Polychloroaromatic Compounds. Part IV.[1] Oxidation of 2- and 4-*N*-Alkylaminotetrachloropyridines and Nucleophilic Substitution of Tetrachloronitropyridines

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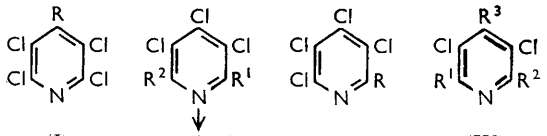
The reaction of pentachloropyridine and its 1-oxide with various primary amines is described. The resulting 2- and 4-*N*-alkylamino-derivatives are oxidised with peroxytrifluoroacetic acid to give nitroso-, nitro-, and amino-compounds for which a mechanism is suggested. The 2- and the 4-nitrotetrachloropyridine are found to suffer nucleophilic displacement of the nitro-group. The synthetic use of these nitro-compounds for producing inaccessible 2- or 4-substituted tetrachloropyridines such as the 4-morpholino-compound is discussed.

We have recently reported [2] that tetrachloro-4-piperidinopyridine (I; R = $C_5H_{10}N$) is oxidised by trifluoroperacetic acid at room temperature to give predominantly the 4-nitroso- and the 4-amino-derivative (I; R = NO or NH_2) and a little of the hydroxylamine (I; R = ONC_5H_{10}), the latter owing to rearrangement of an intermediate *N*-oxide. With hot peroxyacid only the 4-nitro-compound (I; R = NO_2) was, however, obtained. The 4-dimethylamino-derivative (I; R = NMe_2) yielded a mixture of the 4-nitroso-, and 4-amino-, and the 4-methylamino-compound (I; R = NO, NH_2, or NHMe) when treated with trifluoroacetic acid and 30% hydrogen peroxide in the cold. The last compound which arises from demethylation *via* its *N*-oxide (I;

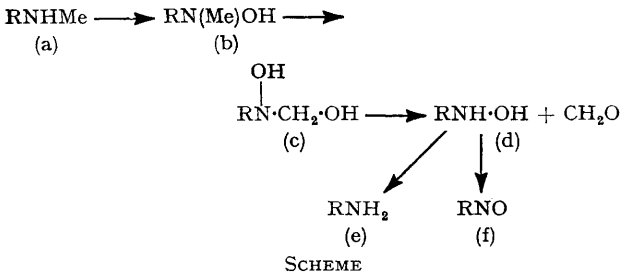


(I) (II) (III) (IV)

[1] Part III, J. D. Cook, B. J. Wakefield, H. Heaney and J. M. Jablonski, *J. Chem. Soc.* (*C*), 1968, 2727.

[2] Part I, S. M. Roberts and H. Suschitzky, *J. Chem. Soc.* (*C*), 1968, 1537.

R = $Me_2N \longrightarrow O$) by a Polonovski-type reaction [3] appears to be the precursor of the 4-amino- and the 4-nitroso-compound since both can be produced from the methylamino-derivative (I; R = NHMe) under the conditions of the reaction but are themselves not affected by cold trifluoroperacetic acid. The oxidation of the secondary amine (I; R = NHMe) is thus visualized to involve the sequence outlined (*cf.* Scheme; R = C_5Cl_4N). The hydroxylamine (b), the oxidation product of a secondary amine,[4] is further oxidised to the carbinolamine (c) which must readily hydrolyse to the pyridylhydroxylamine (d). This in turn yields, as expected under the reaction conditions, the nitroso-compound [5] (f) and the amine (e) the latter feasibly by an incomplete Bamberger hydroxylamine rearrangement [6] owing to a blocked *para*-position. This is partly borne out by the observation that pentachlorophenylhydroxylamine smoothly rearranges to pentachloroaniline in a similar way.[7]



SCHEME

The *N*-alkylaminotetrachloropyridines (I; R = NHMe, ·NHEt, ·$NHCH_2Ph$, and ·$NH·[CH_2]_3Me$) were prepared readily from pentachloropyridine and the requisite primary amine in boiling dioxan. The resulting mixture of the 2- and the 4-isomer with the latter predominating was readily separable on a silica column. Details of the reactions are given in the Table. When pentachloropyridine was heated in ethanol at 180° in a stirred autoclave with an excess of n-butylamine 2,4-di-(n-butylamino)trichloropyridine constituted 94% of the products with only 6% of the 2,6-isomer being formed.

By contrast, pentachloropyridine-1-oxide (II; R^1 = R^2 = Cl) reacted with primary aliphatic amines at room temperature to give exclusively the 2-derivative [*e.g.* (II; R^1 = ·NHMe or ·NHEt, R^2 = Cl) (molar ratio 1 : 2)] in dioxan, ethanol, or benzene. With excess of the amine under reflux in dioxan, only the 2,6-disubstituted derivatives [*e.g.* (II; R^1 = R^2 = NHMe)] were formed. Thus the outcome of substitution in the *N*-oxide with primary amines is unaffected by the nature of the solvent owing to the over-riding nucleophilicity of the 2(6) positions in contrast to the behaviour of the parent compound (I; R = Cl). This is in complete harmony with the orientation pattern which we observed for secondary amines.[8] Deoxygenation of the *N*-oxides occurred smoothly with phosphorus trichloride in hot chloroform to give the corresponding *N*-alkyl substituted derivatives.

Orientation of the above described secondary amines was achieved by oxidation with trifluoroacetic acid and 30% hydrogen peroxide. The 4-*N*-alkylaminopyridines (I; R = NHMe, NHEt, $NH·Bu^n$, or $NHCH_2Ph$) gave the 4-nitroso-compound (I; R = NO) while the 2-*N*-alkylpyridines (III; R = *s*-amine) gave tetrachloro-2-nitropyridine in good yield. Reduction of the nitro- or nitroso-compound with stannous chloride gave the corresponding and known [9] aminochloropyridines thereby confirming the structure of the various *N*-alkylamino-derivatives. Oxidation of the 4-*N*-alkylamino-derivatives with trifluoroacetic acid and 15% hydrogen peroxide gave predominantly 4-aminotetrachloropyridine with very little nitroso-compound. In this case the hydroxylamine (d; Scheme) does not appear to suffer further oxidation under these milder conditions but again undergoes a ' frustrated ' Bamberger hydroxylamine rearrangement [(d) ⟶ (e)] which regenerates the amine.

The 2- and the 4-tetrachloronitropyridine can be prepared in many ways. For instance, the 4-nitro-compound (I; R = NO_2) was produced by treatment of various 4-t-aminopyridines (I; R = NMe_2 or $C_5H_{10}N$)

Products from pentachloropyridine and primary amines (molar ratio 1 : 2) after 18 hr. reflux in dioxan

Amine	4-Isomer (%)	2-Isomer (%)	M.p. (b.p./mm.)	Total yield (%)	Found (%) C	Found (%) H	Found (%) N	Formula	Required (%) C	Required (%) H	Required (%) N
$MeNH_2$	68		130°	77	28·8	1·65	11·3	$C_6H_4Cl_4N_2$	29·3	1·6	11·4
		32	106		29·3	1·8	11·4				
$EtNH_2$	68		69	77	32·6	2·3	10·6	$C_7H_6Cl_4N_2$	32·3	2·3	10·8
		32	61		32·8	2·6	11·2				
Bu^nNH_2 *	71		220/25	82·5	38·0	3·85	9·6	$C_9H_{10}Cl_4N_2$	37·5	3·5	9·7
		29	170/25		37·5	3·6	9·3				
$PhCH_2·NH_2$	73		125	77	44·2	2·85	8·6	$C_{12}H_8Cl_4N_2$	44·7	2·5	8·7
		27	76		44·8	2·3	8·4				

* The isomer ratio reported in the literature (W. T. Flowers, R. N. Haszeldine, and S. A. Majid, *Tetrahedron Letters*, 1967, 2503) is 25% of the 4-isomer and 75% of the 2-isomer.

[3] M. Polonovski and M. Polonovski, *Bull. Soc. Chim. France*, 1927, **47**, 1190; O. Meth-Cohn and H. Suschitzky, *J. Chem. Soc.*, 1963, 4666.

[4] K. M. Ibne-Rasa and J. O. Edwards, *J. Amer. Chem. Soc.*, 1962, **84**, 763; H. O. House, ' Modern Synthetic Reactions,' W. A. Benjamin, New York, 1965, p. 129.

[5] A. Baeyer and V. Villiger, *Ber.*, 1900, **33**, 1569; J. D'Ans and A. Kneip, *Ber.*, 1915, **48**, 1136.

[6] C. K. Ingold, ' Structure and Mechanism in Organic Chemistry,' G. Bell and Sons Limited, London, 1953, p. 621.

[7] I. Collins and H. Suschitzky, unpublished observations.

[8] S. M. Roberts and H. Suschitzky, *Chem. Comm.*, 1967, 893.

[9] W. J. Sell and F. W. Dootson, *J. Chem. Soc.*, 1898, 777.

or of 4-aminotetrachloropyridine with trifluoroacetic acid and 30% hydrogen peroxide under reflux. Oxidation of 4-aminotetrachloropyridine with cold trifluoroacetic acid and 90% hydrogen peroxide also gave the 4-nitro-compound as did a solution of tetrachloro-4-nitrosopyridine with trifluoroacetic and 30% hydrogen peroxide when heated for 10 min. By contrast tetrachloro-2-nitropyridine is obtained under milder conditions namely, by oxidation of the 2-amino-compound (III; R = NH_2) with trifluoroacetic acid and 30% hydrogen peroxide at room temperature.

Anionic replacement of a nitro-group in preference to halogen in chloro- and fluoro-nitro-aromatic compounds is well documented.[10] We also found that the 2-nitro- and the 4-nitro-group in the corresponding tetrachloropyridines is more readily replaced by nucleophiles than any active chlorine atoms. For instance, when tetrachloro-4-nitropyridine and pentachloropyridine were made to compete for piperidine, the 4-piperidino-compound (I; R = $C_5H_{10}N$·) was the only substitution product, thereby revealing the 4-nitropyridine (I; R = NO_2) to be the more reactive compound since pentachloropyridine gives the 2-piperidino-compound. This is noteworthy since the order of reactivity in the corresponding fluoropyridines appears to be the reverse (*i.e.*, $C_5F_5N > C_5F_4N_2O_2$) at least towards methoxide.[11] In our case methoxide would not be useful in determining the relative reactivities of the two pyridines since both would yield the 4-methoxy-compound (I; R = MeO) and the separation of unchanged starting material would present difficulty. When a mixture of the 2- and the 4-nitrotetrachloropyridine were made to react with piperidine in a similar way the nitro-groups were found to be of comparable reactivity.

Tetrachloro-2-nitropyridine (III; R = NO_2) reacted with various nucleophiles ($C_5H_{11}N$, MeO^-, morpholine), with (or without) a solvent with replacement of the nitro-group to give the corresponding 2-substituted tetrachloropyridines. This procedure is obviously the method of choice for directing small nucleophiles into the 2-position since they attack pentachloropyridine almost exclusively in the 4-position.[8,12,13] For instance, tetrachloro-2-methoxypyridine can be made in quantitative yield from the 2-nitro-compound while pentachloropyridine and methoxide gave preferentially the 4-methoxy-isomer.[14]

Similarly, various nucleophiles (piperidine, pyrrolidine, perhydroazepine, MeO^-, or OH^-) reacted with the 4-nitro-isomer (I; R = NO_2) with expulsion of the nitro-group to give the corresponding 4-substituted derivatives. These results are again of wider synthetic interest since they open up a route to 4-substituted tetrachloropyridines with bulky substituents, which react with pentachloropyridine invariably in the 2-position.[2,13] Morpholine behaved differently towards the 4-nitropyridine yielding a mixture of trichloro-2-morpholino-4-nitropyridine (IV; R^1 = Cl, R^2 = morpholino, R^3 = NO_2), and the elusive tetrachloro-4-morpholinopyridine [8] (I; R = morpholino). With a fourfold excess of morpholine a mixture of trichloro-2,4-dimorpholino- (IV; R^1 = Cl, R^2 = R^3 = morpholino) and dichloro-2,6-dimorpholino-4-nitropyridine (IV; R^1 = R^2 = morpholino-, R^3 = NO_2) was obtained.

Apart from the synthetic applications demonstrated above, the chloronitropyridines are also useful for preparing authentic 2- and 4-substituted tetrachloropyridine for structural assignment.

EXPERIMENTAL

Reaction of Pentachloropyridine with Primary Amines.—(*a*) Pentachloropyridine (5·0 g.) and the appropriate primary amine (molar ratio 1 : 3) dissolved in dioxan (150 ml.) were kept under reflux for 18 hr. The cooled solution was diluted with water (650 ml.) and extracted with chloroform several times. The combined extracts were evaporated to dryness. The residue was chromatographed in light petroleum (b.p. 60—80°) on a silica column. The first fractions contained the 2- and later fractions the 4-substituted derivatives. For composition of product mixtures and data of isomers see Table.

(*b*) Pentachloropyridine (10 g.) and n-butylamine (11·7 g.) (*i.e.* a molar ratio 1 : 4) and ethanol (100 ml.) were heated and stirred at 180° in a 1-l. autoclave for 16 hr. The cooled solution was diluted with water and then extracted with chloroform; the extracts were worked up as in (*a*). Elution of the silica column gave first the 2,6-*di*-(*n-butylamino*)-*isomer* (0·4 g.) (Found: C, 48·5; H, 6·2; N, 12·8. $C_{13}H_{20}Cl_3N_3$ requires C, 48·1; H, 6·2; N, 12·9%). Later fractions gave the 2,4-*di*-(*n-butylamino*)-*isomer* (6·3 g.) (Found: C, 48·6; H, 6·3; N, 13·1%).

Reaction of Pentachloropyridine-1-oxide with Primary Amines.—(*a*) Pentachloropyridine-1-oxide [8] (1·0 g.) was dissolved in dioxan and the required primary amine (molar ratio 1 : 2) was added to the solution at room temperature. The reaction mixture was kept for 16 hr. then diluted with water (150 ml.) and extracted with chloroform several times. The combined extracts were evaporated under reduced pressure and the residue was crystallised to give 3,4,5,6-*tetrachloro-2-methylaminopyridine-1-oxide* (75%), m.p. 152° (Found: C, 27·9; H, 1·8; N, 11·1. $C_6H_4Cl_4N_2O$ requires C, 27·5; H, 1·5; N, 10·7%). The 2-*ethylamino-analogue* (85%) had m.p. 115° (Found: C, 30·6; H, 2·45; N, 10·0. $C_7H_6Cl_4N_2O$ requires C, 30·4; H, 2·2; N, 10·15%). Deoxygenation of the 1-oxides occurred practically quantitatively when a chloroform solution (15 ml.) of the 1-oxide (1·0 g.) was heated with phosphorus trichloride (1·0 ml.) for 1 hr. under reflux. The mixture was cooled, water (10 ml.) was added, and the reaction mixture was made alkaline (2N-sodium hydroxide). The chloroform layer was run off, dried and the solvent removed to give the product. T.l.c. of the products showed no 4-substituted isomers.

(*b*) Pentachloropyridine-1-oxide (1·0 g.) and the required amine (molar ratio 1 : 5) were kept as a dioxan solution

[10] V. S. F. Berckmans, A. F. Holleman, *Rec. Trav. Chim.*, 1925, **44**, 851; G. C. Finger, C. W. Kruse, *J. Amer. Chem. Soc.*, 1956, **78**, 6034; Z. Talik, T. Talik, and A. Puszynski, *Roczniki Chem.*, 1965, **39**, 601; T. Talik and Z. Talik, *ibid.*, 1966, **40**, 1187.

[11] R. D. Chambers, J. Hutchinson, and W. K. R. Musgrave, *J. Chem. Soc.* (*C*), 1966, 220.

[12] A. Roedig and K. Grohe, *Chem. Ber.*, 1965, **98**, 923.

[13] W. T. Flowers, R. N. Haszeldine, and S. A. Majid, *Tetrahedron Letters*, 1967, 2503.

[14] A. Roedig, K. Grohe, D. Klatt, *Chem. Ber.*, 1966, **99**, 2818.

under reflux for 16 hr. Extraction with chloroform of the diluted reaction mixture (water) followed by evaporation of the solvent yielded the 2,6-disubstituted products. *3,4,5-Trichloro-2,6-di(methylamino)pyridine-1-oxide* (100%) had m.p. 154° (Found: C, 33·1; H, 3·5; N, 16·0. $C_7H_8Cl_3N_3O$ requires C, 32·75; H, 3·1; N, 16·4%). The *2,6-di(butylamino)-analogue* (65%) had m.p. 47·5° (Found: C, 45·9; H, 6·0; N, 12·1. $C_{13}H_{20}Cl_3N_3O$ requires C, 45·8; H, 5·9; N, 12·3%). Deoxygenation was accomplished as under (*a*) to give *3,4,5-trichloro-2,6-dimethylaminopyridine* (83%), m.p. 127° (Found: C, 34·6; H, 3·2; N, 17·3. $C_7H_8Cl_3N_3$ requires C, 34·9; H, 3·3; N, 17·5%) and the 2,6-di-(n-butylamino)-analogue identical with the specimen obtained above.

Oxidation of 4-t-Aminotetrachloropyridines.—(*a*) The 4-piperidino-compound (I; R = $C_5H_{10}N$) (1·0 g.) was oxidised with trifluoroacetic acid (25 ml.) and 30% hydrogen peroxide (3 ml.) under reflux for 16 hr. The solution was cooled, diluted with water, and extracted with chloroform. The extract was dried and chromatographed on a silica column with benzene to give *2,3,5,6-tetrachloro-4-nitropyridine* (0·51 g., 57%), m.p. 70·5° (ethanol) (Found: C, 22·5; H, 0·0; N, 10·7. $C_5Cl_4N_2O_2$ requires C, 22·9; H, 0·0; N, 10·7%).

(*b*) By a similar procedure in the cold for 24 hr. tetrachloro-4-dimethylaminopyridine (1·5 g.) gave a mixture of tetrachloro-4-nitrosopyridine [2] (0·2 g.), m.p. 181°, tetrachloro-4-methylaminopyridine (*cf.* Table) (0·5 g.), and 4-aminotetrachloropyridine.[9]

Oxidation of 4-s-Aminotetrachloropyridines.—The 4-s-aminopyridine (1·5 g.) was dissolved in chloroform (20 ml.) and trifluoroacetic acid (25 ml.) containing 30% hydrogen peroxide (5 ml.) and was stirred for 16 hr. at room temperature. After this time the insoluble material was filtered off and identified as the 4-nitroso-compound (*ca.* 80%). The filtrate was diluted with water and the chloroform layer was separated and its products chromatographed as described previously. It yielded starting material, a little 4-aminotetrachloropyridine (*ca.* 6%), and a little 4-nitroso-compound.

When oxidation was carried out with 15% hydrogen peroxide under similar conditions the main product was the 4-amino-compound (*ca.* 80%) and some starting material separable on a silica-gel column with light petroleum (b.p. 60—80°) as eluant.

Other Preparations of Tetrachloro-4-nitropyridine.—(*a*) The nitroso-compound was oxidised with boiling trifluoroacetic acid and 30% hydrogen peroxide for 0·5 hr. and the reaction mixture was worked up in the usual way. The yield was 90%.

(*b*) 4-Aminotetrachloropyridine when treated with trifluoroacetic acid and 30% hydrogen peroxide for 16 hr. under reflux gave the nitro-compound (53%).

(*c*) The 4-amino-compound was stirred with trifluoroacetic acid (10 ml.) chloroform (10 ml.) and hydrogen peroxide (90%; 1 ml.) for 16 hr. to give the 4-nitro-compound (65%).

Tetrachloro-2-nitropyridine.—(*a*) Any of the 2-s-amino-compounds (1·0 g.) listed in the Table were oxidised in a mixture of chloroform (20 ml.), trifluoroacetic acid (20 ml.), and 30% hydrogen peroxide (5 ml.) at room temperature for 16 hr. The work up was similar to that described for other oxidations and yielded the *2-nitropyridine* (*ca.* 75%), m.p. 139° (ethanol) (Found: C, 23·2; H, 0·0; N, 10·7. $C_5Cl_4N_2O_2$ requires C, 22·9; H, 0·0; N, 10·7%).

(*b*) 2-Aminotetrachloropyridine [7] was oxidised as described in (*a*) to give tetrachloro-2-nitropyridine (70%).

Relative Reactivity of Tetrachloro-4-nitro- and Pentachloro-pyridine.—To a mixture of the nitro-compound (1·0 g.) and pentachloropyridine (1·0 g.) (molar ratio 1 : 1·05) in dry benzene (50 ml.) was added a molar deficiency of piperidine (0·85 ml.; 1 mol.) in benzene and the resultant mixture was kept at room temperature for 6 hr. The benzene mixture was then washed with water to remove piperidine hydrochloride. The benzene layer was dried, the solvent was removed and the residue was chromatographed in light petroleum (b.p. 60—80°) on a silica column. Nearly all pentachloropyridine (95%) was recovered together with tetrachloro-4-piperidinopyridine (1·0 g.; 88% based on nitropyridine). No tetrachloro-2-piperidinopyridine, the expected product from pentachloropyridine, was detectable by t.l.c. The 4-nitro-compound is thus considerably more reactive.

Relative Reactivity of the Tetrachloro-2-nitro- and -4-nitropyridine.—By a similar experiment it was found that the 2-nitro- and the 4-nitro-tetrachloropyridine are of comparable reactivity towards piperidine.

Nucleophilic Substitution of Tetrachloro-4-nitropyridine.—(*a*) The 4-nitro-compound (1·0 g.) dissolved in benzene (25 ml.) was made to react with various nucleophiles (*ca.* 1 mol.) at room temperature for 12 hr. The benzene solution was extracted with water to remove unchanged nucleophile and any salt formed and the benzene layer was separated and worked up. Yields of the substitution products in brackets were as follows: 4-piperidino- (90%), 4-pyrrolidino- (70%), and 4-perhydroazepino- (42%).

(*b*) The 4-nitro-compound (0·54 g.) was made to react with sodium (0·07 g.) dissolved in methanol (50 ml.) under reflux for 10 hr. The mixture was evaporated to dryness and the residue was extracted with ether. From the extracts tetrachloro-4-methoxypyridine [12] (75%), m.p. 116°, was obtained. A similar experiment with sodium hydroxide (0·2 g.) in ethanol (25 ml.) carried out under reflux for 16 hr. gave tetrachloro-4-hydroxypyridine [15] (80%), m.p. 233°.

(*c*) A benzene solution (35 ml.) of the 4-nitro-compound (0·5 g.) and morpholine (0·33 g.) corresponding to a molar ratio 1 : 2 was kept under reflux for 16 hr. The reaction mixture was worked up as under (*a*) and chromatographed on a silica column with benzene to give *tetrachloro-4-morpholinopyridine* (0·21 g.), m.p. 118° (Found: C, 35·5; H, 2·8; N, 9·2. $C_9H_8Cl_4N_2O$ requires C, 35·8; H, 2·65; N, 9·3%) and *3,5,6-trichloro-2-morpholino-4-nitropyridine* (0·18 g.), m.p. 107° (Found: C, 34·8; H, 2·5; N, 12·95. $C_9H_8Cl_3N_3O_3$ requires C, 34·6; H, 2·6; N, 13·4%). It reacted with piperidine in dioxan to give the known trichloro-2-morpholino-4-piperidinopyridine (80%) which confirmed structural assignment.

With a molar ratio of nitro-compound to morpholine of 1 : 4 and a reflux period of 72 hr. the products obtained as above were *3,5,6-trichloro-2,4-dimorpholinopyridine* (24%), m.p. 131° (Found: C, 44·2; H, 4·5; N, 12·0. $C_{13}H_{16}Cl_3N_3O_2$ requires C, 44·25; H, 4·5; N, 11·9%) and *3,5-dichloro-2,6-dimorpholino-4-nitropyridine* (32%), m.p. 224° (Found: C, 43·0; H, 4·4; N, 15·25. $C_{13}H_{16}Cl_2N_4O_4$ requires C, 43·0; H, 4·4; N, 15·4%). The latter compound on being heated with sodium ethoxide in ethanol for 6 hr. gave the same product as was obtained from the known trichloro-2,6-dimorpholinopyridine [2] and sodium ethoxide

[15] H. J. Den Hertog, J. Maas, C. R. Kolder, W. P. Combé, *Rec. Trav. chim.*, 1953, **74**, 63.

in ethanol, namely, *dichloro-4-ethoxy-2,6-dimorpholinopyridine*, m.p. 136° (Found: C, 49·6; H, 5·9; N, 11·4. $C_{15}H_{21}Cl_2N_3O_3$ requires C, 49·7; H, 5·8; N, 11·6%) thus establishing the structure.

Nucleophilic Substitution of Tetrachloro-2-nitropyridine.—(*a*) The 2-nitro-compound was made to react with piperidine and morpholine under identical conditions to those described for the 4-nitro-isomer under (*a*). 2-Piperidino- and 2-morpholino-tetrachloropyridine were obtained in 60% yield.

(*b*) Reaction with sodium methoxide in methanol was as for the 4-nitro-compound [*cf.* (*b*) above] and gave a quantitative yield of *tetrachloro-2-methoxypyridine*, m.p. 91° (Found: C, 29·0; H, 1·15; N, 5·6. $C_6H_3Cl_4NO$ requires C, 29·05; H, 1·2; N, 5·7%).

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