in converting such a precursor to purines. It appears therefore that this coenzyme functions in combining a single carbon unit into the pyrimidine ring.

THE CLAYTON BIOCHEMICAL INSTITUTE AND THE DEPARTMENT OF CHEMISTRY THE UNIVERSITY OF TEXAS AUSTIN, TEXAS

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RECEIVED MARCH 5, 1947

PARTIAL HYDROLYSIS OF SILICON TETRACHLORIDE

Sir:

The existence of an homologous series of silicon oxychlorides of the general formula $Si_nO_{n-1}Cl_{2n+2}$ was established, and the first seven members isolated and identified in 1941 by Schumb and Holloway.[1] Single members, including the cyclic tetramer, $Si_4O_4Cl_8$, had also been prepared by others by various means,[2,3,4] but the method referred to[1] gave a complete series of homologs which were separable by distillation.

More recently it was mentioned in a review article[5] that the first two members of the series of oxychlorides had been prepared in this Laboratory by the partial hydrolysis of silicon tetrachloride in dilute, anhydrous diethyl ether solution by means of addition of moist ether, according to the equations

$$2SiCl_4 + H_2O = Si_2OCl_6 + 2HCl$$
$$3SiCl_4 + 2H_2O = Si_3O_2Cl_8 + 4HCl$$

Half a mole of silicon tetrachloride was dissolved in about 200 g. of anhydrous ether in a three-necked flask immersed in ice water. One quarter mole of water measured from a buret was separately dissolved in the minimum amount of anhydrous ether (about 350 g.). The wet ether was introduced by means of a separatory funnel, into the silicon tetrachloride solution at a rate of about two drops a second, with constant stirring.

There was no observable change within the flask during the reaction, but on standing overnight a very small deposit of a white solid, presumably silica, accumulated in the flask. The ether was evaporated off on a steam-bath, leaving about twenty milliliters of oily residue. This oil was fractionated at a pressure of fifteen mm. Two of the fractions were analyzed gravimetrically for chloride.

Fraction	Boiling point, °C.	%Cl Found	Calculated
II	50–53	76.69	76.76 for Si_2OCl_6
III	75.5–76.5	70.90	70.94 for $Si_3O_2Cl_8$

Better yields of higher boiling residue were obtained when the reaction was carried out at the temperature of solid carbon dioxide.

All attempts to carry out the partial hydrolysis of silicon tetrachloride in the vapor phase were unsuccessful.

The partial alcoholysis of $SiCl_4$ in ether solution with ethyl alcohol has also been accomplished in a similar manner, to give the following compounds.

Compound	Boiling point, °C.	%Cl Found	%Cl Calculated
$C_2H_5OSiCl_3$	102–104	58.83	59.28
$(C_2H_5O)_2SiCl_2$	137–138	37.59	37.52
$(C_2H_5O)_3SiCl$	156.5	17.53	17.86

Similar reactions with ammonia, dihydric alcohols, such as ethylene glycol, and hydrogen sulfide, in place of water, are to be attempted, as well as the partial hydrolysis, thiohydrolysis, ammonolysis, and alcoholysis of other non-metal halides, such as boron trichloride.

DEPARTMENT OF CHEMISTRY MASSACHUSETTS INST. OF TECH. CAMBRIDGE, MASSACHUSETTS

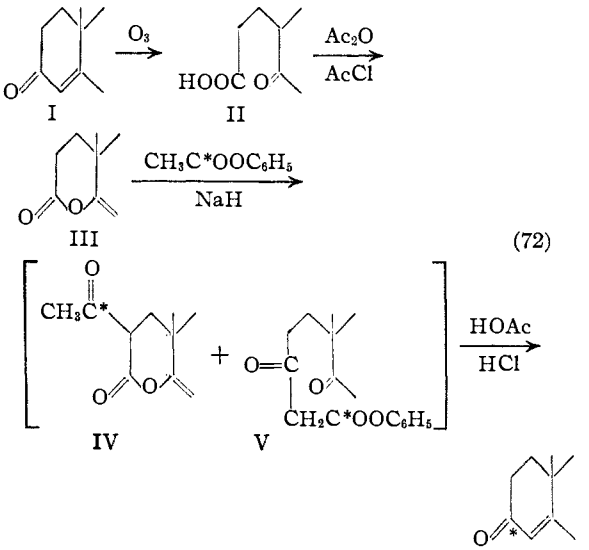
W. C. SCHUMB A. J. STEVENS

RECEIVED MARCH 1, 1947

RADIOACTIVE CHOLESTENONE[1]

Sir:

In connection with recent studies of the intermediary metabolism of the steroid hormones and the relation of these substances to various forms of cancer,[2] a method has been devised for the preparation of steroids containing isotopic carbon in ring A. Cholestenone was used as a model in the following series of reactions in which C^{14} was employed.



The yield of keto acid II obtained by ozonization of cholestenone (I) was considerably im-

(1) Schumb and Holloway, THIS JOURNAL, **63**, 2753 (1941).

(2) Friedel and Ladenberg, *Compt. rend.*, **66**, 539 (1868); *Ann.*, **147**, 355 (1868).

(3) Troost and Hautefeuille, *Bull. soc. chim.*, [2] **13**, 213 (1870); **16**, 243 (1871); **19**, 255 (1873) **35**, 360 (1881); *Ann. chim. phys.*, [5] **7**, 452 (1876).

(4) Rheinboldt and Wisfeld, *Ann.*, **517**, 197 (1935).

(5) Schumb, *Chem. Rev.*, **31**, no. 3, 590 (1942).

(1) This work was supported by funds provided by the American Cancer Society on the recommendation of the Committee on Growth of the National Research Council.

(2) (a) Dobriner, *et al.*, *Science*, **99**, 494 (1944); (b) Heilman and Kendall, *Endocrinology*, **34**, 416 (1944).

proved over that reported previously[3] by the addition of hydrogen peroxide following decomposition of the ozonide and by treatment of the residual neutral fraction with periodic acid. The keto acid melted at 154–154.5° and was smoothly converted to an enol-lactone (III), m. p. 94–94.5°, by heating with acetic anhydride and acetyl chloride. *Anal.* Calcd. for $C_{26}H_{42}O_2$: C, 80.77; H, 10.95. Found: C, 81.00; H, 10.89.

Condensation of III with phenyl acetate in the presence of sodium hydride gave an oily mixture which was simultaneously hydrolyzed, decarboxylated and cyclized by the action of acid or base. Chromatography furnished a pure product, m. p. 80–80.6°, which was identified as cholestenone by mixed m. p. determination and measurements of specific rotation (+80.1 ± 2°) and ultraviolet absorption (λ max 241.5 mμ, log ε 4.18).

It is evident that intermediates IV and V can both lead to cholestenone, and that in either case the methyl-carbon of phenyl acetate will occupy the 4-position in the reformed ring. The carbonyl group of phenyl acetate, however, will be incorporated only by the reaction of phenyl acetate with the enol-lactone anion to give IV. The use of carboxyl-labeled phenyl acetate thus provides a method for determining the relative amounts of the two intermediates.

This reagent was prepared from sodium acetate containing C^{14} in the carboxyl group[4] and had a specific activity of 4.20×10^4 counts/min./mmole.[5] Condensation with III yielded cholestenone with a specific activity of 3.79×10^4 counts/min./mmole.[5] These results indicate that about 90% of the cholestenone obtained is derived from intermediate IV.

Details of the above observations will be published shortly. The use of methyl-labeled phenyl acetate and extension of the method to other steroids has been undertaken.

(3) Bolt, *Rec. trav. chim.*, **57**, 906 (1938).

(4) Ruben, Allen and Nahinsky, THIS JOURNAL, **64**, 3050 (1942).

(5) The author is indebted to Dr. Warren W. Miller of the Radioactivity Center, M. I. T., for activity measurements. For a description of the counting method see Miller, *Science*, **105**, 123 (1947).

DEPARTMENT OF CHEMISTRY
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RECEIVED FEBRUARY 21, 1947

NEW BOOKS

Modern Developments of Chemotherapy. By E. HAVINGA, H. W. JULIUS, H. VELDSTRA and K. C. WINKLER. (Monographs on the Progress of Research in Holland.) Elsevier Publishing Co., Inc., 215 Fourth Ave., New York 3, N. Y., 1946. xi + 175 pp. Illustrated. 14.5 × 21 cm. Price, $3.50.

This monograph presents in lucid and concise form the results of research in Holland during the last war on sulfanilamide derivatives and other therapeutic agents. Many of the products synthesized were similar to those obtained in other countries and the data bearing on these and the foreign literature relating to similar products are presented as a critical study in an interesting and readable form. The critical analysis of these results is provocative even if the conclusions are not always convincing.

Considerable attention is given to the mechanism of the action of the sulfanilamide compounds, evidence being presented from both bacteriological and physical chemical investigations. Much of this evidence is similar to that obtained by other investigators but in certain instances novel conclusions are reached. Stress is laid on the importance of adsorption in determining the relative activities of the several sulfa drugs. The validity of this evidence cannot be assessed without having further information concerning the methods employed in obtaining the data. From the data one cannot say with certainty that activity is entirely dependent upon adsorption for sulfanilamide, the parent substance, is listed as showing no adsorption on the organisms used in the experiment within the experimental error of the method. Again, other investigators have shown that organisms grown on media containing metanilamide or orthanilamide retain more of these drugs than the same organisms grown on sulfanilamide under similar conditions, yet the former are inactive in inhibiting the growth of the organisms in comparison with sulfanilamide.

The chapter on physico-chemical investigations in particular is well written and deserves careful consideration.

In the chapter on chemical investigations the sulfanilamide compounds studied are given in Table 3 beginning on page 120 and should follow paragraph 3 on page 119.

Biochemical investigations beginning in the middle of page 119 are continued on page 134.

Chapter 4 deals with pharmacological, immunological and clinical investigations, while Chapter 5 covers the researches on antibiotic substances and related mycotherapy.

The monograph is a valuable contribution to the literature of chemotherapy.

M. L. CROSSLEY

Medical Biochemistry. By MARK R. EVERETT, Ph.D., Professor of Biochemistry, University of Oklahoma School of Medicine. Second edition, completely revised. Paul B. Hoeber, Inc., Medical Book Department of Harper and Brothers, 49 East 33rd Street, New York 16, N. Y., 1946. xii + 767 pp. 106 tables. 16.5 × 24 cm. Price, $7.00.

"Medical Biochemistry" is intended to serve as "a modern, concise and correlated survey of biochemical knowledge" primarily for students of medicine, and as a convenient reference volume for more general use. The subject matter is divided into ten chapters dealing successively with acid–base relations, colloids, enzymes and oxidation, digestion, lipides, carbohydrates, proteins, prosthetic radicals of nucleoproteins and chromoproteins,