The Chemistry of the Pyrrocolines. Part VIII. Alkyl Derivatives.*

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[Reprint Order No. 6042.]

Several alkylpyrrocolines have been prepared and their reactions with methyl and ethyl iodide studied. Complete substitution in the 5-membered ring can lead to both 1 : 1 : 2 : 3- and 1 : 2 : 3 : 3-tetra-alkylpyrrocolinium iodides, which may be distinguished by their ultraviolet absorption spectra.

THE most useful method for the preparation of 2-substituted pyrrocolines is that of Tschitschibabin (*Ber.*, 1927, **60**, 1607) in which the adduct (II) from a suitably substituted pyridine (I) and an α-halogeno-ketone is treated with a base, usually hot sodium hydrogen carbonate solution. Since many simple pyrrocolines are unstable in air it is often convenient to prepare the intermediate (II) in bulk and convert it into the pyrrocoline (III) as required.

The reaction of bromo-ketones with pyridines is often vigorous (cf. Holliman and Schickerling, *J.*, 1951, 914) but can be moderated by diluting the mixture with acetone. With this modification 2-methyl-, 1 : 2-dimethyl-, and 2 : 3-dimethyl-pyrrocoline were prepared in appreciably improved yields, and a good yield of 2 : 7-dimethylpyrrocoline was also obtained. No diluent was required in the first stage of the syntheses of 1 : 2 : 3-trimethyl- and 3-benzyl-2-methyl-pyrrocoline. No more than 5% of 2 : 5-dimethylpyrrocoline could be obtained from 2 : 6-lutidine and bromo- or chloro-acetone, but poor

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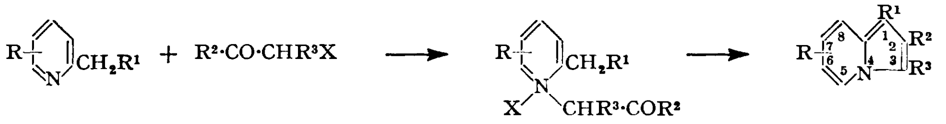
* Part VII, *J.*, 1955, 1504.

yields appear to be not unusual in the synthesis of 5-substituted pyrrocolines (cf. Tschitschibabin, *loc cit.*; Buu-Hoï and Khoi, *Compt. rend.*, 1950, **230**, 967). Stable chloroplatinates were readily obtained from most of the pyrrocolines, and chloroplatinates also proved useful for the characterisation of the quaternary intermediates (II) in those cases where the pure bromides were not isolated.

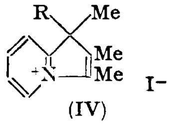
The 2-ethylpyridine required for the preparation of 1 : 2-dimethyl- and 1 : 2 : 3-trimethyl-pyrrocolines was prepared by Wolff–Kishner reduction of 2-acetylpyridine



(I) (X = Cl or Br) (II) (III)

(Prostenik and Balling, *Arkiv Kemi*, 1946, **18**, 11). The Clemmensen procedure, by which Furst (*J. Amer. Chem. Soc.*, 1949, **71**, 3550) obtained 2-ethylpyridine in about 80% yield, in our hands gave only 1-2′-pyridylethanol. Späth and Galinovsky (*Ber.*, 1938, **71**, 721) similarly obtained an alcohol from 3 : 5-dimethyl-2-propionylpyridine. 1-2′-Pyridylethanol gave a crystalline hydrogen phthalate and an oily phenylurethane, but the picrate of the latter, like that of the parent alcohol, was crystalline. We confirm that 1-2′-pyridylethanol has the physical properties recently recorded by Bullitt and Maynard (*J. Amer. Chem. Soc.*, 1954, **76**, 1370) and agree with their conclusion that the solid described by Pinner (*Ber.*, 1901, **34**, 4241) cannot have been this alcohol.

Alkylation of pyrrocolines was first reported by Scholtz (*Ber.*, 1912, **45**, 1718; *Arch. Pharm.*, 1913, **251**, 666) who obtained methiodides of di- and tri-methylpyrrocolines by the action of methyl iodide at 120° on pyrrocoline and 7-methylpyrrocoline respectively. Rossiter and Saxton (*J.*, 1953, 3654) prepared another trimethylpyrrocoline methiodide, m. p. 197·5°, together with a trace of 1 : 2 : 3-trimethylpyrrocoline, by heating 2-methylpyrrocoline with methyl iodide. Under essentially similar conditions we obtained a product, m. p. 180—181°, which also arose by the action of methyl iodide on 1 : 2 : 3-trimethylpyrrocoline at room temperature.



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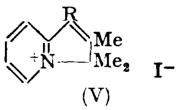

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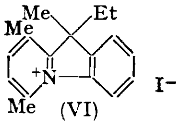

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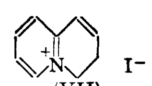


(VII)

Saxton (*J.*, 1951, 3239) adduced evidence that 1 : 3 : 5 : 7-tetramethylpyrrocoline methiodide contained a *C*-alkylated cation of the pyridinium type. Possible structures of such a type for 1 : 2 : 3-trimethylpyrrocoline methiodide are (IV and V; R = Me), but no decision can be made between these alternatives on chemical grounds. However, the elegant experiments of Robinson and Saxton (*J.*, 1952, 976), in which the ethiodide of 1 : 5 : 8-trimethyl-2 : 3-benzopyrrocoline was shown to be identical with the methiodide of 1-ethyl-5 : 8-dimethyl-2 : 3-benzopyrrocoline, both having structure (VI), suggested a possible means of establishing the structures of 1 : 2 : 3-trialkylpyrrocoline alkiodides in which the alkyl substituents were not all alike.

Heating 1 : 2 : 3-trimethylpyrrocoline with ethyl iodide under pressure apparently gave a mixture of isomers, $C_{13}H_{18}NI$, which after many crystallisations afforded one pure component, m. p. 199—200°. The same compound was obtained from 1-ethyl-2 : 3-dimethylpyrrocoline and methyl iodide and is therefore formulated as 1-ethyl-1 : 2 : 3-trimethylpyrrocolinium iodide (IV; R = Et), whilst an isomer which accompanied it in larger amount in the methylation is regarded as 1-ethyl-2 : 3 : 3-trimethylpyrrocolinium iodide (V; R = Et). These two isomers exhibited markedly different ultraviolet absorption (see Fig. 1), whilst further evidence for the presence of the 1 : 2-double bond in the isomer (V; R = Et) is afforded by the similarity of its spectrum to that of the pyridocolinium iodide (VII) (Boekelheide and Gall, *J. Amer. Chem. Soc.*, 1954, **76**, 1832). The only pure product which could be isolated from the reaction of 3-ethyl-2-methylpyrrocoline with an excess of methyl iodide was formulated as 3-ethyl-1 : 2 : 3-trimethylpyrrocolinium iodide because of the resemblance of its spectrum to that of the iodide (V; R = Et) (see Fig. 1).

The methiodide, m. p. 180—181°, of 1 : 2 : 3-trimethylpyrrocoline exhibited ultraviolet absorption of a type intermediate between those of 1 : 1 : 2 : 3- and 1 : 2 : 3 : 3-tetra-alkylpyrrocolinium iodides. It was probably a mixture or molecular compound of (IV and V; R = Me), whilst the form, m. p. 197·5°, the isolation of which by Rossiter and Saxton (*loc. cit.*) we have been unable to repeat, may have been one of the pure components. An "intermediate" type of absorption was also shown by an incompletely purified product from 1 : 2 : 3-trimethylpyrrocoline and ethyl iodide, in which 1-ethyl-1 : 2 : 3-trimethylpyrrocolinium iodide (V; R = Et) was probably accompanied by some of the 3-ethyl compound.

FIG. 1. FIG. 2.

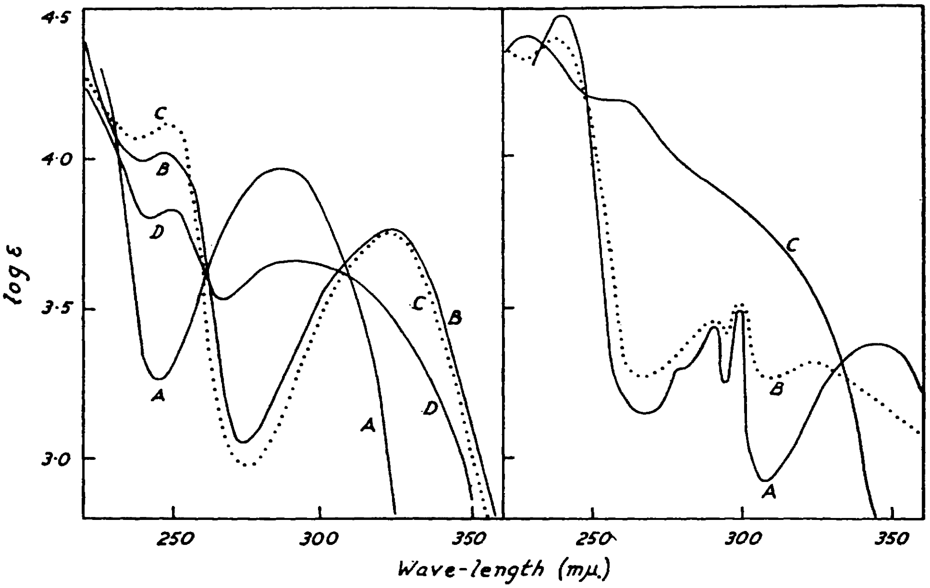
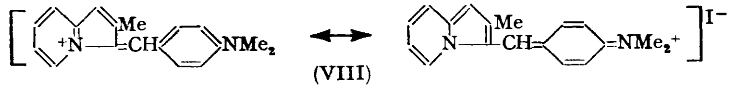


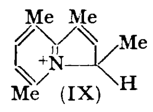
FIG. 1. *Absorption spectra of* (A) 1-*ethyl*-1 : 2 : 3-, (B) 1-*ethyl*-2 : 3 : 3-, *and* (C) 3-*ethyl*-1 : 2 : 3-*trimethylpyrrocolinium iodide, and* (D) 1 : 2 : 3-*trimethylpyrrocoline methiodide, m. p.* 180—181°, *all in* EtOH.

FIG. 2. *Absorption spectra of* (A) 2-*methylpyrrocoline in ethanol,* (B) 2-*methylpyrrocoline hydriodide in ethanol, and* (C) 2-*methylpyrrocoline hydriodide in ethanolic* 0·08N-HCl.

Rossiter and Saxton (*loc. cit.*) have reported that the crystals which separate when 2-methylpyrrocoline is kept in methyl iodide at room temperature are 2-methylpyrrocoline hydriodide, the methylation product apparently remaining in solution as the free base. We obtained the same hydriodide, m. p. 169—172° (decomp.), by the action of either ethyl iodide or hydriodic acid on 2-methylpyrrocoline. Deposition of the corresponding hydriodides, rather than the methiodides, was also observed when 1 : 2-, 2 : 5-, and 2 : 7-dimethylpyrrocolines were kept with methyl iodide. Treatment of 2-methylpyrrocoline hydriodide with *p*-dimethylaminobenzaldehyde gave a deep blue dye, presumably (VIII) or the 1-substituted isomer. Other pyrrocolines containing free 1- or 3-positions also gave intense blue or violet Ehrlich colours with *p*-dimethylaminobenzaldehyde in the presence of mineral acid (cf. Clemo, Fox, and Raper, *J.*, 1953, 4173).



(VIII)



(IX)

The striking similarity of the spectrum recorded by Saxton (*loc. cit.*) for 1 : 3 : 5 : 8-tetramethylpyrrocoline in ethanolic hydrochloric acid to those of the 1 : 2 : 3 : 3-tetra-alkylpyrrocolinium iodides indicates (IX) as the probable structure of the 1 : 3 : 5 : 8-tetramethylpyrrocolinium ion. On the other hand, the spectrum of 2-methylpyrrocoline hydriodide in ethanol chiefly resembled that of 2-methylpyrrocoline itself (see Fig. 2), owing no doubt to dissociation. Inclusion of a large excess of hydrogen chloride radically altered the absorption curve, but there was still no resemblance to the spectra of either the 1 : 1 : 2 : 3- or the 1 : 2 : 3 : 3-tetra-alkylpyrrocolinium iodides, so that the 2-methylpyrrocolinium ion was presumably not a single species.

EXPERIMENTAL

2-*Methylpyrrocoline.*—A mixture of α-picoline (120 ml.) and bromoacetone (167 g.) in acetone (200 ml.) was refluxed for 1 hr., external heating being initially unnecessary, and allowed to cool. 1-Acetonyl-2-methylpyridinium bromide (237 g., 84%) was collected and washed with acetone; a specimen crystallised from ethanol had m. p. 196° as reported by Tschitschibabin (*loc. cit.*) (Found: C, 46·6; H, 5·1; N, 6·0; Br, 34·7. Calc. for $C_9H_{12}ONBr$: C, 47·0; H, 5·3; N, 6·1; Br, 34·7%). A solution of the bromide (23 g.) and sodium hydrogen carbonate (20 g.) in water (500 ml.) was heated to boiling and the resulting 2-methylpyrrocoline distilled in steam. An ether extract of the distillate was dried and evaporated to leave the cream-coloured product (12·5 g., 95%), m. p. 59° after sublimation *in vacuo* (Found: N, 11·0. Calc. for C_9H_9N: N, 10·7%).

1 : 2-*Dimethylpyrrocoline.*—Refluxing 2-ethylpyridine (6·5 ml.) and bromoacetone (7·7 g.) in acetone (12 ml.) for 90 min. gave deliquescent crystals of 1-acetonyl-2-ethylpyridinium bromide (11·0 g., 80%). A little of this salt in water was treated with chloroplatinic acid to give the dull yellow insoluble 1-*acetonyl*-2-*ethylpyridinium chloroplatinate*, m. p. 210° (decomp.) (Found: Cl, 28·2; Pt, 26·5. $C_{20}H_{28}O_2N_2Cl_6Pt$ requires Cl, 28·9; Pt, 26·5%). Ring-closure of the bromide as described for 2-methylpyrrocoline gave 1 : 2-dimethylpyrrocoline (92%), a resublimed specimen having m. p. 63° (Found: N, 9·9. Calc. for $C_{10}H_{11}N$: N, 9·7%). Rossiter and Saxton (*loc. cit.*) reported m. p. 57—58°. The insoluble yellow *chloroplatinate* melted indefinitely from 214° after becoming discoloured above 175° (Found: Cl, 30·7; Pt, 28·1. $C_{20}H_{24}N_2Cl_6Pt$ requires Cl, 30·4; Pt, 27·9%).

2 : 3-*Dimethylpyrrocoline*, m. p. 37°, was similarly prepared in 61% yield from α-picoline and 1-bromoethyl methyl ketone (Found: N, 9·5. Calc. for $C_{10}H_{11}N$: N, 9·7%). Orange needles of 2-*methyl*-1-(1-*methyl*-2-*oxopropyl*)*pyridinium chloroplatinate*, m. p. 201° (decomp.), were prepared from the intermediate bromide (Found: C, 32·2; H, 4·2; N, 4·1. $C_{20}H_{28}O_2N_2Cl_6Pt$ requires, C, 32·6; H, 3·8; N, 3·8%).

2 : 7-*Dimethylpyrrocoline.*—A solution of 2 : 4-lutidine (27·3 ml.) and bromoacetone (33·3 g.) in acetone (50 ml.) was refluxed for 15 min., a heavy oil separating, and set aside overnight. After removal of acetone *in vacuo* the frothy residue was dissolved in water and the solution was clarified by ether-extraction. Addition of chloroplatinic acid to a little of the aqueous phase gave pale orange microcrystals of 1-*acetonyl*-2 : 4-*dimethylpyridinium chloroplatinate*, m. p. 219—220° (decomp.) (Found: C, 32·6; H, 4·1; Pt, 26·6. $C_{20}H_{28}O_2N_2Cl_6Pt$ requires C, 32·6; H, 3·8; Pt, 26·5%). The bulk of the aqueous solution was diluted to about 600 ml., treated with sodium hydrogen carbonate (60 g.), and distilled. 2 : 7-*Dimethylpyrrocoline* (24·4 g., 70%) was isolated from the steam-distillate in the usual way and after vacuum-sublimation formed colourless plates, m. p. 78—79°, which became black within 36 hr. (Found: N, 9·3. $C_{10}H_{11}N$ requires N, 9·7%). The orange-yellow *chloroplatinate* decomposed above 200° (Found: C, 34·2; H, 3·0; Pt, 28·2. $C_{20}H_{24}N_2Cl_6Pt$ requires C, 34·3; H, 3·4; Pt, 27·9%).

2 : 5-*Dimethylpyrrocoline.*—A solution of 2 : 6-lutidine (44 ml.) and bromoacetone (52·5 g.) in acetone (60 ml.) was refluxed for 30 min. and worked up as described for 2 : 7-dimethylpyrrocoline, to give 2 : 5-dimethylpyrrocoline (2·72 g.), b. p. 116—117°/14 mm. Tschitschibabin (*loc. cit.*) reported b. p. 238—240°.

1 : 2 : 3-Trimethylpyrrocoline was prepared as described by Rossiter and Saxton (*loc. cit.*), b. p. 135—137°/13 mm. The intermediate 2-*ethyl*-1-(1-*methyl*-2-*oxopropyl*)*pyridinium bromide* crystallised from ethanol–ether in colourless needles, m. p. 182—184° (Found: C, 50·7; H, 6·2; N, 5·5; Br, 31·2. $C_{11}H_{16}ONBr$ requires C, 51·1; H, 6·2; N, 5·4; Br, 31·0%). 1 : 2 : 3-*Trimethylpyrrocoline chloroplatinate*, a pale yellow microcrystalline powder, had m. p. 179° (decomp.) (Found: N, 4·1; Cl, 29·8; Pt, 26·7. $C_{22}H_{28}N_2Cl_6Pt$ requires N, 3·9; Cl, 29·2; Pt, 26·8%).

3-*Benzyl*-2-*methylpyrrocoline*.—α-Picoline (8·9 ml.) and 1-bromo-2-phenylethyl methyl ketone (Janetzky and Verkade, *Rec. Trav. chim.*, 1945, **64**, 129) (20·5 g.) were heated on the steam-bath for 8 hr., then cooled and diluted with water. Sodium hydrogen carbonate was added to pH 7 and the solution was clarified by extraction with chloroform. A small portion of the aqueous solution was acidified with hydrochloric acid and treated with chloroplatinic acid to give a cream-coloured precipitate of 1-(1-*benzyl*-2-*oxopropyl*)-2-*methylpyridinium chloroplatinate*, decomp. >170° (Found: N, 3·2; Cl, 24·2; Pt, 21·7. $C_{32}H_{36}O_2N_2Cl_6Pt$ requires N, 3·2; Cl, 23·9; Pt, 22·0%). The bulk of the aqueous solution was treated with a further 16 g. of sodium hydrogen carbonate and refluxed for 4 hr., then cooled and extracted with chloroform. Evaporation of the dried extracts gave 3-*benzyl*-2-*methylpyrrocoline* as a crude brown powder (5·06 g.) which was best purified by sublimation at 0·05 mm. (bath, 80—120°), giving colourless crystals, m. p. 93—94° (Found: C, 87·1; H, 6·9; N, 6·2. $C_{16}H_{15}N$ requires C, 86·8; H, 6·8; N, 6·3%). The *chloroplatinate* had m. p. 178—179° (decomp.) (Found: N, 3·2; Cl, 25·0; Pt, 22·8. $C_{32}H_{32}N_2Cl_6Pt$ requires N, 3·3; Cl, 25·0; Pt, 22·9%).

1-2′-*Pyridylethanol*.—Clemmensen reduction of 2-acetylpyridine was carried out as described by Furst (*loc. cit.*) for the preparation of 2-ethylpyridine, but the product was 1-2′-pyridylethanol (67%), b. p. 204—211°/754 mm. A redistilled specimen had b. p. 207°/747 mm. (Found: C, 68·0; H, 7·7; N, 11·0. Calc. for C_7H_9ON: C, 68·3; H, 7·4; N, 11·4%). The *picrate*, prepared in benzene, crystallised from a little ethyl acetate as yellow prisms, m. p. 85—86° (Found: C, 44·4; H, 3·4; N, 15·8. $C_{13}H_{12}O_8N_4$ requires C, 44·3; H, 3·4; N, 15·9%). A solution of the alcohol (3·4 g.) and phthalic anhydride (4·1 g.) in acetone (15 ml.) was refluxed for 90 min., then cooled to give colourless crystals of the *hydrogen phthalate* (4·42 g.), m. p. 134—136° after further crystallisation from acetone (Found: C, 66·6; H, 5·0; N, 5·0%; equiv., 268. $C_{15}H_{13}O_4N$ requires C, 66·4; H, 4·8; N, 5·2%; equiv., 271). The oily phenylurethane, obtained by heating the alcohol (1 g.) with phenyl *iso*cyanate (0·96 g.) in dry toluene under reflux for 30 hr., gave a *picrate* (2·84 g.) which crystallised from ethanol in yellow prisms, m. p. 164—166° (Found: C, 51·0; H, 3·7; N, 14·8. $C_{20}H_{17}O_9N_5$ requires C, 51·0; H, 3·6; N, 14·9%).

1 : 2 : 3-*Trimethylpyrrocoline Methiodide*.—(*a*) 2-Methylpyrrocoline (3·1 g.), methyl iodide (6 ml.), and methanol (10 ml.) were heated at 120° for 7 hr., then evaporated *in vacuo*. Trituration of the residue with dry acetone gave 1 : 2 : 3-trimethylpyrrocoline methiodide (4·83 g. in two crops), which formed lemon-yellow prisms (from ethanol) or needles (from *iso*propanol), m. p. 180—181° (Found: C, 48·1; H, 5·3; N, 4·9; I, 41·8. Calc. for $C_{12}H_{16}NI$: C, 47·9; H, 5·4; N, 4·7; I, 42·1%). (*b*) 1 : 2 : 3-Trimethylpyrrocoline (3·7 g.) was kept in methyl iodide (15 ml.) for 1 week, then diluted with dry ether. The dark supernatant liquid was decanted and the crystals (2·73 g.) were washed successively with ether and acetone, then crystallised from ethanol or *iso*propanol; they had m. p. 180—181° alone or mixed with the product from (*a*).

1 : 2 : 3-*Trimethylpyrrocoline Ethiodide*.—1 : 2 : 3-Trimethylpyrrocoline (3·8 g.), ethyl iodide (10 ml.), and ethanol (10 ml.) were heated at 120° for 8 hr. Crystals separated from the strongly cooled solution, and further crops were obtained by evaporating the filtrate and triturating the residue with acetone. Recrystallisation from acetone–ether to constant m. p. gave the mixed isomers of the ethiodide as cream-coloured crystals (2·4 g.), m. p. 179—180° (Found: C, 49·7; H, 6·2; N, 4·2. Calc. for $C_{13}H_{18}NI$: C, 49·5; H, 5·8; N, 4·4%). Three crystallisations from ethanol followed by ten from *iso*propanol gave 1-*ethyl*-1 : 2 : 3-*trimethylpyrrocolinium iodide* (0·26 g.) as cream-coloured needles, m. p. 199—200° (Found: C, 49·8; H, 6·2; N, 4·3%).

Reaction of 1-*Ethyl*-2 : 3-*dimethylpyrrocoline with Methyl Iodide*.—1-Ethyl-2 : 3-dimethylpyrrocoline (Part VII, *J.*, 1955, 1504) (3 g.), methyl iodide (3 ml.), and methanol (8 ml.) were heated at 110—120° for 7 hr., then evaporated *in vacuo*. Trituration of the sticky residue with dry acetone gave a cream-coloured powder (1·3 g., 24%), m. p. 197°, which crystallised from ethanol in needles, m. p. 199—200°, identical with 1-ethyl-1 : 2 : 3-trimethylpyrrocolinium iodide from the previous experiment. The dark green acetone filtrate was diluted with dry ether (35 ml.) to precipitate an oil which crystallised when rubbed. The pale green solid (2·65 g., 48%) was collected and washed with acetone–ether, m. p. 158—166°. Crystallisation from acetone–ether gave straw-coloured crystals of 1-*ethyl*-2 : 3 : 3-*trimethylpyrrocolinium iodide*, m. p. 179° (Found: C, 49·5; H, 5·6; N, 4·4%).

Reaction of 3-*Ethyl*-2-*methylpyrrocoline with Methyl Iodide*.—3-Ethyl-2-methylpyrrocoline (Part VIII, *loc. cit.*) (5 g.), methyl iodide (5 ml.), and methanol (12 ml.) were heated at 120° for 7 hr., then evaporated *in vacuo*. The green gum crystallised when rubbed with dry acetone, and addition of dry ether to the filtrate gave more solid. The combined crops (2·95 g.;

m. p. 157—162°) crystallised from acetone–ether in cream-coloured tablets of 3-*ethyl*-1 : 2 : 3-*trimethylpyrrocolinium iodide*, m. p. 167—168° (Found: C, 49·2; H, 5·8; N, 4·7; I, 40·0. $C_{13}H_{18}NI$ requires C, 49·5; H, 5·8; N, 4·4; I, 40·3%). As in most of the other alkylations, low-melting material could be recovered from the mother-liquors but could not be purified.

Reaction of Dimethylpyrrocolines with Cold Methyl Iodide.—2 : 5-Dimethylpyrrocoline (2·7 g.) in ether (15 ml.) was kept with methyl iodide (4 ml.) for 1 week, during which an oil slowly separated. The oil solidified when rubbed with acetone, and the resulting powder (0·65 g.) was crystallised from ethanol, to give pale green platelets of 2 : 5-*dimethylpyrrocoline hydriodide*, m. p. 188° (decomp.) (Found: C, 43·9; H, 4·7; N, 5·1; I, 46·0. $C_{10}H_{12}NI$ requires C, 44·0; H, 4·4; N, 5·1; I, 46·5%).

Similarly 2 : 7-dimethylpyrrocoline and methyl iodide gave 2 : 7-*dimethylpyrrocoline hydriodide*, green needles (from ethanol), m. p. 171—172° (Found: C, 44·2; H, 4·8; N, 5·0%). 1 : 2-*Dimethylpyrrocoline hydriodide*, similarly prepared, crystallised from ethanol–ether in pale blue-green needles, m. p. 177—179° (Found: C, 44·2; H, 4·8; N, 4·8%). All these hydriodides gave blue or violet dyes when treated with *p*-dimethylaminobenzaldehyde in alcohol. They also regenerated the parent pyrrocolines when treated with cold sodium hydroxide solution.

3(or 1)-p-*Dimethylaminobenzylidene*-2-*methylpyrrocolinium Iodide.*—A solution of 2-methylpyrrocoline (2·62 g.) and *p*-dimethylaminobenzaldehyde (2·98 g.) in ethanol (60 ml.) was treated with 55% hydriodic acid (2·8 ml.) and after 15 min. the deep blue *dye* (8·4 g.) was collected, washed with ethanol, and dried at 100°/4 mm. (Found: C, 55·4; H, 5·2; N, 7·3. $C_{18}H_{19}N_2I$ requires C, 55·4; H, 4·9; N, 7·2%). The product (no definite m. p.) was soluble in hot nitromethane but could not be recrystallised; it was only very sparingly soluble in most other solvents.

We thank Dr. L. J. Edwards for the ultraviolet absorption measurements, Dr. J. Kenyon, F.R.S., for his interest, and the Directors of Beecham Research Laboratories Limited for the provision of laboratory facilities and materials.

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[*Received, January 20th,* 1955.]