770. *A Series of ω-Dimethylaminoalkylphenols.*

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The first four members of the series 4-chloro-2-(ω-dimethylamino-*n*-alkyl)-3 : 5-dimethylphenol have been prepared.

It was desired to study the effect on germicidal properties of introducing a quaternary nitrogen atom into a phenolic germicide, using *n*-alkylene groups of various lengths to separate the nitrogen atom from the phenol nucleus. 4-Chloro-3 : 5-dimethylphenol was chosen as the phenol, on account of its ease of preparation and high germicidal activity. The more active 2 : 4-dihalogenated phenols were not used, as 2 : 4 : 6-trisubstitution leads to a marked loss of activity against many micro-organisms.

Apart from the dimethylaminomethyl compounds,[1] few phenols substituted by a

[1] B.P. 458,033/1936.

straight alkyl chain with a tertiary nitrogen atom on the terminal carbon atom have been described. von Braun [2] prepared the higher homologues of hordenine, 4-2′-dimethylaminoethylphenol, with three, four, and five carbon atoms in the alkylene chain, using starting materials obtained by degradation of piperidine derivatives.[3] The *ortho*-isomer of hordenine has also been prepared by von Braun.[2]

We have prepared 4-chloro-2-dimethylaminomethyl-3 : 5-dimethylphenol by the Mannich reaction. For the three higher homologues the successful routes involved the reduction of the dimethylamide of an ω-carboxyalkyl derivative.

The 2-dimethylaminoethyl analogue was made by chloromethylation of 4-chloro-3 : 5-dimethylanisole, conversion into the cyanomethyl compound, hydrolysis to the phenylacetic acid, then reduction of the dimethylamide of this acid by lithium aluminium hydride, followed by demethylation. An attempt to prepare 4-chloro-2-chloroacetyl-3 : 5-dimethylphenol (for conversion into 4-chloro-2-dimethylaminoacetyl-3 : 5-dimethylphenol) by Fries rearrangement of 4-chloro-3 : 5-dimethylphenyl chloroacetate failed, cyclisation to the coumaranone taking place. 4-Chloro-2-dimethylaminoacetyl-3 : 5-dimethylanisole was then prepared but Wolff–Kishner reduction of this ketone failed. An attempt to prepare the phenylacetic acid by an azlactone synthesis from 4-chloro-2-formyl-3 : 5-dimethylphenol also failed, cyclisation to the acetamidocoumarin occurring (cf. Shaw, McMillan, and Armstrong [4]). Application of the azlactone synthesis to 4-chloro-2-formyl-3 : 5-dimethylanisole was then considered, but the yield of the latter was very low.

The 3-dimethylaminopropyl analogue was prepared from the β-arylpropionic acid made by decarboxylation of the benzylmalonic acid obtained from the chloromethylated anisole. Here, also, preparation *via* the dimethylaminoacyl derivative was attempted, but the β-chloropropionate of 4-chloro-3 : 5-dimethylphenol also cyclises under Fries rearrangement conditions, the product being identical with the chromanone prepared from ethyl β-(4-chloro-3 : 5-dimethylphenoxy)propionate.

The 4-dimethylaminobutyl analogue was prepared from the γ-arylbutyric acid obtained by Clemmensen reduction of the reaction product of 4-chloro-3 : 5-dimethylanisole and succinic anhydride. In a minor variation, the γ-arylbutyric acid was demethylated before conversion into the dimethylamide and subsequent reduction. It was here noticed that demethylation with hydriodic acid removed the nuclear chlorine atom and pyridine hydrochloride was afterwards used for all demethylations. Reduction of the methoxy-acid (after esterification) to the 4-arylbutanol was also carried out. Demethylation of this by pyridine hydrochloride is accompanied by introduction of a pyridinium quaternary nitrogen atom on the terminal carbon atom of the alkyl chain. Demethylation of this alcohol by hydrobromic acid failed, the only reaction being formation of the bromide.

4-Chloro-2-dimethylaminomethyl-3 : 5-dimethylphenol readily formed a crystalline quaternary salt with methyl iodide but not with benzyl chloride. Its 3-dimethylaminopropyl homologue did not readily form a crystalline salt with either reagent. The 2-dimethylaminoethyl and 4-dimethylaminobutyl derivatives, however, form crystalline derivatives rapidly with both these halides. This alternation, which is presumably due to variation of the degree of interaction between the nitrogen atom and the phenolic hydroxyl group, is reflected in the melting points of the four amines.

These amines and their quaternary salts showed poor to moderate antibacterial activity *in vitro*, the activity increasing with the length of the alkyl group. The test organisms were *B. mycoides*, *S. aureus*, and *B. coli*.

Experimental

4-Chloro-2-dimethylaminomethyl-3 : 5-dimethylphenol.—4-Chloro-3 : 5-dimethylphenol (31·2 g.), dimethylamine (60 ml.; 15% aqueous solution), and water (30 ml.) were stirred at 20°.

[2] von Braun and Deutsch, *Ber.*, 1912, **45**, 2504.
[3] von Braun, *ibid.*, 1910, **43**, 2837.
[4] Shaw, McMillan, and Armstrong, *J. Org. Chem.*, 1956, **21**, 601.

Aqueous formaldehyde (15 g.; 40%) was run in during 15 min., a slight temperature rise occurring. After 2 hours' stirring, the mixture was heated for 1 hr. at 95°. After cooling, the sticky solid was filtered off, purified by reprecipitation from dilute acid, and isolated in ether. The crude *base* (40·5 g.) recrystallised from methanol (50 ml.) as needles (35·3 g.), m. p. 65—66° (Found: C, 61·3; H, 7·6. $C_{11}H_{16}ONCl$ requires C, 61·8; H, 7·5%).

3-*Chloro*-6-*methoxy*-2 : 4-*dimethylbenzyl Chloride*.—A mixture of 4-chloro-3 : 5-dimethylanisole (17 g.), paraformaldehyde (3·5 g.), and 32% hydrochloric acid (50 ml.) was shaken for 48 hr. at 22°. The sticky *chloride* (18·4 g.) was filtered off, washed with water, and crystallised from light petroleum (b. p. 60—80°), forming prisms (15·7 g.), m. p. 91—92° (Found: C, 54·8; H, 5·4. $C_{10}H_{12}OCl_2$ requires C, 54·4; H, 5·4%).

3-*Chloro*-6-*methoxy*-2 : 4-*dimethylbenzyl Cyanide*.—The foregoing benzyl chloride (6·6 g.) in hot ethanol (7 ml.) was added to boiling water (2 ml.) containing sodium cyanide (2 g.). The mixture was boiled for 2 hr., then diluted with water, and the reddish, sticky *cyanide* (6·1 g.) filtered off, washed with water, and crystallised from light petroleum (b. p. 60—80°), forming off-white needles (3·6 g.), m. p. 120—122° (Found: C, 63·0; H, 5·8. $C_{11}H_{12}ONCl$ requires C, 63·0; H, 5·7%).

3-*Chloro*-6-*methoxy*-2 : 4-*dimethylphenylacetic Acid*.—The cyanide (2·2 g.) was boiled for 1½ hr. with acetic acid (3 ml.), concentrated sulphuric acid (3 ml.), and water (3 ml.). A clear solution was obtained at one stage of the heating but two phases later separated. On addition of water (10 ml.), the *arylacetic acid* (2·2 g.) readily solidified. It formed stout prisms (from toluene), m. p. 164—166° (Found: C, 57·7; H, 5·5. $C_{11}H_{13}O_3Cl$ requires C, 57·7; H, 5·5%).

3-*Chloro*-6-*methoxy*-2 : 4-*dimethylphenyl*-NN-*dimethylacetamide*.—The above acid (2·3 g.) was heated with thionyl chloride (5 ml.) in boiling benzene (20 ml.) for 1 hr. The residue after removal of benzene and excess of chloride was added in benzene (10 ml.) to dimethylamine solution (15 ml.; 12% in benzene). After 16 hr. water was added. Crystallisation of the residue (2·6 g.) from the benzene layer from light petroleum (b. p. 60—80°) gave the *dimethylamide* as prisms, m. p. 130—131° (Found: C, 61·4; H, 7·1. $C_{13}H_{18}O_2NCl$ requires C, 61·1; H, 7·0%).

4-*Chloro*-2-2′-*dimethylaminoethyl*-3 : 5-*dimethylphenol*.—The above dimethylamide (1·9 g.), in partial solution in ether (50 ml.) was added to lithium aluminium hydride (0·32 g.) in ether (25 ml.). After 1 hr. at room temperature the mixture was boiled for a further hour, then decomposed with 2% aqueous sodium hydroxide (9 ml.). The oily amine (2 g.) was isolated by means of ether.

The amine (1·5 g.) was heated at 180—190° for 3 hr. with pyridine hydrochloride (5 g.). 10% Aqueous sodium carbonate (50 ml.) was then added and the sand-coloured solid (1·3 g.), m. p. 90—120°, filtered off and recrystallised from light petroleum (b. p. 60—80°), to give 4-*chloro*-2-2′-*dimethylaminoethyl*-3 : 5-*dimethylphenol* (0·7 g.). It formed large, off-white prisms, m. p. 130—132°, from aqueous methanol (Found: C, 62·7; H, 7·6. $C_{12}H_{18}ONCl$ requires C, 63·3; H, 7·9%).

5-*Chloro*-4 : 6-*dimethylcoumaranone*.—4-Chloro-3 : 5-dimethylphenol (144·5 g.) and chloroacetyl chloride (82·7 g.) were heated at 95° for 10 hr. After 2 days the product was dissolved in ether (500 ml.) and washed with alkali. The residue (108 g.) from the ether formed white needles (82·3 g.), m. p. 50—52°, from light petroleum (b. p. 60—80°). This chloroacetate (25 g.) was heated with powdered aluminium chloride (5 g.) quickly to 120° and then from 120° to 155° during 10 min. The product was decomposed with ice and hydrochloric acid, and the sticky solid (4·2 g.) filtered off. 5-*Chloro*-4 : 6-*dimethylcoumaranone* (3·5 g.) separated in pale-yellow needles, m. p. 137—140°, from light petroleum (b. p. 60—80°) (Found: C, 60·9; H, 4·2; Cl, 18·1. $C_{10}H_9O_2Cl$ requires C, 61·1; H, 4·6; Cl, 18·1%). The compound gave an orange-red precipitate with alcoholic 2 : 4-dinitrophenylhydrazine sulphate and did not contain readily hydrolysable chlorine.

4-*Chloro*-2-*chloroacetyl*-3 : 5-*dimethylanisole*.—4-Chloro-3 : 5-dimethylanisole (5·7 g.) and chloroacetyl chloride (3·8 g.) in carbon disulphide (40 ml.) were stirred at 0° while aluminium chloride (2·5 g.) was added during 10 min. After 2 hr. at 0°, the mixture was allowed to warm to room temperature. Ice (15 g.) and 32% hydrochloride acid (10 ml.) were added. Extraction with ether (60 ml.) gave 4-*chloro*-2-*chloroacetyl*-3 : 5-*dimethylanisole* (2·3 g.), m. p. 130—131°, which formed colourless plates, m. p. 133—135°, from benzene (Found: C, 53·6; H, 4·8. $C_{11}H_{12}O_2Cl_2$ requires C, 53·4; H, 4·9%).

4-*Chloro*-2-*dimethylaminoacetyl*-3 : 5-*dimethylanisole*.—The above chloro-ketone (20 g.) was

mixed with a cold 18% solution of dimethylamine in benzene (80 ml.). After 4 days at room temperature, excess of hydrochloric acid was added and the product liberated from the acid extracts with dilute alkali as a brown oil (17·5 g.). A sample (1·3 g.) was treated with saturated methanolic hydrogen chloride (2 ml.); *4-chloro-2-dimethylaminoacetyl-3 : 5-dimethylanisole hydrochloride* (0·7 g.) separated in prisms, m. p. 210° to 225° according to the rate of heating (Found: C, 54·0; H, 6·6; N, 4·6. $C_{13}H_{19}O_2NCl_2$ requires C, 53·5; H, 6·5; N, 4·8%).

3-Acetamido-6-chloro-5 : 7-dimethylcoumarin.—A mixture of glycine (0·8 g.), acetic anhydride (1 ml.), and acetic acid (2 ml.) was gently boiled for 3 min. 4-Chloro-2-formyl-3 : 5-dimethylphenol (1·8 g.) [5] was then added at 95°, followed by acetic anhydride (3 ml.) and sodium acetate (0·8 g.). After 2 hr. at 95°, the crystalline *acetamidocoumarin* (0·1 g.) was filtered off and recrystallised from dioxan, forming needles, subliming above 330° (Found: C, 58·4; H, 4·7. $C_{13}H_{12}O_3NCl$ requires C, 58·5; H, 4·5%). The compound did not dissolve readily in boiling 2N-sodium hydroxide.

4-Chloro-2-formyl-3 : 5-dimethylanisole.—4-Chloro-3 : 5-dimethylanisole (85·2 g.), *N*-methylformanilide (96 g.), and phosphorus oxychloride (102·3 g.) were warmed for 12 hr. at 95°. The bulk of the anisole was unchanged but a little aldehyde was extracted with aqueous sodium hydrogen sulphite. *4-Chloro-2-formyl-3 : 5-dimethylanisole* formed pale yellow needles (1·3 g.), m. p. 106—107°, from methanol (Found: C, 60·4; H, 5·8. $C_{10}H_{11}O_2Cl$ requires C, 60·5; H, 5·5%).

3-Chloro-6-methoxy-2 : 4-dimethylbenzylmalonic Acid.—Sodium (0·6 g.) was stirred in boiling toluene (30 ml.) while ethyl malonate (4·2 g.) was added during 20 min. The mixture was cooled and 3-chloro-6-methoxy-2 : 4-dimethylbenzyl chloride (5·7 g.) in toluene (20 ml.) added during 30 min. After 2 hr. at room temperature, the mixture was boiled for 1 hr. Water (25 ml.) was added and the toluene layer separated and evaporated, to give the substituted malonic ester (8·2 g.), forming prisms, m. p. 77—78°, from light petroleum (b. p. 60—80°). The ester (8 g.) was heated in methanol (15 ml.) with a solution of sodium hydroxide (8 g.) in water (30 ml.), at 95° for 2 hr. Hydrochloric acid (16%; 40 ml.) was then added and the mixture boiled for 10 min. This *malonic acid* (6·6 g.) formed sheaves of needles, m. p. 166—168° (decomp.), from a large volume of water (Found: C, 54·1; H, 5·0. $C_{13}H_{15}O_5Cl$ requires C, 54·4; H, 5·2%).

β-(3-Chloro-6-methoxy-2 : 4-dimethylphenyl)propionic Acid.—The crude malonic acid (5 g.) was heated at 180—190° for 1 hr. and the decarboxylated product heated with 10% aqueous sodium hydroxide (20 ml.). Neutral material which was extracted into benzene gave off-white prisms, m. p. 130—131°, from methanol (Found: C, 61·7; H, 5·9; Cl, 17·8%). Acidification of the alkaline liquors gave the *arylpropionic acid* (2·5 g.), which forms prisms, m. p. 116—117°, from benzene (Found: C, 59·8; H, 6·1. $C_{12}H_{15}O_3Cl$ requires C, 59·4; H, 6·2%).

The *dimethylamide*, made in the usual way, formed off-white prisms, m. p. 104—105°, from light petroleum (b. p. 60—80°) (Found: C, 62·6; H, 7·2. $C_{14}H_{20}O_2NCl$ requires C, 62·3; H, 7·4%).

4-Chloro-2-3'-dimethylaminopropyl-3 : 5-dimethylphenol.—This *phenol* (1 g.) was prepared from the above dimethylamide (1·5 g.) as described for the 2-dimethylaminoethyl compound. It formed pale cream prisms, m. p. 100—102°, from light petroleum (Found: C, 64·1; H, 8·3. $C_{13}H_{20}ONCl$ requires C, 64·6; H, 8·3%).

6-Chloro-5 : 7-dimethylchromanone.—(*a*) 4-Chloro-3 : 5-dimethylphenol (192·6 g.) and β-chloropropionyl chloride (156·3 g.) were heated for 12 hr. at 95°. The resulting brown oil was dissolved in ether (700 ml.) and washed with alkali. Evaporation of the ether gave the crude β-chloropropionate (269 g.) which formed prisms (206·4 g.), m. p. 51—52°, from light petroleum (b. p. 60—80°).

This ester (10 g.) was heated with powdered aluminium chloride (10 g.) at 130° for 1 hr. The mixture was decomposed with ice (40 g.) and 32% hydrochloric acid (40 ml.). The semisolid product was isolated in ether and distilled, to give a yellow oil (3·6 g.), b. p. 124—127°/1 mm., which largely solidified. This *chromanone* formed prisms, m. p. 70—71°, from light petroleum (b. p. 60—80°) (Found: C, 62·8; H, 5·4. $C_{11}H_{11}O_2Cl$ requires C, 62·7; H, 5·2%).

(*b*) 4-Chloro-3 : 5-dimethylphenol (78·3 g.), ethyl acrylate (50 g.), and sodium 4-chloro-3 : 5-dimethylphenoxide (4·3 g.) were heated at 95° for 40 hr. The product was poured into water (100 ml.) containing acetic acid (0·5 ml.), and the liberated oil was isolated in ether and fractionally distilled. The fraction, b. p. 123—140°/0·2 mm. (44·3 g.), was collected and recrystallised from light petroleum (b. p. 60—80°), forming prisms (32·5 g.), m. p. 46—49°. This

[5] Duff, *J.*, 1941, 549.

ethyl β-(4-chloro-3 : 5-dimethylphenoxy)propionate (2 g.) was heated for 1 hr. at 95° with concentrated sulphuric acid (10 ml.). The solid obtained after dilution with water recrystallised from ethanol as prisms (0·7 g.), m. p. 68—69° undepressed on admixture with the product from (*a*) (Found: C, 63·2; H, 5·3%). The 2 : 4-*dinitrophenylhydrazone* formed orange spangles, m. p. 265°, from acetic acid (Found: C, 51·9; H, 3·7. $C_{17}H_{15}O_5N_4Cl$ requires C, 52·2; N, 3·8%).

β-(3-*Chloro*-6-*methoxy*-2 : 4-*dimethylbenzoyl*)*propionic Acid.*—A solution of 4-chloro-3 : 5-dimethylanisole (152·8 g.) and succinic anhydride (91 g.) in nitrobenzene (1100 ml.) was stirred at 0° while aluminium chloride (244 g.) was added during 3 hr. at 0—10°. Next day the clear, dark yellow solution was treated with ice (2 kg.) and 32% hydrochloric acid (1 l.). The off-white solid (201·7 g.) was filtered off and washed with water and ether. The dried *acid*, m. p. 172—180°, was pure enough for the next step. Recrystallised from toluene, then aqueous ethanol, it formed colourless prisms, m. p. 178—181° (Found: C, 57·2; H, 5·7. $C_{13}H_{15}O_4Cl$ requires C, 57·7; H, 5·5%).

γ-(3-*Chloro*-6-*methoxy*-2 : 4-*dimethylphenyl*)*butyric Acid.*—The foregoing benzoylpropionic acid (11 g.) was added to zinc amalgam (22 g.), water (16 ml.) and 32% hydrochloric acid (44 ml.), and the mixture boiled for 12 hr. (Weaker acid is unsatisfactory.) The crude *butyric acid* (10·8 g.) was recovered from the liquors and from the amalgam by repeated ether-extractions and formed plates (7·8 g.), m. p. 117—118° (from aqueous methanol) (Found: C, 60·8; H, 6·6. $C_{13}H_{17}O_3Cl$ requires C, 60·8; H, 6·6%). The *dimethylamide* was made in the usual way from the acid (4 g.) and formed hexagonal plates (2·6 g.), m. p. 83—86°, from ether (Found: C, 63·2; H, 7·6. $C_{15}H_{22}O_2NCl$ requires C, 63·5; H, 7·8%).

4-*Chloro*-2-4′-*dimethylaminobutyl*-3 : 5-*dimethylphenol.*—By the usual methods, the above dimethylamide (2·6 g.) gave successively 4-chloro-2-4′-dimethylaminobutyl-3 : 5-dimethylanisole as a colourless oil (2·3 g.), and the corresponding *phenol* (2·0 g.), m. p. 154—158°.

A sample of the latter was recrystallised from benzene and then from aqueous ethanol, whence it formed prisms, m. p. 159—160° (Found: C, 65·9; H, 8·7. $C_{14}H_{22}ONCl$ requires C, 65·8; H, 8·6%).

γ-(3-*Chloro*-6-*hydroxy*-2 : 4-*dimethylphenyl*)*butyric Acid.*—Demethylation of γ-(3-chloro-6-methoxy-2 : 4-dimethylphenyl)butyric acid (2 g.) with pyridine hydrochloride gave the corresponding *hydroxy-acid* (1·7 g.), m. p. 145—150°. It formed leaflets (1·4 g.), m. p. 151—153°, from toluene (Found: C, 59·8; H, 6·3. $C_{12}H_{15}O_3Cl$ requires C, 59·4; H, 6·2%).

Heating a mixture of the acid (10 g.) with 30% aqueous dimethylamine (30 ml.) gradually to 190° and keeping the mixture at this temperature for 1½ hr. gave the *dimethylamide*, off-white prisms (4·4 g.), m. p. 182—183° (from methanol) (Found: C, 62·2; H, 7·6. $C_{14}H_{20}O_2NCl$ requires C, 62·3; H, 7·4%).

Extraction of this dimethylamide (1·4 g.) from a Soxhlet apparatus into lithium aluminium hydride (0·26 g.) and boiling ether gave 4-chloro-2-4′-dimethylaminobutyl-3 : 5-dimethylphenol (0·9 g.), m. p. and mixed m. p. 159—160°.

γ-(2-*Hydroxy*-4 : 6-*dimethylphenyl*)*butyric Acid.*—γ-(3-Chloro-6-methoxy-2 : 4-dimethylphenyl)butyric acid (5·7 g.) was boiled for 15 min. with 66% hydriodic acid (60 ml.) and water (10 ml.). On cooling, γ-(2-*hydroxy*-4 : 6-*dimethylphenyl*)*butyric acid* (4·2 g.) separated. It formed leaflets, m. p. 130—132°, from toluene (Found: C, 68·9; H, 7·4. $C_{12}H_{16}O_3$ requires C, 69·2; H, 7·7%).

The *dimethylamide* (0·4 g.), made by heating the acid (1 g.) with aqueous dimethylamine as described above for the chloro-analogue, formed off-white needles, m. p. 179—181°, from methanol (Found: C, 71·2; H, 8·9; N, 6·1. $C_{14}H_{21}O_2N$ requires C, 71·4; H, 8·9; N, 6·0%). This reaction probably proceeds *via* the lactone of the hydroxy-acid. When acetylation of the hydroxy-acid was attempted, lactonic material was the main product; with cold aqueous dimethylamine this gave a mixture of the above dimethylamide and the dimethylamine salt of the hydroxy-acid.

Methyl γ-(3-*Chloro*-6-*methoxy*-2 : 4-*dimethylphenyl*)*butyrate.*—The methoxy-acid (5 g.) was heated in boiling methanol (50 ml.) for 5 hr. with concentrated sulphuric acid (0·5 ml.). The *methyl ester* (4 g.), isolated in the usual way, formed needles, m. p. 41°, from light petroleum (b. p. 40—60) (Found: C, 61·9; H, 6·9. $C_{14}H_{19}O_3Cl$ requires C, 62·1; H, 7·0%).

4-(3-*Chloro*-6-*methoxy*-2 : 4-*dimethylphenyl*)*butan*-1-*ol.*—The foregoing methyl ester (3·9 g.) in ether (20 ml.) was added during 15 min. to lithium aluminium hydride (0·53 g.) in ether (40 ml.). After 1 hr. at room temperature, the mixture was boiled for 2 hr., then cooled, and

3% aqueous sodium hydroxide (2·5 ml.) was added. The ether solution was filtered and on evaporation yielded the *butanol* (3·5 g.) which formed waxy needles, m. p. 61°, from light petroleum (b. p. 60—80°) (Found: C, 64·8; H, 7·8. $C_{13}H_{19}O_2Cl$ requires C, 64·3; H, 7·8%). Its p-*nitrobenzoate* formed pale yellow prisms, m. p. 89—91°, from ethanol (Found: C, 61·4; H, 5·7. $C_{20}H_{22}O_5NCl$ requires C, 61·3; H, 5·6%).

The alcohol (2 g.) was boiled with 48% hydrobromic acid (10 ml.) for 2 hr. The dark oil was extracted into ether. Alkali removed only traces of phenols. The residue from the ether crystallised on trituration with light petroleum to a grey solid (0·6 g.), m. p. 105—115°. This *bromide* separated in colourless needles, m. p. 124°, from benzene (Found: C, 48·1; H, 5·6. $C_{13}H_{18}OClBr$ requires C, 48·9; H, 5·7%).

1-[4-(3-*Chloro*-6-*hydroxy*-2 : 4-*dimethylphenyl*)*butyl*]*pyridinium Chloride*.—4-(3-Chloro-6-methoxy-2 : 4-dimethylphenyl)butanol (1 g.) was heated at 190° with pyridine hydrochloride (2·5 g.) for 5 hr. The mixture was dissolved in warm methanol (3 ml.). On cooling, prisms (0·35 g.), m. p. 186—190°, of the *quaternary salt* separated (Found: C, 62·8; H, 6·6. $C_{17}H_{21}ONCl_2$ requires C, 62·6; H, 6·4%).

4-(3-*Chloro*-6-*hydroxy*-2 : 4-*dimethylphenyl*)*butan*-1-*ol*.—γ-(3-Chloro-6-hydroxy-2 : 4-dimethylphenyl)butyric acid (1·9 g.) in partial solution in ether (20 ml.) was added to lithium aluminium hydride (0·7 g.) in ether (60 ml.). After 16 hr. the mixture was boiled for 2 hr., then treated with aqueous alkali in the usual way. The *phenolic alcohol* (1 g.) formed needles, m. p. 105—106°, from light petroleum (b. p. 80—100°) (Found: C, 63·0; H, 7·9. $C_{12}H_{17}O_2Cl$ requires C, 63·0; H, 7·4%).

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