ous) and pure cafesterol (5 mg.) also on mice, but we could not observe estrus.

The results of these experiments demonstrate clearly that neither cafesterol and oxcafestandiol A nor any other part of coffee-oil have any estrogenic activity.

Acknowledgments.—We are indebted to D. Koch-Weser (Endoquimica Laboratories S. Paulo, Director Prof. Dr. K. H. Slotta) and to Prof. Dr. J. Ribeiro do Vale (Instituto Butantan, S. Paulo) for their kind help in performing the physiological experiments.

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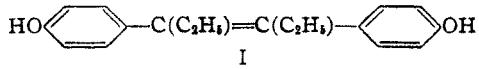
RECEIVED MARCH 11, 1943

STUDIES ON THE PREPARATION OF SYNTHETIC SEX HORMONES. II. CONCERNING SOME DERIVATIVES OF HEXESTROL

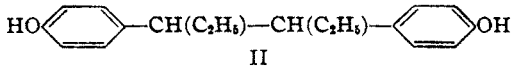
Sir:

That relatively simple synthetic organic compounds exhibit oestrogenic activity is now well known. Of especial interest in this connection are diethylstilbestrol (I)



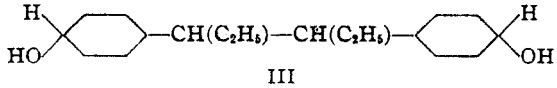
I

and hexestrol (II)



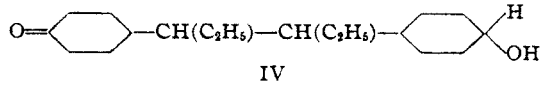
II

A good method for the preparation of the *meso* form of this latter compound has already been described in the first part of this series.[1] We now wish to report the preparation of certain of its derivatives. The starting material for this study was one of the isomeric perhydrohexestrols (III),



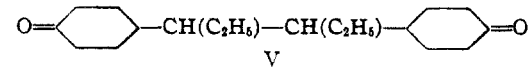
III

m. p. 167° prepared by hydrogenation of *meso*-hexestrol and kindly furnished to us by Merck and Co., Inc., Rahway, New Jersey. By means of partial oxidation with chromic acid we have converted this substance to the keto-alcohol (IV)



IV

and to the diketone (V)



V

This was achieved in the following manner: the diol (III) in pyridine was treated with the theoretical quantity of acetic anhydride necessary to acetylate one hydroxyl group. From the mixture of products so obtained the bulk of the unchanged diol was first precipitated by the addition of ether, and the ether soluble fraction, which consisted primarily of the mono- and diacetates, was then oxidized in the cold by a slight excess of chromic acid in acetic acid. Cold hydrolysis of the oxidation product with alcoholic sodium hydroxide gave a mixture of the diol and keto-alcohol contaminated with a small amount of the diketone. After conversion of the hydroxylic substances to the corresponding acid succinates (by the action of succinic anhydride in boiling pyridine for one hour) the acid esters were dissolved in dilute aqueous carbonate solution, freed from the diketone (V), m. p. 80°, by washing with ether, and precipitated by the addition of mineral acid. Cold alkaline hydrolysis of the esters gave the keto-alcohol contaminated with some diol. The former compound was then converted to the water soluble sodium bisulfite complex and the diol removed from the aqueous solution by filtration. Decomposition of the bisulfite complex with sodium carbonate gave the pure keto-alcohol (IV), m. p. 70°; semicarbazone, m. p. 146°. On treatment of the keto-alcohol with boiling acetic anhydride the acetate, m. p. 66°, was obtained; semicarbazone, m. p. 161°.

Further transformations of the keto-alcohol (IV) are now under investigation and will be reported in a later, more complete communication.

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RECEIVED APRIL 20, 1943

CRYSTALLINE β-D-GLUCO-L-TALO-OCTOSE (SYN. D-GLUCO-α-L-TALO-OCTOSE)

Sir:

The epimeric pair of acids, D-gluco-L-gala-octonic[1] and D-gluco-L-talo-octonic, results from the application of Kiliani's cyanohydrin synthesis to D-gluco-D-gulo-heptose.[2] The reduction of the lactones of these acids with sodium amalgam by

(1) Bernstein and Wallis, THIS JOURNAL, **62**, 2871 (1940).

(1) Concerning this nomenclature see Hudson, THIS JOURNAL, **60**, 1537 (1938).

(2) Emil Fischer, *Ann.*, **270**, 64 (1892).