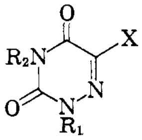
Table I



No.	R_1	R_2	X	Mp, °C	Yield, %	Formula	Calcd, % C	H	N	X	Found, % C	H	N	X
I	H	CH_3	Br	190–191	85	$C_4H_4BrN_3O_2$	23.32	1.96	20.40	38.79	23.24	2.04	20.70	28.70
II	CH_3CO	CH_3	Br	111.5–113[a]	87	$C_6H_6BrN_3O_3$	29.05	2.44	16.94	32.22	29.17	2.65	17.07	32.02
III	CF_3CO	CH_3	Br	135–136[b]	83	$C_6H_3BrF_3N_3O_3$	23.86	1.00	13.91		23.73	1.26	14.15	
IV	CH_3	CH_3	Br	105–106	88	$C_5H_6BrN_3O_2$	27.29	2.75	19.10	36.32	27.19	3.00	18.80	36.24
V	CH_3	CH_3	F	130–131[c]	34[d]	$C_5H_6FN_3O_2$	37.74	3.80	26.41	11.94	37.82	3.84	26.14	11.93
VI	$(C_6H_5)_2CH$	$(C_6H_5)_2CH$	Br	183–185[e]	60[f]	$C_{29}H_{22}BrN_3O_2$	66.42	4.23	8.01	15.24	66.65	4.35	7.74	15.05

[a] Crystallized from C_6H_6–CCl_4. [b] Recrystallizing and remelting at 183°. [c] Recrystallizing and remelting at 138°. Purified by sublimation. [d] Crude product. [e] Crystallized from absolute ethanol. [f] Crude product, mp 176–178°.

dry dioxane was treated with 4.6 g (25 mmoles) of diphenyldiazomethane[8] in 20 ml of dry dioxane and stirred overnight at 90°.[9] After evaporation of this mixture to dryness, the crude product (VI) was obtained.

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(8) J. H. Ford, "Organic Syntheses," Coll. Vol. III, John Wiley and Sons, Inc., New York, N. Y., 1955, p 35.

(9) Methodology of M. Prystas and F. Šorm, *Collection Czech. Chem. Commun.*, **27**, 1578 (1962).

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cis-1-(3-Dimethylaminopropyl)-2,3-pentamethylenetetrahydroquinoline

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The useful antidepressant clinical activity of imipramine suggested the synthesis of the title compound as a variation on the basic heterocyclic system. However, the only activity of note uncovered was the antagonism of ethanol depression and death in mice.

Experimental Section[1]

2,3-Pentamethylenecinchoninic acid:[2] mp 302–303° (lit.[2] mp 291–292°); 95% yield; $\lambda_{\max}^{\text{Nujol}}$ 2.95, 3.75, 4.30, 4.97, 6.29 μ.

2,3-Pentamethylenequinoline:[2] mp 91–92.5° (lit.[2] mp 93.5°); 93% yield; $\lambda_{\max}^{\text{Nujol}}$ 6.25, 6.43, 6.72 μ.

***cis*-Tetrahydro-2,3-pentamethylenequinoline.**[3]—2,3-Pentamethylenequinoline was reduced with tin and HCl or catalytically (PtO_2, H_2) to give, in either case, an oil which was shown by tlc to consist of starting material and a new component. The oil was treated with benzoyl chloride under Schotten–Baumann conditions to give *cis*-1-benzoyl-2,3-pentamethylenetetrahydroquinoline, mp 142–146° (33% yield based on the quinoline). A recrystallized sample melted at 145–146.5° (lit. mp 145–146°,[3a] 146.5°[3b]); $\lambda_{\max}^{CHCl_3}$ 6.16, 6.37, 6.72, 7.19, 7.37 μ. The benzamide was hydrolyzed by refluxing it in a mixture of KOH, ethanol, and water for 45 hr. Work-up afforded a 94% yield of a clear oil which showed one spot on tlc, and was used as such; $\lambda_{\max}^{CHCl_3}$ 2.92, 6.30, 6.38, 6.78, 6.94 μ. A portion of the base was converted to the hydrochloride, mp 141–144° (lit.[3] mp 143–145°).

***cis*-1-(3-Dimethylaminopropyl)-2,3-pentamethylenetetrahydroquinoline Hydrochloride.**—To a suspension of 1.75 g (0.076 mole) of sodamide in 175 ml of liquid NH_3 was added 12.5 g (0.062 mole) of *cis*-tetrahydro-2,3-pentamethylenequinoline in 25 ml of ether. After allowing this mixture to stir for 1 hr, there was added a solution of 3-dimethylaminopropyl chloride (liberated from 23.5 g, 0.15 mole, of the corresponding hydrochloride) in 10 ml of ether over a 15-min period. The resultant mixture was stirred for 1.5 hr and then allowed to stand overnight, whereby NH_3 evaporated. Water was then added, the layers were separated, and the aqueous phase was extracted several times with ether. The combined organic portions were dried ($MgSO_4$), filtered, and concentrated under reduced pressure. The residual oil was distilled, and the main fraction [bp 155–160° (0.2 mm)] amounted to 9.0 g (51%). This yellow oil showed one component (not the starting material) on tlc; $\lambda_{\max}^{CHCl_3}$ 6.28, 6.70, 6.90 μ. The oil was converted to the hydrochloride to give 7.1 g of crude solid. Recrystallization from ethanol–ether gave 4.3 g, mp 155–157° dec, and 0.8 g, mp 153.5–156° dec. An analytical sample, prepared from this latter material, melted at 155.5–157.5° dec; $\lambda_{\max}^{KBr}$ 3.79, 4.10, 6.26, 6.68, 7.34, 7.82 μ; $\lambda_{\max}^{EtOH}$ 258, 311 mμ ($\epsilon \times 10^{-3}$ 17.6, 3.35).

Anal. Calcd for $C_{19}H_{31}ClN_2$: C, 70.67; H, 9.68; N, 8.68. Found: C, 70.84; H, 9.66; N, 8.83.

Acknowledgment.—We wish to express our appreciation to the S. E. Massengill Co., Bristol, Tenn., for instituting and supporting this work.

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(1) Melting points were determined on a Thomas–Hoover capillary apparatus and are corrected.

(2) W. Borsche, *Ann.*, **377**, 122 (1910).

(3) (a) T. Masamune, *J. Am. Chem. Soc.*, **79**, 4418 (1957); (b) S. G. P. Plant and R. J. Rosser, *J. Chem. Soc.*, 1840 (1930).

Preparation of Substituted Diaminopropanols

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In a search for compounds that might be useful hypotensive agents a series of N-substituted diamino-2-propanols have been prepared[1] (Tables I and II).

Experimental Section

Analysis of Reactions and Compounds by Means of Thin Layer Chromatography (Tlc).—Aluminum oxide was used as an adsorbent.[2] The spotted plates were developed by means of an acetone–hexane mixture (2:5 v/v), and the plates were exposed to HNO_3 fumes.

Synthesis of Substituted Diaminopropanols.—Substituted 1-anilino-3-chloropropanols were prepared from aromatic primary amines and epichlorohydrin by procedures previously reported.[3] These were usually isolated as picrates and regenerated by means of saturated LiOH. The halo compound was immediately

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(1) *Cf.* B. J. Ludwig, W. A. West, and D. W. Farnsworth, *J. Am. Chem. Soc.*, **76**, 2893 (1954).

(2) Camag, Arthur H. Thomas Co., Philadelphia, Pa.