chloroform–petroleum ether, a bright red crystalline solid was obtained, m.p. 170–171° (lit.,[2] m.p. 170–171°). Mixed melting point determination with an authentic sample[2] showed no depression, m.p. 170–171.5°.

Anal. Calcd. for $C_{17}H_{20}N_4O_4$: C, 59.29; H, 5.85; N, 16.27. Found: C, 59.11; H, 5.71; N, 16.05. Ultraviolet absorbtion (ethanol) λ_{max} 386.5 mμ, ϵ_{max} 27,700.

The semicarbazone derivative prepared by standard procedure[12] formed colorless crystals from methanol, m.p. 183–184.5°. No depression was observed on mixed melting point with a sample prepared from the ketone obtained from the selenium dioxide dehydrogenation of *trans*-bicyclo[5.4.0]undecan-5-one, m.p. 183–184°.

Anal. Calcd. for $C_{12}H_{19}N_3O$: C, 65.13; H, 8.65; N, 18.99. Found: C, 65.01; H, 8.50; N, 18.71.

Conversion to the Dienone (XV).—0.5 g. of Bicyclo-[5.4.0]undec-10-en-5-one, 0.5 g. of *N*-bromosuccinimide, and 25 ml. of anhydrous carbon tetrachloride were refluxed for 2 hr. The solution was filtered and evaporated. The residue was treated with lutidine to effect debromination. After the usual work-up, the residual ketone was chromatographed over activated alumina in 1:1 ether–petroleum ether to yield a small amount of colorless oil, assigned to structure XV on the basis of the ultraviolet absorption (ethanol) λ_{max} 247.5 mμ, ϵ_{max} 8700.

The dinitrophenylhydrazone derivative was recrystallized from chloroform–petroleum ether, m.p. 192–193° (lit.,[2] m.p. 191–192°).

The semicarbazone derivative was recrystallized from methanol, m.p. 204.5–205° (lit.,[2] m.p. 205–206°).

Anal. Calcd. for $C_{12}H_{17}N_3O$: C, 65.73; H, 7.82; N, 19.16. Found: C, 65.98; H, 8.08; N, 18.91.

Bicyclo[5.4.0]undec-4-en-5-one (X).—*Anal.* Calcd. for $C_{11}H_{16}O$: C, 80.44; H, 9.81. Found: C, 80.29; H, 9.65. Ultraviolet absorption (ethanol) λ_{max} 241.5 mμ, ϵ_{max} 9300.

The dinitrophenylhydrazone derivative was prepared by standard procedure.[12] After recrystallization from chloroform–petroleum ether, a red crystalline solid was obtained, m.p. 177–178°. Mixed melting point with bicyclo[5.4.0]-undec-6-en-5-one from selenium dioxide dehydrogenation showed no depression, m.p. 177–178°.

Anal. Calcd. for $C_{17}H_{20}O_4$: C, 59.29; H, 5.85; N, 17.27. Found: C, 59.24; H, 5.92; N, 16.23. Ultraviolet absorption (ethanol) λ_{max} 388.5 mμ, ϵ_{max} 24,200.

The semicarbazone derivative formed colorless crystals from methanol, m.p. 190–191°. Mixed melting point with bicyclo[5.4.0]undec-6-en-5-one from selenium dioxide dehydrogenation showed no depression, m.p. 190–191°.

Anal. Calcd. for $C_{12}H_{19}N_3O$: C, 65.13; H, 8.65; N, 18.99. Found: C, 65.27; H, 8.91; N, 19.20.

Conversion to the Dienone XI.—Conversion to the dienone was carried out by the same procedure as described above for bicyclo[5.4.0]undec-10-en-5-one. Ultraviolet absorption (ethanol) λ_{max} 241.5 mμ, ϵ_{max} 5300.

The dinitrophenylhydrazone derivative was recrystallized from chloroform–petroleum ether, m.p. 200–202° (lit.,[2] 201–202°). Ultraviolet absorption (ethanol) λ_{max} 383 mμ, ϵ_{max} 22,000.

The semicarbazone was recrystallized from methanol, m.p. 224–225° (lit.,[2] 224–225°).

Anal. Calcd. for $C_{12}H_{17}N_3O$: C, 65.73; H, 7.82; N, 19.16. Found: C, 65.79; H, 7.91; N, 19.36.

Acknowledgment.—The authors wish to express their gratitude to Mr. Norbert Helmer, Analytical Research Department, National Aniline Division, Allied Chemical and Dye Corp., for some of the ultraviolet spectral data reported in this communication, and to Dr. Richard K. Hill of Princeton University for his suggestions during the course of this investigation.

3-Phenylcyclobutylamine. II

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Alkylation of diethyl malonate with 2-phenylpropane-1,3-diol di-*p*-toluenesulfonate followed by saponification gave 3-phenylcyclobutane-1,1-dicarboxylic acid which was decarboxylated to *cis*- and *trans*-3-phenylcyclobutanecarboxylic acids. These acids were degraded to *cis*- and *trans*-3-phenylcyclobutylamine, respectively. The dicarboxylic acid was degraded to 3-phenylcyclobutanone, the oxime of which gave *cis*-3-phenylcyclobutylamine[1] on reduction. This configurational assignment is based on the conversion of *cis*-3-phenylcyclobutanol,[8] via its tosylate ester, to *trans*-3-phenylcyclobutyl azide and hence to *trans*-3-phenylcyclobutylamine.

One of the two geometrical isomers of 3-phenylcyclobutylamine has been prepared[1] by reduction of 3-phenylcyclobutanone oxime with lithium aluminum hydride, but its configuration was not known. In an attempt to determine the configuration of this amine, and to provide the other isomer for pharmacological comparison, alternative synthetic routes to the amine have been investigated. These methods were also to provide access to an intermediate, 3-phenylcyclobutanone (V), by a potentially less hazardous route than that using dichlorodifluoroethylene previously employed.[2]

Diethyl phenylmalonate (I) was reduced to 2-phenylpropane-1,3-diol (II) with lithium aluminum hydride.[3] The di-*p*-toluenesulfonate (III) of II, on alkylating diethyl malonate, gave diethyl 3-phenylcyclobutane-1,1-dicarboxylate, which was saponified to 3-phenylcyclobutane-1,1-dicarboxylic acid (IV). This acid was degraded to 3-phenyl-

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[1] A. Burger and R. Bennett, *J. Med. Pharm. Chem.*, **2**, 1269 (1960).

[2] J. D. Roberts, A. Bruce Kline, and H. E. Simmons, *J. Am. Chem. Soc.*, **75**, 4765 (1953).

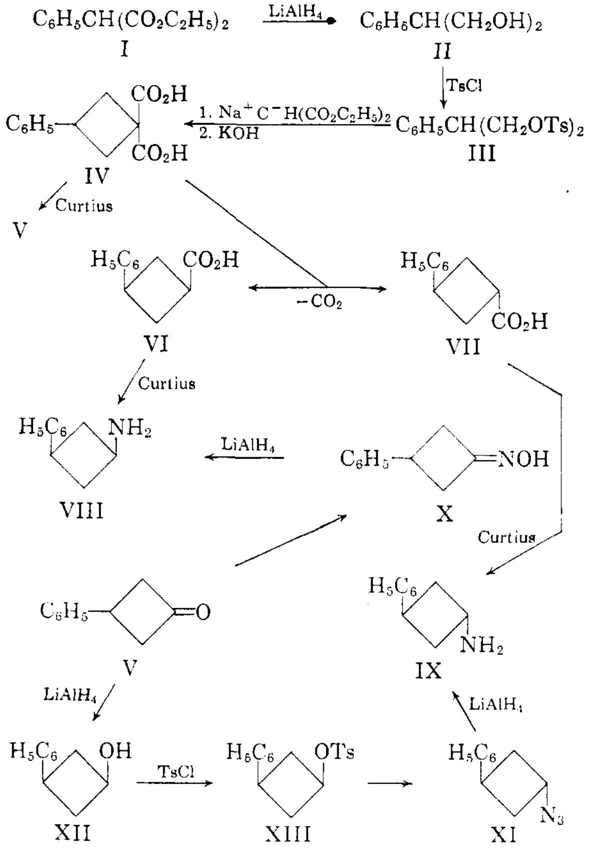
[3] This method is more convenient than the high pressure hydrogenolysis described by H. Adkins and H. R. Billica, *J. Am. Chem. Soc.*, **70**, 3131 (1948).

cyclobutanone (V) by a modified[4] Curtius procedure analogous to that employed for the preparation of the isomeric 2-phenylcyclobutanone from 2-phenylcyclobutane-1,1-dicarboxylic acid.[5] The semicarbazone of 3-phenylcyclobutanone (V) thus obtained was identical with an authentic sample stemming from the preparation by the method of Roberts.[2]

Decarboxylation of IV gave a mixture of *cis*- and *trans*-3-phenylcyclobutanecarboxylic acids (VI and VII) which was partially separated by chromatography. The acid eluted first from the column was a solid, m.p. 65–66°, and has been assigned the *trans* configuration (VII); the other acid (*cis*, VI) was an oil. Each of these acids was degraded to the corresponding amine (VIII and IX), respectively. The amine from the oily acid proved to be identical with the 3-phenylcyclobutylamine (VIII) obtained by Burger and Bennett[1] by reduction of 3-phenylcyclobutanone oxime (X) with lithium aluminum hydride.

Several attempts to ozonize the monocarboxylic acids VI and VII to the respective *cis*- and *trans*-cyclobutane-1,3-dicarboxylic acids[6] gave only oily products and a trace of solid material which could not be adequately purified. Chromatography and sublimation of the solid led to a substance melting over a range above the melting point of *cis*-cyclobutane-1,3-dicarboxylic acid, but well below that of the *trans* isomer. A number of experiments designed to avoid over-ozonization by shortening the reaction time remained inconclusive. Control runs ozonizing *trans*-2-phenylcyclopropanecarboxylic acid to *trans*-cyclopropane-1,2-dicarboxylic acid proceeded in excellent yields; the difficulties encountered in the 1,3-cyclobutane series are the harder to explain as the truxillic acids have been ozonized successfully to cyclobutane-1,2,3,4-tetracarboxylic acids.[7] The failure of our present ozonizations forced us to find another method to assign configurations to VI and VII.

Roberts and Manatt[8] have prepared the same isomer of 3-phenylcyclobutanol (XII) by lithium aluminum hydride reduction of 3-phenylcyclobutanone as well as by catalytic hydrogenation of 2-chloro-3-phenylcyclobut-2-enol. This 3-phenylcyclobutanol was assigned the *cis* configuration because *cis* addition of hydrogen must occur from the less hindered side of the cyclobutene ring, away from the hydroxyl group. In 3-phenylcyclobutanone the phenyl group sterically hinders the approach of lithium aluminum hydride, which can attack only from one side. It is most likely that the reduction of 3-phenylcyclobutanone oxime (X) would take the same steric course to give *cis*-3-phenylcyclobutylamine (VIII). Confirmation of this assumption has now been obtained in the conversion of *cis*-3-phenylcyclobutanol (XII) into the *trans*-amine (IX) isomeric with that formed by reduction of the oxime X. In our sequence, 3-phenylcyclobutanol (XII) was prepared by lithium aluminum hydride reduction of 3-phenylcyclobutanone.[8] The oily *p*-toluenesulfonate (XIII) of XII was treated with an alcoholic aqueous solution of sodium azide[9] to give a liquid azide XI which was reduced with lithium aluminum hydride. Only one isomer of 3-phenylcyclobutylamine was obtained by this route, the acetyl derivative of which was identical with *N*-(3-phenylcyclobutyl)acetamide from the Curtius degradation of the solid acid VII. This result is in keeping with our configurational assignments provided that inversion occurs in the formation of the azide XI.



Further support of these configurations was provided by nuclear magnetic resonance measurements of VI and VII. The measurements confirm the configurations of these compounds and are in good agreement with analogous signals produced by *cis*- and *trans*-3-benzyloxycyclobutanecarboxylic acids, respectively, which will be published later.

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(4) J. Weinstock, *J. Org. Chem.*, **26**, 3511 (1961).

(5) C. Beard and A. Burger, *J. Org. Chem.*, **26**, 2335 (1961).

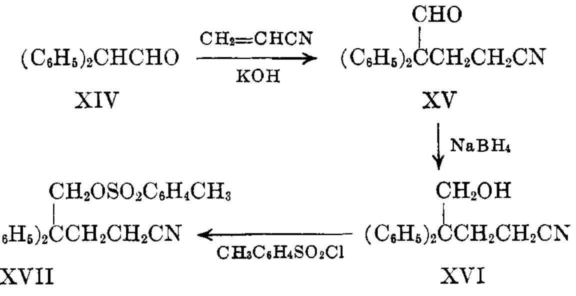
(6) D. H. Deutsch and E. R. Buchman, *Experientia*, **6**, 562 (1950); details by private communication from Prof. E. R. Buchman.

(7) R. Criegee and H. Höver, *Chem. Ber.*, **93**, 2521 (1960).

(8) Private communication; see also S. Manatt, dissertation, California Institute of Technology, 1960.

(9) For a previous use of this displacement reaction in the cyclobutane series, see M. Avram, C. C. Nenitzescu, and M. Maxim, *Chem. Ber.*, **90**, 1424 (1957).

In an attempt to synthesize 3,3-diphenylcyclobutylamine *via* 3,3-diphenylcyclobutanecarboxylic acid, we followed a route analogous to that used to prepare 3,3-dimethylcyclobutanecarboxylic acid.[10] A Michael addition of diphenylacetaldehyde (XIV) to acrylonitrile in the presence of potassium hydroxide gave 2,2-diphenyl-4-cyanobutyraldehyde (XV), which was reduced with sodium borohydride to 2,2-diphenyl-4-cyanobutanol (XVI). This oily product was converted to a solid toluenesulfonate (XVII), but cyclization of this ester with potassium isobutoxide to 3,3-diphenylcyclobutyl cyanide could not be effected.



Experimental[11]

2-Phenylpropane-1,3-diol (II).—Diethyl phenylmalonate (I) (295 g., 1.25 moles) was added, under nitrogen, to a stirred solution of lithium aluminum hydride (57 g., 1.5 moles) in ether (1.5 l.) at a rate to maintain refluxing, and refluxing and stirring were continued for another 2.5 hr. The cooled mixture was decomposed with 100 ml. of water and 2 l. of 15% sulfuric acid, the ether layers from two batches were combined, washed, and dried over sodium sulfate. Fractionation furnished 212 g. (56%) of an oil, b.p. 134–139°/0.7 mm. (lit. b.p.[3] 136–137°/2 mm.). The diol solidified on cooling, and was used directly in the next step.

2-Phenylpropane-1,3-diol Di-*p*-toluenesulfonate (III).—To a solution of 2-phenylpropane-1,3-diol (9.7 g., 64 mmoles) in 50 ml. of dry pyridine was added, below 10°, 25.4 g. (133 mmoles) of *p*-toluenesulfonyl chloride. After 12 hr. at 20° the mixture was treated with cold hydrochloric acid, and the resulting solid filtered off. Recrystallization from ethanol or propanol gave 24.9 g. (85%) of colorless crystals, m.p. 127.5–128°.

Anal. Calcd. for $C_{23}H_{24}O_6S_2$: C, 59.97; H, 5.25. Found: C, 59.88; H, 5.22.

3-Phenylcyclobutane-1,1-dicarboxylic Acid (IV).—A solution of 2-phenylpropane-1,3-diol di-*p*-toluenesulfonate (198 g., 0.43 mole) and 75 g. (0.47 mole) of diethyl malonate in 1 l. of dry dioxane was warmed almost to reflux under a cover of nitrogen, and a suspension of sodium hydride (19.6 g., 0.43 mole; 52.6% in mineral oil) in 100 ml. of dioxane was added dropwise over 80 min. at a rate to cause spontaneous refluxing. After stirring and refluxing for 1 hr., a further 19.6 g. of sodium hydride in 200 ml. of dioxane was added with great care over a period of 3 hr. After another 15 hr. of refluxing, the dioxane was removed under reduced pressure, the residue treated with water, extracted with ether, and the combined ether extracts were dried over magnesium sulfate. Fractionation gave 83.5 g. of diethyl 3-phenylcyclobutane-1,1-dicarboxylate, b.p. 126.5–129.5°/0.32 mm., contaminated with mineral oil from the sodium hydride. The ester was saponified in a refluxing solution of 53 g. of potassium hydroxide in 154 ml. of 50% aqueous ethanol for 3 hr., solvent was removed under reduced pressure, and the residue taken up in water. The aqueous solution was washed twice with ether, cooled, acidified with concd. hydrochloric acid, and the precipitated solid filtered off. Recrystallization from water yielded 60 g. (63.4%) of acid, m.p. 181–182° dec. An analytical sample, from chloroform, had the same melting point.

Anal. Calcd. for $C_{12}H_{12}O_4$: C, 65.44; H, 5.49. Found: C, 65.47; H, 5.44.

3-Phenylcyclobutanone (V).—A solution of 1.1 g. (5 mmoles) of 3-phenylcyclobutane-1,1-dicarboxylic acid in 2 ml. of acetone and 2 ml. of water was treated with 1.2 g. (12 mmoles) of triethylamine in 10 ml. of acetone, then with ethyl chloroformate (1.30 g., 12 mmoles), and later with sodium azide (1.0 g., 15 mmoles) under the conditions specified by Weinstock.[4] Conversion of the bisazide to the bisethyl carbamate followed by hydrolysis with 2% sulfuric acid, steam distillation, and ether extraction yielded 45% of ketone. The semicarbazone, m.p. 210.5–212°, was identical (mixture melting point) with an authentic sample.[2]

***cis*- and *trans*-3-Phenylcyclobutanecarboxylic Acids (VI and VII).**—3-Phenylcyclobutane-1,1-dicarboxylic acid (10 g.) was decarboxylated by heating in a metal bath at 200–210° for 30 min. The resulting oil (8.0 g.) was chromatographed on a 100-mesh silica column (63 × 3.3 cm.) under *ca.* 20 mm. positive pressure, using 10% of ether in hexane as the eluent and collecting 100-ml. fractions. After obtaining *ca.* 0.1 g. of oil from fractions No. 7–8, 1.7 g. of sticky solid was collected (fractions No. 15–18). The volume of fractions was then increased to 500 ml., and oily material was thus obtained. The last 0.9 g. of oil was eluted with 50% ether–hexane.[12]

The solid (*trans-*) acid was pressed on a porous plate and recrystallized from hexane, yielding 1.2 g. of material, m.p. 65–66°.

Anal. Calcd. for $C_{11}H_{12}O_2$: C, 74.97; H, 6.86. Found: C, 75.06; H, 6.76.

The *p*-toluidide was prepared by treating the acid VII (m.p. 62–64°) (0.15 g.) with a solution of 0.5 ml. of thionyl chloride in 1 ml. of benzene, and after 24 hr. at 20° removing volatile fractions at 20° under vacuum. The residue was dissolved in 10 ml. of ether, treated with 0.3 g. of *p*-toluidine, and the mixture shaken twice with 10 ml. of 2 *N* hydrochloric acid. The ether layer was separated, washed, dried over magnesium sulfate, and evaporated. The residue crystallized from benzene–hexane, m.p. 133–133.5°.

Anal. Calcd. for $C_{18}H_{19}NO$: C, 81.47; H, 7.22. Found: C, 81.43; H, 7.04.

In a similar manner, the oily (*cis-*) acid gave a *p*-toluidide, m.p. 137–138.5°.

Anal. Calcd. for $C_{18}H_{19}NO$: C, 81.47; H, 7.22. Found: C, 81.26; H, 7.14.

A mixture of the two toluidides melted at 116.5–119.5°.

***trans*-3-Phenylcyclobutylamine (IX).**—A solution of triethylamine (0.91 g., 9 mmoles) in 15 ml. of acetone was added, at 0–5°, to a stirred solution of 1.41 g. (8 mmoles) of *trans*-3-phenylcyclobutanecarboxylic acid in 3 ml. of 50% aqueous acetone. By the procedure previously described,[4] and using 1.09 g. (10 mmoles) of ethyl chloroformate and 0.8 g. (12 mmoles) of sodium azide, the oily azide was obtained and rearranged in 20 ml. of dry toluene at 100°. The isocyanate was hydrolyzed with 6 ml. of 11 *N* hydrochloric acid at 100° for 15 hr. and worked up. The amine (0.82 g., 69.5%) distilled at 70–70.5°/0.75 mm. The hydrochloride was prepared in dry ether and recrystallized from ethanol–ethyl acetate, m.p. 178–180°.

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[10] M. Julia and A. Rouault, *Bull. soc. chim. France*, 1833 (1959).

[11] All melting points are corrected, boiling points uncorrected. Microanalyses by Miss Winkie Sheffield, and by Alfred Bernhard, Mühlheim (Ruhr), Germany.

[12] Since the infrared spectra of the solid and oily 3-phenylcyclobutanecarboxylic acids showed only minor differences in contrast to the 2-isomers,[5] separation based on infrared spectra could not be used in this case.

Anal Calcd. for $C_{10}H_{13}N \cdot HCl$: C, 65.37; H, 7.68; N, 7.63. Found: C, 65.52; H, 7.69; N, 7.85.

The acetyl derivative melted at 118–118.5°.

Anal. Calcd. for $C_{12}H_{15}NO$: C, 76.15; H, 7.99; N, 7.40. Found: C, 76.00; H, 7.88; N, 7.07.

***cis*-3-Phenylcyclobutylamine (VIII).**—A sample of the (oily) *cis*-3-phenylcyclobutanecarboxylic acid (0.18 g., 1 mmole) was degraded as described above. The acetyl derivative of the resulting amine melted at 124.5–126.5°, and this melting point remained undepressed by admixture of a sample of the *N*-(3-phenylcyclobutyl)acetamide prepared by Burger and Bennett[1] from the amine obtained by reduction of 3-phenylcyclobutanone oxime with lithium aluminum hydride. The infrared spectra of the two *N*-acetyl derivatives were also identical, but different from that of the *trans*-*N*-acetyl derivative (m.p. 118–118.5°, above).

***trans*-3-Phenylcyclobutylamine from *cis*-3-Phenylcyclobutanol.**—*cis*-3-Phenylcyclobutanol (XII) was prepared[8] by reduction of pure 3-phenylcyclobutanone with lithium aluminum hydride in 88% yield. It boiled at 121–122°/3 mm., n^{30}_D 1.5395. A solution of XII (1.85 g., 12.5 mmoles) in 6 ml. of dry pyridine was treated with 2.48 g. (13 mmoles) of *p*-toluenesulfonyl chloride at 10° and allowed to stand at 20° for 12 hr. The mixture was poured into excess 2 *N* hydrochloric acid and the oil which separated was extracted into ether. After work-up, the oily *cis*-3-phenylcyclobutyl toluenesulfonate (XIII) weighed 3.35 g. (88%), and was used in the next step.

A solution of XIII (3.33 g.) and sodium azide (1.37 g.) in 5 ml. of ethanol and 2 ml. of water was refluxed for 12 hr., cooled, diluted with water, and extracted with ether. The residue from the washed and dried ether solution weighed 1.28 g. (67%). The azide (XI) boiled at 85.5°/0.85 mm.–91.5°/1.4 mm. A solution of the azide (0.6 g.) in 5 ml. of ether was added to a solution of 0.4 g. of lithium aluminum hydride in 10 ml. of ether at 15–20°. The mixture was stirred at 25–30° for 2 hr., then decomposed with water and 30 ml. of 2 *N* hydrochloric acid and worked up. The oily amine (0.25 g., 49%) yielded an acetyl derivative, m.p. 118–118.5°. The melting point was not depressed by admixture with a sample of the acetyl derivative obtained by degradation of *trans*-3-phenylcyclobutanecarboxylic acid (VII), and the infrared spectra of the two amides were identical. A mixture melting point with *cis*-*N*-(3-phenylcyclobutyl)acetamide (m.p. 125.5–126.5°) was 89–99°.

4-Cyano-2,2-diphenylbutyraldehyde (XV).—Freshly distilled acrylonitrile (11.7 g., 0.22 mole) was added to a rapidly stirred mixture of 39.2 g. (0.2 mole) of diphenylacetaldehyde and 0.5 ml. of 10 *N* potassium hydroxide solution. After spontaneous heating subsided, the mixture was kept at 95–100° for 1 hr., cooled, 3 ml. of 2 *N* hydrochloric acid was added, and the organic material taken up in ether–benzene (1:1). Removal of the solvent from the washed and dried solution left 49.1 g. of solid which was recrystallized from methanol, m.p. 81–82°.

Anal. Calcd. for $C_{17}H_{15}NO$: C, 81.90; H, 6.06; N, 5.62. Found: C, 82.16; H, 5.99; N, 5.56.

4-Cyano-2,2-diphenylbutyl *p*-Toluenesulfonate (XVII).—A solution of 1.76 g. (0.045 mole) of sodium borohydride in 10 ml. of water was added to a solution of 14.9 g. (0.06 mole) of 4-cyano-2,2-diphenylbutyraldehyde in 100 ml. of methanol at a temperature below 25°, and the mixture was allowed to stand at 15–20° for 14 hr. Then 150 ml. of 2 *N* hydrochloric acid was added at 20°, the mixture was diluted with water, and thoroughly extracted with ether. The thick oily residue from the washed and dried ether extracts weighed 14.3 g. This crude 4-cyano-2,2-diphenylbutanol (XVI) was dissolved in 100 ml. of dry pyridine and treated, with stirring at *ca.* 5°, with 12.4 g. (0.065 mole) of *p*-toluenesulfonyl chloride. After being stirred at 20° for 15 hr., the mixture was decomposed with 200 ml. of 10 *N* hydrochloric acid and ice, and the separated solid was washed, filtered, and recrystallized from ethanol, m.p. 111.5–113°. The yield was 13.0 g.

Anal. Calcd. for $C_{24}H_{23}NO_3S$: C, 71.09; H, 5.72. Found: C, 70.81; H, 5.54.

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Pyrolysis of *N*-Methylpyrrole and 2-Methylpyrrole[1]

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N-Methylpyrrole and 2-methylpyrrole were pyrolyzed at various temperatures and the composition of the volatile liquid pyrolysis products was determined. The relative quantities of the major reaction products, 2-methylpyrrole, pyridine and pyrrole, were found to vary with the pyrolysis temperature.

The thermal rearrangement of *N*-methylpyrrole to 2-methylpyrrole and pyridine had been reported in the literature,[2] but little information on the effect of temperature on the product composition was disclosed.

In order to gain further information on the behavior of *N*-substituted pyrroles at high temperatures and to determine conditions which effected their rearrangement, *N*-methylpyrrole was pyrolyzed at 550, 560, 650, and 745° and 2-methylpyrrole was pyrolyzed at 650 and 745°. In 1958, Jacobson, Heady, and Dinneen[3] reported the results of a similar study with *N*-methylpyrrole

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[1] Abstracted from the Ph.D. dissertation of Peter Drenchko, University of Kentucky, 1958.

[2] A. Pictet and A. Steinmann, *Ber.*, **37**, 2792 (1904); W. Reppe, *Ann.*, **596**, 80 (1955).

[3] I. A. Jacobson, Jr., H. H. Heady, and G. U. Dinneen, *J. Phys. Chem.*, **62**, 1563 (1958).