6,8-Dichloro-4-epoxyethyl-2,3-trimethylenequinoline (7).—A soln of 1 g (0.026 mole) of $NaBH_4$ in 10 ml of H_2O and 7 ml of 2 *N* NaOH, was added dropwise over 10 min to a stirred suspension of 4.65 g (0.013 mole) of nearly pure **6** (above) in 50 ml of MeOH. Stirring for an addl 1.5 hr, cooling for 15 min, filtering, and washing with MeOH gave 3.42 g (94.5%) of **7** (mp 134–139°); recrystd from Et_2O–hexane, mp 144–145°; ir (cm^{-1}) 2960, 2980, 3100, none for C=O; nmr ($CDCl_3$), 8.02 (1 H, d), 7.71 (1 H, d), 4.26 (1 H, m), 3.10 (5 H, m), 2.17 (2 H, quintuplet). *Anal.* ($C_{14}H_{11}Cl_2NO$) C, H, N.

5,7-Dichloro-2,3-dihydro-1*H*-cyclopenta[*b*]quinoline-9-(α-di-*n*-butylaminomethyl)methanol·HCl (1).—A suspension of 3.6 g of **7** in 12 ml of Bu_2NH was stirred for 4.5 hr at 105–110°, monitoring disappearance of **7** (4 hr) by tlc (silica gel G, 1:1 Et_2O–hexane). After evapn *in vacuo* of Bu_2NH (60°) the oil (5.1 g), dissolved in 150 ml of Et_2O, was treated with increments of Et_2O·HCl, each sufficient to give 0.2–0.4 g of **1** (each fraction being washed with Et_2O). Fractions 1–4 contd decreasing amts of Bu_2NH·HCl; and 5–8 were largely **1** (2.65 g). Repeated recrystn from EtOH–Et_2O gave 0.5 g, light tan, mp 160–162° dec; ir (cm^{-1}) 3440, 3220 (OH), 2960, 2940, 2880 (CH), 2670, 2620, 2530 ($\overset{+}{N}H$). *Anal.* ($C_{22}H_{30}Cl_2N_2O$·HCl) C, H, N, Cl.

Incidental and Preliminary Experiments. Attempts to add 2-PyLi and MeLi to the 2,3-trimethylenecinchoninic acids were unsuccessful, presumably because of steric interference of the 3-CH_2 group and/or the activity of the 2-CH_2 hydrogens (*cf.* ref 12).

2,3-Trimethylenecinchoninic acid·HCl (11), pptd from Et_2O, mp 252–255° dec, was treated with PCl_5 (steam bath for 30 min, addn of C_6H_6, and reflux for 2 hr), giving a ppt presumed to be the **acid chloride·HCl (12)** (sublimed, 8%, mp 245° dec).

2,3-Trimethylenecinchoninamide (13) was prepd from **12** by treatment with H_2O–NH_3; crystd from EtOH, mp 276–277°; ir (cm^{-1}) 3330 (s), 3140 (s) (NH_2), 1688 (C=O). *Anal.* ($C_{13}H_{14}N_2O$) C, H.

4-Bromoacetyl-2,3-trimethylenequinoline·HBr (14).—CH_2N_2–Et_2O with 3 g of **12** (overnight) gave orange cubes of diazo ketone. Treatment with 10 ml of 48% HBr–H_2O gave **14**; crystd from EtOH; 2.1 g (70%); mp 208° dec; ir (cm^{-1}) 1730 (C=O), 2500 ($\overset{+}{N}H$). *Anal.* ($C_{14}H_{13}Br_2NO$) N.

Derivatives of 2,3-trimethylene-4-quinolones were made by the action of the appropriate aniline on ethyl cyclopentanone-2-carboxylate, cyclizing at 250°, and crystn from EtOH:[3b,13] **15,** (a) 6,8-Cl_2, 26%, mp 305–307° (b) cyclization by refluxing Ph_2O, recrystd, mp 314–315° (lit.[3b] 313°) [*Anal.* ($C_{12}H_9Cl_2NO$) C, H, N]; **16,** 6,8-Me_2, 60%, mp 326–327° [*Anal.* ($C_{14}H_{15}NO$) N]; **17,** 6-Me, 39%, mp 319–322° [*Anal.* ($C_{13}H_{13}NO$) C, H]; **18,** 8-OMe, 26%, mp 212–213° [*Anal.* ($C_{13}H_{13}NO_2$) C, H, N]; **19,** 8-Cl, 21%, mp 269–270° [*Anal.* ($C_{12}H_{10}ClNO$) C, H, N]; **20,** 8-F, 15%, mp 292–293° [*Anal.* ($C_{12}H_{10}FNO$) C, H, N].

4-Bromo-2,3-trimethylenequinolines were prepd by treating the quinolone[13] with $POBr_3$ at 120°; crystd from EtOH: **21** (parent compd), 50%, mp 72–73° [*Anal.* ($C_{12}H_{10}BrN$), C, H, N]; **22,** 6,8-Me_2, from **16,** 69%, mp 124–125° [*Anal.* ($C_{14}H_{14}BrN$) C, H].

4,6,8-Trichloro-2,3-trimethylenequinoline (23) was prepd by refluxing $POCl_3$ on **15,** crystd from EtOH, 80%, mp 160–162°. *Anal.* ($C_{12}H_8Cl_3N$) C, H, N.

Attempted preparation of 4-lithio-2,3-trimethylenequinolines from **21** and **22** by BuLi and addns to 2-pyridaldehyde were unsuccessful, presumably because of the activities of the 2-CH_2 groups.[12]

(12) P. G. Campbell and P. C. Teague, *J. Amer. Chem. Soc.*, **76**, 1371 (1954).

(13) D. K. Blount, W. H. Perkin, Jr., and S. G. P. Plant, *J. Chem. Soc.*, 1975 (1929).

N,*N*^{1}-α,ω-Alkylenebis(nitroacetamides)

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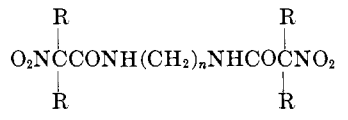
Some bis(nitroacetamides) with the general structure **1** were required for screening as antispermatogenic agents. The amides were readily prepared by heating the appropriate amine with the desired nitro ester without solvent and recrystallizing the resulting solid from a suitable solvent.

The compds prepared are listed in Table I. While

TABLE I



1

	R	*n*	Yield, %	Mp, °C	Rxt solv	Formula[a]
1	H	6	34.1	143–144	CH_3CN	$C_{10}H_{18}N_4O_6$
2	H	8	50.3	147–148	95% EtOH	$C_{12}H_{22}N_4O_6$
3	CH_3	2	12.8	183–185	CH_3CN	$C_{10}H_{18}N_4O_6$
4	CH_3	3	21.7	105–108	C_6H_6–*n*-C_6H_{14}	$C_{11}H_{20}N_4O_6$
5	CH_3	4	14.7	207–208	CH_3CN	$C_{12}H_{22}N_4O_6$
6	CH_3	6	30.6	168–170	CH_3CN	$C_{14}H_{26}N_4O_6$
7	CH_3	8	23.0	138–141	CH_3CN	$C_{16}H_{30}N_4O_6$

[a] All compds were anal. for C, H, N.

no antispermatogenic activity was found in this series anthelmintic activity was discovered. For example, **1** (R = H; *n* = 6) when administered orally to Swiss mice naturally infected with *Aspicularis tetraptera* (pinworm) cleared 100% of the mice (5/5 per dose level) at 100 mg/kg per day for 4 days and **1** (R = H; *n* = 8) cleared 100% of the mice (5/5 per dose level) at 200 mg/kg per day for 4 days; also, **1** (R = CH_3; *n* = 8) cleared 80% of the mice (4/5 per dose level) infected with the tapeworm *Hymenolepis nana* at 400 mg/kg per day for 4 days.

Experimental Section[1]

N,*N*^{1}-**Hexamethylenebis(nitroacetamide).**—Ethyl nitroacetate (11.2 g, 0.0855 mole) was added to hexamethylenediamine (9.94 g, 0.855 mole). The mixt became hot and liquefied, after which a white solid pptd. The mixt was heated for 3 hr on a steam bath. It slowly turned to a thick orange liquid. The mixt was acidified with alcoholic HCl and poured into H_2O. The white solid was collected and recrystd from MeCN, mp 147–148° dec.

The other compds were prepd similarly except that in the case of the compds with no free H α to NO_2, 1 equiv of diamine was treated with 2 equiv of nitro ester and the alcoholic HCl treatment was unnecessary.

(1) Melting points were measured in open capillary tubes in a bath and are corrected.

Tricyclic Heterocycles Derived From 4-Oxo-4,5,6,7-tetrahydrothianaphthenes[1]

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Recently we described the synthesis of a variety of tricyclic heterocycles from 4-oxo-4,5,6,7-tetrahydroin-

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