quinoline have been synthesised by direct chlorination of the corresponding heterocyclic compound or its monochloro-derivative, complexed with aluminium trichloride. Attempts to replace chlorine by fluorine, by using potassium fluoride under a variety of conditions, were unsuccessful.   Hydrolysis of the acridine and phenanthridine derivatives gave the corresponding 9-acridone and phenthridin-6-one, respectively.   The orientation of nucleophilic substitution is established and there is no evidence for competing reductive dechlorination in reactions with amines.

IN this series we have been concerned with synthesis of highly chlorinated and fluorinated derivatives of nitrogen heterocyclic compounds, but have not previously dealt with tricyclic systems.   Here, the syntheses of perchloro-acridine (1) and -phenanthridine (2) are described, together with a preliminary survey of some of their reactions.   Octachloro-9-acridone has been obtained previously by the reaction of acridone with antimony pentachloride [2] and by diazotisation of tetrachloroanthranilic acid,[3] and octafluoro-9-acridone has been obtained by diazotisation of tetrafluoroanthranilic acid [4] and by electrochemical oxidation of 2-aminononafluorobenzophenone.[5]

We have synthesised perchloro-acridine and -phenanthridine by direct chlorination of 9-chloroacridine and 6-chlorophenanthridine, respectively, with aluminium trichloride as catalyst, by using techniques similar to those developed earlier for making chlorinated quinolines and isoquinolines.[6,7]   A major difficulty was encountered, however, in the isolation of perchloroacridine since direct addition of the reaction mixture to iced water, to remove aluminium trichloride, gave a product containing considerable amounts of octachloro-9-acridone.   Nevertheless, it was possible to circumvent this hydrolysis by first dissolving the reaction mixture in dry chloroform and then cooling to —10 °C before addition of cold dry methanol.   Perchloroacridine was then obtained as a precipitate while aluminium alkoxides remained in solution.

Perchloro-acridine and -phenanthridine are soluble in concentrated sulphuric acid but they are only weak bases: we were unable to isolate salts by passing hydrogen chloride into their solutions in chloroform.

Reactions.—We have previously developed a synthesis of fluorinated heterocyclic compounds by reaction of a perchloro-compound with potassium fluoride in an autoclave at elevated temperatures, in the absence of a solvent,[8] and this procedure has been successfully applied to obtain a range of new fluorinated aza- and diaza-benzenes and -naphthalenes.[9]   Nevertheless, we have been totally unsuccessful in applying these pro-

cedures to perchloro-acridine and -phenanthridine.   In reactions at 250 °C and above, for a variety of times, we only obtained black intractable material, and use of potassium fluoride in tetramethylene sulphone,[10] at temperatures up to 200 °C, led to moderate recovery of mixtures containing only partially fluorinated material. The solvent method, however, could not be developed into a preparatively useful procedure for fluorinated acridines and phenanthridines.

(1)   (2)

(3)
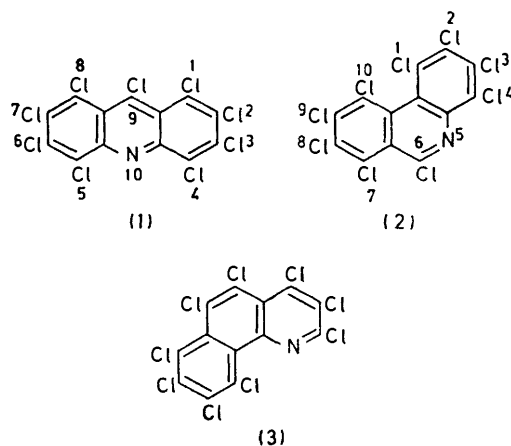
For comparison, perchlorobenzo[h]quinoline (3) was synthesised, as a further example of a perchlorinated tricyclic system.   In this case, however, there are the combined structural features of perchloro-quinoline and -naphthalene, both of which have been successfully fluorinated with potassium fluoride.[6,10]   Nevertheless, in spite of these similarities, attempted fluorination of (3) gave similar results to those described above for reactions with the systems (1) and (2), and this implies that the difficulties stem from the presence of three fused rings.   At this stage, however, the explanation is not clear.

Hydrolysis of perchloroacridine, giving octachloro-9-acridone, occurs more readily than hydrolysis of perchlorophenanthridine.   These are probably acid-induced hydrolyses, occurring *via* nucleophilic attack on

¹ Part XXVIII, G. M. Brooke, R. D. Chambers, W. K. R. Musgrave, R. A. Storey, and J. Yeadon, *J.C.S. Perkin I,* 1976, 162.

² A. Eckert and K. Steiner, *Ber.,* 1914, **47,** 2628.

³ R. Howe, *J. Chem. Soc. (C),* 1966, 478.

⁴ S. Hayashi and N. Ishikawa, *Nippon Kagaku Kaishi,* 1973, **7,** 1319 (*Chem. Abs.,* 1973, **79,** 78,576t).

⁵ C. M. Jenkins, A. E. Pedler, and J. C. Tatlow, *Tetrahedron,* 1971, **27,** 2557.

⁶ R. D. Chambers, M. Hole, B. Iddon, W. K. R. Musgrave, and R. A. Storey, *J. Chem. Soc. (C),* 1966, 2328.

⁷ M. Gordon and D. E. Pearson, *J. Org. Chem.,* 1964, **29,** 329.

⁸ R. D. Chambers, J. Hutchinson, and W. K. R. Musgrave, *J. Chem. Soc.,* 1964, 3573.

⁹ See R. D. Chambers, ' Fluorine in Organic Chemistry,' Wiley–Interscience, New York, 1973, p. 273, and references therein.

¹⁰ G. Fuller, *J. Chem. Soc.,* 1965, 6264.

the *N*-protonated species, and the difference in reactivity implies that perchloroacridine is the stronger base. This was anticipated from the fact that the acridine (1) has no chlorine atom attached to carbon which is α to nitrogen, whereas the phenanthridine (2) has one such chlorine atom and it is known that such an adjacent chlorine atom reduces the base strength of nitrogen significantly.[11]
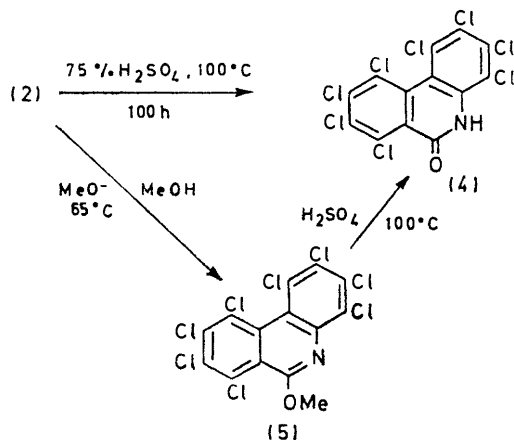
Hydrolysis of perchlorophenanthridine (2) gave the lactam (4), arising from nucleophilic attack at the
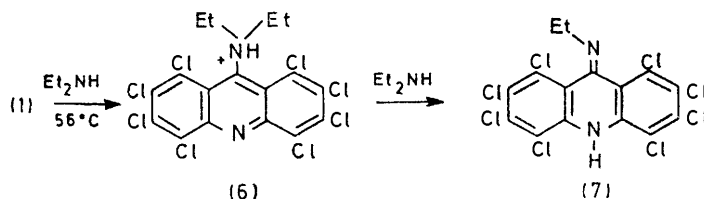
6-position. The i.r. spectrum of (4) shows bands at 3 200 (NH str.) and 1 690 cm⁻¹ (C:O str.), similar to the values for phenanthridin-6-one (1 669)[12] and octachloro-9-acridone (1 665 cm⁻¹). If attack had occurred at a position other than C-6, it is unlikely that the product would exist in the oxo-form. This follows from the fact that, of the various hydroxyphenanthridines studied, only the 6-' hydroxy '-derivative exists in the oxo-form.[12] Also, the presence of a 6-chlorine atom would make it even less likely that a hydroxy-group in the system would exist in the oxo-form, since adjacent chlorine would severely diminish the base strength of nitrogen.[11]

Nucleophilic substitution with sodium methoxide in methanol occurs quite readily at 65 °C, giving exclusive monosubstitution at the expected 9-position in perchloroacridine (1) and at the 6-position in perchlorophenanthridine (2). These positions of substitution were established by hydrolysis of the methoxy-compounds to the corresponding lactams, octachloro-9-acridone and octachlorophenanthridin-6-one (4). Reaction of the phenanthridine (2) with diethylamine gave a diethyl-

amino-derivative, but the acridine (1) gave only an imine (7), presumably produced by Hofmann elimination from the intermediate (6). Competition between nucleophilic aromatic substitution and reductive dehalogenation has been observed in reactions of nitro-activated iodobenzenes with bulky amines,[13] and reduction is the major process in reactions of 3-iodopyridine and 4-bromoisoquinoline with methoxide in methanol.[14] It has been suggested that this arises from competition between electron transfer from the nucleophile and nucleophilic aromatic substitution:
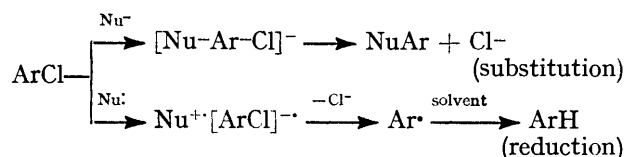
The chlorinated tricyclic systems (1) and (2) should be effective electron acceptors and, indeed, each of these systems reacts with potassium in toluene to give a coloured solution, from which products indicating the occurrence of reduction can be detected. However, reactions of (1) and (2) with the bulky amines t-butylamine and tri-n-butylamine resulted in nucleophilic substitution; reduction products were observed only in trace amounts with the tertiary amine.

Pyrolysis of (2) was attempted, in anticipation of the possible formation of perchlorobiphenylene. However, rearrangement, involving migration of chlorine, occurred, giving the nitrile (8).

EXPERIMENTAL

Spectroscopic data were obtained with the following spectrometers: i.r., Grubb–Parsons Spectromaster; u.v., Unicam SP 800 or SP 8000; mass, A.E.I. MS9 (parent *m/e*

[11] C. D. Johnson, A. R. Katritzky, B. J. Ridgewell, N. Shakir, and A. M. White, *Tetrahedron*, 1965, **21**, 1055.

[12] S. F. Mason, *J. Chem. Soc.*, 1957, 4874.
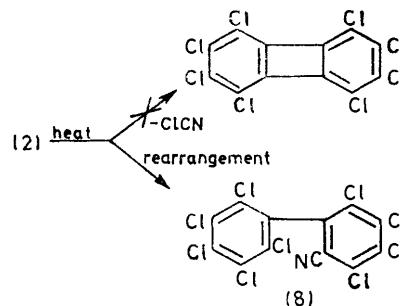
[13] F. Pietra, M. Bartolozzi, and F. Del Cima, *Chem. Comm.*, 1971, 1232.

[14] J. A. Zoltewicz, T. M. Oestreich, and A. A. Sale, *J. Amer. Chem. Soc.*, 1975, **97**, 5889.

values for chloro-compounds recorded refer to [35]Cl; n.m.r., Varian A56/60D.

Except for reactions involving aqueous systems, all apparatus was carefully dried before use. Where indicated with an asterisk full spectroscopic data are available in Supplementary Publication No. SUP 21725 (2 pp.).†

Perchloroacridine (1).—A mixture of 9-chloroacridine (60 g) and finely crushed aluminium chloride (200 g) was heated in a flask which was continually purged with dry nitrogen. At *ca.* 100 °C, a molten complex was formed between the two substances and stirring was begun. The temperature was maintained at 120—160 °C for 48 h while dry chlorine was passed into the flask. The temperature was then raised to 170—190 °C for 48 h while more chlorine was added. Dry chloroform (1 l) was added to the resulting mixture and the system was heated under reflux until all material had passed into solution. This solution was deep red and contained, presumably, a complex between perchloroacridine and aluminium chloride; as far as possible it was only handled under dry nitrogen. After filtering, the solution was cooled to −10 °C and dry methanol (500 ml), also cooled to −10 °C, was added. This mixture was stirred and kept cool until the red colour had disappeared and a yellow precipitate was formed. The precipitate was crude nonachloroacridine (104 g, 75%), m.p. 246—249°. A mixture of crude nonachloroacridine (6 g; containing some octachloro-9-acridone), *NN*-diethylaniline (1.9 g, 12.5 mmol), and phosphoryl chloride (25 ml) was heated under reflux in dry nitrogen for 48 h. The excess of phosphoryl chloride was removed *in vacuo*, *NN*-diethylaniline hydrochloride was removed by sublimation at 110—120° and 0.1 mmHg, and the residual solid was recrystallised (dry xylene) to give yellow-green needles (3.9 g) of *nonachloroacridine*,* m.p. (sealed tube) 273—275° (decomp.) (Found: C, 32.2; Cl, 65.5; N, 2.75%; M^+, 485. $C_{13}Cl_9N$ requires C, 31.9; Cl, 65.2; N, 2.9%; M, 485).

Perchlorophenanthridine (2).—A mixture of 6-chlorophenanthridine (42 g) and finely crushed aluminium chloride (180 g) was heated, in a flask which was continually purged with dry nitrogen, to 120 °C, at which temperature a molten complex was formed and stirring was begun. The temperature was maintained at 120—150 °C for 48 h, and dry chlorine was passed into the flask. The temperature was raised to 160—180 °C for a further 48 h and more chlorine was added. Dry chloroform (1 l) was added to the resulting mixture, which was heated under reflux until all the material had passed into solution. This solution was deep red, containing, presumably, a complex between perchlorophenanthridine and aluminium chloride, and, as far as possible, it was only handled under dry nitrogen. After filtering, the solution was cooled to −10° and dry methanol (750 ml), also cooled to −10 °C, was added. This mixture was stirred and kept cool until the red colour had disappeared and a yellow precipitate was formed, which was crude nonachlorophenanthridine. A mixture of the crude nonachlorophenanthridine (27 g), *NN*-diethylaniline (6 ml, 35 mmol), and phosphoryl chloride (90 ml) was heated under reflux in dry nitrogen, for 4.5 h. The mixture was allowed to cool and the excess of phosphoryl chloride was removed *in vacuo*. *NN*-Diethylaniline hydrochloride was removed by sublimation at 100—120° and 0.1 mmHg, and the residual solid was recrystallised [dry chlorobenzene–petroleum (b.p. 60—80°)] to give pale yellow

† For details of Supplementary Publications, see Notice to Authors No. 7, *J.C.S. Perkin I*, 1975, Index issue.

needles (17 g) of *nonachlorophenanthridine*,* m.p. 255—257°. A sample for analysis was recrystallised further from dry xylene–petroleum (b.p. 60—80°) (1 : 1 v/v) (Found: C, 31.9; Cl, 65.0; N, 3.2%; M^+, 485. $C_{13}Cl_9N$ requires C, 31.9; Cl, 65.2; N, 2.9%; M, 485).

Perchlorobenzo[h]*quinoline* (3).—A mixture of benzo[*h*]-quinoline (50 g) and finely crushed aluminium chloride (180 g) was heated, in a flask which was continually purged with dry nitrogen, to 130 °C, at which temperature a molten complex was formed between the two substances and stirring was begun. The temperature was maintained at 140—160 °C for 48 h, and dry chlorine was passed into the flask. Then the temperature was raised to 160—180 °C for a further 48 h, while more chlorine was added. The hot molten mixture was then cautiously poured onto ice-water (1 500 ml) and stirred. The reddish brown precipitate was a mixture of chlorinated benzo[*h*]quinolines, chiefly pentachlorobenzo[*h*]quinoline and hexachlorobenzo-[*h*]quinoline (90 g, *ca.* 85%), m.p. 140—144° (Found: Cl, 53.2. $C_{13}H_3Cl_6N$ requires Cl, 55.2. $C_{13}H_4Cl_5N$ requires Cl, 50.6%). The partially chlorinated benzo[*h*]quinolines (10 g) and phosphorus pentachloride (150 g) were sealed in an evacuated steel autoclave, heated to 370 °C, and then maintained at this temperature for a total of 6 h. After venting, the autoclave was allowed to cool and opened, and the contents were mixed with ice–water (400 ml) and stirred. The precipitate (6 g, 85%) was then sublimed (0.01 mmHg; 150 °C) to give *nonachlorobenzo*[h]*quinoline* * (3), m.p. 209—211° (Found: Cl, 65.1; M^+, 485. $C_{13}Cl_9N$ requires Cl, 65.2%; M, 485).

Attempted Fluorinations in the Absence of Solvent.—Several reactions were attempted on compounds (1)—(3) but without giving useful products. A number of reactions were carried out under various conditions but by the following general procedure. The substrate (2—30 g) was mixed with dry potassium fluoride (20—200 g) and sealed in a nickel-lined autoclave under vacuum. The autoclave was heated at temperatures ranging from 300 to 470 °C for 1½—24 h. Volatile material was transferred under vacuum into a cold trap, but in every case only materials which were gaseous at room temperature were obtained. The autoclave was cooled and opened, and the dark residue was emptied out. It was not possible to obtain significant amounts of tractable material in any case, despite the use of various extraction procedures or sublimation.

Attempted Fluorinations of Perchlorophenanthridine (2) *in a Solvent.*—Several reactions were attempted, with potassium or caesium fluoride as the fluorinating reagent, but all were without significant success. In the general procedure nonachlorophenanthridine (2—5 g) and the fluorinating agent (15—30 g) were stirred in tetramethylene sulphone at temperatures in the range 160—180°, for 29—90 h, under dry nitrogen. After cooling, the resulting mixtures were poured into water (4 l) and stirred. Solid was filtered off and refluxed with chloroform (200 ml) for 1½ h. After filtering, solvent was removed from the filtrate to leave a pale brown material, which in most cases was a mixture of polychloropolyfluorophenanthridin-6-ones, *m/e* 403, 419, and 435 corresponding to $C_{13}HCl_4F_4NO$, $C_{13}HCl_5F_3NO$, and $C_{13}HCl_6F_2NO$, respectively.

Hydrolysis of Perchloroacridine (1).—Nonachloroacridine (2.0 g) was heated under reflux with water (50 ml) and concentrated hydrochloric acid (50 ml) for 18 h. The green solid produced was octachloro-9-acridone * (1.6 g, 80%), m.p. >370° (Found: C, 32.9; Cl, 60.8; N, 2.8%;

M, 467. Calc. for $C_{13}HCl_8NO$: C, 33.15; Cl, 60.3; N, 3.0%; M, 467).

Hydrolysis of Perchlorophenanthridine (2).—Nonachlorophenanthridine (4.0 g) was stirred with water (30 ml) and concentrated sulphuric acid (120 ml) at 100 °C for 100 h. The solid was filtered from the cooled mixture and washed well with water. Recrystallisation from chlorobenzene gave green *octachlorophenanthridin-6-one* * (4) (2.4 g, 62%), m.p. >350° (Found: Cl, 60.5; M^+, 467. $C_{13}HCl_8NO$ requires Cl, 60.3%; M, 467).

Reaction of Perchloroacridine with Sodium Methoxide.— A mixture of perchloroacridine (2.00 g, 4.09 mmol) in dry methanol (150 ml) and sodium (0.10 g, 4.35 mmol) was heated under reflux for 24 h. The green solid produced was *octachloro-9-methoxyacridine* (1.75 g, 88%), m.p. 244—246° (Found: C, 35.0; H, 0.8; Cl, 58.2; N, 2.6%; M^+, 481. $C_{14}H_3Cl_8NO$ requires C, 34.7; H, 0.6; Cl, 58.5; N, 2.9%; M, 481). The octachloro-9-methoxyacridine (1.00 g) was heated under reflux with water (50 ml) and concentrated hydrochloric acid (50 ml) for 18 h. The solid that was filtered off was identical with the octachloro-9-acridone obtained earlier.

Reaction of Perchlorophenanthridine with Sodium Methoxide.—A mixture of perchlorophenanthridine (1.00 g, 2.04 mmol), dry methanol (40 ml), and sodium (0.06 g, 2.60 mmol) was heated under reflux for 65 h. The resulting mixture was added to water (40 ml), giving a green solid, *octachloro-6-methoxyphenanthridine* (5) (0.82 g, 81%), m.p. 245—247° (Found: C, 34.4; H, 0.6; Cl, 58.9; N, 3.0%; M^+, 481. $C_{14}H_3Cl_8NO$ requires C, 34.7; H, 0.6; Cl, 58.5; N, 2.9%; M, 481). This octachloro-6-methoxyphenanthridine (0.7 g) was heated with water (30 ml) and concentrated sulphuric acid (120 ml) at 100 °C for 100 h. The solid filtered off was identical with the octachlorophenanthridin-6-one (4) obtained earlier.

Reaction of Perchloroacridine with Diethylamine.—Perchloroacridine (1.00 g) was refluxed with diethylamine (70 ml) for 24 h. The resulting mixture was allowed to cool and poured into water (100 ml), and the solid was filtered off. This was recrystallised (chloroform) to give orange crystals of *octachloro-9-ethylimino-9,10-dihydroacridine* (7) (0.34 g, 33%), m.p. 222—224° (Found: Cl, 56.6%; M^+, 494. $C_{15}H_6Cl_8N_2$ requires Cl, 57.0%; M, 494), $\delta_H(C_6H_5Cl)$ 0.9 (CH$_3$) and 6.3 (CH$_2$); $\nu_{max.}(KBr)$ 3 395 (NH) and 1 621 cm^{-1} (C=N).

Reaction of Perchlorophenanthridine with Diethylamine.— Nonachlorophenanthridine (2.0 g) was stirred with refluxing diethylamine (50 ml) for 65 h. The resulting mixture (1.5 g, 70%) was added to water (100 ml) and the solid was recrystallised twice (acetone) to give pale brown *octachloro-6-diethylaminophenanthridine* (0.4 g, 19%), m.p. 131—132° (Found: C, 39.0; H, 1.2; Cl, 54.2; N, 5.1%; M^+, 522. $C_{17}H_{10}Cl_8N_2$ requires C, 38.8; H, 1.9; Cl, 53.95; N, 5.35%; M, 522); $\delta_H(CCl_4)$ 1.3 (CH$_3$) and 3.6 (CH$_2$).

Reaction of Nonachloroacridine with t-Butylamine.—A mixture of nonachloroacridine (1.0 g, 2.0 mmol) and t-butylamine (25 ml) was heated under reflux in dry nitrogen for 24 h. The resulting mixture was allowed to cool and volatile material was removed *in vacuo*. The residual solid was stirred with water (50 ml) for 5 min, filtered off, dried *in vacuo* at 100 °C, and recrystallised [petroleum (b.p. 60—80°)] to give orange crystals of *octachloro-9,10-dihydro-9-t-butylaminoacridine* (750 mg, 72%), m.p. 223—234° (Found: C, 39.1; H, 2.3; Cl, 53.6; N, 5.6%; M^+, 522. $C_{17}H_{10}Cl_8N_2$ requires C, 38.8; H, 1.9; Cl,

53.9; N, 5.3%; M, 522); $\delta_H(CCl_4)$ 1.22 (CH$_3$) and 7.77 (NH); $\nu_{max.}(KBr)$ 3 390 (NH) and 1 640 cm^{-1}. This product (200 mg) was heated under reflux with water (10 ml) and concentrated hydrochloric acid (10 ml) for 22 h. The solid that was filtered off was identical with the octachloro-9-acridone obtained earlier (i.r.).

Reaction of Nonachlorophenanthridine with t-Butylamine. —A mixture of nonachlorophenanthridine (0.85 g, 1.75 mmol) and t-butylamine (50 ml) was heated under reflux beneath dry nitrogen for 24 h. The mixture was allowed to cool and poured into water (100 ml). The resulting suspension was filtered and the residue was washed with water (100 ml) and dried *in vacuo* at 110 °C. T.l.c. in various solvents showed only one component. The solid was twice recrystallised (carbon tetrachloride) to give *octachloro-6-t-butylaminophenanthridine* as a bright yellow solid (0.6 g, 65%), m.p. 239—241° (Found: C, 38.5; H, 1.6; Cl, 54.1; N, 5.5%; M^+, 522. $C_{17}H_{10}Cl_8N_2$ requires C, 38.8; H, 1.9; Cl, 53.9; N, 5.3%; M, 522); $\delta_H(C_6H_5Cl)$ 1.3 (CH$_3$). This product was heated under reflux with water (10 ml) and concentrated hydrochloric acid (40 ml) for 240 h. The solid that was filtered off was identical with the octachlorophenanthridin-6-one obtained earlier (i.r.).

Reaction of Nonachloroacridine with Tri-n-butylamine.—A mixture of nonachloroacridine (1 g, 2.0 mmol) and tri-n-butylamine (50 ml) was heated in an oil-bath at 100 °C, beneath dry nitrogen, for 24 h, while the colour changed from red through yellow to black. The solvent was removed *in vacuo* to yield a brown solid which was then stirred with water (100 ml), filtered off, and dried *in vacuo*. The resulting black solid was fractionally crystallised [xylene–petroleum (b.p. 60—80°)] to yield small quantities of a brown solid. The mass spectrum of this material was complex, but showed only a small peak at 451 ($C_{13}HNCl_8$) and major peaks at 467 (perchloroacridone), and 485 (perchloroacridine) as well as at higher mass numbers such as 668, corresponding to substitution products.

Reaction of Nonachlorophenanthridine (2) *with Tri-n-butylamine.*—Nonachlorophenanthridine (0.7 g, 1.43 mmol) was stirred with dry tri-n-butylamine (40 ml) at 100 °C beneath dry nitrogen for 24 h. The mixture was allowed to cool and the excess of tri-n-butylamine was removed *in vacuo* to yield a brown tar. The i.r. spectrum was different from that of (2) and consistent with a quaternary ammonium salt. This was confirmed by hydrolysis in moist xylene, giving octachlorophenanthridin-6-one (i.r. and mass spectrum) from which tri-n-butylamine hydrochloride was sublimed *in vacuo*. There was no peak in the mass spectrum of the product corresponding to octachlorophenanthridine.

Pyrolysis of Perchlorophenanthridine.—Perchlorophenanthridine (1.0 g) was passed, under vacuum, through a silica tube heated to 800 °C, and the product was collected in a cold trap. This was recrystallised from chloroform and gave *nonachlorobiphenyl-2-carbonitrile* (8) (0.2 g, 20%), m.p. 216—218° (Found: Cl, 65.2%; M^+, 485. $C_{13}NCl_9$ requires Cl, 65.2%; M, 485), $\nu_{max.}(KBr)$ 2 218 (C≡N) and 1 537 cm^{-1}. This nitrile (8) was stirred with water (15 ml) and concentrated sulphuric acid (50 ml) for 100 h, and the resulting solid was removed by filtration and washed well with water. The i.r. spectrum indicated the presence of a carboxy-group ($\nu_{max.}$ 1 690 cm^{-1}), but a pure compound was not isolated.

Reaction of Nonachloroacridine with Potassium.—To the red solution of nonachloroacridine (250 mg, 0.51 mmol) in

dry toluene (25 ml) was added potassium (20 mg, 0.5 mmol), and the mixture was heated under reflux for 25 h. Volatile material was removed *in vacuo* to yield a yellow-green solid mixture of benzyloctachloroacridine (M^+ 541), nona-chloroacridine (M^+ 485), and octachloroacridone (M^+ 467). The mass spectrum also showed a peak at m/e 451, possibly due to octachlorohydroacridine.