53. *New Syntheses of Heterocyclic Compounds. Part V. The Schmidt Rearrangement of* 1 : 3-*Dimethyl-2-azafluorenone.*

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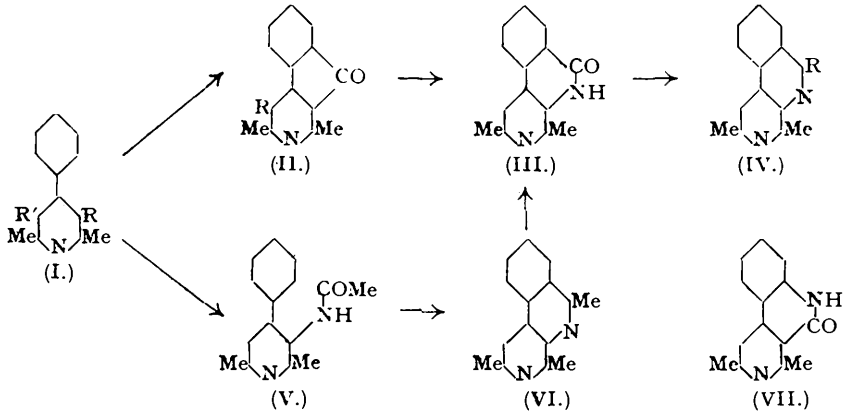
With the object of preparing naphthyridine derivatives suitable for biological examination, 1 : 3-dimethyl-2-azafluorenone (II; R = H) has been submitted to the Schmidt reaction and a *substance*, $C_{14}H_{12}ON_2$, obtained in yields approaching 90%. This compound may be represented by either (III) or (VII). Its formulation as a 9-*hydroxy*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (2-*hydroxy*-3 : 4-*benz*-6 : 8-*dimethyl*-1 : 7-*naphthyridine*) (III) followed from the observation that the same compound was obtained by oxidation of 1 : 3 : 9-*trimethyl*-2 : 10-*diazaphenanthrene* (VI) with chromic acid. 9-Hydroxy-1 : 3-dimethyl-2 : 10-diazaphenanthrene has been converted into the *chloro*-derivative (IV; R = Cl) and hence into the *amino*-, *piperidino*- and *phenoxy*-derivatives.

THE object of the present investigation was to study the ring enlargement of 1 : 3-dimethyl-2-azafluorenone (II; R = H) by the Beckmann and Schmidt rearrangements so as to obtain a hydroxydimethyldiazaphenanthrene (hydroxybenzdimethylnaphthyridine). This was required as part of a wider programme dealing with the relation between chemical constitution and biological action in the naphthyridine series. Such ring systems involving two adjacent pyridine nuclei may well be expected to possess some degree of biological interest. It was therefore not surprising that certain members of the group, prepared in these laboratories, have shown outstanding properties on preliminary examination. The biological results, on completion, will be referred to in a subsequent communication.



The Beckmann rearrangement of fluorenone oxime to phenanthridone was first described by Beckmann and Wegerhoff (*Annalen*, 1889, **252**, 39; cf. Pictet, *Chem. Zeit.*, 1894, **18**, 1822; Kerp, *Ber.*, 1896, **29**, 230). The yield was very low, but was subsequently increased to 30% by Pictet and Gonset (*Arch. Sci. phys. nat.*, 1897, **8**, 3751). Moore and Huntress (*J. Amer. Chem. Soc.*, 1927, **49**, 2618), by carrying out the rearrangement with phosphorus pentachloride in phosphorus oxychloride, still further improved the yield, preparing 7-nitrophenanthridone from 2-nitrofluorenone oxime. Attempts to apply this reaction to the oxime of 1 : 3-dimethyl-2-azafluorenone (Borsche and Hahn, *Annalen*, 1939, **537**, 229) under a variety of experimental conditions were uniformly unsuccessful. Extensive resinification occurred or the starting materials were recovered unchanged. This result may be related to the stereochemical configuration of the oxime since Jeiteles (*Monatsh.*, 1896, **17**, 515) has shown that only one of the two oximes of phenyl 3-pyridyl ketone undergoes a Beckmann rearrangement to give a product hydrolysed to nicotinic acid and aniline [cf. (II; R = H) → (III) described below].

Attention was therefore directed to the Schmidt reaction (*Ber.*, 1924, **57**, 704; 1925, **58**, 2413) whereby cyclic ketones suffer ring enlargement when treated with hydrazoic acid in the presence of a suitable catalyst. This reaction forms the basis for the synthesis of phenanthridones from fluorenones described in B.P. 333,173. It has been further improved and its scope considerably extended by Walls (*J.*, 1935, 1405) who modified the experimental conditions, replacing a benzene solution of hydrazoic acid by a concentrated aqueous solution of the commercially available sodium azide, which was added to a cooled solution of the ketone in concentrated sulphuric acid.

When 1 : 3-dimethyl-2-azafluorenone (Mills, Palmer, and Tomkinson, *J.*, 1924, 2365) (II; R = H) was submitted to the Schmidt reaction, a homogeneous *hydroxydimethyldiazaphenanthrene* was readily obtained in yields approaching 90%. The rearrangement of the unsymmetrical fluorenone (II; R = H) may give rise to either a 9-hydroxy-1 : 3-dimethyl-2 : 10-diazaphenanthrene (III) or to a 9-hydroxy-6 : 8-dimethyl-7 : 10-diazaphenanthrene (VII).* A decision in favour of the former alternative was made possible by a study of the oxidation of 1 : 3 : 9-*trimethyl*-2 : 10-*diazaphenanthrene* (VI), which was obtained fairly readily by the following series of reactions: 4-Phenyl-lutidine dicarboxylic ester (I; R = R′ = CO_2Et) gave a monoester on partial hydrolysis (Hantzsch, *Ber.*, 1884, **17**, 2910) from which 4-phenyl-lutidine-3-carboxylic ester (I; R = CO_2Et, R′ = H) was obtained by distillation at atmospheric pressure. Conversion of this ester to the acid chloride (Borsche and Hahn, *ibid.*, p. 228) followed by treatment with aqueous ammonia gave the *amide*, smoothly converted by hypobromite into 3-*amino*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NH_2, R′ = H). The *acetyl derivative* (V) of this amine failed to undergo ring closure with such condensing agents as anhydrous zinc chloride or phosphorus oxychloride. Fusion with phosphorus pentoxide at 270° (cf. Petrow, Stack, and Wragg, *J.*, 1943, 316) gave 1 : 3 : 9-trimethyl-2 : 10-diazaphenanthrene (VI) in *ca.* 10% yield; but, when the acetyl derivative (V) was heated with phosphorus pentoxide in ψ-cumene the yield of (VI) rose to 30%. 3-*Benzamido*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NHBz, R′ = H) similarly gave 9-*phenyl*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (IV; R = Ph).

Walls (*J.*, 1935, 1405) has shown that oxidation of 9-methylphenanthridines with chromic acid leads to the formation of the corresponding phenanthridones in nearly quantitative yield, and it was therefore expected that (VI), which bears a formal analogy to 9-methylphenanthridine, would behave in a similar way. Oxidation of 1 : 3 : 9-trimethyl-2 : 10-diazaphenanthrene (VI) with chromic acid gave a complex mixture of products from which 9-*hydroxy*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (III) was readily isolated owing to its low solubility in alcohol. This compound proved to be identical in m. p. and mixed m. p. with the rearrangement product of 1 : 3-dimethyl-2-azafluorenone with hydrazoic acid, to which must therefore be assigned the formulation (III).

A parallel series of experiments were also carried out with the object of preparing 4-amino-1 : 3-dimethyl-2-azafluorenone (II; R = NH_2). This compound was required for alkaline fusion when, in analogy with the quantitative conversion of 4-aminofluorenone into phenanthridone (Graebe and Schestakow, *Annalen*, 1895, **284**, 312) it was hoped to obtain a compound represented by formula (III). 4-Phenyl-lutidinedicarboxylic acid monoethyl ester (I; R = CO_2H, R′ = CO_2Et) (Hantzsch, *loc. cit.*) was converted into the *ester-amide via* the acid chloride and hence, by hypobromite treatment, into 3-*amino*-5-*carbethoxy*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NH_2, R′ = CO_2Et). Hydrolysis followed by acetylation gave the *diacetyl derivative* of the corresponding *acid* (I; R = NAc_2, R′ = CO_2H). Attempts to convert this compound into (II; R = NH_2) by ring closure with sulphuric acid were unsuccessful. This failure may have been due to the facile hydrolysis of the diacetyl group. As it was hoped that the benzamido group might prove more resistant to hydrolysis, 3-*benzamido*-5-*carbethoxy*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NHBz, R′ = CO_2H) was prepared by hydrolysis of the corresponding *ester* (I; R = NHBz, R′ = CO_2Et). On submitting this compound to ring closure experiments, the initial action again appeared to be hydrolysis of the benzamido group and further experiments in this direction were abandoned.

Treatment of 9-hydroxy-1 : 3-dimethyl-2 : 10-diazaphenanthrene (III) with phosphorus pentachloride in phosphorus oxychloride suspension in a sealed tube at 180° led to the formation of the corresponding *chloro-compound* (IV; R = Cl), from which the *phenoxy-*, *amino-*, and *piperidino-derivatives* were obtained. Reduction of (IV; R = Cl) with red phosphorus and hydriodic acid in a sealed tube was not successful, the hydroxy compound (III) being regenerated unchanged. Hydrazoic acid was without action on a solution of (III) in concentrated sulphuric acid, there being no evidence for the formation of a tetrazole derivative (cf. ε-leucine lactam → pentamethylenetetrazole, Schmidt, *Ber.*, 1924, **57**, 706).

Experimental.

M. ps. are corrected.

9-*Hydroxy*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (III).—1 : 3-Dimethyl-2-azafluorenone (160 g.) (Mills, Palmer, and Tomkinson, *loc. cit.*) dissolved in concentrated sulphuric acid (1000 ml.) was treated at 0° whilst mechanically stirred with a solution of sodium azide (90 g.) in water (250 ml.), added dropwise over a period of 5 hours. After some hours the mixture was poured on ice (2500 g.). The precipitated sulphate was collected after 12 hours and the base regenerated by treatment with dilute ammonia on the water-bath. After extraction with spirit (1500 ml.) it (90%) is sufficiently pure for conversion into the chloro compound. 9-*Hydroxy*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* formed prismatic

* *Note on nomenclature.*—The nomenclature employed is based upon phenanthrene and not upon benznaphthyridine. Furthermore, the phenanthrene nucleus is so numbered that the substituent in the middle ring is always attached to C_9. This is necessary in order to simplify subsequent discussion on the relationship between structure and biological activity.

needles from nitrobenzene, m. p. 319—320° (Found: C, 75·0; H, 5·4; N, 12·5. $C_{14}H_{12}ON_2$ requires C, 75·0; H, 5·4; N, 12·5%). A saturated alcoholic solution gives a faint but distinct yellow colouration with ferric chloride solution. It is sparingly soluble in aqueous sodium hydroxide, but readily soluble in sodium ethoxide solution.

9-*Chloro*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (IV; R = Cl).—The very finely powdered hydroxy compound (2·0 g.), phosphorus pentachloride (2·5 g.), and phosphorus oxychloride (15 ml.) were heated in a sealed tube at 180° for 4 hours. After 12 hours at room temperature the product was collected, decomposed with ice, thoroughly extracted with 2N-hydrochloric acid, and the base precipitated by addition of ammonium hydroxide. After extraction with chloroform, it (40%) was crystallised (charcoal) from ligroin. 9-*Chloro*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* formed needles, m. p. 153—154° (Found: Cl, 14·4. $C_{14}H_{11}N_2Cl$ requires Cl, 14·6%).

9-*Phenoxy*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (IV; R = OPh).—The chloro compound (1·2 g.) in phenol (5 g.) was heated at 180° for 5 hours. The product was digested with excess sodium hydroxide solution and the insoluble fraction isolated with chloroform. The product was extracted twice with ligroin (20 ml. each portion, b. p. 100—120°). The cooled extracts were decanted from some resinous material, the solvent removed and the residue, in alcohol, treated with picric acid. 9-*Phenoxy*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene picrate* (1·75 g.) formed bright yellow plates from spirit, m. p. 203—203·5° (Found: N, 13·1. $C_{20}H_{16}ON_2,C_6H_3O_7N_3$ requires N, 13·2%). The *base*, regenerated by shaking the picrate with 10% sodium hydroxide solution, formed needles, m. p. 112—113° from ligroin (Found: C, 80·2; H, 5·4; N, 8·8. $C_{20}H_{16}ON_2$ requires C, 80·0; H, 5·3; N, 9·3%).

9-*Amino*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (IV; R = NH_2).—The chloro compound (2·7 g.) with saturated alcoholic ammonia (15 ml.) was heated in a sealed tube at 180—190° for 16 hours. The product was taken to dryness, extracted with water (charcoal) and the filtrate made alkaline with sodium hydroxide. The precipitated base was purified by means of the *picrate*, silky yellow needles, m. p. 261—262° (decomp.), from alcohol (Found: N, 18·6. $C_{14}H_{13}N_3,C_6H_3O_7N_3$ requires N, 18·6%). 9-*Amino*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* formed cubic crystals, m. p. 189·5—190·5°, from ligroin containing a trace of alcohol (Found: C, 75·3; H, 5·9; N, 18·9. $C_{14}H_{13}N_3$ requires C, 75·3; H, 5·8; N, 18·8%). The *dihydrochloride* (60%) formed octahedra from aqueous alcoholic hydrogen chloride, m. p. >310° (Found: Cl, 22·7. $C_{14}H_{13}N_3,2HCl,H_2O$ requires Cl, 22·6%).

9-*Piperidino*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (IV; R = NC_5H_{10}).—The chloro compound (1·5 g.) and piperidine (9 ml.) were heated in a sealed tube at 180° for 5 hours. The product was taken to dryness on the water-bath, stirred with a few drops of sodium hydroxide solution, and the free base extracted with chloroform and treated, in alcoholic solution, with picric acid. 9-*Piperidino*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene picrate* formed yellow plates, m. p. 228—229°, from alcohol (Found: N, 16·2. $C_{19}H_{21}N_3,C_6H_3O_7N_3$ requires N, 16·1%). The *base*, regenerated from the picrate with sodium hydroxide solution, formed octahedra, m. p. 113·5—114·5°, from aqueous methanol (Found: C, 78·2; H, 7·5; N, 14·3. $C_{19}H_{21}N_3$ requires C, 78·3; H, 7·2; N, 14·4%). The *dihydrochloride* separated from alcoholic hydrogen chloride in small needles, m. p. 165—166° (Found: Cl, 18·9. $C_{19}H_{21}N_3,2HCl$ requires Cl, 19·5%).

4-*Phenyl*-2 : 6-*dimethylpyridine*-3-*carboxyamide* (I; R = $CONH_2$, R′ = H).—The corresponding crude acid chloride (27 g., Borsche and Hahn, *loc. cit.*) in warm benzene (50 ml.) was added dropwise to ammonia (150 ml., *d*, 0·880) whilst mechanically stirred and cooled below 10°. After some hours, the product (23 g.) was collected and crystallised from acetone. 4-*Phenyl*-2 : 6-*dimethylpyridine*-3-*carboxyamide* formed plates, m. p. 198—199° (Found: C, 74·3; H, 6·2; N, 12·5. $C_{14}H_{14}ON_2$ requires C, 74·3; H, 6·2; N, 12·4%).

3-*Amino*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NH_2, R′ = H).—The crude amide (90 g.), finely powdered, was added at 0° to a mechanically stirred solution of bromine (67·5 g.) in potassium hydroxide (1125 ml., 10%). After one hour, potassium hydroxide (675 ml. of 10%) was added to the clear solution and the mixture heated for 1½ hours on the water-bath. The base (51 g.) was isolated with benzene (500 ml.) (charcoal). 3-*Amino*-4-*phenyl*-2 : 6-*dimethylpyridine* formed squat needles, m. p. 87—87·5°, from ligroin (Found: C, 78·8; H, 7·1; N, 14·3. $C_{13}H_{14}N_2$ requires C, 78·8; H, 7·1; N, 14·1%).

3-*Acetamido*-4-*phenyl*-2 : 6-*dimethylpyridine* (V).—The amino compound (20 g.), dry pyridine (60 ml.) and acetic anhydride (20 g.) were heated under reflux for 30 minutes. The product was made alkaline with ammonia, transferred to an evaporating basin and taken to dryness on the water-bath. The residue (almost quantitative) was crystallised (charcoal) either from aqueous acetone or from benzene–ligroin. 3-*Acetamido*-4-*phenyl*-2 : 6-*dimethylpyridine* formed flat needles containing solvent of crystallisation, m. p. 162—163° (Found: C, 75·0; H, 6·7; N, 11·5. $C_{15}H_{16}ON_2$ requires C, 75·0; H, 6·7; N, 11·7%).

1 : 3 : 9-*Trimethyl*-2 : 10-*diazaphenanthrene* (VI).—3-Acetamido-4-phenyl-2 : 6-dimethylpyridine (10 g.) dissolved in dry technical ψ-cumene (30 ml.) in a 250 ml. flask was treated with phosphorus pentoxide (30 g.), and the mass rapidly mixed with a glass rod until fairly homogeneous. The mixture was heated under reflux (calcium chloride tube) at 200—210° for 8 hours. The product was decomposed with water, the base precipitated with ammonia, dissolved in spirit and treated with alcoholic picric acid. The picrate, m. p. 242—243° (decomp.), was crystallised once from a very large volume of spirit and the base regenerated with 10% sodium hydroxide solution. 1 : 3 : 9-*Trimethyl*-2 : 10-*diazaphenanthrene* formed long faintly yellow glittering needles, m. p. 132·5—133°, from ligroin (Found: C, 80·8; H, 6·2; N, 12·4. $C_{15}H_{14}N_2$ requires C, 81·1; H, 6·3; N, 12·6%). The *hydrochloride* separated from spirit in small crystals, m. p. >310° (Found for air-dried product: Cl, 12·6. $C_{15}H_{14}N_2,HCl,H_2O$ requires Cl, 12·8%).

9-*Hydroxy*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (III).—1 : 3 : 9-Trimethyl-2 : 10-diazaphenanthrene (1·8 g.), glacial acetic acid (12 ml.), 2N-sulphuric acid (12 ml.), and finely powdered potassium dichromate (4·5 g.) were heated under reflux for 6 hours. The product was poured into excess ammonium hydroxide and the insoluble material collected and crystallised (200 mg.) three times from spirit. 9-*Hydroxy*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* formed silky colourless needles, m. p. 320—321° (Found: C, 74·8; H, 5·3; N, 12·4. $C_{14}H_{12}ON_2$ requires C, 75·0; H, 5·4; N, 12·5%), not depressed in admixture with a sample prepared by the rearrangement of 1 : 3-dimethyl-2-azafluorenone with hydrazoic acid (above).

3-*Benzamido*-4-*phenyl*-2 : 6-*dimethylpyridine*, needles, m. p. 213—214°, from aqueous methyl alcohol (Found: C, 78·7; H, 6·0; N, 9·3. $C_{20}H_{18}ON_2$ requires C, 79·5; H, 6·0; N, 9·3%) was prepared by treating the base (900 mg.) in dry pyridine (2 ml.) with benzoyl chloride (700 mg.) for 1 hour on the water-bath.

9-*Phenyl*-1 : 3-*dimethyl*-2 : 10-*diazaphenanthrene* (IV; R = Ph).—Prepared by ring closure of 3-benzamido-4-phenyl-2 : 6-dimethylpyridine with phosphorus pentoxide in ψ-cumene (as above) separated (30%) with water of crystallisation as felted silky needles, m. p. 131—132°, from aqueous methanol (Found: C, 84·1; H, 5·5; N, 10·0. $C_{20}H_{16}N_2$ requires C, 84·5; H, 5·6; N, 9·9%). The *picrate* formed bright yellow needles, m. p. 206—207°, from alcohol (Found: N, 13·6. $C_{20}H_{16}N_2,C_6H_3O_7N_3$ requires N, 13·6%). The *monohydrochloride* separated from alcohol in stout needles, m. p. >300° (Found: Cl, 10·6. $C_{20}H_{16}N_2,HCl$ requires Cl, 10·8%).

5-*Carbethoxy*-4-*phenyl*-2 : 6-*dimethylpyridine*-3-*carboxyamide* (I; R = $CONH_2$; R′ = CO_2Et).—The corresponding acid-ester (30 g.) (Hantzsch, *loc. cit.*) was gently refluxed with thionyl chloride (100 ml.) for 30 minutes. The product was taken to dryness under reduced pressure on the water-bath and the residual oil, dissolved in benzene (50 ml.), added dropwise to mechanically stirred ammonium hydroxide (150 ml., d, 0·880) cooled below 10°. After 24 hours at the ordinary temperature, the precipitated material was collected and purified from aqueous acetone. 5-*Carbethoxy*-4-

phenyl-2 : 6-*dimethylpyridine*-3-*carboxyamide* (18 g.) formed flat needles, m. p. 180—181° (Found : C, 68·5; H, 6·0; N, 9·6. $C_{17}H_{18}O_3N_2$ requires C, 68·5; H, 6·0; N, 9·4%).

3-*Amino*-5-*carbethoxy*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NH_2, R′ = CO_2Et).—The finely powdered amide (26 g.) was added in one portion to a mechanically stirred ice-cold solution of bromine (15 g.) in potassium hydroxide (250 ml., 10%). After 45 minutes, a further volume of potassium hydroxide (150 ml., 10%) was added to the clear solution. The mixture was heated on the water-bath for 30 minutes and the amide salted out by addition of sodium chloride (100 g.). After some hours at 0°, the crystalline product was collected. The *compound* (50%) formed needles, m. p. 124·5—125·5°, from ligroin (Found : C, 70·8; H, 6·5; N, 10·4. $C_{16}H_{18}O_2N_2$ requires C, 71·1; H, 6·7; N, 10·4%).

3-*Diacetylamino*-5-*carboxy*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NAc_2, R′ = CO_2H).—The above amino-ester (10 g.), potassium hydroxide (6·8 g.), and absolute alcohol (22 ml.) were heated under reflux for 3 hours on the water-bath. The mixture was diluted to *ca.* 250 ml. with absolute alcohol and carbon dioxide passed in until precipitation was complete. The filtrate was taken to dryness on the water-bath and the crystalline residue heated under reflux with acetic anhydride (100 ml.) for 1 hour. The product was taken to dryness on the water-bath under reduced pressure and the residue crystallised from alcohol. 3-*Diacetylamino*-5-*carboxy*-4-*phenyl*-2 : 6-*dimethylpyridine* formed octahedra, m. p. 240—241° (Found : C, 66·4; H, 5·5; N, 8·6. $C_{18}H_{18}O_4N_2$ requires C, 66·3; H, 5·5; N, 8·6%).

3-*Benzamido*-5-*carbethoxy*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NHBz, R′ = CO_2Et).—Crude 3-amino-5-carbethoxy-4-phenyl-2 : 6-dimethylpyridine (58 g.) dissolved in dry pyridine (200 ml.) was treated with benzoyl chloride (30 g.) with cooling. Reaction was completed by heating for 30 minutes on the water-bath. 3-*Benzamido*-5-*carbethoxy*-4-*phenyl*-2 : 6-*dimethylpyridine*, glittering needles (66%) from aqueous methyl alcohol, separated in dimorphic forms having melting points of 156—157° and 170—171° (Found : C, 73·7; H, 6·1; N, 7·4. $C_{23}H_{22}O_3N_2$ requires C, 73·8; H, 5·9; N, 7·5%).

3-*Benzamido*-5-*carboxy*-4-*phenyl*-2 : 6-*dimethylpyridine* (I; R = NHBz, R′ = CO_2H).—The corresponding ester (52·3 g.), potassium hydroxide (8·5 g.), and absolute alcohol (100 ml.) were heated under reflux for 80 hours. The alcohol was removed on the water-bath and the product extracted several times with small quantities of water (28 g. unchanged material recovered). The extracts and washings were acidified with sulphuric acid (8·5 g.) diluted with a little water, and the precipitated acid (19 g.) crystallised from alcohol–light petroleum. The *compound* formed stout needles, m. p. 276—277° (decomp.) (Found : C, 72·8; H, 5·2; N, 8·2. $C_{21}H_{18}O_3N_2$ requires C, 72·8; H, 5·2; N, 8·1%).

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