

ported for 9- $\alpha$ -D-arabofuranosyladenine<sup>7</sup>: m.p. 208°;  $[\alpha]^{17D} +69^\circ$  (c 1.1% in water).

**2,8-Dichloro-9-tetraacetyl- $\beta$ -D-glucopyranosyladenine** from 2,6,8-Trichloro-9-tetraacetyl- $\beta$ -D-glucopyranosylpurine.—The tetraacetyl glucosyltrichloropurine<sup>8</sup> (0.8 g.) was heated in a sealed tube with 15 ml. of ethanolic ammonia (saturated at 0°) for one hour at 100°. The cooled solution was evaporated to dryness and the residue dissolved in 5 ml. of pyridine and 2 ml. of acetic anhydride and kept overnight at room temperature. Ethanol was added, solvents removed under reduced pressure, and the residue crystallized from 4 ml. of glacial acetic acid, giving 0.11 g. (14%) of 2,8-dichloro-9-tetraacetyl- $\beta$ -D-glucopyranosyladenine as colorless needles, m.p. 213–216°, alone or in admixture with an authentic specimen.<sup>8</sup>

*Anal.* Calcd. for C<sub>19</sub>H<sub>21</sub>O<sub>9</sub>N<sub>5</sub>Cl<sub>2</sub>: N, 13.1. Found: N, 13.2.

**2,6,8-Trichloro-9-triacetyl- $\beta$ -D-ribofuranosylpurine**.—A suspension of 4.5 g. of silver 2,6,8-trichloropurine in 120 ml. of xylene was treated with triacetyl D-ribofuranosyl chloride prepared from 4 g. of tetraacetate. The mixture

was refluxed for 1.5 hours, then filtered hot and the filtrate evaporated to dryness. Crystallization of the residue from 75 ml. of ethanol gave 3.9 g. (60%) of fine needles, m.p. 164–165°, raised to 166–168° by recrystallization from ethanol.

*Anal.* Calcd. for C<sub>16</sub>H<sub>15</sub>O<sub>7</sub>N<sub>4</sub>Cl<sub>3</sub>: C, 39.9; H, 3.1; N, 11.6. Found: C, 40.4; H, 3.5; N, 11.5.

**Acknowledgment.**—The authors wish to thank Mr. Roscoe C. Funk, Jr., for the microanalyses and Dr. George Bosworth Brown for his interest in this work. Thanks are due also to Dr. Howard W. Bond of the National Institute of Arthritis and Metabolic Diseases for a gift of 4,6-diamino-5-benzeneazo-2-methylpyrimidine, and to Dr. George H. Hitchings, of the Wellcome Research Laboratories, for a supply of 2,6-diaminopurine hydrochloride.

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[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, BROOKLYN COLLEGE]

## Tertiary Amines Derived from N-(2-Pyridyl, 2-Thiazolyl and 2-Lepidyl)-1,2-diphenylethylamine

BY IRVING ALLAN KAYE AND CHESTER L. PARRIS

N-(2-Pyridyl)-1,2-diphenylethylamine was prepared by the reductive alkylation of 2-benzylideneaminopyridine with benzylmagnesium chloride. The 2-thiazolyl isostere was formed similarly, as well as by total synthesis, and the 2-lepidyl analog by condensing 2-chlorolepidine with 1,2-diphenylethylamine. Although these secondary amines could be alkylated in the presence of lithium amide with several diverse alkyl halides and with styrene oxide to yield products desired for testing as potential antimitotic agents, reaction between 1,2-diphenylethyl chloride and either 2-aminopyridine or N,N-dimethyl-N'-(2-pyridyl)-ethylenediamine under the same conditions yielded *trans*-stilbene as the only isolable product.

Interest in the preparation of compounds related to 1,2-diphenylethylamine has been whetted by the observation that some of these substances show promise as tumor growth-inhibitors<sup>1–7</sup> or as analgesics.<sup>8–13</sup> Since a heterocyclic nucleus frequently enhances or is essential for pharmacological activity, some 1,2-diphenylethylamines, having an amino hydrogen replaced by either a 2-pyridyl, 2-thiazolyl or a 2-lepidyl group, were prepared for evaluation as tumor-necrotizing agents.<sup>14</sup>

The tertiary amines (II Aa-g, II Ba, II Ca)

(1) H. Lettré, M. Albrecht and H. Fernholz, *Naturwissenschaften*, **29**, 30 (1941); H. Lettré and H. Fernholz, *Z. physiol. Chem.*, **278**, 175 (1943); H. Lettré and I. Delitzsch, *ibid.*, **281**, 139 (1944).

(2) J. H. Hartwell and S. R. L. Kornberg, *THIS JOURNAL*, **67**, 1606 (1945).

(3) C. T. Bahner, H. E. Dickson and L. Moore, *ibid.*, **70**, 1982 (1948).

(4) R. E. Lutz, J. A. Freek and R. S. Murphey, *ibid.*, **70**, 2015 (1948).

(5) (a) W. J. P. Neish, *Rec. trav. chim.*, **68**, 337 (1949); (b) **69**, 207 (1950).

(6) L. H. Goodson and H. Christopher, *THIS JOURNAL*, **72**, 358 (1950).

(7) K. Rorig, *J. Org. Chem.*, **15**, 391 (1950).

(8) E. C. Dodds, W. Lawson and P. C. Williams, *Nature*, **151**, 614 (1943); *Proc. Roy. Soc. (London)*, **B132**, 119 (1944); *Nature*, **154**, 514 (1944); E. C. Dodds, W. Lawson, S. A. Simpson and P. C. Williams, *J. Physiol.*, **104**, 47 (1945).

(9) W. D. McPhee and E. S. Erickson, Jr., *THIS JOURNAL*, **68**, 624 (1946).

(10) L. H. Goodson, C. J. W. Wiegand and J. D. Splitter, *ibid.*, **68**, 2174 (1946).

(11) R. B. Moffett and W. M. Hoehn, *ibid.*, **69**, 1792 (1947).

(12) J. Weijlard, K. Pfister, 3rd, E. F. Swanezy, C. A. Robinson and M. Tisher, *ibid.*, **73**, 1216 (1951).

(13) S. Wawzonek and E. M. Smolin, *J. Org. Chem.*, **16**, 746 (1951).

(14) For other examples of this concept, see ref. 5a.

were prepared by alkylating an N-(2-pyridyl, 2-thiazolyl or 2-lepidyl)-1,2-diphenylethylamine (IA, B or C) with a  $\beta$ - or  $\gamma$ -substituted alkyl chloride in the presence of lithium amide<sup>15a</sup> (Method D). A similar reaction, wherein the alkyl halide was replaced by styrene oxide, yielded the tertiary aminoalcohol, II Ai (Method E).<sup>18</sup>

Although this procedure has yielded the homologous 2-pyridyl and 2-lepidyl benzohydrilamines,<sup>15</sup> the only product isolated from the reaction of 1,2-diphenylethyl chloride with either 2-aminopyridine or N,N-dimethyl-N'-(2-pyridyl)-ethylenediamine, in the presence of lithium amide, was *trans*-stilbene. This hydrocarbon has been obtained by others as the major product formed in the reaction between piperidine and 1,2-diphenylethyl bromide; the desired tertiary amine was isolated in only 10% yield.<sup>4</sup> The pyridine and thiazole secondary amines (IA, B) were obtained in good yield by treatment of either 2-benzylideneaminopyridine or 2-benzylideneaminothiazole with benzylmagnesium chloride<sup>11,15b</sup> (Method A). The thiazole amine (I B) was also prepared in excellent yield by total

(15) I. A. Kaye, I. C. Kogon and C. L. Parris, *THIS JOURNAL*, **74**, 403 (1952). (a) A mixture of all three reactants was refluxed and worked up according to the directions given under Method C in the experimental section of this publication. (b) In Method B directions are given for the preparation of several substituted 2-benzohydrilaminopyridines. (c) The steps in the synthesis of this compound are the same as those employed in the preparation of 2-benzohydrilaminothiazole.

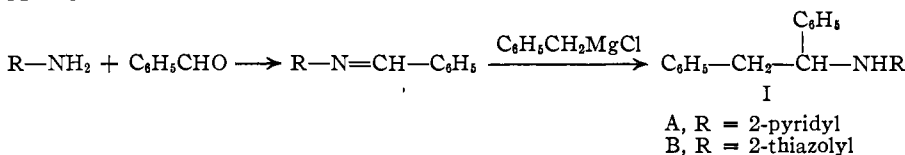
(16) The reaction of epoxides with the lithium derivatives of heterocyclic amines has been studied in this Laboratory. A report of this investigation is in preparation and will substantiate the structure assigned the aminoalcohol, II Ai.

TABLE I  
SECONDARY AND TERTIARY AMINES, C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>-CH(C<sub>6</sub>H<sub>5</sub>)-NRR'

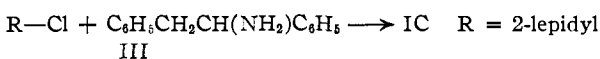
R <sup>a</sup>	R'	Pro- cedure	°C. B.P., Mm.	M.p., °C.	Yield, <sup>b</sup> %	Nitrogen analyses, % Formula Calcd. Found
C <sub>5</sub> H <sub>4</sub> N-	H	A	157-159	0.08	65-66 <sup>c</sup> 185-186.5 <sup>d</sup>	73 C <sub>15</sub> H <sub>16</sub> N <sub>2</sub> 10.21 9.90
C <sub>6</sub> H <sub>2</sub> NS-	H	A(C)	200-202	.60	103.5-104.5 <sup>e</sup>	56(84 <sup>f</sup> ) C <sub>19</sub> H <sub>16</sub> N <sub>2</sub> ·C <sub>6</sub> H <sub>5</sub> N <sub>3</sub> O <sub>7</sub> 13.91 13.61
C <sub>10</sub> H <sub>8</sub> N-	H	B			135-136 <sup>e</sup>	51 <sup>f</sup> C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> S 9.99 10.00
C <sub>6</sub> H <sub>4</sub> N-	CH <sub>2</sub> CH <sub>2</sub> N(CH <sub>3</sub> ) <sub>2</sub>	D	161-163	.05	168.5-169.5 <sup>e,g</sup>	97 C <sub>24</sub> H <sub>27</sub> N <sub>3</sub> 8.28 8.15
C <sub>6</sub> H <sub>4</sub> N-	CH <sub>2</sub> CH <sub>2</sub> N(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub>	D	174-177	.03	129-129.5 <sup>e,g</sup>	97 C <sub>28</sub> H <sub>27</sub> N <sub>3</sub> ·H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> 12.16 12.08
C <sub>6</sub> H <sub>4</sub> N-	(CH <sub>2</sub> ) <sub>2</sub> N(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub>	D	179-183	.07	<sup>h</sup>	97 C <sub>28</sub> H <sub>27</sub> N <sub>3</sub> ·H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> 9.65 9.45
C <sub>6</sub> H <sub>4</sub> N-	CH <sub>2</sub> CH <sub>2</sub> -NC <sub>4</sub> H <sub>8</sub> <sup>a</sup>	D	181-183	.05	183-184 dec. <sup>e,g</sup>	96 C <sub>23</sub> H <sub>23</sub> N <sub>3</sub> 11.25 10.93
C <sub>6</sub> H <sub>4</sub> N-	CH <sub>2</sub> CH <sub>2</sub> NC <sub>4</sub> H <sub>8</sub> O <sup>a</sup>	D	205-207	.11	96.5-97.5 <sup>c</sup> 176.5-177 <sup>e,g</sup>	98 C <sub>25</sub> H <sub>29</sub> N <sub>3</sub> ·H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> 9.06 9.05
C <sub>6</sub> H <sub>4</sub> N-	CH <sub>2</sub> CH <sub>2</sub> N(CH <sub>2</sub> C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub>	D			114-115 <sup>e</sup>	94 <sup>f</sup> C <sub>25</sub> H <sub>29</sub> N <sub>3</sub> ·H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> 10.84 10.63
C <sub>6</sub> H <sub>4</sub> N-	CH <sub>2</sub> CH <sub>2</sub> SCH <sub>3</sub>	D	184-185	.09	74-75 <sup>c</sup>	95 C <sub>22</sub> H <sub>24</sub> N <sub>2</sub> S 8.04 7.99
C <sub>6</sub> H <sub>4</sub> N-	CH <sub>2</sub> CH(OH)C <sub>6</sub> H <sub>5</sub>	E	200-202	.03	<sup>h</sup>	92 C <sub>27</sub> H <sub>23</sub> N <sub>2</sub> O 7.10 7.10
C <sub>6</sub> H <sub>2</sub> NS-	CH <sub>2</sub> CH <sub>2</sub> N(CH <sub>3</sub> ) <sub>2</sub>	D	173-176	.02	142-143 <sup>i</sup>	82 C <sub>21</sub> H <sub>23</sub> N <sub>2</sub> S 11.95 11.78
C <sub>10</sub> H <sub>8</sub> N-	CH <sub>2</sub> CH <sub>2</sub> N(CH <sub>3</sub> ) <sub>2</sub>	D	215-217	.04	171-172 <sup>j</sup>	93 C <sub>25</sub> H <sub>23</sub> N <sub>3</sub> 14.48 14.41
						C <sub>28</sub> H <sub>27</sub> N <sub>3</sub> 10.26 10.01
						C <sub>28</sub> H <sub>27</sub> N <sub>3</sub> ·2C <sub>6</sub> H <sub>5</sub> N <sub>3</sub> O <sub>7</sub> 14.53 14.04

<sup>a</sup> C<sub>5</sub>H<sub>4</sub>N- is 2-pyridyl, C<sub>6</sub>H<sub>2</sub>NS- is 2-thiazolyl, C<sub>10</sub>H<sub>8</sub>N- is 2-lepidyl, -NC<sub>4</sub>H<sub>8</sub> is N-pyrrolidyl and -NC<sub>4</sub>H<sub>8</sub>O is N-morpholino. <sup>b</sup> Yields are based on the weights of distilled products unless otherwise noted. <sup>c</sup> Recrystallized from hexane. <sup>d</sup> Picrate, prepared in ether and recrystallized twice from acetone. <sup>e</sup> Recrystallized from isopropyl alcohol. <sup>f</sup> Yield of crystallized product. <sup>g</sup> Oxalate. <sup>h</sup> Difficulty was experienced in preparing crystalline salts. <sup>i</sup> Picrate, recrystallized from acetone-isopropyl alcohol. <sup>j</sup> Picrate, recrystallized from dioxane.

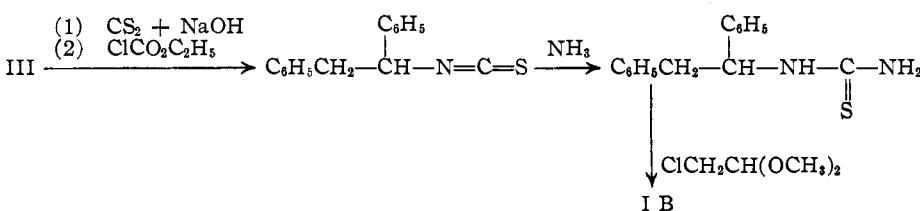
METHOD A



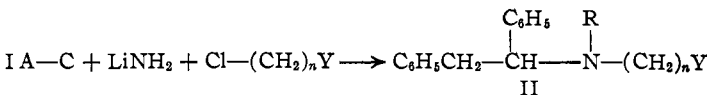
METHOD B



METHOD C

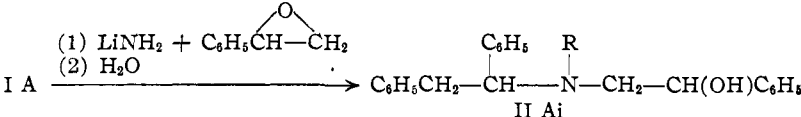


METHOD D



- (a) n = 2, Y = dimethylamino (A) R = 2-pyridyl
- (b) n = 2, Y = diethylamino (B) R = 2-thiazolyl
- (c) n = 3, Y = diethylamino (C) R = 2-lepidyl
- (d) n = 2, Y = N-pyrrolidyl
- (e) n = 2, Y = N-morpholino
- (f) n = 2, Y = dibenzylamino
- (g) n = 2, Y = methylmercapto

METHOD E



synthesis (Method C).<sup>15c</sup> The lepidyl secondary amine (I C) was conveniently prepared by heating 2-chlorolepidine with excess 1,2-diphenylethylamine (Method B).

Compounds I A, II Aa-c, II Ae and II Ba have

shown no evidence of ability to retard the growth of sarcoma 180.<sup>17a</sup> N,N-Dimethyl-N'-(1,2-diphenylethyl)-N'-(2-pyridyl)-ethylenediamine (II Aa) caused tremor-like convulsions in mice; it offered no protection against electric shock.<sup>17b</sup> Since this compound is structurally related to N,N-dimethyl-N'-benzyl-N'-(2-pyridyl)-ethylenediamine (Pyribenzamine), it was screened for antihistaminic activity. In preliminary *in vitro* studies on the isolated guinea pig ileum strip it showed only 0.08% of the activity of Pyribenzamine.<sup>17c</sup> Against acetylcholine it was 0.08% as active as atropine.<sup>17c</sup>

Experimental

All melting points are corrected; boiling points are not. Lithium amide, N,N-dimethyl-N'-(2-pyridyl)-ethylenediamine, β-dimethylaminoethyl chloride hydrochloride and β-diethylaminoethyl chloride hydrochloride were obtained from commercial sources. β-(N-Pyrrolidyl)-ethyl chloride hydrochloride,<sup>18</sup> β-(N-morpholino)-ethyl chloride hydrochloride,<sup>19</sup> β-chloroethyl methyl sulfide<sup>20</sup> and β-dibenzylamino-

(17) The authors wish to thank (a) Dr. C. Chester Stock of the Sloan-Kettering Institute for Cancer Research, (b) Dr. Irwin H. Slater of the School of Medicine and Dentistry at the University of Rochester and (c) Dr. Harold Blumberg and Mr.

Eric Meyer of Endo Products, Inc., for this information.  
(18) J. B. Wright, H. G. Kolloff and J. H. Hunter, *THIS JOURNAL*, **70**, 3098 (1948).  
(19) J. P. Mason and H. W. Block, *ibid.*, **62**, 1443 (1940).  
(20) E. Booth, V. C. F. Burnop and W. E. Jones, *J. Chem. Soc.*, 666 (1944).

ethyl chloride hydrochloride<sup>21</sup> were prepared by previously described methods.

**2-(1,2-Diphenylethyl)-aminopyridine (I A) and -thiazole (I B).** **Method A.**—These compounds were prepared by the method used in the preparation of 2-benzohydrylamino-pyridine.<sup>16b</sup> 2-Benzylideneaminopyridine was prepared by refluxing a cumene solution of benzaldehyde and 2-aminopyridine until water no longer was collected in a moisture trap. The warm solution of the Schiff base was then added to a solution of benzylmagnesium chloride. I B was prepared in the same fashion except that the cumene was replaced by benzene, since the Schiff base appeared to undergo decomposition in the higher-boiling solvent, and the final reflux period was shortened from 4 to 2 hours.

**2-(1,2-Diphenylethyl)-aminolepidine.** **Method B.**—A mixture of 30.2 g. (0.17 mole) of 2-chlorolepidine<sup>22</sup> and 67.1 g. (0.34 mole) of 1,2-diphenylethylamine<sup>23</sup> was heated at a bath temperature of 150° until an exothermic reaction occurred. The bath was removed and the reaction allowed to proceed without moderation until the temperature of the mixture dropped from a peak of 185° to about 100°. Three hundred ml. of dry benzene was added and the mixture refluxed for about 12 hours longer. The hydrochloride of 1,2-diphenylethylamine was separated by filtration and washed with acetone. (The salt melted at 256–258°. In ref. 5(a), the melting point is given as 254–256°.) The solvents were removed from the filtrate and the residue crystallized from hexane. The crude yield was 29.6 g. (52%), m.p. 123–130°. After one recrystallization from ethanol, and two more recrystallizations from isopropyl alcohol, the compound melted at 135–136°.

**1,2-Diphenylethyl Isothiocyanate.**—This compound was prepared by the procedure described in "Organic Syntheses" for the preparation of methyl isothiocyanate.<sup>24</sup> From 59.2 g. (0.3 mole) of 1,2-diphenylethylamine,<sup>23</sup> there was obtained 68.6 g. (96%) of product, collected at 120–121° (0.07 mm.). The analytical sample, prepared by redistillation of the product, gave an unsatisfactory analysis. Although the nitrogen composition was in excess of theory, the compound was used without further purification in the preparation of N-(1,2-diphenylethyl)-thiourea.

*Anal.* Calcd. for C<sub>15</sub>H<sub>13</sub>NS: N, 5.85. Found: N, 6.40.

**N-(1,2-Diphenylethyl)-thiourea.**—A solution of 62.2 g. (0.26 mole) of 1,2-diphenylethyl isothiocyanate in 150 ml. of acetone and 150 ml. of concentrated ammonia water was refluxed one-half hour. During this time a white crystalline solid precipitated. After cooling, the mixture was diluted with 500 ml. of cold water, chilled in an ice-bath for about one-half hour and then filtered. The product, after being washed with water and air-dried, weighed 63.7 g. (96%), m.p. 165–170°. A sample melted at 171–171.5° after 3 recrystallizations from isopropyl alcohol.

*Anal.* Calcd. for C<sub>15</sub>H<sub>13</sub>N<sub>2</sub>S: N, 10.94. Found: N, 10.98.

**2-(1,2-Diphenylethyl)-aminothiazole (I B).** **Method C.**—A mixture of 28.2 g. (0.11 mole) of N-(1,2-diphenylethyl)-

thiourea, 15.0 g. (0.12 mole) of dimethylchloroacetal and 100 ml. of water was heated on a steam-bath for 2.5 hours. Upon the addition of excess dilute sodium hydroxide to the cooled mixture, a brown gum precipitated. The product was extracted several times with ether and the combined ether extracts were dried over anhydrous potassium carbonate. After removal of the solvent, the residue was crystallized by rubbing with hexane. The tan solid weighed 25.8 g. (84%), m.p. 98–102°. After four recrystallizations from isopropyl alcohol, the white product melted at 103.5–104.5°. Mixed with product obtained by Method A of m.p. 103–104°, there was no depression in melting point.

**Tertiary Amines (II Aa-h, II Ba, II Ca).** **Method D.**—The procedure described in a previous publication was used without modification.<sup>15a</sup>

**N-(1,2-Diphenylethyl)-N-(2-pyridyl)-1-phenyl-2-aminoethanol.** **Method E.**—A mixture of 13.9 g. (0.05 mole) of 2-N-(1,2-diphenylethyl)-aminopyridine, 7.2 g. (0.06 mole) of styrene oxide, 1.5 g. (0.06 mole) of lithium amide (of 98% purity) and 100 ml. of dry benzene was refluxed for 24 hours. After cooling to room temperature, the mixture was shaken thoroughly with ca. 500 ml. of water. The aqueous layer was separated and extracted twice with benzene. The benzene extracts were combined with the original benzene layer, the solvent removed by distillation and the residual oil distilled *in vacuo*. There was obtained 18.1 g. (92%) of a dark yellow, very viscous oil which was collected at 200–202° (0.03 mm.).

**1,2-Diphenylethyl Chloride.**<sup>25</sup>—Three hundred and fifty grams (2.94 moles) of purified thionyl chloride was added dropwise, over the course of ca. 1 hour, to a solution of 464.1 g. (2.34 moles) of 1,2-diphenylethanol<sup>26</sup> in 950 ml. of ethylene dichloride. The temperature of the solution was maintained below 10° during the addition. After standing ca. 18 hours, the reaction-mixture was washed twice with water and then with dilute sodium carbonate solution until the washings were distinctly alkaline. The solvent was removed by distillation and the residue distilled *in vacuo*. The greenish-yellow oil weighed 426.4 g. (84%), b.p. 146–149° (5 mm.).

*Anal.* Calcd. for C<sub>14</sub>H<sub>13</sub>Cl: Cl, 16.36. Found: Cl, 15.98.

**Reaction of N,N-Dimethyl-N'-(2-pyridyl)-ethylenediamine and 2-Aminopyridine with 1,2-Diphenylethyl Chloride.**—The procedure outlined in Method E was followed. From 23.0 g. (0.14 mole) of N,N-dimethyl-N'-(2-pyridyl)-ethylenediamine, 32.5 g. (0.15 mole) of 1,2-diphenylethyl chloride and 3.9 g. (0.15 mole) of lithium amide (of 92% purity) in 150 ml. of dry benzene, there was obtained 20.2 g. of distillate, b.p. 124–125° (0.05 mm.), which rapidly solidified in the receiving flask. The solid, after washing with ethanol, melted at 124–125°. The melting point of *trans*-stilbene has been given as 123–124°.<sup>27</sup> Repeating this on the same scale, with 2-aminopyridine in place of N,N-dimethyl-N'-(2-pyridyl)-ethylenediamine, there was obtained 3.2 g. of the hydrocarbon.

**Acknowledgment.**—This investigation was supported by a research grant from the National Cancer Institute, of the National Institutes of Health, U. S. Public Health Service.

BROOKLYN 10, N. Y.

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(21) W. S. Gump and E. J. Nikawitz, *THIS JOURNAL*, **72**, 1309 (1950).

(22) I. A. Kaye, *ibid.*, **71**, 2322 (1949).

(23) N-(1,2-Diphenylethyl)-formamide, prepared by the method of V. J. Webers and W. F. Bruce, *ibid.*, **70**, 1422 (1948), using calcium chloride catalysis as suggested in a footnote of a publication by J. F. Bunnnett and J. L. Marks, *ibid.*, **71**, 1587 (1949), was hydrolyzed without purification to 1,2-diphenylethylamine by the method of F. F. Blicke and M. U. Tsao, *ibid.*, **68**, 905 (1946). The product, distilling at 148–149° (3 mm.), was collected in 85% over-all yield.

(24) M. L. Moore and F. S. Crossley in *Org. Syntheses*, **21**, 81 (1941).

(25) The *d,l*-1,2-diphenylethyl chloride has not been reported previously. P. A. Levine and L. A. Mikeska, *J. Biol. Chem.*, **65**, 507 (1925), prepared the individual optical antipodes.

(26) Prepared by the method of M. Tout and M. Guyard, *Bull. soc. chim. France*, 1086 (1947).

(27) R. L. Shriner and A. Berger in *Org. Syntheses*, **23**, 86 (1943).