2-Ethynyl-2,3,3a,4,5,9b-hexahydro-7-methoxy-3a-methyl-1H-benz[*e*]inden-2-ol (19).—The procedure for preparing **18** was used. Starting with the *trans* isomer of **12** (8.0 g) a yield of 7.8 g (87%) of **19** was obtained, bp 134–137° (0.05 mm). *Anal.* ($C_{17}H_{20}O_2$) C, H.

2,3,3a,4,5,9b-Hexahydro-7-methoxy-2-(2-methyl-1,3-dioxolan-2-yl)-1H-benz[*e*]inden-2-ol (20).—The method of Nieuwland[22] was used to convert **18** into **20**. A solution of redistilled BF_3 etherate (2 ml) and HgO (0.5 g) in 15 ml of dry ethylene glycol was added to a solution of **18** (14.2 g) in 90 ml of dry ethylene glycol cooled to −10°. After the addition was complete, the mixture was allowed to warm to room temperature over a period of several hours, stirred overnight, and hydrolyzed. The crude product was recrystallized from C_6H_6–C_6H_{12} to give **20** (11.5 g, 64%), mp 106–108°. Tlc analysis indicated that only one isomer was present. *Anal.* ($C_{18}H_{24}O_4$) C, H.

2,3,3a,4,5,9b-Hexahydro-7-methoxy-3a-methyl-2-(2-methyl-1,3-dioxolan-2-yl)-1H-benz[*e*]inden-2-ol (21).—The procedure used to prepare **20** from **18** was followed. Starting with **19** (7.9 g) a yield of 8.1 g (91%) was obtained, mp 99–100°. Tlc analysis indicated that only a single isomer was present. *Anal.* ($C_{19}H_{26}O_4$) C, H.

2β-Acetyl-1,2,3,3aβ,4,5,6,8,9,9bβ-decahydro-2α-hydroxy-7H-benz[*e*]inden-7-one (22).—Compound **20** was converted into **22** using a procedure outlined previously[2] for similar compounds. Starting with **20** (6.0 g) a yield of 3.1 g (64%) of **22** was obtained, boiling range 149–152° (0.05 mm). *Anal.* ($C_{17}H_{20}O_3$) C, H.

2α-Acetyl-1,2,3,3a,4,5,6,8,9,9bα-decahydro-2β-hydroxy-3β-methyl-7H-benz[*e*]inden-7-one (23).—The procedure used to prepare **22** was used. Starting with **21** (6.0 g), a yield of 3.5 g (73%) of the product was obtained, after the initial product was purified by column chromatography using silica gel H as adsorbent, followed by redistillation, boiling range 148–150° (0.05 mm). *Anal.* ($C_{16}H_{22}O_3$) C, H.

(22) J. A. Nieuwland, R. R. Vogt, W. L. Foohey, *J. Amer. Chem. Soc.*, **52**, 1018 (1930).

Organic Disulfides and Related Substances. XXVIII. Analogs of *o*-(2-Protoaminoethyldithio)benzoate As Antiradiation Drugs[1a-c]

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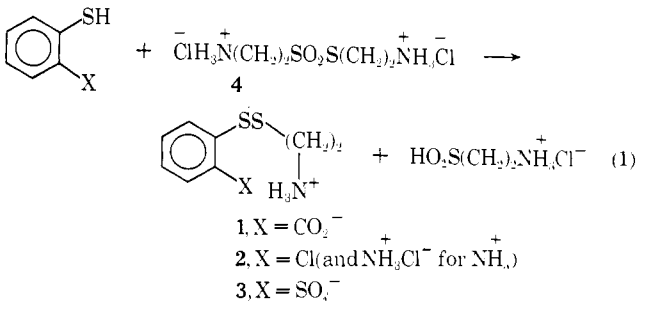
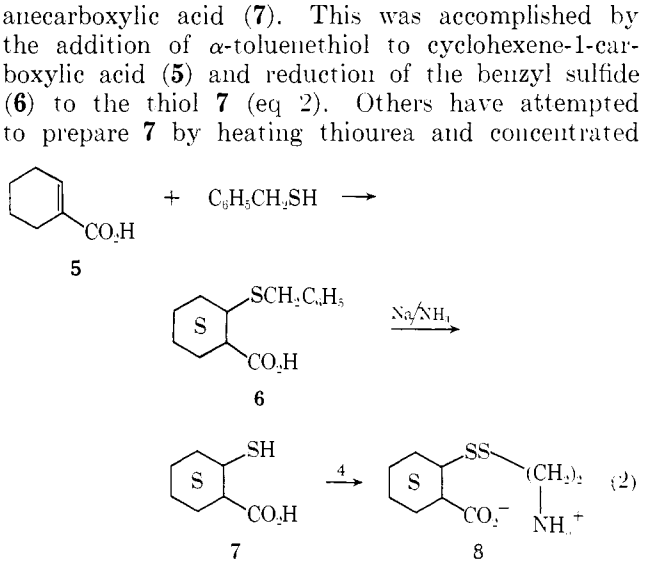
o-(2-Protoaminoethyldithio)benzoate (**1**)[2] has shown promise as an antiradiation drug,[3a] and two analogous compounds, *o*-(2-aminoethyldithio)chlorobenzene·HCl (**2**) and *o*-(2-protoaminoethyldithio)benzenesulfonate (**3**), also have shown activity.[3b] Although several other derivatives, isomers, and analogs have been inactive,[3] the saturated analogs of **1–3** were desired in order to establish whether the aromatic or the aliphatic system would provide the better basis for further extensions. Disulfides **1–3** were prepared by thioalkylation of the appropriate thiol with 2-aminoethyl 2-aminoethanethiolsulfonate·2HCl (**4**), as shown by eq 1,[3] and the same approach seemed feasible for the saturated compounds.

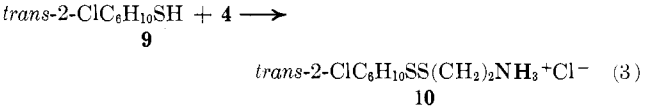


1, X = CO_2^-
2, X = Cl (and NH_3Cl^- for NH_3^+)
3, X = SO_3^-

In order to prepare the cyclohexane analog of **1**, it was necessary to synthesize 2-mercapto-1-cyclohexanecarboxylic acid (**7**). This was accomplished by the addition of α-toluenethiol to cyclohexene-1-carboxylic acid (**5**) and reduction of the benzyl sulfide (**6**) to the thiol **7** (eq 2). Others have attempted to prepare **7** by heating thiourea and concentrated HBr with hexahydrosalicylic acid and then treating with NaOH. However, they were unable to obtain a pure product.[4] Thioalkylation of **7** with **4** gave 2-(2′-protoaminoethyldithio)-1-cyclohexanecarboxylate (**8**). Although the reaction of α-toluenethiol with **5** would seem more likely to give at the outset *trans* addition, and therefore **6** as the *cis* product, the presence of excess hot piperidine in turn would seem likely to have afforded ample opportunity for epimerization to the presumably more stable *trans* isomer of **6**. In any event, tlc of **7** in two systems showed only a single spot, and prolonged treatment with base failed to effect any apparent change. Only single spots also were seen for the mercury (II) mercaptide of **7** and for disulfide **8**. Thus it would seem that a single isomer of **6** resulted, probably the *trans*.



Thioalkylation of the known *trans*-2-chlorocyclohexanethiol (**9**) gave *trans*-2-aminoethyl 2-chlorocyclohexyl disulfide·HCl (**10**), the reduced analog of **2** (eq 3).



Attempts to prepare the saturated analog of **3** were thwarted by a series of unsuccessful attempts to prepare the requisite thiol, 2-mercaptocyclohexanesulfonic acid.

(1) (a) Paper XXVII: L. Field and R. B. Barbee, *J. Org. Chem.*, **34**, 1792 (1969). (b) This investigation was supported by the U. S. Army Medical Research and Development Command, Department of the Army, under Research Contracts No. DA-49-193-MD-2030 and DADA17-69-C-9128. Taken from part of the forthcoming Ph.D. dissertation of P. M. G., Vanderbilt University. (c) Reported in part at the Southeastern Regional Meeting of the American Chemical Society, Tallahassee, Fla., Dec 1968; Abstracts, p 98.

(2) Nomenclature suggested by Dr. F. Y. Wiselogle. See F. G. Bordwell, M. L. Peterson, and C. S. Rondestvedt, Jr., *J. Amer. Chem. Soc.*, **76**, 3945 (1954). Previously **1** was named *o*-(2-aminoethyldithio)benzoic acid;[3] although the dipolar ionic structure of **1** is merely inferred from its behavior, rather than rigorously proved, the "proto" nomenclature seems to be a justifiable simplication.

(3) (a) R. R. Crenshaw and L. Field, *J. Org. Chem.*, **30**, 175 (1965); (b) L. Field and H. K. Kim, *J. Med. Chem.*, **9**, 397 (1966).

(4) J. F. Burke, and M. W. Whitehouse, *Biochem. Pharmacol.*, **14**, 1039 (1965).

In the preparation of **9**, concentrated HCl was used to open the episulfide ring of cyclohexene sulfide, but a similar approach using H_2SO_3 gave intractable polymeric material. All attempts to effect the ring opening with $NaHSO_3$ also gave polymer.

Thioalkylation of the known α-mercaptocinnamic acid (**11**) with **4** gave α-(2-protoaminoethyldithio)cinnamate (**12**; eq 4). Disulfide **12** was of interest as

$$C_6H_5CH{=}C(SH)CO_2H + \mathbf{4} \longrightarrow \underset{\mathbf{11}}{} \qquad (4)$$

$$C_6H_5CH{=}C(SSCH_2CH_2NH_3^+)CO_2^- \qquad \underset{\mathbf{12}}{}$$

an antiradiation drug since its resemblance to **1** is clear and, particularly, since the method used to synthesize **11** lends itself to the introduction of electron-donating or -withdrawing substituents into the aromatic moiety. Thiol **11** is thought to have a *trans* relationship of the aryl and carboxyl groups,[5] and **12** therefore probably is the *trans* isomer also.

The antiradiation activities of disulfides **1**, **2**, **3**, **8**, **10**, and **12** are listed in Table I. These were determined

TABLE I
ANTIRADIATION ACTIVITIES OF 2-PROTOAMINOETHYLDITHIO COMPOUNDS[a]

Compd	LD_{50}, mg/kg[b]	Drug dose, mg/kg	Vehicle[c]	pH of solution	Survival, 30 days, %[d]
1[e,g]	325	225	CMCTw	7.4[h]	60 (85)
		112	CMCTw	7.4[h]	0
2[e,i]	200	50	NaCl	7.2[h]	7
		25	NaCl	7.2[h]	0
3[f,i]	>1000	600	CMCTw	6.7	67 (87)
		300	CMCTw	6.7	33
8[f,i]	>600	250	CMCTw	6.1	0
		125	CMCTw	6.1	0
10[e,j]	300	50	NaCl	4.5	0
		25	NaCl	4.5	0
12[f,i]	350	100	CMCTw	6.1	0
		50	CMCTw	6.1	0

[a] For details of testing not given in other footnotes, see ref 6. [b] Approximate LD_{50} for the compound in mice; *cf.* ref 6. [c] CMCTw = suspension or solution in 0.3% carboxymethyl-cellulose plus 0.1% Tween 80; NaCl = suspension or solution in physiological saline. [d] Fifteen mice with 10 controls, to permit comparison; the figure in parentheses is the *best* survival seen in *any* tests. [e] Drug administered 15 min prior to irradiation. [f] ^{60}Co γ irradiation. [g] In a check at low dosage (6 mice, 6 controls), the survival was 17% (X-irradiation, 20 min after injection of 141 mg/kg of **1** in polyethylene glycol at pH 6). [h] Adjusted pH value. [i] Drug administered 30 min prior to irradiation. [j] X-Ray irradiation.

as described previously,[6] through the kindness of Drs. D. P. Jacobus, T. R. Sweeney, and E. A. Steck, and of Miss Marie Grenan, of the Walter Reed Army Institute of Research, Washington, D. C. The syntheses of **1**–**3** were reported elsewhere,[3] but the complete testing results have not been published previously; we feel that the results of Table I supersede an earlier report[3b] that **3** afforded "good" protection at 50 mg/kg or less.

Since the cyclohexyl compounds **8** and **10** were inactive, in contrast to the benzenoid compounds **1**–**3**, the benzenoid system evidently is much the more promising. The disappointing inactivity of the interesting prototype **12** shows that the benzenoid system also is more attractive than the cinnamyl modification having the 1,1-arrangement of **12**.

(5) M. Nishio and T. Ito, *Agr. Biol. Chem.* (Tokyo), **29**, 1119 (1965).

(6) L. Fiehl, B. J. Sweetman, and M. Bellas, *J. Med. Chem.*, **12**, 624 (1969).

Experimental Section[7]

Starting Materials. Thiolsulfonate **4**[8] and thiols **9**[9] and **11**[10] were prepared according to reported procedures. All other materials were used as purchased.

Cyclohexene-1-carboxylic Acid (5). In a modification of a procedure of Marvel,[11] a mixture of cyclohexanecarboxylic acid (192 g, 1.5 mol), PCl_3 (4 ml), and Br_2 (240 g, 1.5 mol) was heated (60–80°, 17 hr). More Br_2 (10 ml) then was added, and heating was continued until no Br_2 color was evident (7 hr more). Recrystallization from petroleum ether gave α-bromocyclohexanecarboxylic acid (253 g, 81%; mp 54–57°, lit.[12] mp 61°).

α-Bromocyclohexanecarboxylic acid (170 g, 0.82 mol) was heated (4.5 hr, 65°) with KOH (120 g, 2.14 mol) in MeOH (675 ml). The reaction mixture was cooled and diluted with H_2O (300 ml), acidified (pH 1, concentrated HCl), and extracted with Et_2O. The extract was dried ($MgSO_4$). Evaporation of the Et_2O gave **5** as a yellow oil (77 g, 72%); sharp ir bands at 1648 (>C=C<) and 1690 (CO_2H) cm^{-1}. This **5** was used without purification.

2-Benzylthio-1-cyclohexanecarboxylic Acid (6). In a method based on one of Schulz and du Vigneaud,[13] α-toluenethiol (64 g, 0.52 mol) and **5** (63 g, 0.50 mol) were heated under reflux (12 hr) in piperidine (130 ml). The yellow solution was cooled, acidified (pH 1, concentrated HCl), and extracted with Et_2O (200 ml). The ethereal solution was washed, first with H_2O (100 ml) and then with 10% $NaHCO_3$ (500 ml) and with 5% KOH (500 ml). The basic solutions were acidified and extracted (Et_2O). The Et_2O solutions were washed, dried, and concentrated to give **6** in both cases (29 g and 53 g, respectively; 64% yield). Since **6** was an oil, it was characterized as the *p*-toluidide (**13**); recrystallized from DMF–H_2O and from EtOH–H_2O, **13** had constant mp 120–121°; ir (KBr) 3320, 2950, 1665, 1610, 1525, 1450, 820, and 700 cm^{-1}. *Anal.* ($C_{21}H_{25}NOS$) C, H, S.

2-Mercapto-1-cyclohexanecarboxylic Acid (7). The benzyl sulfide **6** (24.68 g, 98.6 mmol) was dissolved in 600 ml of redistilled liquid NH_3. Enough dry Na (8.61 g, 374 g-atoms) was added to maintain a blue solution for 0.5 hr while the solution was stirred mechanically with a glass paddle. NH_4Cl (*ca.* 4 g) then was added to dispel the blue color, and the NH_3 was swept away with a stream of dry N_2. The white powdery residue was washed with 100 ml of Et_2O and then was dissolved in 50 ml of H_2O. The solution was acidified to pH 1 with 10% HCl, and extracted three times with 50-ml portions of Et_2O. The extracts were dried (Na_2SO_4) and evaporated to give a yellow oil (11.95 g, 76%). Distillation gave **7** as a colorless oil (bp 160–165° (12 mm), $n^{25}D$ 1.5098), with appropriate ir absorption. The **7** solidified to a waxy solid (mp 36–39°) on standing. The (Brinkmann MN-Polygram cellulose powder, developed with 5-PrOH–H_2O–NH_4OH, 8:1:1, *ca.* 25°) showed only a single spot under uv light (R_f 0.68). Likewise, on Brinkmann MN-Polygram MN silica gel ($CHCl_3$–EtOH, 9:1, *ca.* 25°) only one spot was observed (R_f 0.78); nmr (CCl_4, δ 0.70–2.50 (m, 9), 2.50–3.83 (m, 2), 12.13 (s, 1), 2 protons exchangeable with D_2O). *Anal.* ($C_8H_{14}O_2S$) C, H, S.

To determine whether **7** could be epimerized, a sample (0.08 g, 0.5 mmol) was stirred (120 hr at *ca.* 25° with KOH (0.06 g, 1.1

(7) Melting points, determined in capillary tubes using a Hershberg-type stirred-liquid apparatus, are corrected. Boiling points are uncorrected. Elemental analyses were by Galbraith Microanalytical Laboratories, Knoxville, Tenn. Ir spectra were obtained using a Beckman Model IR10 spectrophotometer with films of liquids and KBr pellets of solids; bands reported were at least of medium intensity. Nmr spectra were obtained using a Varian Model A-60 spectrometer (Me$_4$Si); purchase of this instrument was assisted by Departmental NSF Grant GP-1683. Solvents were evaporated under reduced pressure using a rotary evaporator. Except where otherwise stated, tlc spots were developed by exposure to I_2 vapor in a sealed container. Where analyses are indicated only by symbols of the elements, analytical results for those elements were within ±0.4% of the theoretical values.

(8) L. Field, T. C. Owen, R. R. Crenshaw, and A. W. Bryan, *J. Amer. Chem. Soc.*, **83**, 4414 (1961).

(9) (a) C. C. A. Culvenor, W. Davies, and N. S. Heath, *J. Chem. Soc.*, 282 (1949); (b) E. E. van Tamelen, *J. Amer. Chem. Soc.*, **73**, 3444 (1951), reported that *trans*-**9** is obtained by the procedure of ref 9a.

(10) E. Campaigne and R. E. Cline, *J. Org. Chem.*, **21**, 32 (1956).

(11) C. S. Marvel, "Organic Syntheses," Coll. Vol. III, John Wiley & Sons, Inc., New York, N. Y., 1955, p 848.

(12) J. v. Braun, *Ber.*, **67B**, 218 (1934).

(13) H. Schulz and V. du Vigneaud, *J. Amer. Chem. Soc.*, **88**, 5015 (1966).

mmol) in H_2O (10 ml). Acidification gave thiol **7**, which showed no change by ir or tlc.

Mercury (II)Bis(2-carboxy-1-cyclohexyl Mercaptide) (14).—Thiol **7** (20 mg, 0.12 mmol) in 2 ml of EtOH was treated with an excess of 10% $Hg(CN)_2$ solution in EtOH. The mercaptide **14** precipitated on cooling, and recrystallization from EtOH–H_2O gave colorless plates (13.6 mg, 42%); mp 160–161°; ir (KBr) 3300–2400, 1690, 1440, 1400, 1240, and 1200 cm^{-1}, tlc on Brinkmann MN Polygram (polyamide) developed in MeOH at *ca.* 25° showed only a single spot under uv light (R_f 0.24). *Anal.* ($C_{14}H_{22}HgO_4S_2$) C, H, S.

2-(2'-Protoaminoethyldithio)-1-cyclohexanecarboxylate (8).—Thiol **7** (1.02 g, 6.4 mmol) in 6 ml of EtOH was added during 10 min to a stirred solution of **4** (1.64 g, 6.4 mmol) in 4 ml of 1:7 EtOH–H_2O. After 4 hr at *ca.* 25°, a cold solution of KOH (0.71 g, 12.7 mmol) in 5 ml of H_2O was added, and the mixture was stirred at 0° for 0.3 hr. In order to initiate precipitation, Et_2O (1 ml) was added. After 2 hr at 0° a white solid separated. Disulfide **8** was collected and washed with H_2O and with EtOH. A white powder resulted (0.86 g, 57%); mp 217–219° dec. Recrystallization from H_2O (100°) gave **8** as colorless plates with mp 230° dec. Tlc of **8** on Eastman Chromagram Type K301R (silica gel) developed with EtOH–H_2O–NH_4OH (25:3:4)[14] at *ca.* 25° showed only a single spot (R_f 0.55); ir (KBr) 3200–2200, 1610, 1510, 1400, and 1275 cm^{-1}. *Anal.* ($C_9H_{17}NO_2S_2$) C, H, N, S.

***trans*-2-Aminoethyl 2-Chlorocyclohexyl Disulfide·HCl (10).**—The thiol **9** (1.51 g, 10 mmol) and **4** (2.57 g, 10 mmol) were stirred in 20 ml of 2:1 EtOH–H_2O for 0.5 hr at *ca.* 25°. Evaporation below 30° gave a white residue, which was dissolved in 25 ml of H_2O and was extracted with 50 ml of Et_2O to remove unchanged **9**. The aqueous layer then was shaken with 50 ml of Et_2O while 10 ml of an iced aqueous solution of KOH (1.7 g, 30 mmol) was added. The H_2O layer was extracted twice more with Et_2O. Each organic layer was backwashed with H_2O and immediately shaken with the same portion of 1.0 ml of 12 *N* HCl in 10 ml of H_2O cooled in an ice bath. The HCl solution then was treated with 5 ml more of 12 *N* HCl. Precipitation of **10** occurred immediately. The crude **10** was isolated by filtration, washed with hexane (20 ml), and carefully dried in a desiccator ($CaCl_2$). Disulfide **10** was washed with Me_2CO and dried under reduced pressure; 0.53 g (20%), mp 148–150°.

Tlc of **10** on Eastman Chromagram (Type K301R) developed with 95% EtOH at *ca.* 25° showed only a single spot (R_f 0.42); ir (KBr) 3200–2300, 1575, 1500, 1440, and 725 cm^{-1}. *Anal.* ($C_5H_{17}Cl_2NS_2$) C, H, Cl, S.

α-(2-Protoaminoethyldithio)cinnamate (12).—Thiol 11 (23.00 g, 0.13 mol) in 200 ml of EtOH was added with stirring to **4** (33.00 g, 0.13 mol) in 120 ml of 1:1 EtOH–H_2O. The mixture was stirred for 4 hr at *ca.* 25°. Evaporation below 30° gave a paste, which was dissolved in 100 ml of H_2O. A cold solution of KOH (14.1 g, 0.25 mol) in 100 ml of H_2O was added slowly. A red oil separated. The mixed oil and H_2O were washed with 100 ml of Et_2O. The aqueous layer was decanted from the oil, which then solidified. Recrystallization from H_2O (100°) gave **12** as a red-orange powder having mp 135–138° dec; 9.27 g (28%); ir (KBr) 3100–2200, 1600, 1540, 1450, 1340, 850, 780, 750, and 680 cm^{-1}. An identical sample (by ir) was recrystallized repeatedly from H_2O to give **12** having constant mp 136–138° dec. *Anal.* ($C_{11}H_{13}NO_2S_2$) C, H, N, S.

(14) D. Braun and H. Geenen, *J. Chromatog.*, **7**, 56 (1962).

Effect of Organic Compounds on Reproductive Processes. VIII. Methanesulfonyloxyacetyl Derivatives of Diamines

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For some time we have been interested in the effects of various alkylating agents on the reproductive processes of houseflies and mice. Certain *N,N*-bis(aziridineacetyl)-α,ω-diamine derivatives were effective as chemosterilants for houseflies[1] and some acted as sterilants for male mice.[2] These amides seemed to be less toxic than the corresponding urea derivatives[3] and we were anxious to replace the aziridine alkylating function with another one, namely, the methanesulfonate group. A series of these derivatives was synthesized from the corresponding hydroxyacetyl derivatives and evaluated for its effects on the reproduction of houseflies and mice. Tables I and II summarize the chemical data on the carbomethoxyacetyl and hydroxyacetyl intermediates and Table III summarizes the data on the final methanesulfonates.

TABLE I

BIS(CARBOMETHOXYACETYL)DIAMINES, $CH_3OCOCH_2CONH(CH_2)_nNHCOCH_2OCOCH_3$

n	Yield, %	Mp, °C	Recrystn solvent	Formula[a]
6	37	187.5–188.5	H_2O	$C_{14}H_{24}N_2O_6$
7	44	159–160	H_2O	$C_{15}H_{26}N_2O_6$
8	47	147.5–148.5	Dioxane + H_2O	$C_{16}H_{28}N_2O_6$
9	47	116–118	Dioxane + H_2O	$C_{17}H_{30}N_2O_6$
10	49	117.5–119	EtOH + H_2O	$C_{18}H_{32}N_2O_6$
11	60	95.5–97	EtOH + H_2O	$C_{19}H_{34}N_2O_6$[b]
12	37	122.5–124.5	Dioxane + H_2O	$C_{20}H_{36}N_2O_6$
p-$CH_2C_6H_4CH_2$		214–216.5	Dioxane + H_2O	$C_{16}H_{20}N_2O_6$[c]
m-$CH_2C_6H_4CH_2$	36	142.5–143.5	MeOH – Et_2O	$C_{16}H_{20}N_2O_6$

[a] All compounds were analyzed for C, H, N. [b] C: calcd, 59.0; found, 58.5. [c] C: calcd, 57.2; found, 57.7.

TABLE II

BIS(HYDROXYACETYL)DIAMINES, $HOCH_2CONH(CH_2)_nNHCOCH_2OH$

n	Yield, %	Mp, °C[a]	Formula[d]
6	25	126–129	$C_{10}H_{20}N_2O_4$
7	78	115–116	$C_{11}H_{22}N_2O_4$
8	75	116–117.5	$C_{12}H_{24}N_2O_4$
9	82	126–127	$C_{13}H_{26}N_2O_4$
10	83	122.5–123.5	$C_{14}H_{28}N_2O_4$
11	93	122–123	$C_{15}H_{30}N_2O_4$
12	74	129.5–131[b]	$C_{16}H_{32}N_2O_4$
p-$CH_2C_6H_4CH_2$	95	225–227[c]	$C_{12}H_{16}N_2O_4$

[a] All recrystallizations were from H_2O except for those noted. [b] Recrystallized from EtOH–H_2O. [c] Recrystallized from dioxane–H_2O. [d] See Table I, footnote *a*.

TABLE III

BIS(METHANESULFONYLOXYACETYL)DIAMINES, $H_3CSO_2OCH_2CONH(CH_2)_nNHCOCH_2OO_2SCH_3$

Compd	*n*	Yield, %	Mp, °C[a]	Formula[b]
1	7	31	103.5–106	$C_{13}H_{26}N_2O_8S_2$
2	8	39	127–129	$C_{14}H_{28}N_2O_8S_2$
3	9	33	109–113	$C_{15}H_{30}N_2O_8S_2$
4	10	33	120–124	$C_{16}H_{32}N_2O_8S_2$
5	11	57	115–118	$C_{17}H_{34}N_2O_8S_2$
6	12	75	127–130	$C_{18}H_{36}N_2O_8S_2$

[a] Recrystallized from Me_2CO–Et_2O. [b] See Table I, footnote *a*.

Experimental Section

***N,N'*-Bis(carbomethoxyacetyl)-α,ω-alkylenediamines.**—A solution of carbomethoxyacetyl chloride (0.04 mol) in 100 ml of C_6H_6 was added slowly to the diamine (0.02 mol) dissolved in 100 ml of C_6H_6, and 5 g of anhydrous K_2CO_3 was suspended in the same solvent. The reaction mixture was stirred at room tem-

(1) W. A. Skinner, H. C. Tong, T. E. Shellenberger, and G. W. Newell, *J. Med. Chem.*, **8**, 647 (1965).

(2) W. A. Skinner and H. C. Tong, *Experientia*, **24**, 924 (1968).

(3) W. A. Skinner, M. Cory, and J. I. DeGraw, *J. Med. Chem.*, **10**, 1186 (1967).