wise degradation[4] of the fatty acid was carried out to determine the position of the radioactive label.

TABLE II

SYNTHESIS AND DEGRADATION OF BUTYRIC-4-C^{14} ACID

Compound or carbons	Specific radioactivity,[a] counts/min./mg. $BaCO_3$ Calcd.	Found
Glutamic-1,2-C_2^{14} acid	...	7.50
3-(Formyl-C^{14})-propionic acid	4.69[b]	...
Butyric-4-C^{14} acid	4.69	4.72
Carbon 1	0.0	0.05
Carbon 2	0.0	0.04
Carbon 3	0.0	0.24
Carbon 4	18.8	18.1

[a] See footnote for Table I. [b] Calculated from specific activity of glutamic-1,2-C_2^{14} acid.

The data of Tables I and II indicate that under the conditions of the Huang-Minlon modification[2] of the Wolff–Kishner reduction there occurred no detectable rearrangement of the carbon skeleton of pyruvic and 3-formylpropionic acids. It is believed that the appreciable radioactivity found for carbon 3 of butyric-4-C^{14} acid was probably not caused by the reduction or the degradation procedure, but may have been introduced during the synthesis of the labeled starting material.[5]

Experimental

Propionic-2-C^{14} Acid.—Carbonyl-labeled pyruvic acid (1.0 mmole) prepared by the method of Anker[6] was purified by partition chromatography. The aqueous solution of the sodium salt (0.85 mmole) obtained from the partition column was concentrated to a volume of 5 ml. in a 200-ml. flask. Redistilled diethylene glycol (25 ml.), 5 ml. of an 85% hydrazine solution and 1 g. of potassium hydroxide were added and the solution was refluxed for one hour. Water was then distilled off until the temperature reached 190°, and heating under reflux was continued for one hour. The reaction mixture was cooled, acidified with sulfuric acid and steam distilled. The steam distillate was concentrated and propionic acid was obtained in aqueous solution, as the sodium salt, by partition chromatography.[7] The chromatographic step was required in order to separate propionic acid from traces of acetic acid formed by thermal decomposition of the solvent. Propionic acid was identified by its position on the chromatogram, Duclaux distillation and preparation of the *p*-bromophenacyl ester, m.p. 59–60°; yield 73% of the theoretical based on sodium pyruvate.

Butyric-4-C^{14} Acid.—Because of its instability, 3-(formyl-C^{14})-propionic acid was prepared as needed by treating glutamic-1,2-C_2^{14} acid with an equimolecular amount of chloramine T at 50°.[8] It was found that this reagent converted glutamic acid quantitatively to 3-formylpropionic acid. The latter was not isolated from solution but was identified and weighed as the *p*-nitrophenylhydrazone, m.p. 178–180°, and the 2,4-dinitrophenylhydrazone, m.p. 198–200°. The aqueous solution of 3-(formyl-C^{14})-propionic acid was taken directly for the Wolff–Kishner reduction as described for propionic-2-C^{14} acid. Butyric-4-C^{14} acid was isolated by partition chromatography[7] and identified by its position on the chromatogram, Duclaux distillation and preparation of the *p*-bromophenacyl ester, m.p. 63–64.5°; yield 55% of theory based on glutamic acid.

The authors wish to acknowledge the technical assistance of Miss M. V. Long and Mrs. B. A. Gwin.

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Preparation of Sodium Ferrate(VI)

BY LOUIS T. OCKERMAN AND JAMES M. SCHREYER

Numerous wet methods for the preparation of solutions of sodium ferrate, Na_2FeO_4, have been reported. Foster,[1] Thiesse,[2] Rosell,[3] and Grube and Gmelin[4] prepared such solutions by different oxidation methods, but were unable to isolate any solid Na_2FeO_4.

Wallace and Fleck[5] prepared crystalline Na_2FeO_4 by fusion of Na_2O_2 in an iron crucible. The crystals were described as probably being pure Na_2FeO_4, although no analysis was reported.

In view of the failure of previous investigators to prepare crystalline Na_2FeO_4 by wet methods, the authors made a further study of the problem.

Experimental

Chlorine gas was passed into a solution containing 30 g. of solid NaOH per 75 ml. of water until the increase in weight amounted to 20 g. The temperature was maintained below 20° by means of an ice-bath. After the dissolution of 70 g. of solid NaOH, the solution was filtered. The beaker containing the solution was replaced in the ice-bath and 20 g. of ferric nitrate was added. The solution was cooled to 10–15° and saturated by adding solid NaOH. The solution was filtered through a fritted glass filter and the black mass was air dried by continued suction. A small volume of benzene was drawn through the mass on the filter, followed by 3 portions of 95% ethanol. Each portion was left in contact with the black mass for only a few minutes. The product was finally dried with a few milliliters of ethyl ether. A calcium chloride drying tube was attached to the mouth of the filter during the final drying operation.

The solid product obtained by the above procedure gave a purple color characteristic of the ferrate ion when added to water.

Although the sample appeared to be highly contaminated with hydrous ferric oxide, analysis by the chromite method[6] showed 41.38% Na_2FeO_4.

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Steroids. XXI.[1] Δ^7-Androstene-3β,17β-diol

BY F. NEUMANN, G. ROSENKRANZ, J. ROMO AND CARL DJERASSI

Steroids with an isolated double bond in the 7,8-position are of considerable importance[2,3] for biological experiments and synthetic purposes. Since no such representative is known in the androstane series, we have investigated two obvious synthetic routes as outlined below.

The first approach consisted of catalytic hydrogenation of $\Delta^{5,7}$-androstadiene-3β,17β-diol (IIa) to yield Δ^7-androstene-3β,17β-diol (IVa). The former substance has been prepared before by an

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