Sodium 11-(**N-Ethylanilino**)-**hendecanoate**.—11-Bromohendecanoic acid for this preparation was made as follows: One hundred eighty-four grams (1 mole) of 10-hendecenoic (undecylenic) acid, b.p. 166–168° (10 mm.), was placed in a flask fitted with a stirrer and gas inlet and outlet tubes, and the solution was maintained at 35°. To it was added 0.75 g. of α,α'-azobis-(α,γ-dimethyl-γ-methoxyvaleronitrile),[14] a free radical catalyst with a half-life of 7.7 hours at 35°. Hydrogen bromide was passed in during 1.5 hours until an excess was present. The solution was then evaporated. The crude acid melted at 36–44°. It was recrystallized twice from 500 ml. of petroleum ether (b.p. 30–75°) by cooling the solution to −20° before filtering, and then melted at 49–50°. The yield was 168 g. or 64%.[15]

11-Bromohendecanoic acid (70 g., 0.26 mole) and ethylaniline (109 g., 0.9 mole) were heated on a steam-bath for 12 hours. To the mixture was added 22 g. of sodium hydroxide in 50 ml. of water and the liberated ethylaniline was removed by steam distillation. The solution was evaporated to dryness.

11-[**N-Ethyl-***p*-(*p*-**nitrophenylazo**)-**anilino**]-**hendecanoic Acid**.—The above dry salt was dissolved in 800 ml. of glacial acetic acid. *p*-Nitroaniline (38.4 g., 0.28 mole) was diazotized and added to the acetic acid solution. After addition of 80 g. of sodium acetate the mixture was allowed to stand for 0.5 hour. The red dye was filtered off, washed with 50% acetic acid and then with water. The yield was 104 g. or 87%, based on 11-bromohendecanoic acid. The compound was recrystallized from methanol in 81% return to give red plates melting at 130–131°.

(14) J. A. Robertson, U. S. Patent 2,586,995 (Feb. 26, 1952).

(15) *Cf.* J. C. Smith, *Chemistry & Industry*, 461 (1938).

Anal. Calcd. for $C_{26}H_{34}N_4O_4$: N, 12.3. Found: N, 12.2, 12.2.

11-(*p*-**Amino-N-ethylanilino**)-**hendecanoic Acid Sulfate**.—Reduction of the dye was carried out as described for the corresponding valerate. The solution of the reduction mixture was filtered, evaporated to dryness, and extracted with 250 ml. of hot water to remove *p*-phenylenediamine. The water-insoluble amino acid was converted to its sulfate in ethanol and recrystallized from ethanol; m.p. 147–149°, yield 90%.

Anal. Calcd. for $C_{19}H_{32}N_2O_2 \cdot H_2SO_4$: N, 6.65. Found: N, 6.55, 6.56.

Exposure and Development.—Films were given a time-scale exposure by means of a sector wheel. They were then developed for 10 min. at 20° in a solution containing, per liter, 0.0125 mole of developer, 10 g. of sodium sulfite, 25 g. of sodium carbonate and 2 g. of potassium bromide. Following development, the films were washed, fixed and bleached by conventional means. Spectral curves were obtained on a GE recording spectrophotometer and yellow densities were read at the wave length of maximum absorption.

Acknowledgments.—The author wishes to thank Dr. J. P. Weiss and Mr. V. W. Blanchard of the du Pont Photo Products Department for carrying out the photographic tests, and Dr. F. C. McGrew for helpful discussions.

WILMINGTON, DELAWARE

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF THE UNIVERSITY OF ROCHESTER]

2,2-Diphenyl-3,3-dimethylethylenimine and Related Compounds

BY HENRY M. KISSMAN,[1] D. S. TARBELL AND JOHN WILLIAMS

RECEIVED JANUARY 17, 1953

The preparation of 2,2-diphenyl-3,3-dimethylethylenimine (I) by the action of phenylmagnesium bromide or phenyllithium on isobutyrophenone oxime is described. The identity of I is established through an alternate synthesis by ring closure from 1-chloro-1,1-diphenyl-2-amino-2-methylpropane (VII) and through acid-catalyzed cleavage to 1,1-diphenyl-2-amino-2-methylpropanol (III).

The present study of the synthesis and properties of 2,2-diphenyl-3,3-dimethylethylenimine (I) was undertaken in connection with the problem of the resolvability of the trivalent nitrogen atom in the ethylenimine ring.[2] It was also hoped that this completely substituted ethylenimine would be more stable than the ones previously studied.[2] The best method of preparation was found to be the action of phenylmagnesium bromide or phenyllithium at elevated temperatures on isobutyrophenone oxime.[3] When a mixture of the two stereoisomers of isobutyrophenone oxime[4] was allowed to react with excess phenylmagnesium bromide in refluxing toluene there was isolated from the reaction mixture a small amount of aniline[5] and a high-boiling oil; this solidified to form colorless plate-like crystals which softened at 60° and melted at 185°. The hydrochloride of this compound could be recrystallized to a constant m.p. of 227–228°, and it had the composition required for the hydrochloride of I.

The action of phenyllithium on isobutyrophenone oxime in boiling toluene gave a mixture of basic products, which could be separated by chromatography and distillation into aniline and a yellow oil. The oil had the same boiling point as the product from the phenylmagnesium bromide reaction but it could not be made to solidify. It had the elementary composition required for the ethylenimine I. An analysis and neutral equivalent determination on the hydrochloride confirmed this result but the salt melted at a somewhat lower temperature than the hydrochloride of the product obtained in the first reaction. The phenylurea derivative II of the products of both reactions had the same m.p.'s and

(1) Beaunit Mills Fellow, 1949–1950.

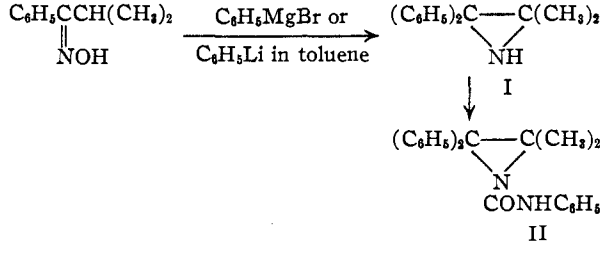
(2) Previous papers in this series: (a) H. M. Kissman and D. S. Tarbell, THIS JOURNAL, **74**, 4317 (1952); (b) D. S. Tarbell and D. K. Fukushima, *ibid.*, **68**, 2499 (1946); (c) T. L. Cairns, *ibid.*, **63**, 871 (1941).

(3) This procedure was discovered by (a) J. Hoch, *Compt. rend.*, **198**, 1865 (1934), and has been extended to the preparation of a number of aryl-substituted ethylenimines by Campbell; (b) K. N. Campbell, B. K. Campbell, J. F. McKenna and E. P. Chaput, *J. Org. Chem.*, **8**, 103 (1943); (c) K. N. Campbell, B. K. Campbell, L. G. Hess and I. J. Schaffner, *ibid.*, **9**, 184 (1944).

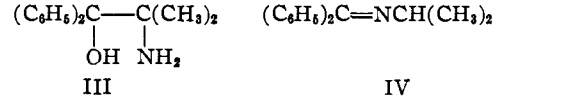
(4) H. M. Kissman and J. Williams, THIS JOURNAL, **72**, 5323 (1950).

(5) J. Hoch (*Compt. rend.*, **203**, 799 (1936)) explains the formation of aniline in the reaction of propiophenone oxime and phenylmagnesium bromide by assuming a rearrangement of the oxime to the corresponding anilide. Aniline would be formed in a reaction of this amide with more of the Grignard reagent.

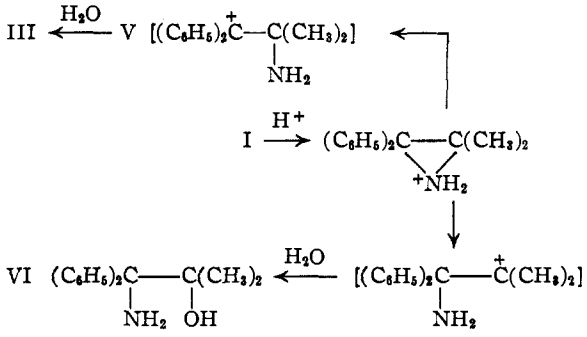
mixed m.p.'s. The possibility of a dimeric structure in either case was excluded by a molecular weight determination carried out on the phenylthiourea derivative of the product obtained from the phenyllithium reaction. The results agreed with the proposed structure I.



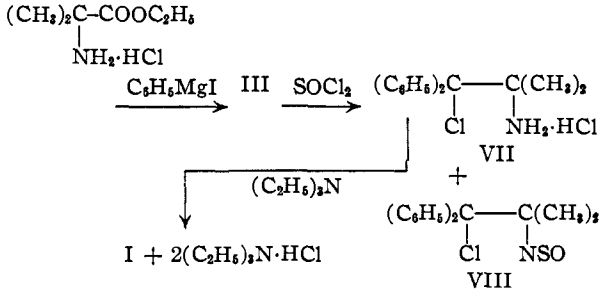
Additional proof for this structure and some explanation for the discrepancies in the physical characteristics of the products was found through acid hydrolysis experiments. The product from the phenylmagnesium bromide reaction could be hydrolyzed to give only one crystalline substance, which was shown to be 1,1-diphenyl-2-amino-2-methylpropanol (III) by an independent synthesis (see below). The oil obtained from the phenyllithium reaction yielded on hydrolysis not only this aminoalcohol but also a small amount of benzophenone. The isolation of this ketone indicated that the ethylenimine formed in the phenyllithium reaction had, in all probability, been contaminated with traces of the Schiff base IV. This substance is isomeric with I and would, therefore, not change the analyses values but its presence would certainly affect the melting points of the free base and its hydrochloride. It might also be the cause of the yellow color of the reaction product. Campbell[6] has shown that Schiff bases such as IV can be formed from ethylenimines at elevated temperatures and in the presence of basic catalysts. Apparently, phenyllithium is more likely to bring about such a rearrangement than phenylmagnesium bromide.



The hydrolysis of I was surprisingly difficult; the compound was recovered unchanged when refluxed with 3 *N* sulfuric acid for 30 minutes, and ring opening was finally brought about by boiling with 9 *N* sulfuric acid. The great stability of I in the presence of acid must, of course, be attributed to the high degree of substitution on both carbon atoms of the ethylenimine ring.[7] The formation of III rather than the isomeric 3-amino-3,3-diphenyl-2-methylpropanol-2 (VI) in the hydrolysis of the ethylenimine I is probably due to the greater stability of the carbonium ion V and is in line with previous reports in the literature.[3]



It seemed reasonably certain at this point that the product obtained in the phenylmagnesium bromide reaction was indeed 2,2-diphenyl-3,3-dimethylethylenimine, but because of the high m.p. of the compound,[8] it was desirable to find an alternate method of synthesis in order to establish this structure without ambiguity. It was possible to do this in very poor yield through ring closure of 1-chloro-1,1-diphenyl-2-amino-2-methylpropane (VII). The aminoalcohol III was prepared by the addition of ethyl α-aminoisobutyrate hydrochloride to phenylmagnesium iodide, and conversion to the halide VII was effected in low and variable yield by thionyl chloride in chloroform.[9] Compound VII was isolated as the hydrochloride and could not be transformed into the free base in aqueous systems without hydrolysis of the halide group and re-formation of the aminoalcohol III. Ring closure was finally brought about with excess triethylamine in chloroform. There were isolated from the reaction mixture, 1.5 equivalents of triethylamine hydrochloride and an ether soluble substance[10] whose hydrochloride melted at 230° and did not depress the melting point of the hydrochloride of the base obtained in the reaction of phenylmagnesium bromide and isobutyrophenone oxime. A neutral equivalent determination on the salt also agreed with the proposed structure. The phenylurea derivatives of the two bases were shown to be identical by mixed m.p.'s.



Various attempts to prepare I through the base-

(6) B. K. Campbell and K. N. Campbell, *J. Org. Chem.*, **9**, 178 (1944).

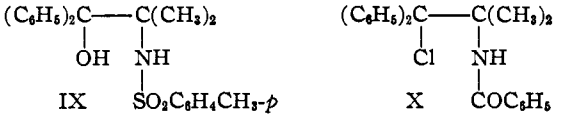
(7) We experienced similar difficulties in attempts to cleave the corresponding epoxide, 2,2-diphenyl-3,3-dimethylethylene oxide (Mme. Ramart-Lucas and M. E. Salmon-Legagneur, *Bull. soc. chim.*, [4] **45**, 730 (1929)). This compound could not be made to react with ammonia under pressure or with sodamide in boiling toluene. It was inert toward ammonium chloride, benzylamine hydrochloride and 6 *N* hydrochloric acid at room temperature.

(8) Campbell[3b] reported the m.p.'s of 2,2-diphenyl-3-ethylethylenimine and 2,2-diphenyl-3-methylethylenimine as 45 and 72°, respectively.

(9) This method gave rise not only to the hydrochloride of VII but also to a hexane-soluble substance which contained nitrogen, chlorine and sulfur, and to which was assigned on the basis of an analysis the provisional structure of the thionylamine VIII. In many runs this compound was the only reaction product. Thionylamines were studied by A. Michaelis, *Ann.*, **274**, 173, 259 (1893).

(10) The crude base melted at 193° but it was only available in such small amounts that it could not be properly purified as such. Mixed m.p. determinations were, therefore, carried out on the nicely crystalline hydrochloride and phenylurea derivatives.

catalyzed elimination of *p*-toluenesulfonic acid[11] from the sulfonamide IX failed. Neither was it possible to synthesize the N-benzoyl derivative of I by direct ring closure of 1-chloro-1,1-diphenyl-2-(N-benzoylamino)-2-methylpropane (X) in analogy to a synthesis reported by Kharasch and Priestley.[12]



IX X

Experimental[13]

2,2-Diphenyl-3,3-dimethylethylenimine (I). A. From Isobutyrophenone Oxime and Phenylmagnesium Bromide.—The reaction was carried out as described by Campbell and his co-workers[3b] from propiophenone oxime and phenylmagnesium bromide. Thirty-three grams (0.2 mole) of isobutyrophenone oxime and 1 mole of phenylmagnesium bromide were used. The reaction mixture was hydrolyzed with a solution of ammonium chloride and extracted with ether. The ether extracts were dried over calcium sulfate and saturated with hydrogen chloride. The precipitate which formed was filtered off, washed with ether, and recrystallized from dry acetone. It weighed 8 g. and melted at 192–193°. It was dissolved in concentrated ammonium hydroxide (45 ml.) and the solution was extracted with ether. The ether solution after drying over calcium sulfate was freed from solvents by evaporation. The residual oil was fractionated to yield two substances.

One boiled at 49–51° (1 mm.) and gave a **hydrochloride** which, after one recrystallization from acetone, melted at 193–194° and after two more recrystallizations from the same solvent did not depress the melting point of aniline hydrochloride (196–198°).

The second fraction distilled at 128–129° (1 mm.). On standing, this material crystallized partially to form large plates which melted at 181–185° with softening at 60°.[14]

A hydrochloride of this material was prepared in an ether solution saturated with hydrogen chloride. After recrystallization from acetone, this salt melted at 227–228°.

Anal. Calcd. for $C_{16}H_{18}NCl$ (hydrochloride of I): C, 73.96; H, 6.99. Found: C, 73.36; H, 6.42.

A phenylurea derivative II of this compound melted at 133–135° after sintering at 127°. It was recrystallized from hexane.

B. From Isobutyrophenone Oxime and Phenyllithium.—A solution of phenyllithium was prepared from 3.45 g. (0.5 mole) of lithium shavings and 39.25 g. (0.25 mole) of bromobenzene in 200 ml. of ether. All of the ether was replaced by 200 ml. of dry toluene. To the boiling mixture was then added, dropwise, 8.7 g. (0.05 mole) of isobutyrophenone oxime in 100 ml. of toluene over a period of 3 hours. Refluxing was continued for 1 hour after addition. The mixture was allowed to cool and was then poured into ice-water. A dark, extremely reactive, residue which had covered the bottom of the reaction vessel was slowly decomposed by the addition of wet ether. These ether washings were added to the water mixture, which was then extracted with 600 ml. of ether. The combined ether layers were evaporated and the residue was freed from benzene and toluene by evaporation *in vacuo*. The black oil, which remained, was dissolved in anhydrous ether and the solution was saturated with hydrogen chloride. The resulting precipitate was filtered off, quickly washed with ether and immediately dissolved in concentrated ammonia (80 ml.). This mixture was then extracted with 450 ml. of ether. The organic extracts were dried over potassium carbonate, freed from solvent by evaporation, and distilled. A forerun of 0.8 g., boiling at 50–90° (1 mm.), consisted mostly of aniline. This was identified by its *tribromo derivative* (m.p. 116–118°). The main fraction distilled at 130–140° (1 mm.), and weighed 3.8 g. Approximately 3 g. of tarry residue remained in the flask. The distillate was dissolved in 15 ml. of hexane and chromatographed on alumina. Elution with hexane yielded 0.5 g. of isobutyrophenone oxime (m.p. 57–58°). Chloroform eluted 2.7 g. (24%) of a yellow oil which was redistilled and boiled at 128–132° (0.8 mm.), $n^{22}D$ 1.5776.

Anal. Calcd. for $C_{16}H_{17}N$: C, 86.05; H, 7.67. Found: C, 86.21; H, 7.63.

A hydrochloride of this material prepared in ether and recrystallized from chloroform–ether melted at 193–194°.

Anal. Calcd. for $C_{16}H_{18}NCl$: C, 73.97; H, 6.99. Found: C, 74.14; H, 7.07.

A phenylurea derivative was prepared from the free base and phenyl isocyanate. After four recrystallizations from hexane it sintered at 127° and melted at 134–135°.

Anal. Calcd. for $C_{23}H_{22}N_2O$: C, 80.67; H, 6.48. Found: C, 80.97; H, 6.71.

This substance did not depress the melting point of the phenylurea derivative which had been obtained from the reaction of isobutyrophenone oxime and phenylmagnesium bromide.

A phenylthiourea derivative melted at 93–94°. It was recrystallized from hexane. The compound slowly decomposed during successive recrystallizations and no good analytical sample could be obtained. A molecular weight determination in camphor was run on a sample which had been recrystallized twice.

Anal. Calcd. for $C_{32}H_{22}N_2S$: mol. wt., 358.5. Found: mol. wt., 348.5, 333.4, 348.5.

Hydrolysis of 2,2-Diphenyl-3,3-dimethylethylenimine to 1,1-Diphenyl-2-amino-2-methylpropanol (III).—The ethylenimine from the phenyllithium reaction (0.4 g.) was refluxed with 10 ml. of 9 *N* sulfuric acid for 2 hours. The solution was cooled and extracted with 50 ml. of ether. The ether extract yielded 0.021 g. of benzophenone, which was identified by the m.p. (236–239°) of its 2,4-dinitrophenylhydrazone.[15] The acid solution was neutralized with sodium bicarbonate and extracted with 100 ml. of ether. The combined ether extracts were dried, more of the ether was removed, and 2 ml. of hexane was added to the residue. The white solid which formed at once was centrifuged free of solvent and recrystallized twice from hot hexane: the yield was 0.36 g. (83%); m.p. 118–120°; the hydrochloride decomposed at 219–222°. A mixed m.p. of the base with an authentic sample of the aminoalcohol III, prepared as below, showed no depression, and the hydrochlorides behaved similarly.

The solid ethylenimine obtained from isobutyrophenone oxime and phenylmagnesium bromide was hydrolyzed in the same manner; no benzophenone could be isolated but the aminoalcohol III was obtained in 87% yield.

1,1-Diphenyl-2-amino-2-methylpropanol (III).—α-Aminoisobutyric acid hydrochloride[16] was esterified with ethanol by the method recently described for the methyl ester.[17] Dry ethyl α-aminoisobutyrate hydrochloride (6.7 g.) was added in small portions to a refluxing solution of phenylmagnesium iodide, prepared from 6.1 g. (0.25 mole) of magnesium and 49.7 g. (0.24 mole) of iodobenzene in 125 ml. of dry ether. After completed addition (2 hours), the solution was allowed to reflux for 1 hour. The cooled mixture was then hydrolyzed with ice and ammonium chloride and extracted with 1 l. of ether. The ether solution was dried with potassium carbonate and all but 100 ml. of the solvent was removed by evaporation. A white solid crystallized during this process. It was filtered; the filtrate, which contained dark-colored tars, was discarded. The white solid was recrystallized four times from alcohol. There were obtained 4.89 g. (50%) of large prisms which melted at 124–125°.

Anal. Calcd. for $C_{16}H_{19}NO$: C, 79.63; H, 7.94. Found: C, 79.59; H, 7.71.

A hydrochloride of this compound was prepared in ether saturated with hydrogen chloride. It was recrystallized several times from absolute ethanol–ether; m.p. 221–222° (dec.).

Anal. Calcd. for $C_{16}H_{20}NOCl$: C, 69.40; H, 7.28. Found: C, 69.24; H, 7.14.

(11) G. M. Bennett and M. M. Hafez, *J. Chem. Soc.*, 287 (1941).

(12) M. S. Kharasch and H. M. Priestley, THIS JOURNAL, **61**, 3425 (1939).

(13) Melting points uncorrected; analyses by Micro-Tech Laboratories and Mrs. G. Sauvage.

(14) After standing in a closed vial for 2 years, the m.p. of this substance had dropped to 40°. This was unaccompanied by a visible change in the crystal structure.

(15) N. R. Campbell, *Analyst*, **61**, 393 (1936).

(16) J. H. Billman and E. E. Parker, THIS JOURNAL, **66**, 538 (1944).

(17) S. M. McElvain and E. H. Pryde, *ibid.*, **71**, 327 (1949).

1-Chloro-1,1-diphenyl-2-amino-2-methylpropane Hydrochloride (VII).—The hydrochloride of 1,1-diphenyl-2-amino-2-methylpropanol (III) (0.82 g., 0.002 mole) was suspended in 20 ml. of dry chloroform. Thionyl chloride was added (2 ml.) and the mixture was refluxed for 4 hours. Solvent and excess thionyl chloride were then taken off under vacuum. The dark residue was dissolved in chloroform and treated three times with charcoal. This removed most of the color. The chloroform was again removed *in vacuo* and the gummy residue was mixed with 100 ml. of anhydrous ether and allowed to stand for 3 days. A crystalline precipitate (0.17 g.) which had formed during that time was filtered off. It melted at 216–218° and was identified as starting material. Agitation of the ether solution caused precipitation of a white, amorphous powder which after two recrystallizations from chloroform–ether melted at 160–161°. This material weighed 0.413 g. (47%).

Anal. Calcd. for $C_{16}H_{19}NCl_2$: C, 65.09; H, 6.46. Found: C, 65.03; H, 6.32.

Using the same conditions and proportions of reactants, there could sometimes be isolated an ether-soluble material which melted at 79°. Elementary analysis showed that the compound contained labile chlorine (it gave an immediate precipitate with silver nitrate), sulfur, and non-basic nitrogen. For an analytical sample, the substance was crystallized from hexane, m.p. 79–80°.

Anal. Calcd. for $C_{16}H_{18}NSOCl$ (VIII): C, 62.83; H, 5.27. Found: C, 63.10; H, 5.40.

Cyclization of 1-Chloro-1,1-diphenyl-2-amino-2-methylpropane Hydrochloride.—The hydrochloride of 1-chloro-1,1-diphenyl-2-amino-2-methylpropane (VII) (0.1464 g., 0.0005 mole) was dissolved in 10 ml. of chloroform and 2 ml. of dry triethylamine. The solution was refluxed for 3 hours and then evaporated to dryness under vacuum. The residue was mixed with 50 ml. of dry ether. This left 0.11 g. (0.0008 mole) of a white solid undissolved. The substance melted at 253° and was identified as triethylamine hydrochloride by a mixed m.p. The ether solution was evaporated to dryness. There remained a small amount (0.079 g.) (70%) of solid material which melted approximately at 193°. This substance was too soluble in most solvents to permit purification. It was converted into a **hydrochloride** which after three recrystallizations from chloroform–ether melted at 230–231°. This salt did not depress the m.p. of the hydrochloride of the solid base which had been obtained by the action of phenylmagnesium bromide on isobutyrophenone oxime.

The hydrochloride was titrated potentiometrically with standard base giving a neutral equivalent of 254.5, compared to the calculated value of 259.8.

The remainder of the hydrochloride was converted to the free base, and the phenylurea was prepared from it in hexane; after four recrystallizations from hexane, it melted at 133–135°, and did not depress the m.p. of the phenylurea derivative obtained from I prepared by other methods.

1,1-Diphenyl-2-(*p*-toluenesulfonamido)-2-methylpropanol (IX).—To a solution of 1,1-diphenyl-2-amino-2-methylpropanol (III) (0.62 g., 0.0025 mole) in 25 ml. of dry pyridine was added 0.5 g. (0.0026 mole) of *p*-toluenesulfonyl chloride. The mixture was refluxed for 2 hours and then evaporated *in vacuo*. The dark residue was taken up in chloroform. This solution was washed with 75 ml. of water and dried over calcium sulfate. It was then boiled twice with portions of calcined Superfiltrol to remove most of the color. Heptane was added and the chloroform was removed by distillation. A precipitate formed on cooling; 0.25 g. (40%), m.p. 160–161°. This material did not form a hydrochloride with ethereal hydrogen chloride.

Anal. Calcd. for $C_{23}H_{25}NSO_3$: C, 69.84; H, 6.37. Found: C, 69.84; H, 6.48.

1,1-Diphenyl-2-(N-benzoylamino)-2-methylpropanol.—Ethyl α-(N-benzoylamino)-isobutyrate was prepared by Fischer esterification of the acid,[16] and melted at 118–119°; the reported value[18] is 123°. This compound (4.4 g.), suspended in 100 ml. of ether, was added to a solution of phenylmagnesium iodide prepared from 12.6 g. of iodobenzene and 1.6 g. of magnesium and the mixture was refluxed for 3 hours. It was then hydrolyzed with ice and ammonium chloride and extracted with ether. After being dried over sodium sulfate, the solution was partially evaporated. This caused the precipitation of a substance which crystallized in large plates from ethanol; m.p. 175–176°. Complete evaporation of the ether solution left a solid residue which, after one crystallization from ethanol, formed silky needles. This substance seemed to change crystal structure at 140° (hot-stage) and finally melted at 175–176°. A mixed melting point with the other form was 175–176°. Repeated recrystallizations of the needles always brought about the formation of some of the plate-like crystals. The two forms were separated manually for the preparation of analytical samples. The total amount of product (both forms) was 5.82 g. (73%).

Anal. Calcd. for $C_{23}H_{22}NO$: C, 79.97; H, 6.71. Found (plates): C, 79.73; H, 6.89; (needles): C, 80.65; H, 7.06.

The identity of the two substances was shown by their ultraviolet absorption spectra. Neither form reacted with gaseous hydrogen chloride in ether.

1-Chloro-1,1-diphenyl-2-(N-benzoylamino)-2-methylpropane (X).—A solution of the above carbinol (either form) (7.56 g.) in 100 ml. of benzene was refluxed with 5 cc. of thionyl chloride for two hours. The solution was then treated with 1 g. of charcoal and filtered. Evaporation of the solvent left a residue which, after two recrystallizations from chloroform–heptane, weighed 6.24 g. (89%) and melted at 144–145°.

Anal. Calcd. for $C_{23}H_{22}ClNO$: C, 75.91; H, 6.09. Found: C, 75.78; H, 6.08.

With 10% potassium hydroxide, this compound did not cyclize but formed instead the carbinol, 1,1-diphenyl-2-(N-benzoylamino)-2-methylpropanol.

ROCHESTER, N. Y.

(18) E. Mohr, *J. prakt. Chem.*, **81**, 70 (1910).