



lents of ozone at -30° . The methylene chloride was replaced with glacial acetic acid and the ozonide decomposed with zinc dust. After separation of the zinc, the acetic acid solution was treated with chromic acid in acetic acid at 20° for three hours and the excess chromic acid was decomposed with sodium bisulfite solution. The reaction mixture was poured into water and the precipitate of the bisnor acid (I), m.p. $226-237^\circ$, was separated by filtration. For analysis, the acid (I) was crystallized from ether-hexane, m.p. $240-243^\circ$. The yield of pure acid (I) was 55% of the theoretical.

Anal. Calcd. for $\text{C}_{28}\text{H}_{34}\text{O}_7$: C, 69.69; H, 7.10; Found: C, 69.73; H, 7.10.

Treatment of the acid (I) with diazomethane in methylene chloride gave the maleic anhydride adduct of methyl 3(β)-acetoxybisnor-5,7,9-cholatrienate (IV), m.p. $246-248^\circ$. *Anal.* Calcd. for $\text{C}_{29}\text{H}_{36}\text{O}_7$: C, 70.14; H, 7.31. Found: C, 69.98; H, 7.48. After saponification, methylation with diazomethane, and acetylation with acetic anhydride, the trimethyl ester of the maleic acid adduct of 3(β)-acetoxybisnor-5,7,9-cholatrienic acid (V) was obtained, m.p. $193-195^\circ$. *Anal.* Calcd. for $\text{C}_{31}\text{H}_{42}\text{O}_8$: C, 68.61; H, 7.80. Found: C, 68.66; H, 7.65. The trimethyl ester of the maleic acid adduct of 3(β)-acetoxy-9,11-oxido-bisnor-5,7-choladienic acid (VI) was prepared by treating the acetoxytrimethyl ester (V) with monoperphthalic acid. It was crystallized from ether, m.p. $208-209.5^\circ$. *Anal.* Calcd. for $\text{C}_{31}\text{H}_{42}\text{O}_9$: C, 66.64; H, 7.58. Found: C, 66.76; H, 7.64.

The 9,11-oxido compound was also prepared from the methyl ester of I by perphthalic acid treatment and had a melting point of $263-265^\circ$ (block), which is in fair agreement with that reported for the methyl ester of II by Bergmann and Stevens.¹

Details of this work, together with further conversions of compound I, will be published at a later date.

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DEGRADATIVE STUDIES ON STREPTOMYCIN

Sir:

Degradation of dihydrostreptomycin with barium hydroxide under conditions which convert streptidine to streptamine¹ yielded an amorphous, antibiotically inactive product containing barium chloride. This substance was acetylated with pyridine and acetic anhydride to a crystalline compound (I), m. p. $261.5-262.5^\circ$, $[\alpha]^{25}_D -84^\circ$ (*c* 1, water).

Anal. Calcd. for $\text{C}_{16}\text{H}_{24}\text{O}_{12}\text{N}_3(\text{CH}_3\text{C})(\text{COCH}_3)_{10}$: C, 50.92; H, 6.25; N, 4.57; O-acetyl, 7.61 cc. of 0.1 *N* NaOH per 100 mg. $\text{CH}_3\text{-C}$, 32.3; mol. wt., 919.9. Found: C, 50.64; H, 6.17; N, 4.47; O-acetyl,² 7.37 cc.; $\text{CH}_3\text{-C}$,³ 32.3; mol. wt., 920 (Rast).

Methanolysis of I with subsequent reacylation yielded hexaacetylstreptamine,^{1,4} transition point¹ 250° , m. p. $341-345^\circ$, N, 6.61% (calcd. 6.50), and methyl pentaacetyldihydro- α -L-streptobiosamide,⁵⁻⁸ m. p. $194-195^\circ$, unchanged on admixture with a specimen prepared from dihydrostreptomycin trihydrochloride, $[\alpha]^{25}_D -120^\circ$ (*c* 0.5, chloroform). I is designated decaacetyldideguanyldihydrostreptomycin. It was found to be readily soluble in methanol, water and hot ethanol, sparingly so in chloroform, ethyl acetate and ethanol, and insoluble in benzene and ethyl ether.

Aqueous solutions of N,N,N-tetraacetyldideguanyldihydrostreptomycin (II), N¹,N²-diacetylstreptamine (III) and N-acetyldihydro- α -L-streptobiosaminide (IV) were prepared by partial deacetylation of the aforementioned acetyl derivatives with 0.05 *N* sodium hydroxide in water-dioxane. These N-acetates were subjected to oxidation with a large excess of buffered periodate at pH 4.9 and 20.0° and showed the following

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oxidant consumption (in moles per mole of compound) at 120, 240 and 550 minutes, respectively: II, 2.55, 3.2, 4.0; III, 2.0, 2.4, 3.0; IV, 1.1, 1.6, 2.0. These data prove the presence in II of an α -glycol which is not in III, and this α -glycol is present in the streptamine moiety of II. The presence of such a glycol group indicates that streptobiosamine is attached at C4 of streptidine, thus con-

firming the results of Folkers and co-workers.⁹

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(9) F. A. Kuehl, Jr., R. L. Peck, C. E. Hoffhine, Jr., Elizabeth W. Peel and K. Folkers, *THIS JOURNAL.*, **69**, 1234 (1947).

(10) Bristol Laboratories Research Fellow of the Ohio State University Research Foundation (Project 224).

NEW BOOK

The Chemistry of the Carbon Compounds. BY VICTOR VON RICHTER. Edited by the late Professor Richard Anschütz. Vol. IV. "The Heterocyclic Compounds," By F. Reindel (translated by M. F. Darken) and "Organic Free Radicals" by Ludwig Anschütz (translated by A. J. Mee). Newly translated from the Twelfth German Edition. Elsevier Publishing Co., Inc., 215 Fourth Ave., New York 3, N. Y., 1947. xv + 498 pp. 14.5 × 22.5 cm. Price, \$12.00.

This volume is a literal translation of the sections on Heterocyclic Compounds and Free Radicals in the twelfth German edition of this celebrated treatise. The appearance of Volume IV makes the entire twelfth edition available in English. Users will find the new volume to be a concise and, except for the more recent work, a comprehensive guide to the literature of the subjects treated.

REYNOLD C. FUSON

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June 10, 1948—July 10, 1946

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