(10.0 g) was dissolved in a mixture of 60 ml of AcOH and 10 ml of ethanedithiol, 1 ml of BF_3-etherate was added, and the solution was allowed to stand at room temperature for 8 min. It was poured onto a mixture of ice and H_2O giving an oily mass which solidified after about 1 hr. The solid was recovered by filtration and washed thoroughly (H_2O, $NaHCO_3$) and the filter cake was pressed dry overnight by means of a rubber dam. The crude product (13.43 g) was dissolved in hot acetone, treated with Norit, and crystallized from Me_2CO-petroleum ether to yield 8.56 g of material, mp 170–172°. Recrystallization from Me_2CO–H_2O gave an analytical sample, mp 173.2–175.0°. *Anal.* ($C_{21}H_{38}O_2S_2$) C, H.

11β-Hydroxy-11α-methyl-5β-pregnan-20-one.—11β-Hydroxy-11α-methyl-5β-pregnane-3,20-dione 3-ethylene mercaptal suspended in 400 ml of 75% EtOH was heated at reflux overnight with about 40 g of Raney Ni. After cooling, the nickel was filtered and the filtrate was evaporated to about 50 ml, whereupon 2.34 g of material crystallized. Recrystallization from petroleum ether afforded an analytical sample, mp 121.2–123.0°. *Anal.* ($C_{22}H_{38}O_2$) C, H.

Acknowledgment.—We are indebted to Mr. Arlen J. Taylor for technical assistance, to Drs. J. L. Johnson and W. A. Struck and associates for the various physical measurements and the microanalytical determinations, and to Dr. P. H. Seay for the pharmacologic tests.

New Compounds

Some Derivatives of 1-Aminoadamantane

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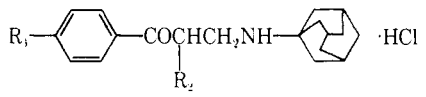
Recent interest in the pharmacology of 1-aminoadamantane and especially its application as an antiviral agent[1-3] prompted us to prepare some 1-(3,3-diarylpropylamino)adamantanes (Tables I–III) for testing for antiviral and also for CNS activity.

TABLE I

1-(3-ARYL-3-OXOPROPYLAMINO)ADAMANTANE HYDROCHLORIDES

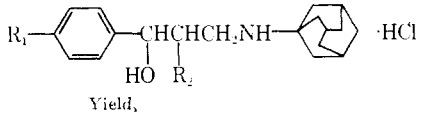


R_1	R_2	Yield, %	Mp, °C	Solvent[a]	Formula[b]
H	H	62.5	215	A	$C_{19}H_{26}ClNO$
H	CH_3	52.4	188–190	B	$C_{20}H_{28}ClNO$
Cl	H	56.5	225–226	C	$C_{19}H_{25}Cl_2NO$
NO_2	H	82.4	233–235	B	$C_{19}H_{25}ClN_2O_3$
CH_3O	H	41.4	205–207	B	$C_{20}H_{28}ClNO_2$
CH_3	H	66.0	213	C	$C_{20}H_{28}ClNO$

[a] Solvents: A, MeOH–Et_2O; B, MeOH–EtOAc; C, $CHCl_3$–EtOAc. [b] All compounds were analyzed for C, H, Cl, N.

TABLE II

1-(3-ARYL-3-HYDROXYPROPYLAMINO)ADAMANTANE HYDROCHLORIDES

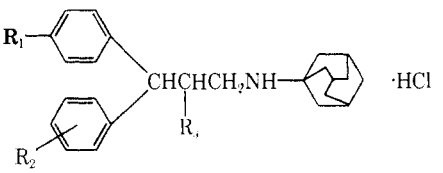


R_1	R_2	Yield, %	Mp, °C	Solvent[a]	Formula[b]
H	H	90.0	299–301	A	$C_{19}H_{28}ClNO$
H	CH_3	93.5	306–307	B	$C_{20}H_{30}ClNO$
Cl	H	87.5	316–318	C	$C_{19}H_{27}Cl_2NO$
NO_2	H	86.6	309–310	C	$C_{19}H_{27}ClN_2O_3$
CH_3O	H	72.0	86–87	D	$C_{20}H_{29}NO_2$[c]

[a] Solvents: A, MeOH–Et_2O; B, MeOH; C, MeOH–EtOAc; D, aqueous MeOH. [b] All compounds were analyzed for C, H, Cl, N except where noted otherwise. [c] This product was isolated and analyzed as a free base (for C, H, N only).

TABLE III

1-(3,3-DIARYLPROPYLAMINO)ADAMANTANE HYDROCHLORIDES



R_1	R_2	R_3	Yield, %	Mp, °C[a]	Formula[b]
H	H	H	76.2	302–304	$C_{25}H_{32}ClN$
H	H	CH_3	51.0	272	$C_{26}H_{34}ClN$
Cl	H	H	67.7	304–306	$C_{25}H_{31}Cl_2N$
Cl	Cl	H	63.0	322	$C_{25}H_{30}Cl_3N$
CH_3O	CH_3O	H	23.0	305	$C_{27}H_{36}ClNO_2$
CH_3	CH_3	H	73.9	315	$C_{27}H_{36}ClN$
H	OH	H	65.0	319–320	$C_{25}H_{32}ClNO$

[a] All products were recrystallized from MeOH–EtOAc. [b] All compounds were analyzed for C, H, Cl, N.

Experimental Section[4]

General Procedures. Mannich Condensation.—A mixture of aminoadamantane hydrochloride (prepared from adamantane[5] by the method of Stetter, *et al.*[6]) (1.0 mol), the appropriate alkyl aryl ketone (1.1 mol), 37% aqueous HCHO (1.5 mol), and concentrated HCl (1 ml) was heated under reflux for 4 hr. After standing at room temperature for 3 more hr, the reaction mixture was diluted (Me_2CO–Et_2O) and the crystalline reaction product was filtered off and purified by crystallization.

Reduction.—The hydrochloride of the Mannich ketone (1.0 mol) was dissolved in six times its weight of 80% aqueous MeOH, some aqueous NaOH was then added in order to liberate the free base, then $NaBH_4$ (0.5 mol) and the reaction mixture was allowed to stand at room temperature for 3 hr. After dilution (H_2O) the reaction product was extracted into $CHCl_3$, dried (Na_2SO_4), and filtered and dry HCl was introduced into the

(1) G. C. Schild and R. N. P. Sutton, *Brit. J. Exptl. Pathol.*, **46** (3), 263 (1965).

(2) R. R. Grunert, J. W. McGahen, and W. L. Davies, *Virology*, **26**, 262 (1965).

(3) E. D. Stanley, R. E. Muhlonn, L. W. Akers, and G. G. Jackson, *Ann. N.Y. Acad. Sci.*, **130**, 44 (1965).

(4) Where analyses are indicated only by symbols of the elements or functions, analytical results obtained for those elements or functions were within ±0.4% of the theoretical values. Microanalyses were performed by Dr. C. Daessle, Organic Microanalysis, Montreal. The ir spectra were recorded on a Perkin-Elmer 237 instrument. The melting points were determined in capillaries on a Mel-Temp apparatus and are uncorrected.

(5) P. von R. Schleyer, M. M. Donaldson, R. D. Nicholas, and C. Cupas, *Org. Syn.*, **42**, 8 (1962).

(6) H. Stetter, M. Schwarz, and A. Hirschhorn, *Chem. Ber.*, **92**, 1629 (1959); H. Stetter, J. Mayer, M. Schwarz, and K. Wulff, *ibid.*, **93**, 226 (1960).

filtrate. The solvent was removed *in vacuo* and the residue was purified by crystallization.

$NaBH_4$ reduction of 1-[3-(*p*-tolyl)-3-oxopropylamino]adamantane and subsequent treatment with HCl yielded the corresponding alcohol hydrochloride, mp 262–263°, which did not give the correct elemental analysis even after repeated recrystallizations and whose ir spectrum, however, left no doubt about its identity [ν_{max} (Nujol) 3375 (OH), 2700–2450 (NH_2^+), and 1590 cm^{-1} (aromatic)]. Condensation of this intermediate with toluene under Friedel–Crafts conditions produced readily the expected 3,3-ditolyl derivative.

Friedel–Crafts Reaction.—To a suspension of the amino alcohol hydrochloride (1.0 mol) in about ten times its weight of the appropriate aromatic hydrocarbon, anhydrous $AlCl_3$ (1.5 mol) was added in small portions and the reaction mixture was heated to 80–90°, where it was kept for 30 min. After cooling to room temperature it was poured into a mixture of equal amounts of ice, H_2O, and concentrated HCl. The product, which separated in crystalline form, was filtered off and purified by crystallization.

Acknowledgment.—Adamantane and 1-aminoadamantane were prepared by Dr. M. C. Bankiewicz.

6H-Indeno[2,1-*g*]quinolines

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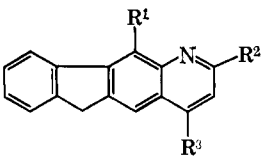
Interest in the antitumour activity of ellipticine has prompted studies on isomeric systems.[1] Linear indenoquinolines are simple analogs of the corresponding pyridocarbazoles and we report here the synthesis for evaluation of the hitherto unknown 6H-indeno[2,1-*g*]quinoline (I, $R^1 = R^2 = R^3 = H$) and some methyl homologs by the Skraup and Doebner procedures.



I

Experimental Section

Melting points were measured using an Electrothermal electrically heated block and are uncorrected. Uv spectra were measured in EtOH using Unicam SP500 and SP800B spectrophotometers.

6H-Indeno[2,1-*g*]quinoline (I, $R^1 = R^2 = R^3 = H$).—A Skraup reaction on 3-fluorenamine[2] with glycerol, I_2, and polyphosphoric acid at 180°[3] gave the parent indenoquinoline,[4] mp 142–143° (C_6H_6–petroleum ether (bp 60–80°)). *Anal.* ($C_{16}H_{11}N$) C, H. The **picrate** had mp 254–255° dec (from 2-methoxyethanol). *Anal.* ($C_{22}H_{14}N_4O_7$) C, H.

11-Methyl-6H-indeno[2,1-*g*]quinoline (I, R^1 = Me; $R^2 = R^3$ = H), prepared similarly from 4-methyl-3-fluorenamine,[2] formed needles, mp 120–121° (from EtOH). *Anal.* ($C_{17}H_{13}N$) C, H. The **picrate** formed needles, mp 237° dec (from 2-methoxyethanol). *Anal.* ($C_{23}H_{16}N_4O_7$) C, H, N.

2-Methyl-6H-indeno[2,1-*g*]quinoline (I, R^2 = Me; $R^1 = R^3$ = H).—3-Fluorenamine was refluxed with pyruvic acid in EtOH and the precipitated acid (I, R^1 = H, R^2 = Me, R^3 = CO_2H), mp *ca.* 350°, was decarboxylated by heating with soda lime. The distillate gave I[5] as needles, mp 147–148° from EtOH. *Anal.* ($C_{17}H_{13}N$) C, H.

(1) A. N. Fujiwara, E. M. Acton, and L. Goodman, *J. Med. Chem.*, **10**, 126 (1967).

(2) J. Davey, B. R. T. Keene, and G. C. Mannering, *J. Chem. Soc., C*, 120 (1967).

(3) Other conditions (*e.g.*, the sulfomix procedure) offered no advantage.

(4) Cyclization at C-2 rather than C-4 is to be expected; the nature of the product is confirmed by the close similarity of its uv spectrum [λ_{max} 213, 262, 311, 327, and 342 mμ (log 4.62, 4.68, 4.00, 4.12, and 4.26)] to that of the product of the following reaction, which can only be 11-methyl-6H-indeno[2,1-*g*]quinoline [λ_{max} 214, 266, 313, 325, and 341 (log 4.60, 4.71, 4.08, 4.11, and 4.11)].

(5) Uv spectrum closely similar to that of the 11-methyl isomer.

Substituted Quinazolone Hydrazides as Possible Antituberculous Agents

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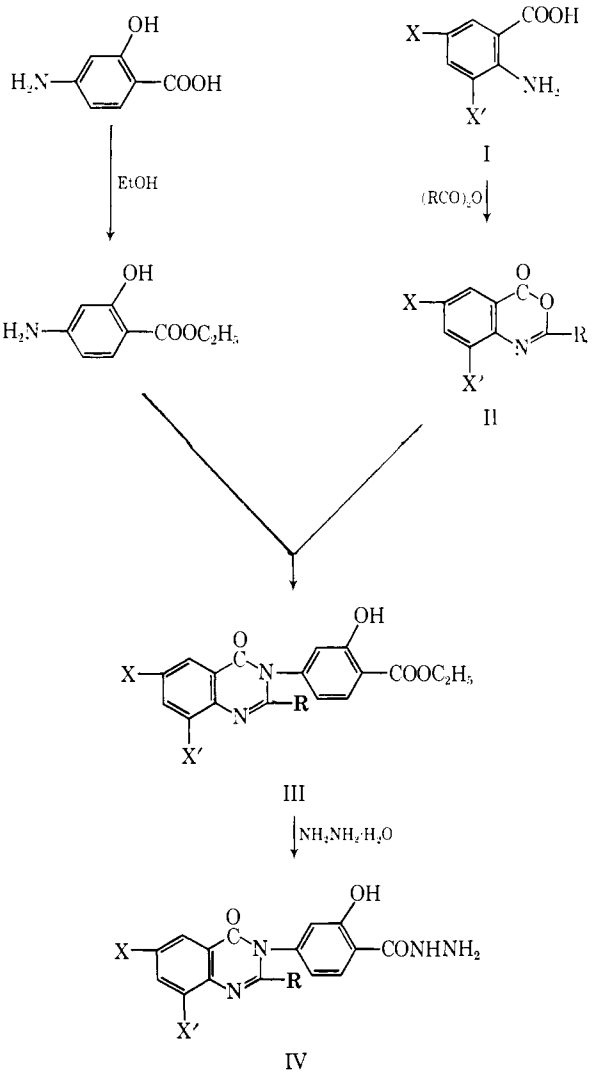
The therapeutic use of isonicotinic acid hydrazide (isoniazid) and *p*-aminosalicylic acid (PAS) as antituberculous agents is well documented. A search for newer antituberculous compounds led us to synthesize some substituted quinazolone hydrazides having structural similarity to PAS. Similar quinazolone hydrazides have also been shown to inhibit rat liver mitochondrial monoamine oxidase.[1,2] Substituted quinazolone hydrazides were synthesized by the route outlined in Scheme I.

SCHEME I



(1) S. S. Parmar and R. C. Arora, *Can. J. Chem.*, **44**, 2100 (1966).

(2) S. S. Parmar and R. C. Arora, *J. Med. Chem.*, **10**, 1182 (1967).