

### 229. Some Derivatives of 3-Aminoquinoline.

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Certain derivatives of 3-aminoquinoline, having the 2-position either unsubstituted or substituted by methyl or phenyl, and 6:7-positions either unsubstituted or substituted by dimethoxy or methylenedioxy have been prepared. The hydrochlorides and methochlorides of these derivatives have been subjected to biological tests. Certain 2-styryl derivatives of 3-aminoquinoline quaternary salts have also been prepared.

ALTHOUGH many derivatives of quinoline have useful chemotherapeutic properties, derivatives of 3-aminoquinoline have been little investigated. We therefore thought it useful to prepare a series of such compounds in which the 2-position was either unsubstituted or substituted by methyl or phenyl, and the 6:7-positions were either unsubstituted or substituted by dimethoxy or methylenedioxy and to subject the hydrochlorides and methochlorides of these bases to chemotherapeutic tests.

Also, in view of the antiseptic properties of many styryl derivatives of quinoline, certain styryl derivatives of 3-aminoquinoline have been prepared.

3-Aminoquinoline is best prepared either by the action of ammonia on 3-bromoquinoline (Renshaw and Friedman, *J. Amer. Chem. Soc.*, 1939, **61**, 3320) or by the reduction of 3-nitroquinoline (*loc. cit.*) obtained either by the condensation of *o*-aminobenzaldehyde with methazonic acid (G.P. 335,197) or from 2-nitro-2-formyl-ethylideneaniline (Uhle and Jacobs, *J. Org. Chem.*, 1945, **10**, 76). 3-Nitro-6:7-dimethoxy- and -6:7-methylenedioxy-quinolines have been obtained by condensing methazonic acid with 6-aminoveratraldehyde and 6-aminopiperonal, respectively. The reduction of the latter proceeded smoothly to the corresponding 3-amino compound; but the reduction of the former was less satisfactory.

3-Amino-2-phenylquinoline was prepared by the method of Petrow, Stack, and Wragg (*J.*, 1943, 316). Attempts to prepare 3-phenylaminoquinoline by the condensation of 3-aminoquinoline with bromobenzene, of 3-bromoquinoline or 3-hydroxyquinoline with aniline, or of 3-aminoquinoline hydrochloride with aniline were unsuccessful. Attempts to prepare 3-methylaminoquinoline by condensing 3-aminoquinoline with *p*-toluenesulphonyl chloride followed by methylation and hydrolysis failed on account of the difficulty of the

final hydrolysis. Substitution of mesitylenesulphonyl chloride in place of *p*-toluenesulphonyl chloride gave only slightly more promising results, so this line was not pursued further (cf. Cocker, *J.*, 1937, 1695).

3-Aminoquinaldine was prepared by the method of Lawson, Perkin, and Robinson (*J.*, 1924, 125, 634) and the corresponding 6:7-methylenedioxy derivative by that of Berlingozzi and Napolitano (*Atti R. Accad. Lincei*, 1923, 32, 339; *Chem. Zentr.*, 1924, I, 1383) by condensing 6-aminopiperonal with acetylphthalimide, followed by hydrolysis. 3-Amino-6:7-dimethoxyquinaldine was similarly prepared from 6-aminoveratraldehyde.

Di-(3-quinaldyl)urea was prepared by the action of phosgene on 3-aminoquinaldine (Stark and Hofmann, *Ber.*, 1913, 46, 2697). Di-(3-quinolyl)urea has been prepared by the interaction of 3-aminoquinoline hydrochloride and urea in aqueous solution.

The conversion of all the above bases into the methochlorides was accomplished smoothly by the usual methods, with the exception of the urea derivatives.

3-Dimethylaminoquinaldine methiodide was obtained by the interaction of 3-aminoquinaldine with methyl iodide (3 mols.) and sodium carbonate (1 mol.); and this was converted into the methochloride. The methiodide condensed readily in alcoholic solution in the presence of piperidine, with *p*-dimethylaminobenzaldehyde and *p*-acetamidobenzaldehyde to give the corresponding styryl compounds. A styryl compound was also prepared by condensing *p*-dimethylaminobenzaldehyde with 3-acetamidoquinaldine methosulphate, followed by hydrolysis.

By the oxidation of 3-acetamidoquinaldine with selenium dioxide, 3-acetamidoquinoline-2-aldehyde was obtained (cf. Kaplan, *J. Amer. Chem. Soc.*, 1941, 63, 2654). Attempts to convert this into derivatives of 2:3:3':2'-pyridoquinoline met with little success.

The hydrochlorides and methochlorides were tested against *Trypanosome rhodesiense*, *T. congolense*, and *T. cruzi*; in no case was there any activity, except in that of 3-amino-2-*p*-dimethylaminostyrylquinoline methochloride which had a very slight activity against *T. rhodesiense*, but no action against *T. congolense* or *T. cruzi*.

#### EXPERIMENTAL.

**3-Nitro-6:7-methylenedioxyquinoline.**—6-Aminopiperonal (4.5 g.) was dissolved in hot alcohol (25 c.c.), the solution rapidly cooled to room temperature, and treated with a solution of methazonic acid (4.5 g.) (Steinkopf, *Ber.*, 1909, 42, 2031) in alcohol (7 c.c.) and concentrated hydrochloric acid (0.7 c.c.). After standing for 2 days at room temperature, the red solid was separated, washed with alcohol, and recrystallised from methanol-acetone (charcoal), giving 3.8 g., m. p. 188–190° (Found: C, 55.5; H, 2.9.  $C_{10}H_8O_4N_2$  requires C, 55.1; H, 2.8%).

**3-Amino-6:7-methylenedioxyquinoline.**—Crystalline sodium sulphide (10.8 g.) was dissolved in water (10.8 c.c.), and concentrated hydrochloric acid (4.5 c.c.) cautiously added with cooling. The resulting solution was added during 5 minutes to a boiling solution of the above nitro compound (3.3 g.) in alcohol (100 c.c.), and the mixture refluxed for a further 15 minutes. Most of the alcohol was distilled away, dilute sodium hydroxide solution added, and the resulting solid (1.02 g., m. p. 180°) collected, and recrystallised from benzene, giving pale yellow prisms of the base, m. p. 187° (Found: C, 64.3; H, 4.25.  $C_{10}H_8O_2N_2$  requires C, 63.8; H, 4.25%). The *monohydrochloride* separated from alcohol-water as pale yellow crystals, m. p. 276° (decomp.) (Found: Cl, 15.4.  $C_{10}H_8O_2N_2 \cdot HCl$  requires Cl, 15.8%).

The base (2 g.) in glacial acetic acid (5 c.c.) was heated for 10 minutes on the water-bath with acetic anhydride (5 c.c.). On pouring the resulting solution into water, the *acetyl* derivative (2.34 g., m. p. 233–234°) was precipitated; this was crystallised from methanol-water and obtained as colourless prisms, m. p. 236° (Found: C, 62.8; H, 4.25.  $C_{10}H_8O_3N_2$  requires C, 62.6; H, 4.35%). The latter (2.25 g.) was heated with methyl *p*-toluenesulphonate (1.8 g.) for 2 hours at 150°, with occasional stirring, the melt extracted with hot methanol, and hot acetone added to the resulting solution. The solid (3 g.) which separated on cooling, was recrystallised from methanol, giving 3-acetamido-6:7-methylenedioxyquinoline metho-*p*-toluenesulphonate as yellow crystals, m. p. 273° (decomp.). The crude solid (1.7 g.) was refluxed for 1 hour with a mixture of concentrated hydrochloric acid (3 c.c.) and water (3 c.c.), the solution evaporated to dryness under reduced pressure, the residue dissolved in hot water, neutralised with potassium carbonate, and a solution of potassium iodide (6 g.) in hot water added. The solid (1.2 g., m. p. 270° decomp.), which separated on cooling, was recrystallised from methanol-water, giving 3-amino-6:7-methylenedioxyquinoline methiodide as orange-yellow prisms, m. p. 268°. The crude compound (0.9 g.) was refluxed with excess of freshly precipitated silver chloride in aqueous methanol for 11 hours, and the filtrate and washings evaporated to dryness under reduced pressure. The residue, recrystallised from methanol, gave 3-amino-6:7-methylenedioxyquinoline methochloride (0.4 g.) as yellow needles, m. p. 294° (decomp.) (Found: Cl, 14.5.  $C_{11}H_{11}O_2N_2 \cdot Cl$  requires Cl, 14.9%).

**3-Amino-6:7-methylenedioxyquinaldine Methochloride.**—3-Acetamido-6:7-methylenedioxyquinaldine (1.37 g.) (Berlingozzi and Napolitano, *loc. cit.*) was heated with methyl *p*-toluenesulphonate (1.1 g.) for 2 hours at 150°, the melt extracted with hot methanol, and the product (2.2 g., m. p. 165°) precipitated with ether. When recrystallised from acetone-methanol, this gave 3-acetamido-6:7-methylenedioxyquinaldine metho-*p*-toluenesulphonate as yellow prisms, m. p. 193–195°. The crude solid (2.2 g.) was hydrolysed with hydrochloric acid as previously described, and converted into 3-amino-6:7-methylenedioxyquinaldine methiodide (1.55 g.), which separated from methanol-water as orange-yellow needles, m. p. 285° (decomp.). By boiling for 12 hours with excess of silver chloride in aqueous methanol, this (1.5 g.) was converted into 3-amino-6:7-methylenedioxyquinaldine methochloride, which separated from methanol as yellow crystals, m. p. 288° (decomp.) (0.9 g.) (Found: Cl, 13.9.  $C_{13}H_{13}O_2N_2 \cdot Cl$  requires Cl, 14.1%).

**3-Amino-6:7-methylenedioxyquinaldine monohydrochloride** separated from alcohol-water as pale yellow crystals, m. p. 272° (decomp.) (Found: C, 51.3; H, 5.0; Cl, 13.5.  $C_{11}H_{11}O_2N_2 \cdot HCl \cdot H_2O$  requires C, 51.3; H, 5.05; Cl, 13.3%).

**3-Nitro-6:7-dimethoxyquinoline** was prepared from 6-aminoveratraldehyde (3 g.) (Rilliet, *Helv. Chim. Acta*, 1922, 5, 547) as described for the preparation of 3-nitro-6:7-methylenedioxyquinoline. The crude reddish-brown solid (1.72 g., m. p. 205°) was recrystallised from acetone-alcohol (charcoal), giving yellow needles (1 g.), m. p. 213° (Found: C, 56.6; H, 4.5; N, 12.4.  $C_{11}H_{10}O_4N_2$  requires C, 56.4; H, 4.3; N, 12.0%).

The above nitro compound in glacial acetic acid solution was hydrogenated using Adams's catalyst (hydrogen uptake: 3 mols.). The filtered solution was evaporated to dryness in a vacuum, the residue treated with dilute sodium hydroxide solution, the resulting precipitate warmed with acetic anhydride, the solution poured into water, and made alkaline with ammonia. The resulting solid, after being crystallised twice from methanol-water, gave colourless needles, m. p.

166—167°, apparently of 3-diacetamido-6:7-dimethoxyquinoline (Found: C, 61.95; H, 5.85; N, 9.35.  $C_{18}H_{16}O_4N_2$  requires C, 62.55; H, 5.55; N, 9.7%).

**3-Amino-6:7-dimethoxyquinaldine.**—A solution of potassium hydroxide (4 g.) in water (16 c.c.) was added to a solution of 6-aminoveratraldehyde (1.44 g.) and acetylphthalimide (1.6 g.) (Gabriel and Colman, *Ber.*, 1902, **35**, 3805) in alcohol (32 c.c.), and the mixture allowed to stand at room temperature for 3 days. The bulk of the alcohol was distilled away, and the residue acidified with dilute acetic acid. The 3-phthaloylamino-6:7-dimethoxyquinaldine (2.8 g.), which separated, when recrystallised from alcohol-water, had m. p. 238°. The crude material (2.6 g.) was refluxed for 2 hours with a mixture of concentrated hydrochloric acid (20 c.c.) and water (10 c.c.) and, after cooling, was made alkaline with sodium hydroxide; the resulting solid (1.1 g.) was separated, washed with water, and recrystallised from benzene, giving the *base* as pale yellow prisms, m. p. 184° (Found: C, 66.1; H, 6.5; N, 13.0.  $C_{12}H_{14}O_2N_2$  requires C, 66.1; H, 6.4; N, 12.8%). The *monohydrochloride* separated from alcohol-water as pale yellow crystals, m. p. 285° (decomp.) (Found: C, 56.15; H, 5.9; N, 11.05; Cl, 13.9.  $C_{12}H_{14}O_2N_2 \cdot HCl$  requires C, 56.6; H, 5.9; N, 11.0; Cl, 13.95%).

The *base* (2 g.) was heated in the water-bath for 5 minutes with acetic anhydride (5 c.c.), the mixture poured into water and made alkaline with ammonia. The *acetyl* derivative (2.26 g., m. p. 189—191°) separated and was crystallised from methanol-water as colourless crystals, m. p. 191° (Found: C, 64.8; H, 6.1.  $C_{14}H_{16}O_3N_2$  requires C, 64.4; H, 6.15%). The crude *acetyl* derivative (2 g.) was heated with methyl *p*-toluenesulphonate (1.5 g.) for 2 hours at 150°, the melt extracted with hot methanol, and ether added to the cooled solution. The resulting solid (2.9 g.) was collected, hydrolysed as before, and converted first into the *methiodide* (1.72 g.), which separated from methanol as yellow needles, m. p. 258°, and then into the *methochloride*, which crystallised from methanol as yellow prisms, m. p. 260° (decomp.) (1 g.) (Found: Cl, 13.2.  $C_{13}H_{17}O_2N_2Cl$  requires Cl, 13.2%).

**3-Amino-2-phenylquinoline Methochloride.**—3-Amino-2-phenylquinoline (2.25 g.) (Petrow, Stack, and Wragg, *loc. cit.*) was heated with acetic anhydride (5 c.c.) for 10 minutes in the water-bath, poured into water, and ammonia added. The diacetyl derivative (2.6 g., m. p. 172°) was filtered off, and washed with water; John (*J. pr. Chem.*, 1931, **131**, 346) gives m. p. 173°. The dry-solid was refluxed in dry benzene (50 c.c.) for 10 hours with dimethyl sulphate (1.1 c.c.), and the solid (1.9 g., m. p. 196—199°) which separated on cooling was recrystallised from methanol-acetone, giving 3-acetamido-2-phenylquinoline *methosulphate* as pale yellow prisms, m. p. 200—201°. The latter (1.7 g.) was hydrolysed in the usual manner, and converted first into 3-amino-2-phenylquinoline *methiodide*, 1.5 g., m. p. 221° (decomp.), and then into the *methochloride*, which crystallised from methanol-acetone as pale yellow prisms, m. p. 230° (decomp.) (0.8 g.) (Found: Cl, 13.1.  $C_{16}H_{15}N_2Cl$  requires Cl, 13.1%).

**3-Dimethylaminoquinaldine Methochloride.**—3-Aminoquinaldine (2.08 g.), methyl iodide (5.6 g.), anhydrous sodium carbonate (1.4 g.), and water (40 c.c.) were heated together under reflux for 4½ hours on the water-bath. The solid which separated on cooling was crystallised twice from methanol and then from water, giving 3-dimethylaminoquinaldine *methiodide* (2 g.) as bright yellow prisms, m. p. 240—241° (Found: N, 8.9.  $C_{13}H_{17}N_2I$  requires N, 8.5%). This was converted, in the usual manner, into the *methochloride*, which separated from methanol-acetone as yellow crystals (1.1 g.), m. p. 230° (decomp.) (Found: Cl, 14.9.  $C_{13}H_{17}N_2Cl$  requires Cl, 15.0%).

**3-Nitroquinoline.**—Renshaw and Friedmann (*loc. cit.*), repeating the method described in G.P. 335,197 (*Chem. Zentr.*, 1921, II, 962), obtained a yield of only 22%. We have obtained a yield of approximately 48% by adding concentrated hydrochloric acid (0.7 c.c.) to a solution of methazonic acid (3.5 g.) and *o*-aminobenzaldehyde (3.2 g.) in alcohol (9 c.c.). After standing for 2 days at room temperature, the 3-nitroquinoline separated (2.2 g.), m. p. 126°.

**3-Aminoquinoline Methochloride.**—3-Acetamidoquinoline (0.92 g.) (Renshaw and Friedmann, *loc. cit.*) in dry benzene (10 c.c.) was refluxed for 7 hours with dimethyl sulphate (0.5 c.c.). The resulting solid (1.47 g., m. p. 167°) on recrystallisation from methanol yielded 3-acetamidoquinoline *methosulphate* as yellow prisms, m. p. 171°. The crude solid was hydrolysed, as before, and converted first into 3-aminoquinoline *methiodide* (orange-yellow prisms from methanol-water, m. p. 187°) and then into the *methochloride* (yellow crystals, m. p. 190°, from acetone-methanol).

**3-Aminoquinaldine Methochloride.**—3-Acetamidoquinaldine (3 g.) (Lawson, Perkin, and Robinson, *loc. cit.*) in dry benzene (45 c.c.) was refluxed for 6 hours with dimethyl sulphate (1.5 c.c.). After cooling, the solid (4.3 g., m. p. 178°) was separated and washed with benzene; on crystallisation from methanol, this gave 3-acetamidoquinaldine *methosulphate* as pale yellow prisms, m. p. 190—191°. The crude solid (4.2 g.) was hydrolysed in the usual way, and converted first into 3-aminoquinaldine *methiodide* (3.6 g.), which separated from methanol as yellow prisms, m. p. 241° (decomp.), and then into the *methochloride* (1.8 g.) which separated from a small volume of methanol as hygroscopic yellow crystals, m. p. 236° (decomp.) (Found: Cl, 16.7.  $C_{11}H_{13}N_2Cl$  requires Cl, 17.0%).

**3-Amino-2-p-dimethylaminostyrylquinoline Methochloride.**—A mixture of 3-acetamidoquinaldine *methosulphate* (1.6 g.), *p*-dimethylaminobenzaldehyde (0.8 g.), methanol (10 c.c.) and piperidine (0.4 c.c.) was refluxed for 18 hours, and ether added to the resulting solution, after concentration. The precipitated gum was washed with ether, and then refluxed for 2 hours with a mixture of concentrated hydrochloric acid (2.5 c.c.) and water (3 c.c.), the resulting solution evaporated to dryness under reduced pressure, the residue dissolved in water, neutralised with ammonia, and an aqueous solution of potassium iodide added. The precipitate, after recrystallisation from methanol, gave 3-amino-2-p-dimethylaminostyrylquinoline *methiodide* (0.7 g.) as dark red needles, with blue reflex, m. p. 322° (decomp.) (Found: C, 56.0; H, 4.9.  $C_{20}H_{22}N_3I$  requires C, 55.7; H, 5.1%). In the usual manner, this was converted into the *methochloride* (0.5 g.) which separated from a small volume of methanol as dark red crystals with a greenish reflex, m. p. 235° (decomp.) (Found: Cl, 10.2.  $C_{20}H_{22}N_3Cl$  requires Cl, 10.45%).

**3-Dimethylamino-2-p-acetamidostyrylquinoline Methochloride.**—3-Dimethylaminoquinaldine *methiodide* (0.84 g.), *p*-acetamidobenzaldehyde (0.45 g.), methanol (15 c.c.) and piperidine (4 drops) were heated together for 12 hours on the water-bath. The solid (0.77 g.) which separated on cooling crystallised from methanol as yellow needles, m. p. 337—339° (decomp.), of 3-dimethylamino-2-p-acetamidostyrylquinoline *methiodide*. When hydrolysed by boiling with hydrochloric acid, this gave the corresponding aminostyryl compound, which separated from methanol as bright red crystals, m. p. 301—302°. The above crude yellow solid was converted into the *methochloride* in the usual way, giving 0.4 g. of yellow crystals from m. p. 331—335° (decomp.) (Found: Cl, 9.2.  $C_{22}H_{24}ON_3Cl$  requires Cl, 9.3%).

**3-Dimethylamino-2-p-dimethylaminostyrylquinoline Methiodide.**—A mixture of 3-dimethylaminoquinaldine *methiodide* (0.33 g.), *p*-dimethylaminobenzaldehyde (0.17 g.), methanol (10 c.c.), and piperidine (2 drops) was refluxed for 10 hours, and the solid (0.1 g.) which separated on cooling was recrystallised from methanol, giving dark red crystals, m. p. 273° (decomp.) (Found: I, 27.6.  $C_{22}H_{22}N_3I$  requires I, 27.65%).

**3-Acetamidoquinoline-2-aldehyde.**—3-Acetamidoquinaldine (2.28 g.), freshly prepared selenium dioxide (1.28 g.), dioxan (20 c.c.), and water (1 c.c.) were refluxed together for 1½ hours; the resulting solution was evaporated to dryness from the water-bath, under reduced pressure, the residue extracted with hot benzene (charcoal), the extract concentrated and allowed to cool. The resulting solid (1 g., m. p. 167—171°), on recrystallisation from benzene, afforded the *aldehyde* as pale yellow prisms, m. p. 171° (Found: C, 67.5; H, 5.1.  $C_{12}H_{10}O_2N_2$  requires C, 67.30; H, 4.7%). The *phenylhydrazone*, obtained by heating the *aldehyde* with phenylhydrazine in dilute acetic acid, crystallised from alcohol as pale yellow needles, m. p. 241—242° (Found: C, 72.0; H, 5.0.  $C_{18}H_{16}ON_4$  requires C, 72.1; H, 5.05%). The *oxime*, prepared by refluxing the *aldehyde* with hydroxylamine hydrochloride and calcium carbonate in alcohol-water, dis-

tiling off the alcohol, adding water to the residue, and collecting the resulting solid, separated from alcohol as colourless crystals, m. p. 215° (Found: C, 62.3; H, 4.4.  $C_{12}H_{11}O_2N_2$  requires C, 62.9; H, 4.8%).

*Di-(3-quinoly)-urea*.—3-Aminoquinoline hydrochloride (0.57 g.) and urea (0.3 g.) were refluxed together in water (7 c.c.) for 7 hours, the mixture allowed to cool, and the resulting solid (0.3 g.) collected. The product, after recrystallisation from alcohol (in which it is sparingly soluble) or from pyridine (in which it is readily soluble), had m. p. 296° (Found: C, 72.25; H, 4.8; N, 17.6.  $C_{19}H_{14}ON_4$  requires C, 72.6; H, 4.5; N, 17.8%). When this *base* (0.2 g.) was heated with methyl *p*-toluenesulphonate (0.25 g.) for 2 hours at 160°, the product extracted with methanol, precipitated with ether, and crystallised from methanol-acetone, the *di-(metho-p-toluenesulphonate)* was obtained as pale yellow prisms, m. p. 250–255° (decomp.) (Found: C, 61.1; H, 4.85.  $C_{35}H_{34}O_7N_4S_2$  requires C, 61.2; H, 4.85%).

*Di-(3-quinaldyl)-urea*.—The following was found to be a convenient modification of the method of Stark and Hoffmann (*loc. cit.*). 3-Aminoquinaldine (2 g.) was dissolved in alcohol-free chloroform (25 c.c.), and a solution of phosgene (0.5 mol.) in xylene added. After standing for 2 days at room temperature, the product was separated, washed with chloroform, dried on the water-bath, warmed with dilute sodium hydroxide solution, separated, washed with water, and dried, giving 2 g., m. p. ca. 280°.

*3-p-Toluenesulphonmethylamidoquinaldine*.—3-Aminoquinaldine (2.12 g.) and *p*-toluenesulphonyl chloride (2.6 g.) were dissolved by warming in acetone (15 c.c.) and pyridine (4 c.c.) added to the warm solution. After standing for 2½ hours at room temperature, the mixture was filtered, the acetone distilled off from the filtrate, water added to the residue; the resulting gum, on stirring, quickly solidified (3.15 g., m. p. 191–198°). This, on recrystallisation from alcohol gave 3-*p*-toluenesulphonamidoquinaldine as colourless prisms, m. p. 202–203° (Found: C, 65.9; H, 5.7.  $C_{17}H_{16}O_2N_2S$  requires C, 65.4; H, 5.15%). The latter (3.1 g., m. p. 191–198°) was dissolved in a mixture of 2*N*-sodium hydroxide solution (5 c.c.) and water (10 c.c.), and shaken for 4½ hours with dimethyl sulphate (0.9 c.c.). The resulting precipitate (2.85 g., m. p. 116–118°) was recrystallised from alcohol, giving 3-*p*-toluenesulphonmethylamidoquinaldine as colourless prisms, m. p. 127° (Found: C, 66.2; H, 5.5.  $C_{18}H_{18}O_2N_2S$  requires C, 66.25; H, 5.5%).

*3-Mesitylenesulphonmethylamidoquinaldine*.—A solution of 3-aminoquinaldine (1.6 g.) and mesitylenesulphonyl chloride (2.5 g.) in acetone (40 c.c.) and pyridine (15 c.c.) was refluxed (water-bath) for 2½ hours, the acetone distilled off, and the product (1.2 g.) precipitated with water. This 3-*mesitylenesulphonamidoquinaldine* separated from alcohol as colourless leaflets, which changed to a white powder on drying, m. p. 178° (Found: C, 67.35; H, 5.9.  $C_{19}H_{20}O_2N_2S$  requires C, 67.05; H, 5.9%). Methylation of this material (1 g.) with dimethyl sulphate (0.4 g.) and dilute sodium hydroxide solution gave 3-*mesitylenesulphonmethylamidoquinaldine* (0.8 g.), which separated from alcohol-water as colourless plates, m. p. 148–149° (Found: C, 67.9; H, 6.1.  $C_{20}H_{20}O_2N_2S$  requires C, 67.75; H, 6.2%). By hydrolysis of this with sulphuric acid (50%) for 5 hours at 130°, followed by steam-distillation, basification and extraction with ether an oil was obtained, from which a very small amount of a compound, separating from benzene-ligroin as colourless prisms, m. p. 71°, was isolated.

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