Method C. **1-(2-Hydroxy-5-methoxybenzyl)-4-phenylpiperazine (VII).**—To an ice-cold mixture of 24.8 g. (0.2 mole) of 1-hydroxy-4-methoxybenzene and 32.4 g. (0.2 mole) of 1-phenylpiperazine in 90 ml. of ethanol and 50 ml. of water was added 20 ml. of 30% aqueous formaldehyde solution. After stirring for 48 hr. at room temperature, crystalline VII was filtered and recrystallized.

Method D. **1-(3,4,5-Trimethoxybenzyl)-4-phenylpiperazine Hydrochloride (X).**—A solution of 23.05 g. (0.1 mole) of 3,4,5-trimethoxybenzoyl chloride and 16.2 g. (0.1 mole) of 1-phenylpiperazine in 150 ml. of anhydrous chloroform was refluxed for 2 hr. and evaporated to dryness to give a solid which was recrystallized from a chloroform–toluene mixture (1:1) to give **1-(3,4,5-trimethoxybenzoyl)-4-phenylpiperazine hydrochloride** in 60% yield; m.p. 216°.

Anal. Calcd. for $C_{26}H_{25}ClN_2O_4$: C, 61.13; H, 6.41; Cl, 9.03. Found: C, 60.9; H, 6.4; Cl, 9.4.

The hydrochloride was converted quantitatively to the free base by alkalinization of an aqueous solution and recrystallization from isopropyl ether; m.p. 134–135°.

Anal. Calcd. for $C_{29}H_{24}N_2O_4$: C, 67.39; H, 6.79. Found: C, 67.7; H, 6.75.

This base was also prepared by mixing a solution of 4.6 g. (0.02 mole) of 3,4,5-trimethoxybenzoyl chloride in 10 ml. of anhydrous chloroform with a solution of 3.24 g. (0.02 mole) of 1-phenylpiperazine and of 1.6 g. of pyridine in 10 ml. of anhydrous chloroform. After standing for 5 days at room temperature and washing twice with 20 ml. of water, the chloroform was removed *in vacuo*. The crystalline residue was recrystallized from isopropyl ether to give the amide in 50% yield, m.p. and mixture m.p. with the above sample 134–136°.

A solution of 35.6 g. (0.1 mole) of the above amide in anhydrous ether was reduced with 0.1 mole of lithium aluminum hydride in anhydrous ether to give **1-(3,4,5-trimethoxybenzyl)-4-phenylpiperazine** in 75% yield, b.p. 180–185° (0.1 mm.).

Anal. Calcd. for $C_{29}H_{36}N_2O_3$: C, 70.15; H, 7.65. Found: C, 70.2; H, 7.65.

To a solution of 17.1 g. (0.05 mole) of this disubstituted piperazine in 50 ml. of anhydrous chloroform was added a solution of 0.11 mole of 2 *N* absolute ethanolic hydrogen chloride. The solvent was evaporated *in vacuo* to give impure 1-(3,4,5-trimethoxybenzyl)-4-phenyl piperazine dihydrochloride which was added to 200 ml. of water, boiled under reflux until completely dissolved, and filtered hot. On cooling, pure crystalline monohydrochloride salt (X) was deposited,[9] m.p. 270°. Recrystallization from absolute ethanol gave an analytical sample; sublimation was observed on a hot stage microscope at 218–220°.

Method E. **1-Benzyl-4-(4-pyridyl)piperazine (XIX).**—A solution of 88 g. (0.5 mole) of 1-benzylpiperazine, 28.4 g. (0.25 mole) of 4-chloropyridine,[10] and 200 ml. of anhydrous xylene was refluxed for 20 hr. After cooling, 1-benzylpiperazine hydrochloride was separated and the xylene was evaporated to dryness to give XIX which was recrystallized.

(9) Such a hydrolysis of dihydrochloride to monohydrochloride salts by boiling water was observed in some other *N,N'*-disubstituted piperazines when one of the substituents was a phenyl or a substituted phenyl group.

(10) J. P. Wibaut and F. W. Broekman, *Rec. trav. chim.*, **58**, 885 (1939).

Substituted 2,3-Dihydro-1,5-benzothiazepin-4(5H)-ones and 3,4-Dihydro-2-phenyl-(2H)-1,6-benzothiazocin-5(6H)-ones

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Received February 15, 1963

The synthesis of substituted 2,3-dihydro-1,5-benzothiazepin-4(5H)-ones and their alkylation is described. The preparation of 3,4-dihydro-2-phenyl-2H-1,6-benzothiazocin-5(6H)-one and 6-(2-dimethylaminoethyl)-3,4-dihydro-2-phenyl-2H-1,6-benzothiazocin-5(6H)-one hydrochloride is also reported. Three of these compounds were found to be highly effective in calming rats with lesions in the septal area of the brain.

In extension of our studies on substituted 2-phenyl-1,4-benzothiazin-3(4H)-ones,[1] we have prepared a number of related 2,3-dihydro-1,5-benzothiazepin-4(5H)-ones (Table II) and 3,4-dihydro-2-phenyl-2H-1,6-benzothiazocin-5(6H)-ones.

The intermediate 2,3-dihydro-1,5-benzothiazepin-4(5H)-ones (Table I) were obtained by heating 2-aminobenzenethiol (or 2-amino-4-chlorobenzenethiol) with the appropriate cinnamic, phenylcrotonic, or furanacrylic acid according to a procedure used for the preparation of 2,3-dihydro-2-methyl-1,5-benzothiazepin-4(5H)-one and the 2-phenyl analog.[2]

The compounds listed in Table II were obtained by addition of a slurry of the appropriate 1,5-benzothiazepin-4(5H)-one in toluene to a slurry of sodamide in toluene; the resulting solution was treated with the corresponding basically-substituted alkyl chloride and the mixture maintained usually at 60–65° for 3 hr. The yield in this alkylation reaction is dependent on the stability of the benzothiazepin-4(5H)-one to ring cleavage under the reaction conditions and the reactivity of the alkyl halide. The alkylation of 2,3-dihydro-2-phenyl-1,5-benzothiazepin-4(5H)-one with 2-dimethylaminoethyl chloride gave a 50% yield of purified product (**3**, Table II); whereas the reaction with the less reactive 3-dimethylaminopropyl chloride gave only a 9% yield of **6** and 65% of 2′-(3-dimethylaminopropylthio)cinnamanilide.[3] The formation of the latter product was not surprising since treatment of 2,3-dihydro-2-phenyl-1,5-benzothiazepin-4(5H)-one with 10% potassium hydroxide was reported to yield 2′-mercaptocinnamanilide.[2]

Because of the low reactivity of 2-(*N*-benzyl-*N*-methylamino)ethyl chloride under the above conditions, the corresponding bromide was used in the reaction with 2,3-dihydro-2-phenyl-1,5-benzothiazepin-4(5H)-one to give **5**.

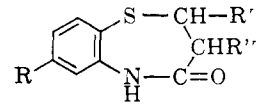
The homologous 8-membered ring system, 3,4-dihydro-2-phenyl-1,6-benzothiazocin-5(6H)-one, was prepared as shown on the following page.

(1) J. Krapcho, A. Szabo, and J. Williams, *J. Med. Chem.*, **6**, 214 (1963).

(2) W. H. Mills and J. B. Whitworth, *J. Chem. Soc.*, 2738 (1927).

(3) An alternate synthesis of 2′-(3-dimethylaminopropylthio)cinnamanilide and the biological activity of this compound has been reported by J. Krapcho, B. Rubin, A. M. Drungis, E. R. Spitzmiller, C. F. Turk, J. Williams, B. N. Craver, and J. Fried, *J. Med. Chem.*, **6**, 219 (1963).

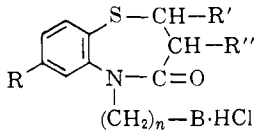
TABLE I

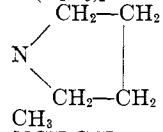


No.	R	R′	R″	M.p., °C.[a]	Yield, %	Formula	Analyses, % Nitrogen Calcd.	Nitrogen Found	Sulfur Calcd.	Sulfur Found
A	H	C_6H_5	CH_3	236–238	22	$C_{16}H_{15}NOS$	5.20	5.48	11.90	12.04
B	H	CH_3	C_6H_5	206–207	41	$C_{16}H_{15}NOS$	5.20	5.19	[b]	
C	H	4-(Cl)C_6H_4	H	204–205	18	$C_{15}H_{12}ClNOS$	4.83	5.09	[c]	
D	H	4-(CH_3)C_6H_4	H	200–202	23	$C_{16}H_{15}NOS$	5.20	5.23	11.90	11.61
E	H	2-(CH_3O)C_6H_4	H	220–222	31	$C_{16}H_{15}NO_2S$	4.91	4.91	11.24	11.08
F	H	4-(CH_3O)C_6H_4	H	120–122	47	$C_{16}H_{15}NO_2S$	4.91	5.26	11.24	10.99
G	H	3,4-(CH_3O)$_2C_6H_3$	H	160–162	23	$C_{17}H_{17}NO_3S$	4.44	4.63	10.16	9.94
H	H	2-Furyl	H	154–156	16	$C_{13}H_{11}NO_2S$	5.71	5.81	13.07	12.98
I	Cl	C_6H_5	H	232–233	35	$C_{15}H_{12}ClNOS$	4.83	4.84	[d]	

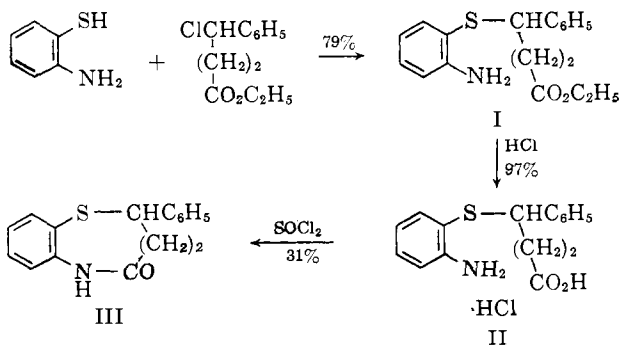
[a] Solvents for crystallization: A, absolute alcohol; B and G, acetonitrile; C, D, and E, dimethylformamide; F, 95% alcohol; H, isopropyl alcohol; I, butanone. [b] Calcd.: C, 71.33; H, 5.61. Found: C, 71.36; H, 5.36. [c] Calcd.: Cl, 12.26. Found: 12.15. [d] Calcd.: Cl, 12.23. Found: 12.48.

TABLE II



No.	R	R′	R″	n	B	M.p., °C.[a]	Yield, %[b]	Formula	Analysis, % Chlorine Calcd.	Chlorine Found	Nitrogen Calcd.	Nitrogen Found
1	H	CH_3	H	2	$N(C_2H_5)_2$	175–176	36	$C_{16}H_{25}ClN_2OS$	10.78	10.73	8.52	8.47
2	H	C_6H_5	H	2	$N(CH_3)_2$	222–224	50	$C_{19}H_{23}ClN_2OS$	9.77	9.55	7.72	7.64
3	H	C_6H_5	H	2	$N(C_2H_5)_2$	178–180	74	$C_{21}H_{27}ClN_2OS$[c]	9.07	8.94	7.17	7.18
4	H	C_6H_5	H	2		193–195	12	$C_{21}H_{25}ClN_2OS$	9.12	8.89	7.20	7.22
5	H	C_6H_5	H	2	$NCH_2C_6H_5$	168–170	46	$C_{25}H_{27}ClN_2OS$	8.07	7.88	6.38	6.11
6	H	C_6H_5	H	3	$N(CH_3)_2$	127–129	9	$C_{20}H_{25}ClN_2OS$	9.41	9.33	7.43	7.55
7	H	C_6H_5	CH_3	2	$N(CH_3)_2$	231–233	74	$C_{20}H_{25}ClN_2OS$	9.41	9.33	7.43	7.66
8	H	C_6H_5	CH_3	3	$N(CH_3)_2$	169–171	73	$C_{21}H_{27}ClN_2OS\cdot H_2O$[d]	8.67	8.68	6.85	7.02
9	H	CH_3	C_6H_5	2	$N(CH_3)_2$	239–240	39	$C_{20}H_{25}ClN_2OS$[e]	9.41	9.19	7.43	7.48
10	H	CH_3	C_6H_5	3	$N(CH_3)_2$	214–216	62	$C_{21}H_{27}ClN_2OS$	9.06	8.96	7.16	7.08
11[f]	H	4-(Cl)C_6H_4	H	2	$N(CH_3)_2$	205–206	13	$C_{19}H_{22}Cl_2N_2OS$	17.84	17.95	7.05	7.03
12	H	4-(CH_3)C_6H_4	H	2	$N(CH_3)_2$	220–222	25	$C_{20}H_{25}ClN_2OS$	9.41	9.24	7.43	7.11
13	H	2-(CH_3O)C_6H_4	H	2	$N(CH_3)_2$	118–120	10	$C_{20}H_{25}ClN_2O_2S\cdot H_2O$[g]	8.63	8.64	6.82	6.48
14	H	4-(CH_3O)C_6H_4	H	2	$N(CH_3)_2$	210–212	19	$C_{20}H_{25}ClN_2O_2S$	9.02	8.78	7.13	6.95
15	H	3,4-(CH_3O)$_2C_6H_3$	H	2	$N(C_2H_5)_2$	179–181	38	$C_{23}H_{31}ClN_2O_3S$	7.86	7.84	6.21	6.36
16	H	2-Furyl	H	2	$N(CH_3)_2$	196–198	16	$C_{17}H_{21}ClN_2O_2S$	10.05	9.99	7.94	7.82
17	Cl	C_6H_5	H	2	$N(CH_3)_2$	180–181	44	$C_{19}H_{22}Cl_2N_2OS$[h]	17.84	17.62	7.05	6.83

[a] These salts were crystallized from acetonitrile except 1 and 10 (ethanol–ether); 4 (ethanol); and 5, 6, 13, and 16 (butanone). [b] These yields are the result of single experiments, most of the reactions having been carried out at 60–65° for 3 hr. The exceptions to these reaction conditions are in the preparation of compounds 1 (110°, 2 hr.), 5 (room temperature, 15 hr.), 7 (90–95°, 4 hr.), 10 (110°, 3 hr.), and 9 (85–90°, 2 hr.). [c] The free base melted at 78–81° (crystallized from hexane). *Anal.* Calcd. for $C_{21}H_{26}N_2OS$: N, 7.90. Found: N, 8.06. [d] Calcd.: C, 61.67; H, 7.15. Found: C, 61.78; H, 7.26. [e] The free base melted at 95–97° (crystallized from hexane). *Anal.* Calcd. for $C_{20}H_{24}N_2OS$: N, 5.20. Found: N, 5.36. [f] This material and compound C of Table I were prepared by J. Williams. [g] Calcd.: C, 58.45; H, 6.62. Found: C, 58.46; H. 6.97. [h] The free base melted at 125–126° (crystallized from cyclohexane); *Anal.* Calcd. for $C_{19}H_{21}ClN_2OS$: Cl, 9.82; N, 7.76. Found: Cl, 10.05; N, 7.68.



The alkylation of this material with 2-dimethylaminoethyl chloride at 80° for 4 hr. gave a 53% yield of 6-(2-dimethylaminoethyl)-3,4-dihydro-2-phenyl-2H-1,6-benzothiazocin-5(6H)-one hydrochloride (IV).

The ability of compounds to calm rats with lesions in the septal area of the brain is a useful method for evaluation of their psychosedative properties; for example, chlorodiazepoxide shows high activity in this test.[4] Compound IV and those listed in Table II were evaluated in this test procedure; **2, 3,** and **6** showed a high order of activity. Compound **3** was found to be about twice as potent as chlorodiazepoxide when ad-

(4) L. O. Randall, W. Schallek, G. A. Heise, E. F. Keith, and R. E. Bagdon, *J. Pharmacol. Exptl. Therap.*, **129**, 163 (1960).

ministered by the i.p. route.[5] This material was also tested for its ability to inhibit the tremors produced by 1,4-dipyrrolidino-2-butyne and was about 1/3 as active as 4-(2-diethylaminoethyl)-2-phenyl-1,4-benzothiazin-3(4H)-one hydrochloride.[1] The latter compound showed no effect in the septal rat at 15 times the effective dose of **3.**

Experimental

Melting points are corrected. Infrared spectra were recorded on a Perkin–Elmer Model 21 spectrometer.

2,3-Dihydro-2-phenyl-1,5-benzothiazepin-4(5H)-one.—A mixture of 250 g. (2.0 moles) of 2-aminobenzenethiol and 300 g. (2.0 moles) of cinnamic acid was heated at 160–180° for 1 hr. Water (36 ml.) distilled from the mixture during this period. After cooling to 90°, the oily product was poured onto 1.1 l. of warm acetonitrile to give 304 g. of crystalline material; m.p. 156–162°. Recrystallization from 4 l. of acetonitrile yielded 180 g. (35%) of colorless product; m.p. 176–177° (reported[2] m.p. 177°); λ_{max}^{Nujol} 3.15, 5.95 μ. The yield in this reaction is higher when carried out on a smaller scale.

5-(2-Dimethylaminoethyl)-2,3-dihydro-2-phenyl-1,5-benzothiazepin-4(5H)-one Hydrochloride.—A suspension of 7.8 g. (0.2 mole) of sodamide in 500 ml. of toluene was treated with a slurry of 50.8 g. (0.2 mole) of 2,3-dihydro-2-phenyl-1,5-benzothiazepin-4(5H)-one[2] in 500 ml. of toluene. The mixture became a clear solution after stirring for 5 min. at room temperature. This solution was treated with 25.0 g. (0.23 mole) of 2-dimethylaminoethyl chloride in 110 ml. of toluene and then heated to 55° during the course of 1 hr. The mixture became turbid and was then maintained at 60–65° for 3 hr., cooled to 20°, and treated with 150 ml. of water. The organic phase was washed with 100 ml. of water and then added to 300 ml. of cold *N* hydrochloric acid. The unreacted starting material which separated from the mixture was removed by filtration (11.5 g., m.p. 174–175°). The aqueous phase was cooled, treated with 60 ml. of 30% sodium hydroxide solution, and the mixture extracted 3 times with 600-ml. portions of ether. After drying over magnesium sulfate, the ether was evaporated to give 43 g. of residue. Part of this material (40.6 g.) was dissolved in 500 ml. of anhydrous ether, the insoluble material (1.5 g.) was discarded, and solvent evaporated to yield 39 g. of pale yellow oil. A solution of 36.5 g. of this base in 50 ml. of ethanol was treated with an equivalent quantity of hydrogen chloride in 20 ml. of ethanol. Dilution of this solution to 500 ml. with ether gave 39.5 g. (80%, based on reacted starting material) of colorless solid; m.p. 195–205°. After crystallization from 600 ml. of acetonitrile, the colorless product weighed 24.3 g. (50%); m.p. 222–224°; λ_{max}^{Nujol} 3.8, 4.32, 5.98 μ.

By substitution of 28.0 g. of 3-dimethylaminopropyl chloride for the 2-dimethylaminoethyl chloride in the above preparation, there was obtained 65 g. of pale yellow base. Trituration of this material with 1.4 l. of hot hexane (in 3 portions) followed by cooling yielded 44.0 g. (65%) of **2′-(3-dimethylaminopropylthio) cinnamanilide** as a colorless solid, m.p. 82–83°; λ_{max}^{Nujol} 3.02, 6.0, 6.12, 6.53 μ.

Anal. Calcd. for $C_{29}H_{34}N_2OS$: C, 70.54; H, 7.11; N, 8.23; S, 9.39. Found: C, 70.39; H, 7.44; N, 8.35; S, 9.45.

The hexane filtrate was concentrated and the residue fractionated to give 11.4 g. (17%) of **5-(3-dimethylaminopropyl)-2,3-dihydro-2-phenyl-1,5-benzothiazepin-4(5H)-one** as a pale yellow distillate, b.p. 210–212° (0.1 mm.).

Anal. Calcd. for $C_{20}H_{24}N_2OS$: N, 8.23. Found: N, 8.02.

The hydrochloride salt of this material (**6**, Table II), after crystallization from 25 ml. of butanone, weighed 6.7 g. (9%); m.p. 127–129°; λ_{max}^{Nujol} 3.85, 4.05, 6.0 μ.

2-(N-Benzyl-N-methylamino)ethyl Bromide Hydrobromide.—This material was obtained in 51% yield by the interaction of N-benzyl-N-methylaminoethanol with 48% hydrobromic acid,[6] m.p. 145–147°. Crystallization from butanone did not change the melting point.

Anal. Calcd. for $C_{10}H_{15}Br_2N$: Br, 51.72; N, 4.53. Found: Br, 51.86; N, 4.68.

The free base of this material was used in the preparation of **5.**

Ethyl 4-Chloro-4-phenylbutyrate.—A solution of 38.0 g. (0.24 mole) of γ-phenyl-γ-butyrolactone[7] in 50 ml. of benzene was reacted with thionyl chloride and subsequently treated with ethanolic hydrogen chloride according to the method described for the preparation of ethyl 4-chloro-4-methylheptoate[8] to give 43 g. (81%) of product; b.p. 106–108° (0.3 mm.).

Anal. Calcd. for $C_{12}H_{15}ClO_2$: Cl, 15.64. Found: Cl, 15.46.

Ethyl 4-(2-Aminophenylthio)-4-phenylbutyrate (I).—A solution of 20.0 g. (0.088 mole) of ethyl 4-chloro-4-phenylbutyrate in 115 ml. of ethanol was added to a solution containing 11.0 g. (0.088 mole) of 2-aminobenzenethiol, 3.6 g. (0.09 mole) of sodium hydroxide, and 235 ml. of 90% ethanol. The mixture was refluxed for 2 hr. and the major portion of the solvent evaporated. The residue was cooled and extracted three times with 200-ml. portions of ether. The combined ether extract was washed with 50 ml. of water and dried over magnesium sulfate. After evaporation of the solvent, the residue was distilled to give 22 g. (79%) of product; b.p. 172–179° (0.1–0.2 mm.).

Anal. Calcd. for $C_{18}H_{21}NO_2S$: N, 4.44; S, 10.16. Found: N, 4.59; S, 10.77.

4-(2-Aminophenylthio)-4-phenylbutyric Acid Hydrochloride (II).—A mixture of 10.0 g. (0.32 mole) of I and 50 ml. of 20% hydrochloric acid was heated on a steam bath for 30 min. After cooling, the crystalline product was filtered and air-dried on filter paper; yield 10.0 g. (97%); m.p. 162–164°.

Anal. Calcd. for $C_{16}H_{18}ClNO_2S$: neut. equiv., 324. Found: neut. equiv., 320.

The free amino acid was obtained by shaking a mixture of 10 g. of the hydrochloride, 50 ml. of water, and 200 ml. of ether. The ether phase was washed 3 times with 20-ml. portions of water, dried over magnesium sulfate, and then evaporated to give 7.5 g. (85%) of pale yellow solid; m.p. 101–103°.

Anal. Calcd. for $C_{16}H_{17}NO_2S$: neut. equiv., 287; S, 11.16. Found: neut. equiv., 290; S, 11.37.

3,4-Dihydro-2-phenyl-2H-1,6-benzothiazocin-5(6H)-one (III).—A mixture of 8.5 g. (0.026 mole) of II, 135 ml. of chloroform, and 26 ml. of thionyl chloride was refluxed for 45 min. The solvent and excess thionyl chloride were removed under reduced pressure and the residue triturated with a mixture of 250 ml. of ether and 100 ml. of water. The solid was filtered, triturated with water, and dried; yield 2.9 g., m.p. 215°. Crystallization of this material from 130 ml. of acetonitrile gave 2.3 g. (31%) of colorless product, m.p. 228–230; λ_{max}^{Nujol} 3.15, 3.28, 5.98 μ. *Anal.* Calcd. for $C_{16}H_{15}NOS$: N, 5.20; S, 11.90. Found: N, 5.20; S, 11.65.

Cyclization of 7.0 g. of the free amino acid by heating at 195–200° for 2 hr. gave 200 mg. of III, m.p. 225–227° (after crystallization from acetonitrile). The melting point showed no depression when this material was mixed with III made *via* thionyl chloride; the infrared spectra were also identical.

6-(2-Dimethylaminoethyl)-3,4-dihydro-2-phenyl-2H-1,6-benzothiazocin-5(6H)-one Hydrochloride (IV).—A mixture of 4.0 g. (0.015 mole) of III, 0.6 g. (0.015 mole) of sodamide, and 2.5 g. (0.023 mole) of 2-dimethylaminoethyl chloride in 130 ml. of toluene was treated according to the above procedure for the corresponding benzothiazepin-4-one except that the reaction mixture was maintained at 80° for 4 hr. A small quantity (0.5 g.) of starting material was recovered from the reaction. The crude product weighed 3.7 g. (76%), m.p. 216–218°. Crystallization from 25 ml. of acetonitrile gave 2.6 g. (53%) of colorless product, m.p. 217–219°; λ_{max}^{Nujol} 3.45, 3.90, 4.08, 6.04 μ.

Anal. Calcd. for $C_{20}H_{25}ClN_2O$: Cl, 9.41; N, 7.43. Found: Cl, 9.26; N, 7.42.

Acknowledgments.—We are indebted to Dr. J. Bernstein for his interest and encouragement during this investigation, to Miss Barbara Keeler for interpretation of the infrared spectra, and to Mr. J. Alicino and his associates for the analyses reported herein.

(5) The authors are indebted to Dr. L. J. Brannick for these data. The detailed pharmacology on these compounds will be reported elsewhere.

(6) According to the general procedure described by F. Cortese, "Organic Syntheses," Coll. Vol. II, John Wiley and Sons, New York, N. Y., 1943, p. 99.

(7) W. L. Meyer and W. R. Vaughan, *J. Org. Chem.*, **22**, 1558 (1957).

(8) J. Cason, C. E. Adams, L. L. Bennett, and V. D. Register, *J. Am. Chem. Soc.*, **66**, 1764 (1944).