S 32. *Aminoalkyl Tertiary Carbinols and Derived Products. Part I. 3-Amino-1 : 1-diphenylpropan-1-ols.*

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3-Amino- and *N-substituted 3-amino-1 : 1-diphenylpropan-1-ols* (V) were prepared from phenylmagnesium bromide and the substituted ethyl β-aminopropionates, obtained by addition of ethyl acrylate to the appropriate amines.

The amino-carbinols were dehydrated to the corresponding *3-amino-1 : 1-diphenylprop-1 enes* (VI) and the latter catalytically reduced to the 3-amino- and *N-substituted 3-amino-1 : 1-diphenylpropanes* (IV).

Quaternary ammonium salts were prepared from the tertiary amines.

The majority of the compounds had spasmolytic and local anæsthetic action. Some showed atropine-like activity of a high order and others had an anti-histamine effect.

The benzhydryl group is common to a number of substituted amines of high pharmacological activity [*inter al.*, the analgesic "Amidone" (I), the antihistamine compound "Benadryl" (II), and the spasmolytics "Trasentin" (III) and "Hoeschst 10,166" (IV; NR^1R^2 = piperidino-)].

$CPh_2(CO\cdot Et)\cdot CH_2\cdot CHMe\cdot NMe_2$ (I.)

$CHPh_2\cdot O\cdot CH_2\cdot CH_2\cdot NMe_2$ (II.)

$CHPh_2\cdot CO_2\cdot CH_2\cdot CH_2\cdot NEt_2$ (III.)

$CHPh_2\cdot CH_2\cdot CH_2\cdot NR^1R^2$ (IV.)

It was thought therefore that the 3-*amino*- and N-*substituted* 3-*amino*-1 : 1-*diphenylpropan*-1-*ols* (V) and the corresponding 3-*amino*-1 : 1-*diphenylprop*-1-*enes* (VI) would constitute a series of potential pharmacological interest.

$CPh_2(OH)\cdot CH_2\cdot CH_2\cdot NR^1R^2$ (V.)

$CPh_2{:}CH\cdot CH_2\cdot NR^1R^2$ (VI).

$CO_2Et\cdot CH_2\cdot CH_2\cdot NR^1R^2$ (VII.)

$Ph\cdot CO\cdot CH_2\cdot CH_2\cdot NR^1R^2$ (VIII.)

A preliminary report on the antispasmodic activity of a similar series of substituted phenylpropylpiperidines appeared after this work was begun (Becker, Anamick, Glenwood, and Miller, *Fed. Proc.*, 1946, **5**, 163). Accounts of the synthesis of similar compounds by methods essentially the same as those now described became available more recently, after the present work had been completed (" Report No. P.B. 981, Office of the Publication Board, Dept. of Commerce, Washington, D.C., pp. 38, 118; F.P. 890,633; B.P. Appl. 2255/47). Except for (V and VI; NR^1R^2 = piperidino) the compounds now described are new.

The amino-carbinols were prepared by the Grignard reaction between phenylmagnesium bromide and the appropriate substituted ethyl β-aminopropionate (VII). The yields were good (about 80% crude product) in the case of the tertiary amino-compounds; a small proportion of the corresponding *N*-substituted ω-aminopropiophenone (VIII) was also isolated. The yields of amino-carbinols were much lower with the primary (VII; $R^1 = R^2 = H$) and the secondary (VIII; $R^1 = H$; R^2 = Me, Et, and CH_2Ph) aminopropionates. A probable explanation is to be found in Breckpot's observation (*Bull. Soc. chim. Belg.*, 1923, **32**, 412) that ethyl β-methylaminobutyrate is converted largely into the β-lactam and non-nitrogenous compounds (but no amino-carbinol) by the action of ethylmagnesium bromide. The yield of amino-carbinol was not improved by the use of phenyl-lithium in place of the Grignard reagent.

The amino-carbinols were crystalline solids which could be distilled without decomposition at low pressures. The *hydrochlorides* were stable in neutral-aqueous solution but were readily dehydrated in acid solution to give a quantitative yield of the corresponding allylamines (VI). The allylamines were normally liquids which could be distilled under reduced pressure without decomposition [except the primary amine (VI; $R^1 = R^2 = H$)] and were readily converted into *hydrochlorides*. 3-*Phenylmethylamino*-1 : 1-*diphenylprop*-1-*ene* (VI; R^1 = Me, R^2 = Ph) was exceptional, degradation occurring in the presence of acids or on attempts to convert it into the methiodide.

Catalytic reduction (palladium–charcoal) of the unsaturated amine hydrochlorides in alcoholic solution furnished the corresponding 3-amino- and N-*substituted* 3-*amino*-1 : 1-*diphenylpropane hydrochlorides* (IV) in good yield. When the reduction was carried out on the free base, the volume of hydrogen absorbed was much in excess of that calculated and the presence of low-boiling products indicated that some fission of the molecule had occurred. In the reduction of dextro-3-N-*methylamphetamino*-1 : 1-*diphenylprop*-1-*ene*, the only product to be isolated was *dextro-N*-methylamphetamine. Several of the substituted propylamines (IV), exemplified by " Hoechst 10,166 " [(IV; NR^1R^2 = piperidino) a component of " Aspasan," used in the treatment of asthma, Schaumann, *Med. u. Chem.*, 1942, **4**, 229], have also been prepared by other methods (Eisleb, *Ber.*, 1941, **74**, 1433; B.I.O.S., 1945, Final Report 116, Item 24, p. 49; Freeman, Ringk, and Spoerri, *J. Amer. Chem. Soc.*, 1947, **69**, 858).

The *N*-disubstituted 3-amino-1 : 1-diphenyl-propan-1-ols, -prop-1-enes and -propanes were readily converted by methyl iodide and by higher alkyl and aralkyl halides into *quaternary ammonium salts*.

The *N*-disubstituted ethyl β-aminopropionates required as starting materials, were prepared in excellent yield [except in the case of *ethyl* β-*pyrrolidinopropionate* (VII; $NR^1R^2 = NC_4H_8$)] by the addition of ethyl acrylate to the appropriate secondary amine (Flürscheim, *J. pr. Chem.*, 1903, **68**, 348; Weisel, Taylor, Mosher, and Whitmore, *J. Amer. Chem. Soc.*, 1945, **67**, 1071). No reaction occurred when *N*-methylaniline and ethyl acrylate were heated together, but the required *ethyl* β-*phenylmethylaminopropionate* (VII; R^1 = Me, R^2 = Ph) was obtained by the general method of Elderfield, Bembry, Kremer, Brody, Hageman, and Head (*J. Amer. Chem. Soc.*, 1946, **68**, 1259) in which a catalytic quantity of acetic acid is added to the mixture.

A less satisfactory yield of the secondary amino-ester, ethyl β-methylaminopropionate (VII; $R^1 = H$, R^2 = Me), resulted from mixing alcoholic methylamine with ethyl acrylate; substantial quantities of methylbis-β-carbethoxyethylamine were formed even when the reaction was conducted at low temperatures (Morsch, *Monatsh.*, 1933, **63**, 220, obtained the corresponding methyl esters in similar yield from methyl acrylate). In agreement with Stork and McElvain

(*J. Amer. Chem. Soc.*, 1947, **69**, 971) benzylamine, in sharp contrast to methylamine, gave the secondary amino-ester (VII; $R^1 = H$, $R^2 = CH_2Ph$) in excellent yield. This favourable reaction is not peculiar to benzylamine, however, as these authors suggest, since ethylamine also gave a high yield (81%) of the secondary amino-ester, *ethyl β-ethylaminopropionate* (VII; $R^1 = H$, $R^2 = Et$).

A preliminary pharmacological examination of the compounds has been made by Dr. A. C. White and his collaborators of the Wellcome Physiological Research Laboratories. Conduction and surface-anæsthetic activity is exhibited in some degree by all the compounds; a large proportion are comparable in activity with procaine and with cocaine respectively. Significant analgesic activity was not observed in this series. Antagonism of spasm induced by carbachol is a general property, the activity being at a maximum in the quaternary ammonium salts, when in some cases it is comparable with that of atropine. The salts have other atropine-like properties, *e.g.*, some are powerful mydriatics. Antihistamine activity (demonstrated by *in vitro* tests and by the protection afforded to guinea-pigs in experimental asthma induced by histamine) was shown by some of the compounds.

The pharmacological examination of these compounds will be described in detail elsewhere.

Experimental.

(Micro-analyses by Mr. A. Bennett; m. p.s are uncorrected.)

Ethyl β-Aminopropionate.—The ester was conveniently prepared according to the method of Weygand (*Ber.*, 1941, **74**, 256) by the hydrogenation of ethyl cyanoacetate in the presence of platinum.

N-*Monosubstituted Ethyl β-Aminopropionates.*—Ethyl acrylate (100 g.) in ethanol (100 c.c.), cooled to −60°, was added to ethylamine (45 g.), cooled to −60°, and the mixture allowed to reach room temperaure during 24 hours. Ethanol was evaporated off and the residual oil distilled under reduced pressure. *Ethyl β-ethylaminopropionate* (VII; $R^1 = H$, $R^2 = Et$) was collected at 74—76°/14 mm. Yield 117 g. (81%) (Found: C, 57·6; H, 10·3; N, 9·7. $C_7H_{15}O_2N$ requires C, 57·9; H, 10·3; N, 9·7%).

Under similar conditions, methylamine and ethyl acrylate gave a 42% yield of ethyl β-methylaminopropionate (VII; $R^1 = H$, $R^2 = Me$), b. p. 78—80°/21 mm. (Gansser, *Z. physiol. Chem.*, 1909, **61**, 42, gives b. p. 58°/8 mm.), together with 31% of methylbis-β-carbethoxyethylamine, b. p. 152—156°/21 mm. (McElvain, *J. Amer. Chem. Soc.*, 1924, **46**, 1724, gives b. p. 136—138°/4 mm.).

Ethyl β-benzylaminopropionate (VII; $R^1 = H$, $R^2 = CH_2Ph$), b. p. 125—128°/1 mm. (yield 83%), was prepared in a similar manner to that described by Stork and McElvain (*loc. cit.*) who quote a yield of 87%, b. p. 133—135°/2 mm.

N-*Disubstituted Ethyl β-Aminopropionates.*—The following were prepared by mixing equimolecular proportions of the appropriate secondary amine and ethyl acrylate and distilling the mixture after 4 days' standing. *Ethyl β-dimethylaminopropionate* (VII; $R^1 = R^2 = Me$), yield 87% (b. p. 56—57°/12 mm. (Found: C, 57·9; H, 10·3; N, 9·8. $C_7H_{15}O_2N$ requires C, 57·9; H, 10·3; N, 9·7%), ethyl β-diethylaminopropionate (VII; $R^1 = R^2 = Et$), yield 85%, b. p. 87—88°/15 mm. (Flürscheim, *loc. cit.*, gives b. p. 83—84°/12 mm.), and ethyl β-di-*n*-propylaminopropionate (VII; $R^1 = R^2 = Pr^n$), yield 81%, b. p. 104—106°/13 mm. (Weisel, Taylor, Mosher, and Whitmore, *loc. cit.*, give b. p. 125—126°/28 mm.).

The following were prepared from an equimolecular mixture of the secondary amine and ethyl acrylate by boiling under reflux for 6 hours and subsequently distilling under reduced pressure. Ethyl β-di-*n*-butylaminopropionate (VII; $R^1 = R^2 = Bu^n$), yield 80%, b. p. 129—130°/19 mm. (Weisel, Taylor, Mosher, and Whitmore, *loc. cit.*, give b. p. 136—137°/16 mm.), ethyl β-piperidinopropionate (VII; $NR^1R^2 = NC_5H_{10}$), yield 85%, b. p. 115—117°/17 mm. (Phillipi and Galter, *Monatsh.*, 1929, **51**, 253, give b. p. 104—106°/12—13 mm.), ethyl β-morpholinopropionate (VII; $NR^1R^2 = NC_4H_8O$), yield 86%, b. p. 118°/16 mm. (Weisel, Taylor, Mosher, and Whitmore, *loc. cit.*, give b. p. 138—140°/25 mm.), *ethyl β-diallylaminopropionate* (VII; $R^1 = R^2 = CH_2{\cdot}CH{:}CH_2$), yield 80%, b. p. 108—110°/15 mm. (Found: C, 66·8; H, 9·6; N, 7·2. $C_{11}H_{19}O_2N$ requires C, 67·0; H, 9·6; N, 7·1%), and *ethyl* dextro-*β-methylamphetaminopropionate* (VII; $R^1 = Me$, $R^2 = CHMe{\cdot}CH_2Ph$), yield 78%, b. p. 165—166°/12 mm. {*hydrogen oxalate*, m. p. 125—126° after crystallisation from ethanol (Found: C, 60·0; H, 7·3; N, 4·3. $C_{15}H_{23}O_2N,C_2H_2O_4$ requires C, 60·2; H, 7·4; H, 4·1%), $[\alpha]_{5461}^{20°}$ +20·2° (*c*, 0·9 in ethanol)}. Under these conditions, pyrrolidine and ethyl acrylate gave only a 40% yield of *ethyl β-pyrrolidinopropionate* (VII; $NR^1R^2 = NC_4H_8$), b. p. 108—110°/22 mm. (Found: C, 62·7; H, 9·6; N, 8·2. $C_9H_{17}O_2N$ requires C, 63·2; H, 9·9; N, 8·2%).

Ethyl acrylate and *N*-methylaniline were recovered unchanged after heating together under reflux in equimolecular proportions. When 10% acetic acid was added and the mixture boiled under reflux for 12 hours, cooled, washed with aqueous sodium hydrogen carbonate, dried by sodium sulphate, and fractionated under reduced pressure, a 66% yield of *ethyl β-phenylmethylaminopropionate* (VII; $R^1 = Me$, $R^2 = Ph$) was obtained, b. p. 98—100°/0·05 mm. (Found: C, 69·8; H, 8·4; N, 6·6. $C_{12}H_{17}O_2N$ requires C, 69·6; H, 8·2; N, 6·8%).

N-*Substituted* 3-*Amino*-1 : 1-*diphenylpropan*-1-*ols* (V).—The general method of preparation employed is illustrated by the following example.

1 : 1-*Diphenyl*-3-*piperidinopropan*-1-*ol* (V; $NR^1R^2 = NC_5H_{10}$). Ethyl β-piperidinopropionate (37 g., 0·2 mol.) in anhydrous ether (50 c.c.) was added gradually to an ether solution of the Grignard reagent made from bromobenzene (94·2 g., 0·6 mol.) and magnesium (14·6 g.) stirred and cooled in a bath kept at 0°. After stirring in the cold for 1 hour and then heating under reflux for 2 hours, the mixture (from which a heavy white solid had separated) was cooled and stirred into crushed ice (100 g.), and aqueous ammonium chloride (25%; 100 c.c.) added. Acetic acid was then added gradually with stirring and cooling until the solution was acidic, and the crude 1 : 1-diphenyl-3-piperidinopropan-1-ol

hydrobromide, which separated as a pale cream solid, was filtered off and washed with ether. A sample of the salt, recrystallised from water and then from ethanol, had m. p. 228° (decomp.) (Found : N, 3·7; Br, 21·4. $C_{20}H_{25}ON,HBr$ requires N, 3·7; Br, 21·3%). The remainder of the hydrobromide was suspended in chloroform, excess ammonia added with shaking, filtered from inorganic material, and the chloroform layer separated, washed with water, and dried by sodium sulphate. On evaporation of the chloroform, the residual crude 1 : 1-diphenyl-3-piperidinopropan-1-ol (40 g., 67% yield) had m. p. 107—112°, which was raised to 120—121° by several recrystallisations from light petroleum (b. p. 60—80°); the total yield of purified material was 33·6 g. (57% yield).

The ethereal and the aqueous layer of the original filtrate and washings were separated. The ethereal layer gave crude diphenyl (2·1 g.) on evaporation. The aqueous layer was basified with excess ammonia, extracted with ether, dried by sodium sulphate, and the ether evaporated. The residual brown oil (7·0 g.) was fractionally distilled under reduced pressure to give ω-piperidinopropiophenone (VIII; $NR^1R^2 = NC_5H_{10}$) (2·1 g.; b. p. 136—138°/10 mm. : hydrochloride, m. p. 188—190°, not depressed on mixing with an authentic specimen; Mannich and Lammering, *Ber.*, 1922, **55**, 3510, give m. p. 192—193°) besides low-boiling material and a crystalline residue (1·6 g.) which was the impure carbinol.

The 3-*amino*- and N-*substituted* 3-*amino*-1 : 1-*diphenylpropan*-1-*ols* (V) included in Table I were prepared in a manner essentially similar to that described above; the recorded yields relate to the purified product.

In the case of the *N*-monosubstituted amino-carbinols, the yields quoted were obtained by conducting the Grignard reaction at −20° for 2 hours, then at 0° for 2 hours; the yields were somewhat lower when these temperatures were exceeded. Under these conditions, quantities of unchanged amino-ester were recovered (*e.g.*, 35% in the case of the benzylamino-compound (V; $R^1 = H$, $R^2 = CH_2Ph$).

In one experiment, applied to ethyl β-methylaminopropionate (VII; $R^1 = H$, $R^2 = Me$), the Grignard reagent was replaced by an equivalent amount of phenyl-lithium and the reaction carried out in an inert atmosphere; the yield of 3-*methylamino*-1 : 1-*diphenylpropan*-1-*ol* (V; $R^1 = H$, $R^2 = Me$) in this case was less than 10%.

In the preparation of 3-*phenylmethylamino*-1 : 1-*diphenylpropan*-1-*ol* (V; $R^1 = Me$, $R^2 = Ph$), the hydrobromide resulting from the acetic acid decomposition of the Grignard complex remained as an oil, and separation was effected by decantation rather than filtration.

In the examples of tertiary amino-carbinols for which a b. p. is recorded in Table I, the crude products (of low m. p. and very soluble in organic solvents) were purified by fractional distillation at low pressures followed by recrystallisation from small volumes of light petroleum (b. p. 40—60°). dextro-3-*Methylamphetamino*-1 : 1-*diphenylpropan*-1-*ol* (V; $R^1 = Me$, $R^2 = CHMe{\cdot}CH_2Ph$) was reluctant to crystallise and was purified by recrystallisation of its *hydrochloride*.

3-*Dimethylamino*-1 : 1-*diphenylpropan*-1-*ol* (V; $R^1 = R^2 = Me$) was prepared also from phenylmagnesium bromide (0·3 mol.) and freshly distilled ω-dimethylaminopropiophenone (XII; $R^1 = R^2 = Me$) (0·2 mol.; b. p. 100°/2·5 mm.; Mannich and Heilner, *Ber.*, 1922, **55**, 356) under conditions identical with those described above, a yield of 57% being obtained.

Amino-carbinol Hydrochlorides (Table II).—Dry hydrogen chloride was led into a solution of the base (*e.g.*, 5·0 g.) in anhydrous chloroform (*e.g.*, 20 c.c.) cooled to 0° until the solution was not more than faintly acidic to Congo-red. Anhydrous ether was added, first gradually with scratching to the point of crystallisation, then in excess to precipitate the hydrochloride. The hydrochlorides were recrystallised with ease, the appropriate solvents being shown in the Table.

In one experiment, 1 : 1-diphenyl-3-piperidinopropan-1-ol (5·0 g.) in chloroform (20 c.c.) was saturated with hydrogen chloride at 0°. The solution became cloudy, and on addition of ether, 1 : 1-diphenyl-3-piperidinoprop-1-ene hydrochloride (VI; $NR^1R^2 = NC_5H_{10}$) (5·0 g.; m. p. 209—210° after recrystallisation from a mixture of acetone and chloroform) was precipitated.

Carbinol Quaternary Ammonium Salts.—The *methiodides* of the *N*-disubstituted 3-amino-1 : 1-diphenylpropan-1-ols were usually readily prepared by adding methyl iodide (2 equivalents) to a cold solution of the tertiary base in anhydrous acetone. The crystalline salts rapidly separated and after standing for 24 hours were obtained in almost quantitative yield. Quaternisation with higher alkyl halides required more vigorous conditions such as boiling under reflux in ethanol solution. In some cases it was necessary to add ether to complete crystallisation of the product (indicated in Table III by, *e.g.*, " ethanol, boil 5 hours : ether ").

N-*Substituted* 3-*Amino*-1 : 1-*diphenylprop*-1-*enes* (VI).—The amino-carbinol (15 g.) was dissolved in concentrated hydrochloric acid (30 c.c.) and glacial acetic acid (100 c.c.) and the solution boiled under reflux for 30 minutes. The solution was then concentrated under reduced pressure, the residue dissolved in water, and the base liberated by addition of excess ammonia and separated by extraction with ether. The ether solution was dried over anhydrous sodium sulphate, the ether evaporated, and the residual oil distilled under reduced pressure to give a substantially quantitative yield of the 3-*amino*- and N-*substituted* 3-*amino*-1 : 1-*diphenylprop*-1-*enes* (Table IV).

The bases were converted into the *hydrochlorides* (Table IV) by a method similar to that adopted for the amino-carbinol hydrochlorides. The hydrochlorides were also obtained directly from the dehydration mixture by evaporation to dryness and recrystallisation of the solid residue.

The *methiodides* (Table V) were prepared by dissolving the tertiary base in anhydrous acetone, adding methyl iodide (2 equivalents) and allowing to stand for 24 hours. In some cases (indicated in Table V) it was necessary subsequently to add ether to complete the crystallisation of the salt. Yields were almost quantitative.

3-*Phenylmethylamino*-1 : 1-*diphenylprop*-1-*ene* (VI; $R^1 = Me$, $R^2 = Ph$). 3-Phenylmethylamino-1 : 1-diphenylpropan-1-ol (7·0 g.) was dissolved in concentrated hydrochloric acid (15 c.c.) and glacial acetic acid (50 c.c.) and heated under reflux for 20 minutes. On working up the pale brown solution as described above, 3-*phenylmethylamino*-1 : 1-*diphenylprop*-1-*ene* was obtained as a viscous pale yellow oil (b. p. 200—204°/0·5 mm., yield 4·8 g., 70%), from which a crystalline hydrochloride could not be obtained.

TABLE I.

N-*Substituted* 3-*Amino*-1 : 1-*diphenylpropan*-1-*ols*, $CPh_2(OH)\cdot CH_2\cdot CH_2\cdot NR^1R^2$ (V).

1 : 1-*Diphenylpropan*-1-*ol*.	NR^1R^2.	Yield, %.	B. p.	M. p.	Recrystallisation solvent.	Formula.	Analysis, Found, %. C.	H.	N.	Required, %. C.	H.	N.
3-*Amino*-	NH_2	33	—	141—143°	Ethanol	$C_{15}H_{17}ON$	79·6	7·6	6·2	79·3	7·5	6·2
3-*Methylamino*-	NHMe	24	148—150°/0·2 mm.	145—146	Ethanol	$C_{16}H_{19}ON$	79·2	7·8	5·9	79·7	7·9	5·8
3-*Ethylamino*-	NHEt	38	—	141—142	Ethanol	$C_{17}H_{21}ON$	80·1	8·1	5·5	80·0	8·2	5·5
3-*Benzylamino*-	$NH\cdot CH_2Ph$	16	—	151—152	Ethanol	$C_{22}H_{23}ON$	83·0	7·3	4·5	83·3	7·2	4·4
3-*Dimethylamino*-	NMe_2	62	—	166	Benzene	$C_{17}H_{21}ON$	79·8	8·1	5·5	80·0	8·2	5·5
3-*Diethylamino*-	NEt_2	56	154/0·2 mm.	53·5	Light petroleum (b. p. 40—60°)	$C_{19}H_{25}ON$	80·5	8·7	4·9	80·6	8·8	4·9
3-*Di*-n-*propylamino*-	NPr^n_2	51	153—154/0·1 mm.	52·5—53·5	Light petroleum (b. p. 40—60°)	$C_{21}H_{29}ON$	80·9	9·0	4·7	81·0	9·3	4·5
3-*Di*-n-*butylamino*-	NBu^n_2	54	157—159/0·1 mm.	41—42	Light petroleum (b. p. 40—60°)	$C_{23}H_{33}ON$	81·6	9·7	4·1	81·4	9·7	4·1
3-*Phenylmethylamino*-	NPhMe	84	—	97	Ethanol	$C_{22}H_{23}ON$	83·4	7·0	4·5	83·3	7·2	4·4
dextro-3-*Methylamphetamino*-	$NMe\cdot CHMe\cdot CH_2Ph$	55	—	57—58	Light petroleum (b. p. 40—60°)	$C_{25}H_{29}ON$	83·3	8·5	4·0	83·6	8·1	3·9
3-*Diallylamino*-	$N[CH_2\cdot CH:CH_2]_2$	60	157—159/0·4 mm.	25—27	Light petroleum (b. p. 40—60°)	$C_{21}H_{25}ON$	81·8	8·0	4·8	82·1	8·1	4·6
3-*Pyrrolidino*-	$N<[CH_2]_3>CH_2$	63	—	171—172	Ethyl acetate	$C_{19}H_{23}ON$	81·3	8·3	5·0	81·1	8·2	5·0
3-Piperidino-	$N<[CH_2]_4>CH_2$	57	—	120—121[a]	Light petroleum (b. p. 60—80°)	$C_{20}H_{25}ON$	81·3	8·3	4·7	81·3	8·5	4·7
3-*Morpholino*-	$N<[CH_2]_4>O$	50	—	106	Ethanol	$C_{19}H_{23}O_2N$	76·8	7·8	4·8	76·8	7·7	4·7

(*a*) B.P. Appln. 2255/47 gives m. p. 115·5—116·5°.

TABLE II.

N-*Substituted* 3-*Amino*-1 : 1-*diphenylpropan*-1-*ol Hydrochlorides*, $CPh_2(OH)\cdot CH_2\cdot CH_2\cdot NR^1R^2,HCl$.

1 : 1-*Diphenylpropan*-1-*ol hydrochloride.*	NR^1R^2.	M. p.	Recrystallisation solvent.	Formula.	Analysis, Found, %. N.	Found, %. Cl.	Required, %. N.	Required, %. Cl.
3-*Amino*-	NH_2	182—184°	Ethyl acetate–ethanol	$C_{15}H_{17}ON,HCl$	5·5	13·5	5·3	13·5
3-*Methylamino*-	NHMe	148—151	Ethyl acetate–ethanol	$C_{16}H_{19}ON,HCl$	5·3	12·9	5·0	12·8
3-*Ethylamino*-	NHEt	176—177	Ethyl acetate–ethanol	$C_{17}H_{21}ON,HCl$	4·9	12·3	4·8	12·2
3-*Benzylamino*-	$NH\cdot CH_2Ph$	203	Ethyl acetate–ethanol	$C_{22}H_{23}ON,HCl$	4·2	10·0	4·0	10·0
3-*Dimethylamino*-	NMe_2	203—205	Ethanol	$C_{17}H_{21}ON,HCl$	5·0	12·4	4·8	12·2
3-*Diethylamino*-	NEt_2	202—203	Ethanol	$C_{19}H_{25}ON,HCl$	4·5	11·0	4·4	11·1
3-*Di*-n-*propylamino*-	NPr^n_2	161	Acetone	$C_{21}H_{29}ON,HCl$	3·9	10·1	4·0	10·2
3-*Di*-n-*butylamino*-	NBu^n_2	108—109	Ethyl acetate	$C_{23}H_{33}ON,HCl$	3·7	9·1	3·7	9·5
3-*Phenylmethylamino*-	NPhMe	170 (decomp.)	Acetone–methanol	$C_{22}H_{23}ON,HCl$	4·1	10·0	4·0	10·0
dextro-3-*Methylamphetamino*-	$NMe\cdot CHMe\cdot CH_2Ph$	207—208[a]	Aqueous ethanol	$C_{25}H_{29}ON,HCl$	3·6	8·8	3·5	9·0
3-*Diallylamino*-	$N[CH_2\cdot CH{:}CH_2]_2$	155—156	Aqueous ethanol	$C_{21}H_{25}ON,HCl$	4·1	10·4	4·1	10·3
3-*Pyrrolidino*-	$N<[CH_2]_3>CH_2$	190—191	Ethyl acetate–ethanol	$C_{19}H_{23}ON,HCl$	4·2	11·3	4·4	11·2
3-Piperidino-	$N<[CH_2]_4>CH_2$	238[b]	Ethanol	$C_{20}H_{25}ON,HCl$	4·2	10·7	4·2	10·7
3-*Morpholino*-	$N<[CH_2]_4>O$	231	Aqueous ethanol	$C_{19}H_{23}O_2N,HCl$	4·2	10·7	4·2	10·6

(*a*) $[\alpha]^{20°}_{5461}$ + 8·2° (*c*, 0·7 in ethanol).
(*b*) F.P. 890,633 gives m. p. 216—217° and B.P. Appln. 2255/47 m. p. 229—230°.

TABLE III.

3 : 3-*Diphenyl*-3-*hydroxypropyl- Quaternary Ammonium Salts*, $CPh_2(OH)\cdot CH_2\cdot CH_2\cdot \overset{+}{N}R^1R^2R^3\ \bar{X}$.

3 : 3-*Diphenyl*-3-*hydroxypropylammonium iodide.*	$\overset{+}{N}R^1R^2R^3\ \bar{X}$.	Solvent.	M. p.	Recrystallisation solvent.	Formula.	Analysis, Found, %. N.	Found, %. I.	Required, %. N.	Required, %. I.
Trimethyl-	$\overset{+}{N}Me_3\ I^-$	Hot acetone	243° (decomp.)	Aqueous ethanol	$C_{18}H_{24}ONI$	3·4	32·1	3·5	32·0
Dimethylethyl-	$\overset{+}{N}Me_2Et\ I^-$	Ethanol, boil 2 hrs.	200—201 (decomp.)	Aqueous ethanol	$C_{19}H_{26}ONI$	3·2	31·0	3·4	30·9
Dimethyl-n-*propyl-* (bromide)	$\overset{+}{N}Me_2Pr^n\ Br^-$	Ethanol, boil 5 hrs.	231—233 (decomp.)	Aqueous ethanol	$C_{20}H_{28}ONBr$	3·9	(Br) 21·6	3·7	(Br) 21·2
Dimethyl-n-*butyl-* (bromide)	$\overset{+}{N}Me_2Bu^n\ Br^-$	Ethanol, boil 5 hrs.	233—235 (decomp.)	Aqueous ethanol	$C_{21}H_{30}ONBr$	3·7	(Br) 20·6	3·6	(Br) 20·4
Phenyldimethyl-	$\overset{+}{N}Me_2Ph\ I^-$	Ethanol	176 (decomp.)	Aqueous ethanol	$C_{23}H_{26}ONI$	2·9	27·4	3·1	27·7
Benzyldimethyl- (chloride)	$\overset{+}{N}Me_2\cdot CH_2Ph\ Cl^-$	Ethanol, boil 2 hrs. : ether	251 (decomp.)	Ethanol	$C_{24}H_{28}ONCl$	3·8	(Cl) 9·4	3·7	(Cl) 9·3
Dimethyl(*phenyl*iso*propyl*)-	$\overset{+}{N}Me_2\cdot CHMe\cdot CH_2Ph\ I^-$	Acetone	226 (decomp.)	Methanol	$C_{26}H_{32}ONI$	3·0	25·2	2·8	25·3
Methyldiethyl-	$\overset{+}{N}MeEt_2\ I^-$	Acetone	198—199	Methanol	$C_{20}H_{28}ONI$	3·2	29·9	3·3	29·9
Triethyl-	$\overset{+}{N}Et_3\ I^-$	Ethanol, boil 5 hrs. : ether	207—208	Aqueous ethanol	$C_{21}H_{30}ONI$	3·3	29·1	3·2	28·9
Methyldi-n-*propyl-*	$\overset{+}{N}MePr^n_2\ I^-$	Acetone	181—183	Aqueous ethanol	$C_{22}H_{32}ONI$	2·9	28·2	3·1	28·0
Methyldi-n-*butyl-*	$\overset{+}{N}MeBu^n_2\ I^-$	Acetone	195—196	Aqueous ethanol	$C_{24}H_{36}ONI$	2·8	26·6	2·9	26·4
Methyldiallyl-	$\overset{+}{N}Me\cdot[CH_2\cdot CH{:}CH_2]_2\ I^-$	Acetone	196—197 (decomp.)	Aqueous ethanol	$C_{22}H_{28}ONI$	3·3	28·6	3·1	28·3
Methyl-3 : 3-*diphenyl*-3-*hydroxypropyl-pyrrolidinium iodide*	$\overset{+}{N}Me{<}[CH_2]_3{>}CH_2\ I^-$	Chloroform : ether	210	Methanol	$C_{20}H_{26}ONI$	3·2	29·7	3·3	30·0
Methyl-3 : 3-*diphenyl*-3-*hydroxypropyl-piperidinium iodide*	$\overset{+}{N}Me{<}[CH_2]_4{>}CH_2\ I^-$	Acetone	214—215	Ethanol	$C_{21}H_{28}ONI$	3·0	29·2	3·2	29·1
Ethyl-3 : 3-*diphenyl*-3-*hydroxypropyl-piperidinium iodide*	$\overset{+}{N}Et{<}[CH_2]_4{>}CH_2\ I^-$	Ethanol, boil 5 hrs. : ether	204—205	Aqueous ethanol	$C_{22}H_{30}ONI$	2·9	28·4	3·1	28·2
Methyl-3 : 3-*diphenyl*-3-*hydroxypropyl-morpholinium iodide*	$\overset{+}{N}Me{<}[CH_2]_4{>}O\ I^-$	Acetone	203—204 (decomp.)	Aqueous ethanol	$C_{20}H_{26}O_2NI$	3·1	28·7	3·2	28·9

TABLE IV.

N-*Substituted* 3-*Amino*-1 : 1-*diphenylprop*-1-*enes and Hydrochlorides*, $CPh_2{:}CH{\cdot}CH_2{\cdot}NR^1R^2$ (VI).

1 : 1-*Diphenylprop*-1-*ene*.	NR^1R^2.	Base, B. p.	Hydrochloride, M. p.	Recrystallisation solvent.	Formula.	Analysis, Found, %. N.	Found, %. Cl.	Required, %. N.	Required, %. Cl.
3-*Amino*-	NH_2	(*a*)	213—215° (decomp.)	Ethyl acetate–ethanol	$C_{15}H_{15}N$, HCl	5·7	14·6	5·7	14·5
3-*Ethylamino*-	NHEt	116—117°/0·15 mm.	178—179 (decomp.)	Ethanol	$C_{17}H_{19}N$, HCl	5·2	13·0	5·1	13·0
3-*Dimethylamino*-	NMe_2	192—193/18 mm.	168—170	Ethanol–acetone	$C_{17}H_{19}N$, HCl	5·2	13·1	5·1	13·0
3-*Diethylamino*-	NEt_2	111/0·05 mm.	146—147	Acetone	$C_{19}H_{23}N$, HCl	4·6	11·6	4·6	11·8
3-*Di*-n-*propylamino*-	NPr^n_2	146—148/0·4 mm.	128—129	Ethyl acetate	$C_{21}H_{27}N$, HCl	4·2	10·9	4·2	10·8
3-*Di*-n-*butylamino*-	NBu^n_2	139—142/0·05 mm.	149—150	Ethyl acetate	$C_{23}H_{31}N$, HCl	4·0	10·1	3·9	9·9
3-*Phenylmethylamino*-	NPhMe	200—204/0·5 mm.(*b*)	—	—	—	—	—	—	—
dextro-3-*Methylamphetamino*-	NMe·CHMe·CH_2Ph	168—170/0·07 mm.(*c*)	Oil(*d*)	—	—	—	—	—	—
3-*Diallylamino*-	$N[CH_2{\cdot}CH{:}CH_2]_2$	134/0·2 mm.(*e*)	Oil	—	—	—	—	—	—
3-*Pyrrolidino*-	$N<[CH_2]_3>CH_2$	125/0·02 mm.	165—167	Ethyl acetate–ethanol	$C_{19}H_{21}N$, HCl	4·7	11·9	4·7	11·9
3-Piperidino-	$N<[CH_2]_4>CH_2$	138/0·1 mm.	209—210(*g*)	Chloroform–acetone	$C_{20}H_{23}N$, HCl	4·4	11·4	4·5	11·3
3-*Morpholino*-	$N<[CH_2]_4>O$	m. p. 70—72°(*f*)	218—219	Ethanol	$C_{19}H_{21}ON$, HCl	4·3	11·6	4·4	11·3

(*a*) Decomposed to a resin on attempted distillation at 0·1 mm.
(*b*) Base (Found : C, 87·3; H, 6·8; N, 4·6. $C_{22}H_{21}N$ requires C, 88·3; H, 7·0; N, 4·7%).
(*c*) Base (Found : C, 88·5; H, 7·7; N, 4·2. $C_{25}H_{27}N$ requires C, 88·0; H, 7·9; N, 4·2%).
(*d*) Acid oxalate, m. p. 163—164° (recrystallised from ethanol), $[\alpha]_{5461}^{20°}$ + 34·0°, (*c*, 1·0 in ethanol) (Found : N, 3·2. $C_{27}H_{29}O_4N$ requires N, 3·2%).
(*e*) Base (Found : C, 86·8; H, 8·1; N, 5·0. $C_{21}H_{23}N$ requires C, 87·2; H, 8·0; N, 4·8%).
(*f*) Recrystallised from ethanol (Found : C, 81·7; H, 7·4; N, 5·0. $C_{19}H_{21}ON$ requires C, 81·7; H, 7·5; N, 5·0%).
(*g*) F.P. 890,633 gives m. p. 204—206°.

TABLE V.

3 : 3-*Diphenylprop*-2-*enyl Quaternary Ammonium Iodides*, $CPh_2{:}CH{\cdot}CH_2{\cdot}\overset{+}{N}R^1R^2R^3\ I^-$.

1 : 1-*Diphenylprop*-1-*ene methiodide.*	$\overset{+}{N}R^1R^2R^3$.	M. p.	Recrystallisation solvent.	Formula.	Analysis, Found, %. N.	Found, %. I.	Required, %. N.	Required, %. I.
3-*Dimethylamino*-	$\overset{+}{N}Me_3$	203—205° (decomp.)	Ethanol	$C_{18}H_{22}NI$	3·7	33·4	3·7	33·5
3-*Diethylamino*-	$\overset{+}{N}MeEt_2$	185—186	Methanol	$C_{20}H_{26}NI$	3·6	31·1	3·4	31·2
3-*Di*-n-*propylamino*-	$\overset{+}{N}MePr^n_2$	157—158[a] (decomp.)	Acetone	$C_{22}H_{30}NI$	3·1	29·3	3·2	29·2
3-*Di*-n-*butylamino*-	$\overset{+}{N}MeBu^n_2$	124—125[a]	Acetone	$C_{24}H_{34}NI$	2·8	27·7	3·0	27·4
3-*Methylamphetamino*-	$\overset{+}{N}Me_2{\cdot}CHMe{\cdot}CH_2Ph$	150—151[a] (decomp.)	Ethyl acetate–methanol	$C_{26}H_{30}NI$	2·9	25·9	2·9	26·3
3-*Diallylamino*-	$\overset{+}{N}Me[CH_2{\cdot}CH{:}CH_2]_2$	149—151 (decomp.)	Aqueous ethanol	$C_{22}H_{26}NI$	2·9	29·6	3·2	29·5
3-*Pyrrolidino*-	$\overset{+}{N}Me{<}[CH_2]_3{>}CH_2$	153—154[a]	Ethyl acetate–ethanol	$C_{20}H_{24}NI$	3·5	31·1	3·5	31·4
3-*Piperidino*-	$\overset{+}{N}Me{<}[CH_2]_4{>}CH_2$	189—190 (decomp.)	Ethanol	$C_{21}H_{26}NI$	3·2	30·3	3·3	30·3
3-*Morpholino*-	$\overset{+}{N}Me{<}[CH_2]_4{>}O$	163—164	Aqueous ethanol	$C_{20}H_{24}ONI$	3·3	30·1	3·3	30·2

(*a*) Ether added to complete crystallisation.

TABLE VI.

N-*Substituted* 3-*Amino*-1 : 1-*diphenylpropanes and Hydrochlorides*, $CHPh_2 \cdot CH_2 \cdot CH_2 \cdot NR^1R^2$ (IV).

1 : 1-*Diphenylpropane.*	NR^1R^2.	Base, B. p. or m. p.	Hydrochloride, M. p.	Recrystallisation solvent.	Formula.	Analysis, Found, %. N.	Found, %. Cl.	Required, %. N.	Required, %. Cl.
3-Amino-	NH_2	—	216—218°[a]	Ethyl acetate–ethanol	$C_{15}H_{17}N,HCl$	5·8	14·3	5·7	14·3
3-*Ethylamino-*	NHEt	—	163—164	Ethanol	$C_{17}H_{21}N,HCl$	5·0	13·2	5·1	12·9
3-*Dimethylamino-* ...	NMe_2	b. p. 183—185°/16 mm. m. p. 44—45°[b]	169—170	Ethyl acetate–methanol	$C_{17}H_{21}N,HCl$	5·0	12·8	5·1	12·9
3-Diethylamino- ...	NEt_2	—	145·5°[c]	Acetone	$C_{19}H_{25}N,HCl$	4·5	11·5	4·6	11·7
3-*Di*-n-*propylamino-*	NPr^n_2	—	114—115	Ethyl acetate	$C_{21}H_{29}N,HCl$	4·2	10·6	4·2	10·7
3-*Di*-n-*butylamino-*	NBu^n_2	—	113—114	Ethyl acetate	$C_{23}H_{33}N,HCl$	3·8	10·0	3·9	9·9
3-*Pyrrolidino-*	$N<[CH_2]_3>CH_2$	b. p. 125/0·02 mm.	135—136	Ethyl acetate–ethanol	$C_{19}H_{23}N,HCl$	4·4	11·8	4·6	11·8
3-Piperidino-	$N<[CH_2]_4>CH_2$	m. p. 40—41°[d]	215—217°[e]	Ethanol–acetone	$C_{20}H_{25}N,HCl$	4·6	11·1	4·4	11·3
3-*Morpholino-*	$N<[CH_2]_4>O$	—	208—209	Ethyl acetate–ethanol	$C_{19}H_{23}ON,HCl$	4·3	11·2	4·4	11·2

(*a*) Freeman, Ringk, and Spoerri, *loc. cit.*, give m. p. 217·5—218·5°.
(*b*) Recrystallised from light petroleum (b. p. 40—60°) (Found : C, 84·8; H, 9·0; N, 5·6. $C_{17}H_{21}N$ requires C, 85·4; H, 8·8; N, 5·9%).
(*c*) Eisleb, *loc. cit.*, quotes m. p. 143—144°.
(*d*) Recrystallised from light petroleum (b. p. 40—60°) (Found : C, 86·0; H, 9·2; N, 5·1. $C_{20}H_{25}N$ requires C, 86·0; H, 9·0; N, 5·0%).
(*e*) Report No. P.B. 981, *loc. cit.*, quotes m. p. 215—216°.

TABLE VII.

3 : 3-*Diphenylpropyl Quaternary Ammonium Iodides*, $CHPh_2 \cdot CH_2 \cdot CH_2 \cdot \overset{+}{N}R^1R^2R^3$ I^-.

1 : 1-*Diphenylpropane methiodide.*	$\overset{+}{N}R^1R^2R^3$.	M. p.	Recrystallisation solvent.	Formula.	Analysis, Found, %. N.	Found, %. I.	Required, %. N.	Required, %. I.
3-*Dimethylamino-*	$\overset{+}{N}Me_3$	179—180°	Ethyl acetate–methanol	$C_{18}H_{24}NI$	3·5	33·0	3·7	33·3
3-*Diethylamino-*	$\overset{+}{N}MeEt_2$	162—163	Aqueous ethanol	$C_{20}H_{28}NI$	3·2	31·0	3·4	31·1
3-*Di*-n-*propylamino-*	$\overset{+}{N}MePr^n_2$	144—145[a]	Ethyl acetate–methanol	$C_{22}H_{32}NI$	3·2	28·7	3·2	29·1
3-*Di*-n-*butylamino-*	$\overset{+}{N}MeBu^n_2$	142—143[a]	Methanol	$C_{24}H_{36}NI$	3·0	27·3	3·0	27·3
3-*Pyrrolidino-*	$\overset{+}{N}Me{<}[CH_2]_3{>}CH_2$	156—157	Ethanol	$C_{20}H_{26}NI$	3·6	31·2	3·4	31·2
3-*Piperidino-*	$\overset{+}{N}Me{<}[CH_2]_4{>}CH_2$	175—176 (decomp.)	Ethanol	$C_{21}H_{28}NI$	3·2	30·3	3·3	30·2
3-*Morpholino-*	$\overset{+}{N}Me{<}[CH_2]_4{>}O$	162—163	Ethyl acetate–methanol	$C_{20}H_{26}ONI$	3·5	29·6	3·3	30·0

(*a*) Ether added to complete crystallisation.

In a similar experiment, in which the refluxing was prolonged, a yellow oil separated and the solution became dark brown. After 2 hours' refluxing the mixture was cooled and the yellow oil (3·3 g.), which became resinous and could not be crystallised, was filtered off. *N*-Methylaniline hydrochloride was the only product to be isolated from the filtrate (m. p. 122—124°, recrystallised from acetone; Menschutkin, *Chem. Zentr.*, 1898, II, 479, gives m. p. 121—122°).

In an attempt to prepare the methiodide, 3-phenylmethylamino-1 : 1-diphenylprop-1-ene (1·0 g.) was dissolved in acetone (5 c.c.) and methyl iodide (1·0 g.) added. The solution became dark red and crystals of phenyltrimethylammonium iodide separated (0·25 g., m. p. 210—213°) (decomp.) (Found : N, 5·0; I, 47·5. Calc. for $C_9H_{14}NI$: N, 5·3; I, 48·3%). Willstätter, Hocheder, and Hug (*Annalen*, 1909, **371**, 27) quote m. p. 210—212°; other literature values vary between 218° and 232°.

N-*Substituted* 3-*Amino*-1 : 1-*diphenylpropanes* (IV).—(a) *Reduction in neutral solution.* The 3-amino- or *N*-substituted 3-amino-1 : 1-diphenylprop-1-ene hydrochloride (*e.g.*, 5 g.) was dissolved in ethanol (*e.g.*, 20 c.c.), palladium–charcoal catalyst (*e.g.*, 2·5 g.) added, and the mixture shaken in an atmosphere of hydrogen at room temperature and pressure. When absorption of hydrogen (*ca.* 10% in excess of the calculated volume) had ceased, usually after 1—2 hours, the catalyst was removed by filtration, and the filtrate evaporated to small bulk. Anhydrous ether was added, first gradually to the point of crystallisation, then in excess to precipitate the 3-amino- or N-*substituted* 3-*amino*-1 : 1-*diphenylpropane hydrochloride* in 75—90% yield. The hydrochlorides included in Table VI were prepared by this method; the *bases* were prepared from the recrystallised hydrochlorides.

(b) *Reduction of the base.* 3-Dimethylamino-1 : 1-diphenylprop-1-ene (VI; $R^1 = R^2 = Me$) (4·3 g.) in ethanol (20 c.c.) and palladium–charcoal (1·5 g.) were shaken in an atmosphere of hydrogen, 600 c.c. (calculated 450 c.c.) being absorbed during 2 hours. The catalyst was removed, the alcohol evaporated, and the residual oil fractionally distilled under reduced pressure. First runnings (b. p. to 183°/16 mm.) amounted to 1·1 g.; 3-*dimethylamino*-1 : 1-*diphenylpropane* (IV; $R^1 = R^2 = Me$) (2·9 g.) was collected at 183—185°/16 mm. and crystallised on cooling.

In the reduction of 3-di-*n*-propylamino-1 : 1-diphenylprop-1-ene (VI; $R^1 = R^2 = Pr^n$) (3·5 g.) the volume of hydrogen absorbed (510 c.c.) similarly was in excess of the calculated (309 c.c.) and the product (2·8 g.) boiled over the range 90—146°/0·4 mm.; hydrochloride, m. p. <100°. Recrystallisation of the crude hydrochloride from ethyl acetate afforded a small quantity (0·5 g.) of 3-*di*-n-*propylamino*-1 : 1-*diphenylpropane hydrochloride* (IV; $R^1 = R^2 = Pr^n$), m. p. 114—115°, identical with that prepared by method (a).

3-Methylamphetamino-1 : 1-diphenylprop-1-ene (VI; $R^1 = Me$, $R^2 = CHMe{\cdot}CH_2Ph$) (3·0 g.) in ethanol (15 c.c.) and palladium–charcoal (1·0 g.) was shaken in an atmosphere of hydrogen, 360 c.c. being absorbed (calculated 205 c.c.). The oil which remained after removal of the catalyst and evaporation of the alcohol distilled over the range 100—140°/18 mm. (small residue); redistillation gave a fraction of b. p. 138—140°/18 mm. which was identified as *dextro-N*-methylamphetamine (hydrochloride, m. p. 174—175°, not depressed by admixture with an authentic specimen).

The *methiodides* of the *N*-disubstituted 3-amino-1 : 1-diphenylpropanes (Table VII) were prepared by adding methyl iodide (2 equivalents) to a solution of the tertiary base in acetone. The salts crystallised out in almost quantitative yield except in those examples, indicated in the Table, in which the addition of ether was necessary.

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