674. *Synthetic Analgesics and Related Compounds. Part V.**
2-*Imino*-3 : 3-*diphenylpyrrolidines.*

By Walter Wilson.

Several γ-benzylmethylamino-cyanides of the amidone series are converted smoothly in boiling hydrochloric acid into 2-iminopyrrolidines and benzyl chloride. The mechanism of the reaction is discussed and also the conversion of the products by nitrous acid into pyrrolid-2-ones. The latter characterise the original 2-iminopyrrolidines.

In Part I (*J.*, 1950, 2173) the 2-iminopyrrolidine structure (II; R = Me, R′ = H) was proposed for a base (m. p. 103°) obtained by heating 3-dimethylamino-1 : 1-diphenylbutyl cyanide (I; R = Me) with ethylenediamine (both as arylsulphonates). It has been reported that 2-iminopyrrolidines are formed by heating salts of γ-dialkylamino-cyanides of the amidone series (Blicke and Zambito, Abs. 111th Meeting Amer. Chem. Soc., 1947, 3K; Blicke, U.S.P., 2,513,279; *Chem. Abs.*, 1951, **45**, 5187). Some possible alternative routes to 2-iminopyrrolidines have now been examined.

The cyanide (III; R = Ph) was readily obtained by condensing 2-methylanilinoethyl chloride with diphenylmethyl cyanide. Attempts to remove the *N*-phenyl group by nitrosation and digestion with alkali failed. The acids (IV; R = H and R = Me) were produced in good yields from methyl acrylate and 1-methacrylate, respectively, and diphenylmethyl cyanide in sodium ethoxide; (IV; R = H) has been obtained previously by a less convenient method using ethyl 3-bromopropionate (Trivedi *et al.*, *J. Univ. Bombay*, 1942, **10**, 135). The amide of (IV; R = H) gave an anomalous product under the conditions of the Hofmann reaction; similarly an anomalous product was obtained from the hydrazide of (IV; R = Me) by the Curtius reaction.

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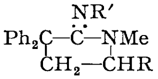
* Part IV, *J.*, 1952, 1321.

The γ-benzylmethylamino-cyanide (III; R = CH_2Ph) was converted smoothly into benzyl chloride and the 2-iminopyrrolidine (II; R = R′ = H) (hydrochloride, m. p. 223·5—224°) in boiling hydrochloric acid. The structure of the product was confirmed by the formation of the known pyrrolid-2-one (VII; R = H) with nitrous acid. The unidentified hydrochloride, m. p. 226—228°, obtained by Morrison and Rinderknecht (*J.*, 1950, 1478) by hydrogenating the hydrochloride of the cyanide (III; R = $CHPh_2$) was probably an impure (hydrated?) form of the same 2-iminopyrrolidine hydrochloride.

$Ph_2C(CN){\cdot}CH_2{\cdot}CHMe{\cdot}NMeR$

(I)



(II)

$Ph_2C(CN){\cdot}CH_2{\cdot}CH_2{\cdot}NMeR$

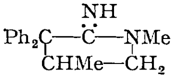
(III)

$Ph_2C(CN){\cdot}CH_2{\cdot}CHR{\cdot}CO_2H$

(IV)

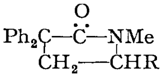
$Ph_2C(CN){\cdot}CHMe{\cdot}CH_2{\cdot}NMe{\cdot}CH_2Ph$

(V)

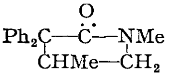


(VI)

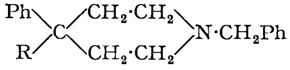
Two isomeric basic cyanides (I; R = CH_2Ph) and (V) were obtained by treating diphenylmethyl cyanide with *N*-benzyl-2-chloro-*N*-methylpropylamine; formation of two isomers in similar reactions has been observed previously (*e.g.*, Bockmuhl and Ehrhart, *Annalen*, 1948, **561**, 52; Schultz, Robb, and Sprague, *J. Amer. Chem. Soc.*, 1947, **69**, 2454; Ofner, *J.*, 1951, 1800). Both basic cyanides were smoothly debenzylated in boiling hydrochloric acid. The resulting 2-iminopyrrolidines (II; R = Me, R′ = H) and (VI) were isolated as the hydrochlorides, which gave the known pyrrolid-2-ones (VII; R = Me) and (VIII) respectively, with nitrous acid. The structures of the two iminopyrrolidines and of the original basic cyanides are therefore established. Cymerman and Gilbert (*J.*, 1952, 3529) has obtained (II; R = Me, R′ = H) from 3-dimethylamino-1 : 1-diphenylbutyl cyanide (I; R = Me) and ammonium benzenesulphonate at 260°; specimens of the base and hydrochloride provided by Dr. Cymerman were identical with the materials obtained here. The compound (m. p. 103°), for which the structure (II; R = Me, R′ = H) was proposed in Part I, is different, and is being examined further. The 2-iminopyrrolidine (II; R = Me, R′ = H) gave an *N*-acetyl derivative, which was *soluble* in dilute acid. This provides independent confirmation of the structure, as the compound (II; R = Me, R′ = Ac) could be basic, but the open-chain isomer (I; R = Ac) would be neutral.



(VII)



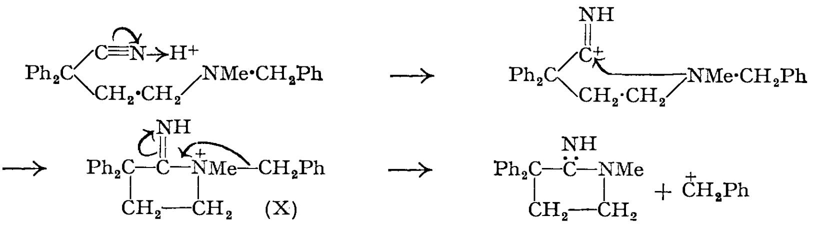
(VIII)



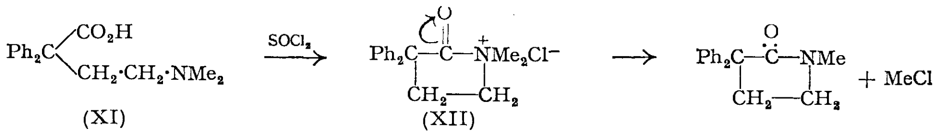
(IX)

The effective reagent in the debenzylating ring closures described is probably the water–hydrogen chloride azeotrope at the boiling point (108·6°/760 mm.; 20·2% HCl by wt.). These conditions are much milder than those customary for the pyrolytic debenzylation of simple tertiary benzylammonium chlorides (*e.g.*, Collie and Schryver, *J.*, 1890, 781, used temperatures of >300°).

It is believed that 2-iminopyrrolidines and benzyl chloride are formed from γ-benzylmethylamino-cyanides by a two-stage process: first, cyclisation, then elimination of the benzyl group from the intermediate cyclic quaternary salt (X):



The first stage is analogous to the well-known formation of amidines from cyanides and amine salts (Oxley and Short, *J.*, 1946, 147, and later papers), whilst the removal of the benzyl cation from (X) would be facilitated by the electromeric displacement $>C\overset{\curvearrowright}{=}NH$. The preferential elimination of benzyl instead of methyl would be expected, as in the simpler cases discussed by Hanhart and Ingold (*J.*, 1927, 1002). Attempts to make the chlorides of acids such as (XI) often yield pyrrolidones (Clarke, Mooradian, Lucas, and Slauson, *J. Amer. Chem. Soc.*, 1949, **71**, 2821; Dupré, Elks, Hems, Speyer, and Evans, *J.*, 1949, 502; Walton, Ofner, and Thorp, *J.*, 1949, 649). In this reaction, it is probable that an intermediate is the cyclic quaternary salt (XII). Elimination of an alkyl group would be facilitated by the $>C\overset{\curvearrowright}{=}O$ displacement, and thus there is a close resemblance to the proposed mechanism of iminopyrrolidine formation :



It would be sterically difficult for the *N*-benzylpiperidyl cyanide (IX; R = CN) to form a cyclic quaternary salt analogous to (X). Indeed, this cyanide was not debenzylated under the usual conditions, but was converted slowly into the corresponding carboxylic acid (IX; R = CO_2H).

The formation of pyrrolid-2-ones from 2-iminopyrrolidines and nitrous acid is difficult to explain in the examples studied because imino-amino-tautomerism is impossible. The following mechanism seems feasible, and recalls the scheme proposed by Barrott, Gillibrand, and Lamberton (*J.*, 1951, 1285) for the alkali-catalysed decomposition of nitramines ($>CH{\cdot}NH{\cdot}NO_2 \longrightarrow >C{:}O + N_2 + H_2O$) :

$$>C{=}NH \xrightarrow{HNO_2} >C\overset{\curvearrowright}{=}N{-}N\overset{\curvearrowright}{=}O \xrightarrow{H^+} >\overset{+}{C}{-}N{=}N{-}OH \xrightarrow{OH} >C(OH){\cdot}N{:}N{\cdot}OH \longrightarrow >C{:}O + N_2 + H_2O$$

EXPERIMENTAL

2-*Methylanilinoethyl Chloride.*—Methylaniline (160·5 g.) and ethylene chlorohydrin (80·5 g.) were heated at 100° under reflux (Laun, *Ber.*, 1884, **17**, 676, used a sealed tube) for 35 hours. Distillation gave 2-methylanilinoethanol (198 g., 88%), b. p. 150°/14 mm., which with phosphorus oxychloride in benzene (Anker and Cook, *J.*, 1944, 490) gave 2-methylanilinoethyl chloride (40%), b. p. 124—127°/19 mm. The latter became intensely blue when kept, and formed a *picrate*, m. p. 108—109·5°, bright yellow prisms from ethanol (Found : C, 45·3; H, 3·85. $C_9H_{12}NCl,C_6H_3O_7N_3$ requires C, 45·1; H, 3·75%).

3-*Methylanilino*-1 : 1-*diphenylpropyl Cyanide.*—Diphenylmethyl cyanide (19·3 g.) in benzene (60 c.c.) was stirred with powdered sodamide (4·7 g.) for 30 minutes at 40°. 2-Methylanilinoethyl chloride (17 g.) in benzene (20 c.c.) was added and the mixture heated at 70—80° for 6 hours. The benzene solution was washed with water then with 3N-hydrochloric acid. The aqueous acid extract was basified, which caused precipitation of 2-methylanilinoethyl chloride (3·6 g.), b. p. 122—126°/18 mm., identified as the picrate. Evaporation of the benzene solution and recrystallisation of the residue from ethanol gave 3-*methylanilino*-1 : 1-*diphenylpropyl cyanide* (25 g.) as white needles, m. p. 143·5—144° (Found : C, 85·05, 85·3; H, 6·5, 6·9; N, 9·1. $C_{23}H_{22}N_2$ requires C, 84·7; H, 6·75; N, 8·6%). The compound was almost insoluble in dilute hydrochloric acid. Nitrous acid gave an unstable green nitroso-derivative, which polymerised rapidly, and gave no amine with aqueous sodium hydroxide.

4-*Cyano*-4 : 4-*diphenylbutyric Acid.*—A solution of diphenylmethyl cyanide (50 g.) in ethanolic sodium ethoxide [from ethanol (60 c.c.) and sodium (6 g.)] was cooled to 0° and methyl acrylate (30 g.) added slowly. The mixture was gently refluxed for 2 hours, then cooled, and 5N-sodium hydroxide (125 c.c.) was added. After 36 hours, the homogeneous mixture was poured into excess of water, and the acid (39·2 g.; m. p. 157—160°) precipitated with hydrochloric acid. It had m. p. 160° after recrystallisation from benzene–light petroleum (b. p. 40—60°) (Trivedi *et al.*, *J. Univ. Bombay*, 1942, **10**, 135, give m. p. 160°). The acid (8·5 g.) with thionyl chloride and then ammonia, gave the *amide* (7 g., 83%), which crystallised from aqueous methanol in glistening flakes, m. p. 129° (Found : N, 10·85. $C_{17}H_{16}ON_2$ requires N, 10·6%) ; treatment with bromine and sodium methoxide did not give the expected basic product.

γ-Cyano-α-methyl-γ : *γ-diphenylbutyric Acid*.—In a condensation similar to the preceding, methyl 1-methylacrylate gave, after recrystallisation of the crude solid from benzene–light petroleum (b. p. 100—120°), the homologous *acid* (89%), m. p. 140·5° (Found : C, 77·45; H, 6·05. $C_{18}H_{17}O_2N$ requires C, 77·5; H, 6·1%). The acid (10 g.) in ethanol (50 c.c.) was mixed with acetyl chloride (2 c.c.), left for 18 hours, then refluxed for 30 minutes. Evaporation gave the *ethyl* ester (8·6 g., 78%), which formed small prisms, m. p. 110°, from benzene–light petroleum (Found : N, 4·6. $C_{20}H_{21}O_2N$ requires N, 4·55%). The ester (1·0 g.) with aqueous-alcoholic 2N-sodium hydroxide (25 c.c.) at 20° for 48 hours gave the original acid (0·85 g.), m. p. 140°. The ester (3·1 g.), when heated under reflux for 8 hours with 90% hydrazine hydrate (4·5 c.c.) in ethanol (10 c.c.), gave the *hydrazide* (2·55 g., 86%), which crystallised from ethanol as glistening prisms, m. p. 195—196° (Found : N, 14·05. $C_{18}H_{19}ON_3$ requires N, 14·35%). Heating the hydrazide (100 mg.) with benzaldehyde (100 mg.) in ethanol (5 c.c.) for 1½ hours gave the *benzylidenehydrazide* (110 mg.), which formed pale yellow prisms, m. p. 199·5—200°, from ethanol (Found : N, 11·05. $C_{25}H_{23}ON_3$ requires N, 11·0%). A normal Curtius reaction did not occur when the hydrazide was treated with nitrous acid ; the product was soluble in alkali.

2-*Benzylaminoethanol*.—A modification of the method of Rumpf and Kwass (*Bull. Soc. chim.*, 1943, **10**, 349) was used. A mixture of 2-hydroxyethylamine (61 g.) and benzyl chloride (63·3 g.) was set aside for 2 weeks, then heated to 100° for 6 hours. Excess of sodium hydroxide solution was added and the oily upper layer distilled, giving 2-benzylaminoethanol (33 g., 44%), b. p. 153—156°/12 mm., and 2-dibenzylaminoethanol (15 g., 25%), b. p. 195—205°/16 mm. (yields are calculated on benzyl chloride).

2-*Benzylmethylaminoethanol*.—(a) From 2-*benzylaminoethanol*. The amino-alcohol (27 g.), paraformaldehyde (5·4 g.), and formic acid (30 c.c. ; *d* 1·2) were heated together at 70—80°, and, when the vigorous reaction subsided, refluxed for 4 hours. The liquid was evaporated to small bulk at 100°/15 mm., and excess of sodium hydroxide added. The base (25 g., 85%), isolated by extraction with ether–benzene, had b. p. 132—133°/12 mm., n_D^{22} 1·5225.

(b) *From benzylmethylamine*. A mixture of benzylmethylamine (24·2 g.) and ethylene chlorohydrin (16·1 g.) was set aside for 3 days, then heated at 100° for 7 hours. Isolation as in (*a*) gave 2-benzylmethylaminoethanol (17 g., 51%), b. p. 129—132°/12 mm., n_D^{22} 1·5270 (cf. Mannich and Kuphal, *Arch. Pharm.*, 1912, **250**, 542, who used a sealed tube for the reaction, and Ofner and Walton, *J.*, 1950, 2166, who used ethylene oxide instead of chlorohydrin).

2-*Benzylmethylaminoethyl Chloride*.—2-Benzylmethylaminoethanol (41 g.) was converted into the crude chloroamine hydrochloride (Sheehan and Mumaw, *J. Amer. Chem. Soc.*, 1950, **72**, 2129; Ofner and Walton, *J.*, 1950, 2165). The crude salt was dissolved in water and filtered through kieselguhr, and 10N-sodium hydroxide (30 c.c.) was added. The chloro-amine (36 g., 79%), b. p. 120°/16 mm., n_D^{18} 1·5238, was isolated by ether. The *picrate* formed needles, m. p. 103—104°, from ethanol (Found : N, 13·65. $C_{10}H_{14}NCl,C_6H_3O_7N_3$ requires N, 13·6%).

3-*Benzylmethylamino*-1 : 1-*diphenylpropyl Cyanide*.—Reaction between 2-benzylmethylaminoethyl chloride, diphenylmethyl cyanide, and sodamide (Walton and Ofner, *J.*, 1950, 2165) gave the cyanide, b. p. 205—212°/0·2 mm., in 81% yield. The hydrochloride was a water-insoluble syrup, and the *picrate* formed glistening flakes, m. p. 161—162°, from ethanol (Found : N, 13·2. $C_{24}H_{44}N_2,C_6H_3O_7N_3$ requires N, 12·3%).

1-*Benzylaminopropan*-2-*ol*.—Propylene oxide (58 g.) was stirred for 24 hours with benzylamine (107 g.) and water (100 c.c.), with cooling when necessary to keep the temperature below 60°. Distillation yielded benzylamine, b. p. 95—100°/11 mm. (16 g., 15%), 1-benzylaminopropan-2-ol, b. p. 145—150°/11 mm. (74 g., 45%), and benzyldi-(2-hydroxypropyl)amine, b. p. 175—195°/11 mm. (60·5 g., 25%) (yields calc. on benzylamine). 1-Benzylaminopropan-2-ol has been made previously by other methods (Uedinck, *Ber.*, 1899, **32**, 969; Staub, *Helv. Chim. Acta*, 1922, **5**, 891).

1-*Benzylmethylaminopropan*-2-*ol*.—(a) *From* 1-*benzylaminopropan*-2-*ol*. The amino-alcohol (74 g.) was heated under reflux for 20 hours with paraformaldehyde (13·5 g.), water (60 c.c.), and formic acid (20 c.c. ; *d* 1·2). More formic acid (60 c.c.) was added, and refluxing continued for 8 hours. Volatile substances were distilled off at 15 mm. and the residue made strongly alkaline with sodium hydroxide solution. The oily layer was removed and distilled, giving 1-*benzylmethylaminopropan*-2-*ol* (65 g., 81%), b. p. 129°/12 mm., n_D^{29} 1·5038 (Found : C, 71·8; H, 9·3. $C_{11}H_{17}ON$ requires C, 73·8; H, 9·5%). The *picrate* crystallised from ether–ethyl acetate as prisms, m. p. 80—80·5° (Found : C, 50·2; H, 5·1. $C_{11}H_{17}ON,C_6H_3O_7N_3$ requires C, 50·0; H, 4·9%).

(b) *From benzylmethylamine.* Propylene oxide (29 g.) was stirred for 20 hours with benzylmethylamine (60·5 g.) and water (50 c.c.), with intermittent cooling to keep the temperature below 50°. Fractionation of the product (two liquid phases) yielded the amino-alcohol (72 g., 88%), b. p. 122°/8 mm.

N-*Benzyl*-2-*chloro*-N-*methylpropylamine*.—The above amino-alcohol (61·7 g.) was converted into the crude dry hydrochloride, which was heated for 3 hours with thionyl chloride (50 c.c.) in chloroform (50 c.c.). Volatile compounds were distilled off under reduced pressure and the residue digested with acetone (100 c.c.), which gave a solid (57·4 g.), m. p. 164—167°. A small portion, recrystallised from ethanol–acetone, gave the pure *hydrochloride* as prisms, m. p. 166—167° (Found: C, 56·3; H, 7·0. $C_{11}H_{16}NCl,HCl$ requires C, 56·4; H, 7·3%). The crude hydrochloride was converted as before into the *chloro-amine* (33 g., 48%), b. p. 130—132°/20 mm., n_{D}^{29} 1·5072 (Found: C, 66·55; H, 7·8. $C_{11}H_{16}NCl$ requires C, 66·9; H, 8·1%). The *picrate* crystallised from ethanol as flakes, m. p. 145° (Found: C, 47·55; H, 4·75. $C_{11}H_{16}NCl$ $C_6H_3O_7N_3$ requires C, 47·8; H, 4·45%).

Reaction of N-*Benzyl*-2-*chloro*-N-*methylpropylamine with Diphenylmethyl Cyanide*.—The cyanide (30 g.), dry benzene (100 c.c.), and powdered sodamide (7·3 g.) were stirred at 30—40° for 30 minutes. The chloro-amine (30·6 g.) in benzene (30 c.c.) was added and the mixture refluxed for 2½ hours. The benzene solution was washed with water, and most of the benzene distilled off. Light petroleum (b. p. 60—80°) was added and the crystals (9 g.), m. p. 99—102°, which slowly formed were isolated and recrystallised from benzene–light petroleum (b. p. 40—60°), giving, as white needles, m. p. 106·5—107·5°, 3-*benzylmethylamino*-2-*methyl*-1 : 1-*diphenylpropyl cyanide* (Found: C, 84·5; H, 7·25; N, 7·8. $C_{25}H_{26}N_2$ requires C, 84·7; H, 7·35; N, 7·9%). This base gave a viscous, oily hydrochloride, insoluble in water but soluble in alcohols. The liquors from which the base had crystallised were evaporated and treated with methanolic hydrogen chloride; a fibrous white solid (13 g.; m. p. 194—198°) was slowly deposited. Recrystallisation from ethanol–ether–acetone gave as a white microcrystalline solid, m. p. 206°, 3-*benzylmethylamino*-1 : 1-*diphenylbutyl cyanide hydrochloride* (Found: C, 76·4; H, 6·95. $C_{25}H_{26}N_2,HCl$ requires C, 77·0; H, 6·9%). The free base was an oil. No attempt was made to find the optimum conditions for separating the isomers.

Conversion of γ-*Benzylmethylamino-cyanides into* 2-*Iminopyrrolidines*.—(*a*) 3-Benzylmethylamino-1 : 1-diphenylpropyl cyanide (3·4 g.) and concentrated hydrochloric acid (40 c.c.) were vigorously refluxed for 48 hours. Steam-distillation and extraction of the distillate with ether afforded benzyl chloride (0·65 g.), characterised by conversion into *S*-benzylthiuronium picrate, m. p. 183—185°. The solution of non-volatile substances was evaporated to dryness and the resulting 2-*imino*-1-*methyl*-3 : 3-*diphenylpyrrolidine hydrochloride* (2·5 g.) recrystallised from ethanol–ether; it formed white needles, m. p. 223·5—224°, which lost solvent of crystallisation and gave a hygroscopic powder on drying at 140°/12 mm. for analysis (Found: C, 70·75; H, 6·6. $C_{17}H_{18}N_2,HCl$ requires C, 71·3; H, 6·65%). The free base was an oil, and the picrate was a syrup, readily soluble in ether. The hydrochloride was dissolved in warm dilute hydrochloric acid, and sodium nitrite added. The resulting neutral solid, recrystallised from aqueous methanol, gave 1-methyl-3 : 3-diphenylpyrrolid-2-one as prisms, m. p. 144° (Dupré, Elks, Hems, Speyer, and Evans, *J.*, 1949, 507, give m. p. 146·5—147°).

(*b*) 2-Benzylmethylamino-2-methyl-1 : 1-diphenylpropyl cyanide (1·7 g.) and concentrated hydrochloric acid (20 c.c.) were refluxed for 8 hours, to give benzyl chloride (0·3 g.) and 2-*imino*-1 : 4-*dimethyl*-3 : 3-*diphenylpyrrolidine hydrochloride* (1·4 g.), solvated prisms, m. p. 239°, from *iso*propyl alcohol–ether; the crystals, after being dried at 140°/12 mm. for analysis, were hygroscopic (Found: C, 72·3; H, 6·8; N, 9·1. $C_{18}H_{20}N_2,HCl$ requires C, 71·9; H, 7·6; N, 9·3%). Hot dilute hydrochloric acid and sodium nitrite gave quantitatively 1 : 4-dimethyl-3 : 3-diphenylpyrrolidone, which formed glistening flakes, m. p. 122° (Walton, Ofner, and Thorp, *J.*, 1949, 654, give m. p. 121—123°); the m. p. of a mixture with the isomeric pyrrolidone from (*c*) was much lower.

(*c*) 3-Benzylmethylamino-1 : 1-diphenylbutyl cyanide hydrochloride (2 g.) and concentrated hydrochloric acid (30 c.c.) refluxed for 5½ hours similarly gave benzyl chloride (0·35 g.) and 2-*imino*-1 : 5-*dimethyl*-3 : 3-*diphenylpyrrolidine hydrochloride* (1·6 g.) as small cubes, m. p. 277°, from ether–*iso*propyl alcohol (Found: C, 71·9; H, 6·6; N, 9·0. $C_{18}H_{20}N_2,HCl$ requires C, 71·9; H, 6·7; N, 9·3%). The *base* formed large transparent prisms, m. p. 94°, from light petroleum (b. p. 60—80°) (Found: C, 81·45; H, 7·6; N, 10·75. $C_{18}H_{20}N_2$ requires C, 81·9; H, 7·6; N, 10·6%). The base was identical (m. p. and mixed m. p.) with a specimen made by Cymerman (*loc. cit.*) by another route, but was not identical with the compound, m. p. 103°, described in Part I. The hydrochloride (0·5 g.) was warmed with acetic anhydride (0·5 c.c.) in pyridine (5 c.c.)

at 30° for 4 hours. The pyridine was distilled off, and the residue made alkaline and extracted with benzene. The syrup obtained by evaporating the benzene, when triturated with light petroleum (b. p. 60—80°), gave a solid (0·18 g.), m. p. 123—125°. Recrystallisation from benzene–light petroleum afforded 2-*acetimido*-1 : 5-*dimethyl*-3 : 3-*diphenylpyrrolidine* as stout opaque rods, m. p. 126·5—127·5° (Found: C, 78·15; H, 7·1. $C_{20}H_{22}ON_2$ requires C, 78·45; H, 7·2%). This acetyl compound was soluble in 2N-hydrochloric acid. The 2-iminopyrrolidine hydrochloride, with hot dilute hydrochloric acid and sodium nitrite, gave quantitatively 1 : 5-dimethyl-3 : 3-diphenylpyrrolid-2-one, m. p. 119·5—120°, m. p. undepressed on admixture with an authentic specimen made by Gardner, Easton, and Stevens's method (*J. Amer. Chem. Soc.*, 1948, **70**, 2906; cf. Walton, Ofner, and Thorp, *J.*, 1949, 653).

Action of Concentrated Hydrochloric Acid on 1-*Benzyl*-4-*phenylpiperid*-4-*yl Cyanide.*—The base (5·3 g.; Part I, p. 2174) and concentrated hydrochloric acid (25 c.c.) were refluxed for 34 hours; no benzyl chloride was formed. The acid was distilled off and the residue treated with excess of sodium hydroxide solution. Unchanged base (1·07 g.; m. p. 69—71°) was filtered off; the filtrate was neutralised with acetic acid, which precipitated 1-benzyl-4-phenylpiperidine-4-carboxylic acid (2·3 g.), which decomposed rapidly above 265° [Eisleb, *Ber.*, 1941, **74**, 1433 gives m. p. 288° (decomp.)]. For identification, the acid (1·75 g.) was refluxed for 6 hours with ethanol (25 c.c.) and concentrated sulphuric acid (5 c.c.), and gave the ethyl ester (1·5 g.), m. p. 72—73° (Eisleb, *loc. cit.*, gives m. p. 73—74°).

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