sulfoxide or some intermediate to produce the dioxa heterocycles or methylenebisamides.

Experimental

Thermal Stability of Dimethyl Sulfoxide.—Dimethyl sulfoxide[11] (78 g., 1.0 mole) was placed in a flask equipped with a Claisen head, condenser, and a receiver cooled in an acetone–Dry Ice bath, and heated at 190° for 72 hr. Paraformaldehyde, 1.5 g. (1.9%), identified by its 2,4-dinitrophenylhydrazone derivative, melting point and mixture melting point with an authentic sample, 165–166° (lit.[12] m.p. 166°), condensed in the Claisen head and 1.40 g. of material collected in the cold trap which consisted of 0.2 g. of ice. The 1.20 g. of liquid was subjected to v.p.c.[13] on a silicone GE-SF-96 column at 100° and with a helium flow rate of 60 cc./min., and was composed of dimethyl sulfide (42 area %), dimethyl disulfide (40 area %), and bismethylthiomethane (18 area %). These compounds were identified by comparison of retention times with authentic material. V.p.c.[13] of the remaining dimethyl sulfoxide under conditions described earlier except at 120° showed the presence of dimethyl sulfone by retention time comparison and peak enhancement with authentic material.

Reaction of Diols in Dimethyl Sulfoxide. A. Ethylene Glycol.—A solution of redistilled ethylene glycol (31 g., 0.50 mole) and dimethyl sulfoxide (158 g., 2.00 mole) was heated for 72 hr. at 190° and produced 64 g. of a distillate collected as in the preceeding experiment. Fractional distillation of this material gave 20 g. (16%) of dimethyl sulfide, b.p. 35–38°, mercuric chloride derivative m.p. 148–149° (lit.[14] b.p. 37.33°, mercuric chloride derivative[15] m.p. 150–151°); 20 g. (54%) of dioxolane, b.p. 73–74°, $n^{20}D$ 1.4000, 2,4-dinitrophenylhydrazone derivative melting point and mixture melting point with formaldehyde 2,4-dinitrophenylhydrazone 165–166°, n.m.r. spectrum[16] singlet 5.22 (2 protons) and singlet 6.24 τ (4 protons) (lit.[10] b.p. 76°, $n^{20}\alpha$ 1.3934, $n^{25}\gamma$ 1.40734, lit.[17] $n^{25}D$ 1.4010); and 12 g. (19%) of dimethyl disulfide, b.p. 104°, $n^{20}D$ 1.5222, n.m.r. spectrum[16] singlet 7.61 τ (lit.[18] b.p. 109.5°, $n^{20}D$ 1.5260). A small amount of bismethylthiomethane was detected by v.p.c. of the initial condensate.

B. 1,2-Propanediol.—The reaction of 1,2-propanediol (15.2 g., 0.200 mole) and dimethyl sulfoxide (109 g., 1.40 moles) at 190° for 48 hr. was processed as above. Fractional distillation of the condensate (32 g.) gave 14.5 g. of 4-methyldioxolane contaminated with dimethyl disulfide. Redistillation of this fraction from sodium produced 12.4 g. (71%) of pure 4-methyldioxolane, b.p. 87–89°, $n^{20}D$ 1.4050 (lit.[10] b.p. 88–89°, $n^{20}\alpha$ 1.40109, $n^{20}\gamma$ 1.41107).

C. 1,3-Propanediol.—A solution of 1,3-propanediol (15.2 g., 0.200 mole) and dimethyl sulfoxide (78 g., 1.0 mole) at 190° for 44 hr. provided 24.5 g. of condensate. The initial distillation gave 13.8 g. of crude 1,3-dioxane which was treated with sodium overnight on a steam bath. Distillation from sodium gave 11.3 g. (64%) of pure 1,3-dioxane, b.p. 104–105°, $n^{20}D$ 1.4168 (lit.[10] b.p. 105°, $n^{20}\alpha$ 1.41652, $n^{20}\gamma$ 1.42730).

Reaction of Amides in Dimethyl Sulfoxide. A. Acetamide.—Using the procedure described under reactions of diols, acetamide (11.8 g., 0.200 mole) and dimethyl sulfoxide (78 g., 1.0 mole) heated at 190° for 36 hr. gave 20 g. of condensate which by v.p.c. (conditions as described in the "Thermal Stability of Dimethyl Sulfoxide") contained dimethyl sulfide, dimethyl disulfide, and bismethylthiomethane. Bismethylthiomethane (4.5 g., 8%), b.p. 148°, $n^{20}D$ 1.5321, disulfone derivative m.p. 144–145° (lit.[19] b.p. 148°, disulfone m.p. 145°), was isolated by distillation. Dimethyl sulfoxide was removed from the reaction mixture and the residue upon crystallization from 95% ethanol gave 7.2 g. (55%) of methylenebisacetamide, m.p. 192–194°, mixture melting point with an authentic sample 194–195° (lit.[9] m.p. 196°). The infrared spectrum was identical with that of an authentic sample.

B. Benzamide.—When a mixture of benzamide (24.2 g., 0.200 mole) and dimethyl sulfoxide (78 g., 1.00 mole) was heated for 34 hr. at 190°, 20 g. of condensate consisting of dimethyl sulfide, dimethyl disulfide, and bismethylthiomethane was isolated. Dimethyl sulfoxide was removed and the residue crystallized from 95% ethanol to give 15.0 g. (60%) of methylenebisbenzamide, melting point and mixture melting point with an authentic sample 217–219° (lit.[9] m.p. 219°). The infrared spectrum was identical with that of an authentic sample.

The product was treated with 2,4-dinitrophenylhydrazine and gave formaldehyde 2,4-dinitrophenylhydrazone, melting point and mixture melting point with an authentic sample 160–163°. When the product was heated with alcoholic potassium hydroxide for 24 hr., benzoic acid (97%) was isolated.

A solution of benzamide (12.1 g., 0.100 mole) and dimethyl sulfoxide (78 g., 1.0 mole) was heated for 9 hr. at 190° while paraformaldehyde (3.0 g., 0.10 mole) was added at 1-hr. intervals until 21.0 g. (0.70 mole) was introduced. After cooling, the reaction mixture was poured into water, filtered, and the solid was recrystallized from 95% ethanol. The yield of methylenebisbenzamide, m.p. 217–219°, was 8.0 g. (63%).

C. Acetanilide.—A solution of acetanilide (27.3 g., 0.200 mole) in dimethyl sulfoxide (78 g., 1.0 mole) was heated at 190° for 24 hr. and processed as before. The condensate (dimethyl sulfide, dimethyl disulfide, and bismethylthiomethane) was 5.5 g., and 24 g. (89%) of unchanged acetanilide, melting point and mixture melting point with an authentic sample 112–114°, was recovered.

Methylenebisbenzamide.—Methylenebisbenzamide, m.p. 217–219°, was prepared in 88% yield from benzonitrile (10.3 g., 0.100 mole), *sym*-trioxane (1.5 g., 0.05 mole), and 38 ml. of 85% sulfuric acid according to the procedure of Magat, Faris, Reith, and Salisbury.[8]

Methylenebisacetamide.—Using the prior procedure,[8] methylenebisacetamide, m.p. 195–196°, was prepared in 46% yield from acetonitrile (0.20 mole) and *sym*-trioxane (0.10 mole) with the following modification. After the reaction mixture was diluted, the acid was neutralized and the solution concentrated before the product crystallized.

(11) The authors wish to thank the Chemical Products Division of the Crown Zellerbach Corp. for making generous samples of this material available for this work.

(12) G. D. Johnson, *J. Am. Chem. Soc.*, **75**, 2720 (1953).

(13) An Aerograph Model A-90P instrument was used.

(14) D. W. Osborne, R. N. Doescher, and D. M. Yost, *J. Am. Chem. Soc.*, **64**, 169 (1942).

(15) W. F. Faragher, J. C. Morrell, and S. Comay, *ibid.*, **51**, 2781 (1929).

(16) N.m.r. spectra were recorded by Mr. R. Daignault on a Varian Associates 60-Mc. high resolution n.m.r. spectrometer, Model V-4300 B, in carbon tetrachloride solution with tetramethylsilane as an internal standard.

(17) H. J. Dauben, Jr., B. Löken, and H. J. Ringold, *J. Am. Chem. Soc.*, **76**, 1362 (1954).

(18) A. I. Vogel and D. M. Cowan, *J. Chem. Soc.*, 18 (1943).

(19) H. Böhme and R. Marx, *Ber.*, **74**, 1667 (1941).

An Attempted Westphalen Rearrangement of a 5β-Hydroxy Steroid[1]

ALEX T. ROWLAND

Department of Chemistry, Gettysburg College, Gettysburg, Pennsylvania

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Numerous studies have been made concerning the Westphalen rearrangement[2] of 5α-hydroxy steroids. These studies have shown that a 6β-substituent is necessary in order for the migration of the C-10 methyl group to C-5 to occur when the alcohol is treated with with an acid catalyst. Recently, Mihina[3] reported the rearrangement of 5α-hydroxy-6β-halo steroids and concluded that a classical carbonium ion intermediate is sufficient to explain the formation of the 5β-methyl-19-nor steroid and by-products. A similar rearrangement of a B-norcholesteryl oxide has been reported by Dauben and co-workers.[4]

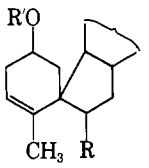
(1) Supported by research grant AM-07105-01 MC from the National Institute of Arthritis and Metabolic Diseases, Public Health Service.

(2) T. Westphalen, *Ber.*, **48**, 1064 (1915).

(3) J. S. Mihina, *J. Org. Chem.*, **27**, 2807 (1962). Pertinent references to the earlier literature are listed in this article.

(4) W. G. Dauben, G. A. Boswell, Jr., W. Templeton, J. W. McFarland, and G. H. Berezin, *J. Am. Chem. Soc.*, **85**, 1672 (1963).

The epimeric system (*i.e.*, 5β-hydroxy steroids) has not been studied in a similar manner. Inspection of a Dreiding model of this system indicates that the C-9 to C-10 bond lies *trans* and backside to the hydroxyl group at C-5. Thus, it might be predicted that rearrangement of this type of steroid would produce a spirane having the interesting structure **1**.



1

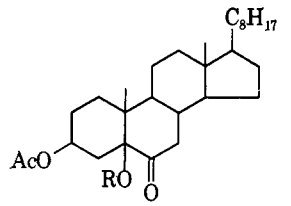
Accordingly, a 5β-cholestane derivative was prepared and treated under conditions which are known to produce rearrangement with the 5α-hydroxy compounds. Reduction of 5β-cholestane-3β,5-diol 6-one 3-monoacetate (Ia)[5] with lithium aluminum tri-*t*-butoxy hydride, followed by acetylation with acetic anhydride–pyridine, yielded 5β-cholestane-3β,5,6β-triol 3,6-diacetate (IIb).[6] It is of interest that the hydroxyl group became *axial* at C-6 instead of *equatorial*, as might have been expected.[7] Saponification of the triol diacetate (IIb) with methanolic potassium hydroxide gave the previously unknown 5β-cholestane-3β,5,6β-triol (IIa).

Treatment of the triol diacetate (IIb) with potassium acid sulfate in acetic anhydride at steam bath temperature for one hour produced a crystalline mixture from which were separated (by chromatography) 43% of cholest-4-ene-3β,6β-diol diacetate (III) and 14% of 5α-cholestan-3β-ol-6-one acetate (IV). The saturated ketone (IV) presumably arose from elimination of the 6α-hydrogen to give the enol acetate, which underwent subsequent hydrolysis and ketonization.

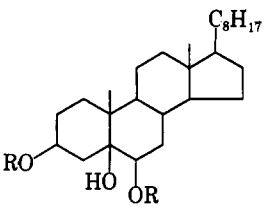
The reaction of 5α-hydroxy-6-keto steroids with acetic anhydride and potassium acid sulfate is known to proceed without a Westphalen rearrangement and to give straightforward acetylation of the 5-hydroxyl function.[8] Treatment of the diolone monoacetate (Ia) under similar conditions gave 5β-cholestane-3β,5-diol-6-one diacetate (Ib) in high yield. The same compound (Ib) was obtained by the acetylation of Ia with an acetic acid–acetic anhydride–*p*-toluenesulfonic acid mixture.

Since neither the 5β-hydroxy-6β-acetoxy nor the 5β-hydroxy-6-keto system promoted a Westphalen rearrangement, Ia was converted by a standard procedure to 5β-cholestane-3β,5-diol-6-one ethylene ketal (V) in order to test this function under the same conditions. When heated in acetic anhydride containing suspended potassium acid sulfate, followed by hydrolysis, infrared analysis indicated that V had been acetylated at the C-3 hydroxyl group and that the C-5 hydroxyl remained unaltered. No evidence was obtained for dehydration of any type.

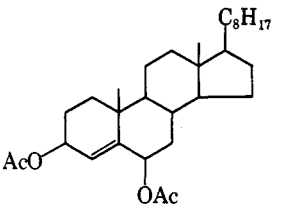
Thus, it appears that 5β-hydroxy steroids do not undergo the Westphalen rearrangement that occurs with the epimeric 5α-hydroxy compounds. The rationale for this probably lies in the internal strain involved in the migration of the C-9 to C-10 bond compared to the elimination of an α-hydrogen from C-4 or C-6.



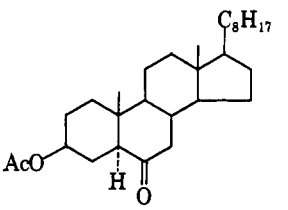
Ia, R=H
Ib, R=Ac



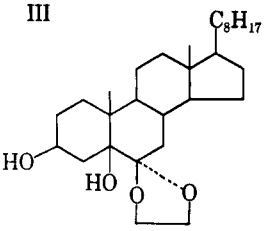
IIa, R=H
IIb, R=Ac



III



IV



V

Experimental[9]

5β-Cholestane-3β,5,6β-triol 3,6-Diacetate (IIb).—To a solution of 500 mg. (1.085 mmoles) of 5β-cholestane-3β,5-diol-6-one 3-monoacetate (Ia)[5a] in 4 ml. of tetrahydrofuran was added 500 mg. of lithium aluminum tri-*t*-butoxy hydride. The solution was stirred magnetically at room temperature for 45 min., at which time the walls of the flask were rinsed with an additional 2 ml. of tetrahydrofuran. After stirring for a further 15 min., the solution was allowed to remain at room temperature for 18 hr. Acidification with 2 *N* hydrochloric acid produced a gelatinous precipitate; water was added, and the product was extracted with three portions of methylene chloride. The combined organic extracts were washed once with water, dried, and evaporated to yield an oil which was treated with 4 ml. of pyridine and 3 ml. of acetic anhydride for 24.5 hr. at room temperature. This solution was added, in portions, to a mixture of 5 ml. of concentrated hydrochloric acid and crushed ice. The precipitated material was filtered, washed well with water, and recrystallized from methanol to give 396 mg. (73%) of IIb, m.p. 157–161°. Recrystallization from acetone–petroleum ether (b.p. 30–60°) yielded 363 mg. of white needles with m.p. 160–162°; $[\alpha]_D$ +9.1° (*c* 1.325), +10.7° (*c* 1.49); λ_{max} 2.79 (w) and 5.74 (s) μ (lit.[10] m.p. 165–167°, $[\alpha]_D$ +16°).

Anal. Calcd. for $C_{31}H_{52}O_5$: C, 73.76; H, 10.38. Found: C, 73.50; H, 10.07.

The homogeneity of IIb was demonstrated by column chromatography (alumina, Merck, acid-washed) and thin layer chromatography (silica gel).

5β-Cholestane-3β,5,6β-triole (IIa).—Five hundred milligrams (0.994 mmole) of IIb was covered with 15 ml. of 0.30 *N* methanolic potassium hydroxide and 13 drops of water. The mixture was heated on the steam bath for 1 hr. (After 0.5 hr., 7 ml. of

(5)(a) A. T. Rowland, *J. Org. Chem.*, **27**, 1135 (1962); (b) Y. Mazur and M. Nussim, *Tetrahedron Letters*, **22**, 817 (1961).

(6) All compounds named according to the Definitive Rules for the Nomenclature of Steroids, *J. Am. Chem. Soc.*, **82**, 5575 (1960).

(7) O. H. Wheeler and J. L. Mateos, *Chem. Ind.* (London), 395 (1957).

(8) B. Ellis and V. A. Petrow, *J. Chem. Soc.*, 1078 (1939).

(9) Melting points are uncorrected. Optical rotations were measured at room temperature in chloroform solutions. Infrared spectra refer to 5% carbon tetrachloride solutions unless otherwise noted. Drying of solutions was accomplished with anhydrous sodium sulfate. Elemental analyses by Micro-Analysis, Inc., Wilmington, Del.

(10) H. B. Henbest and R. A. L. Wilson, *J. Chem. Soc.*, 1958 (1957). These authors prepared IIb by lithium aluminum hydride reduction of 4β,5-oxido-5β-cholestane-3β,6β-diol, followed by acetylation.

methanol was added and after another 20 min. an additional 10 ml. of the 0.30 *N* base was added.) The resulting solution was acidified to litmus with 2 *N* hydrochloric acid, water was added, and the amorphous material which separated soon crystallized and was collected. Two recrystallizations from acetone–water gave 413 mg. (99%) of IIa (which contained water of crystallization), m.p. 123–126°. A chloroform solution of the product was dried, concentrated, and diluted with carbon tetrachloride to yield 281 mg. of IIa as white plates with m.p. 126–128°; $[\alpha]_D$ +25° (*c* 1.845); $\lambda_{max}^{CHCl_3}$ 2.90 (w, br.) μ.

Anal. Calcd. for $C_{27}H_{48}O_3$: C, 77.08; H, 11.50. Found: C, 77.00; H, 11.49.

Reaction of 5β-Cholestane-3β,5,6β-triol 3,6-Diacetate (IIb) with Potassium Acid Sulfate.—A mixture of 500 mg. (0.994 mmole) of IIb, 150 mg. of powdered potassium acid sulfate, and 5 ml. of acetic anhydride was heated on the steam bath for 1 hr., after which time crushed ice was added to the reaction mixture. After standing for 2.5 hr., the crystallline material was collected and dissolved in methylene chloride. The dried solution was evaporated, and the residue was chromatographed on 25 g. of alumina (Merck, acid-washed). Elution with 40–60% benzene–petroleum ether yielded 308 mg. of semicrystalline material which, upon crystallization from methanol, gave 207 mg. (43%) of cholest-4-ene-3β,6β-diol diacetate (III), m.p. 130–135°, $[\alpha]_D$ −13.5° (*c* 2.07). Recrystallization from methanol gave 170 mg. with m.p. 133–133.5°, λ_{max} 5.74 (s) μ. This material did not depress the melting point of authentic III (m.p. 133–133.5°, $[\alpha]_D$ −13°) prepared by the Darzens' dehydration of 5α-cholestane-3β,5,6β-triol 3,6-diacetate and the infrared spectra of the two samples were identical.

The semicrystalline material (115 mg.) that was eluted with 70% benzene–petroleum ether to 100% benzene mixtures was crystallized from methanol to yield 60 mg. (14%) of 5α-cholestan-3β-ol-6-one acetate (IV) as white rods with m.p. 126–128°, $[\alpha]_D$ −20° (*c* 0.75). Recrystallization from methanol gave 31 mg. with m.p. 128–130°. No depression in melting point was observed upon admixture with authentic IV and the infrared spectra of the two samples were identical.

5β-Cholestane-3β,5-diol-6-one Diacetate (Ib). A.—A mixture of 700 mg. (1.52 mmoles) of 5β-cholestane-3β,5-diol-6-one 3-monoacetate (Ia), 7.5 ml. of acetic anhydride, and 7.5 ml. of glacial acetic acid was warmed on the hot plate until solution was complete. One hundred and forty milligrams of *p*-toluenesulfonic acid monohydrate was added to the cooled solution, which was allowed to remain overnight at room temperature. The flask was then cooled in an ice–water mixture as water was added in portions. The precipitated product was collected, washed with water, and recrystallized from methanol containing a small amount of chloroform to yield 700 mg. (92%) of Ib, m.p. 190–193°. Recrystallization from chloroform–petroleum ether gave 653 mg. of small white needles with m.p. 192–193.5°; $[\alpha]_D$ −26° (*c* 1.25), −23° (*c* 1.30); λ_{max} 5.73 (s) and 5.79 (s, sh) μ.

Anal. Calcd. for $C_{31}H_{50}O_5$: C, 74.06; H, 10.02. Found: C, 74.02; H, 9.88.

B.—A mixture of 300 mg. (0.652 mmole) of Ia, 80 mg. of powdered potassium acid sulfate, and 2.5 ml. of acetic anhydride was heated on the steam bath for 80 min. Crushed ice was added to precipitate the product, which was collected, washed with water, and recrystallized from acetone–petroleum ether to give 285 mg. (87%) of Ib, m.p. 191–193°. No depression of the melting point occurred upon admixture with Ib prepared by method A.

5β-Cholestane-3β,5-diol-6-one Ethylene Ketal (V).—To a solution of 1.000 g. (2.17 mmoles) of the diolone monoacetate (Ia) in 80 ml. of toluene were added 10 ml. of redistilled ethylene glycol and 80 mg. of *p*-toluenesulfonic acid monohydrate. The mixture was boiled under reflux for 6.75 hr. (constant water separation) with magnetic stirring, cooled, stirred for a few minutes after the addition of 1 g. of anhydrous potassium carbonate, and diluted with water. The layers were separated and the aqueous phase was saturated with sodium chloride and extracted twice with ether. The combined organic phases were washed with a saturated saline solution and dried. Evaporation of the solvents gave an oil [λ_{max} 2.79–2.89 (w, br), 5.75 (m, sh), 5.84 (m) μ] which was treated with 10 ml. of 0.34 *N* methanolic potassium hydroxide by warming on the hot plate for 0.5 hr. The cooled solution was diluted with water and extracted three times with methylene chloride. The combined extracts were washed with water and dried. Evaporation of the solvent yielded an orange oil which crystallized slowly (upon seeding with V from another run) from cold 95% ethanol to give 576 mg. (57%) of the ketal (V) as pale yellow crystals, m.p. 136–140°. Recrystallization from methanol yielded 501 mg. of pure V, m.p. 140–142°; $[\alpha]_D$ +23.5° (*c* 1.41); λ_{max} 2.82 (w, br.) μ.

Anal. Calcd. for $C_{29}H_{50}O_4$: C, 75.27; H, 10.89. Found: C, 75.33; H, 10.67.

Hydrolysis of the Ethylene Ketal (V).—A solution of 50 mg. (0.108 mmole) of V and 12 mg. of *p*-toluenesulfonic acid monohydrate in 3 ml. of acetone containing 3 drops of water was allowed to stand at room temperature for 22.5 hr. Three milliliters of water was added, most of the acetone was removed by an air stream, and the resulting suspension was extracted three times with methylene chloride. The dried extracts were evaporated to yield an oil whose infrared spectrum was identical to that of 5β-cholestane-3β,5-diol-6-one.[5a]

The oil was dissolved in 0.5 ml. of glacial acetic acid and 0.5 ml. of acetic anhydride and treated with 10 mg. of *p*-toluenesulfonic acid monohydrate for 21 hr. at room temperature. Crushed ice was added to the green solution and, after precipitation was complete, the product was collected and recrystallized from acetone–petroleum ether to give 45 mg. (83%, calculated from V) of the diolone diacetate Ib,[11] m.p. 190–192°. The mixture melting point with Ib prepared from Ia was 191–192.5°.

Reaction of the Ethylene Ketal (V) with Potassium Acid Sulfate.—A mixture of 143 mg. (0.309 mmole) of V, 50 mg. of potassium acid sulfate, and 4 ml. of acetic anhydride was heated on the steam bath for 70 min. Crushed ice was added to the green solution and, after standing overnight, the orange oil that had separated was redissolved by the addition of *ca.* 25 ml. of acetone. The resulting solution was heated on the steam bath for 1.5 hr., as acetone was added periodically to maintain solution. Cooling, followed by the addition of water and two extractions with chloroform gave an oil whose infrared spectrum [λ_{max} 2.82 (w), 2.89 (w), 5.75 (s), and 5.85 (m) μ] indicated incomplete removal of the ketal group and significant acetylation at C-3. The oil was treated in the manner used for the hydrolysis of the ketal (V) and gave another oil whose infrared spectrum [λ_{max} 2.89 (w), 5.75 (s), and 5.85 (s) μ] was identical with that of Ia.[5a] Upon treatment with 80 mg. of potassium acid sulfate in 2 ml. of acetic anhydride on the steam bath for 1 hr., this material yielded a brown oil which, upon two recrystallizations from methanol, gave 35 mg. (23%) of the diolone diacetate (Ib) as brown crystals, m.p. 185–187° (previous softening).

Acknowledgment.—The author is indebted to Thomas Green and Steven Dressner for technical assistance.

(11) The facile hydrolysis of V and subsequent conversion to Ib indicated that no unusual rearrangement occurred in the formation of V from Ia: *cf.* S. Bernstein, M. Heller, and W. S. Allen, *J. Org. Chem.*, **26**, 1333 (1961).

Heterocyclic Derivatives of 3-Aminopropanethiol

E. CAMPAIGNE AND P. K. NARGUND

Department of Chemistry, Indiana University, Bloomington, Indiana

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2-Aminoethanethiol, 3-aminopropanethiol, and their substituted derivatives[2] are known to protect experi-

(1) Contribution No. 1156, from this laboratory. This investigation was supported by the U. S. Army Research and Development Command (Contract No. DA-49-193-MD 2096).

(2)(a) D. G. Doherty and W. T. Burnett, Jr., *Proc. Soc. Exptl. Biol. Med.*, **89**, 312 (1955); (b) S. Akerfeldt, *Acta Chem. Scand.*, **14**, 1980 (1960); D. P. Jacobus, B. Ramsay, R. E. Spalding, and D. C. Dittmer, Abstracts of Papers, presented at the 141st National Meeting of the American Chemical Society, Washington, D. C., March, 1962; (c) T. P. Johnston and A. Gallagher, *J. Org. Chem.*, **26**, 3780 (1961), and references cited therein; (d) H. Bretschneider, *Monatsh.*, **81**, 372 (1950); Holmberg and B. Sörbo, *Nature*, **183**, 832 (1959); B. Sörbo, *Acta Chem. Scand.*, **12**, 1990 (1958); (e) A. Kaluszyner, P. Czerniak, and E. D. Bergmann, *Radiation Res.*, **14**, 23 (1961).