

brown, and the coloring matter (hemin?) had precipitated during the growth test. Comparison of the turbidity in this case with that in a control tube containing the same amounts of hydrolyzed hemoglobin and medium but to which no yeast seeding had been added, showed that the turbidity was due almost entirely to the precipitated coloring matter. The difference between the two turbidities corresponded to 0.07 microgram of β -alanine per four mg. of original hemoglobin, which is much less than the minimum figure of 5.3 micrograms per 4 mg. which would be required by the presence of one β -alanyl unit in the hemoglobin molecule (molecular weight 66,700).

CLAYTON RESEARCH FOUNDATION AND
THE BIOCHEMICAL INSTITUTE
THE UNIVERSITY OF TEXAS
AUSTIN, TEXAS

RECEIVED DECEMBER 26, 1942

The Preparation of Desoxycholic Acid from Cholic Acid

ALEXANDER W. SCHNEIDER¹ AND WILLARD M. HOEHN

In a recent publication by Haslewood² the preparation of desoxycholic acid from cholic acid was presented. The details of the method were not given. It was stated the cholic acid was preferentially oxidized by means of chromic acid to 3,12-dihydroxy-7-ketocholanic acid and this product converted to the corresponding semicarbazone. We assume the semicarbazone was reduced according to the Wolff-Kishner technique to yield desoxycholic acid.

More than two years ago we carried out these particular reactions not only with cholic acid but also with derivatives of it. The data obtained from these investigations enabled Schmidt, Hughes and one of us (W. M. H.) to ascertain the course of the bacterial oxidation of cholic acid.³ Cholic acid, methyl cholate, methyl 3-benzoxy-7,12-dihydroxycholanate⁴ were some of the derivatives used. The compound was dissolved in either acetic acid or a mixture of acetic acid, benzene and water, and a solution of chromic acid in dilute acetic acid added. The mixture of oxidation products was converted to semicarbazones or hydrazones, which were then reduced according to the method of Wolff and Kishner (*cf.* ref. (3)). It was found that concentrated solutions of potassium or sodium hydroxide in methanol could be used instead of an alcoholic sodium alkoxide solution. The desoxycholic acid was isolated from

(1) Present address: Central Soya Company, Decatur, Ind.

(2) Haslewood, *Nature*, **150**, 211 (1942); also *cf.* *C. A.*, **36**, 7029 (1942).

(3) The results of this investigation were presented at the A. C. S. meeting in Memphis, April, 1942.

(4) Hoehn and Mason, *THIS JOURNAL*, **62**, 569 (1940).

the mixture of reduction products in accordance with known methods.

The controlled oxidation of methyl 3-benzoxy-7,12-dihydroxycholanate gave a mixture of products. From this mixture a monosemicarbazone was isolated by virtue of its solubility in methanol. The monosemicarbazone was heated with sodium methoxide in methanol (10 g. of sodium in 100 cc. of methanol) or with sodium or potassium hydroxide in methanol (10% solutions), to a temperature of 170–200° for periods up to six hours. The crude desoxycholic acid prepared by these reactions may be crystallized directly from acetic acid to yield the acetic-choleic acid. Desoxycholic acid is obtained from this complex in the usual manner.⁵ The melting point⁶ for the pure acid was 174–176°. The desoxycholic acid prepared by this procedure still gave a positive Gregory-Pascoe reaction⁷ which is indicative of the presence of cholic acid. The melting point of a mixture of a sample of desoxycholic acid (m. p. 172–173°) (Gane and Ingram, N. Y.) and that prepared by the above method was 172–174°. The $[\alpha]^{26}_D$ of a methanol solution of the desoxycholic acid was observed to be $+57 \pm 1^\circ$.

(5) Sobotka, "Chemistry of Sterids," Williams and Wilkins Co., Baltimore, Md., 1938, p. 77.

(6) Melting points were observed on the Johns melting point block.

(7) L. H. Schmidt, *Am. J. Physiol.*, **120**, 75 (1937).

RESEARCH LABORATORIES

GEORGE A. BREON & COMPANY,

KANSAS CITY, MO.

RECEIVED DECEMBER 17, 1942

Total and Partial Pressures of Binary Mixtures of Dioxane in Benzene at 25°

BY PEYTON C. TEAGUE¹ WITH W. A. FELSING

In an investigation with dioxane, it became necessary to know the partial vapor pressure of a solution of dioxane (diethylene dioxide) in benzene. Since the similarity in structure and polarity indicated solutions approaching ideality, it was decided to cover the entire composition range.

The method used was the differential method of Parks and Schwenck² as modified by Olsen and Washburn³ and by Allen, Lingo and Felsing.⁴ The benzene was purified as described previously⁴ and the dioxane was purified by the method of

(1) Present address: Department of Chemistry, University of Alabama, Tuscaloosa, Ala.

(2) Parks and Schwenck, *J. Phys. Chem.*, **28**, 720 (1924).

(3) Olsen and Washburn, *ibid.*, **41**, 457 (1937).

(4) Allen, Lingo and Felsing, *ibid.*, **43**, 425 (1939).