Spectroscopically pure cesium chloride was bombarded with 85 Mev. protons in the 184-in. Berkeley cyclotron for periods of one to three hours. The induced barium radioactivities, after chemical isolation, were observed with a Geiger counter with a 3 mg./sq. cm. mica window. The decay curve showed half lives of 2.0 ± 0.1 hours and 2.4 ± 0.1 days, as well as longer lived activity due to the well known[2] $Ba^{131}$ and its daughter $Cs^{131}$.

Isolation of cesium from the purified barium (after the 2.0-hour activity had effectively decayed) yielded a mixture of the 31-hour[3] $Cs^{129}$ and 10-day $Cs^{131}$. Subsequent separations of cesium from the same barium yielded only $Cs^{131}$, at a time when the 2.4-day activity was still present. Thus it is probable that the 2.0-hour activity is $Ba^{129}$ produced by the reaction $Cs^{133}$ ($p$, $5n$). The 2.4-day period is not $Ba^{127}$, otherwise it would produce 5.5-hour[3] $Cs^{127}$ as a daughter. The most probable assignment is $Ba^{128}$ from the $p$, $6n$ reaction, but this assignment lacks direct proof.

A mass spectrographic analysis of a purified barium fraction showed a line at mass 129 which was proved to be radioactive by the photographic transfer technique. A second line at mass 128 was too weak to be identified as radioactive. The line at 129 is probably due to the cesium daughter, which had time to grow between the purification and the analysis. Cesium is ionized by the thermal ion source with much better efficiency than is barium. The mass scale was fixed by means of a small amount of stable $Cs^{133}$ added to the sample.

$Ba^{128}$ decays to $Cs^{128}$, which is expected to be short lived[3] and to decay to stable $Xe^{128}$. Our experiments indicate a half life of 30 minutes or less. Thus the radiations we observe for the 2.4-day period include those from both the barium decay and the cesium decay. We have observed positrons of energy limit about 3.0 Mev., electrons of about 0.3 Mev., and gamma rays.

$Ba^{129}$ emits positrons, but we have not characterized its radiations otherwise.

Recently we have learned that Thomas and Wiig have reached some of these same conclusions independently.[4]

We are indebted to the crew of the 184-in. cyclotron for their coöperation in these experiments, and to F. L. Reynolds for assistance with the mass spectrograph.

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(2) G. T. Seaborg and I. Perlman, *Revs. Mod. Phys.*, **20**, 585 (1948).

(3) Fink, Reynolds, and Templeton, *Phys. Rev.*, **77**, 614 (1950).

(4) C. C. Thomas and E. O. Wiig, THIS JOURNAL, **72**, 2818 (1950).

(5) Now at the Knolls Atomic Power Laboratory, Schenectady, N. Y.

## INCORPORATION OF THE CARBON OF FORMALDEHYDE AND FORMATE INTO THE METHYL GROUPS OF CHOLINE[1,2]

*Sir:*

It has been recently demonstrated in this Laboratory that the carbon of methanol may be incorporated, *in vivo*, into the methyl groups of choline.[3] Continuing work along these lines, we have found that the carbon of formaldehyde or of formate may also be utilized. The use of the carbon of bicarbonate for this purpose was not detected.

The compounds, labeled with $C^{14}$, were injected subcutaneously into rats which were kept in an open circuit metabolism apparatus for the collection of the expired carbon dioxide. For a few days prior to the injections and during the experiments the animals were allowed free access to a diet of the following percentage composition: vitamin-free casein 20; cystine 0.4; sucrose 54.6; Osborne and Mendel salt mixture 4; fat (Covo) 19; corn oil (Mazola) 1, containing 4.0 mg. of $\alpha$-tocopherol acetate and 0.1 mg. of 2-methyl-1,4-naphthoquinone, 750 I.U. of vitamin A and 125 I.U. of vitamin D; water-soluble vitamin mixture consisting of thiamine hydrochloride, riboflavin, pyridoxine hydrochloride, nicotinic acid and $p$-aminobenzoic acid, 1 mg. each, calcium $d$-pantothenate 5 mg., inositol 10 mg., folic acid 0.4 mg., biotin 0.01 mg., and sucrose to make 1000 mg. Each animal received 3 micrograms of vitamin $B_{12}$ intraperitoneally at the beginning of the experiment.

Over a period of three days, rat no. 804 received daily, in three subcutaneous injections, 2 ml. of a 0.093 molar solution of $C^{14}$-formaldehyde along with an equimolecular amount of ordinary bicarbonate. The animal was sacrificed three hours after the last injection. Of the $1.30 \times 10^6$ counts per minute injected as radioformaldehyde, $1.06 \times 10^6$ counts per minute were excreted in the expired carbon dioxide. The choline was isolated from the carcass as the choline chloroplatinate (*Anal.* Calcd. for $C_{10}H_{28}N_2O_2 \cdot PtCl_6$: Pt, 31.68. Found: Pt, 31.70). It was degraded to trimethylamine which was isolated as the chloroplatinate (*Anal.* Calcd. for $C_6H_{20}N_2 \cdot PtCl_6$: Pt, 36.96. Found: Pt, 37.08). The specific activities of these compounds were determined as previously described[3] and are given in the table.

Rat no. 808, received daily, in three subcutaneous injections, 2 ml. of a 0.095 molar solution of sodium $C^{14}$-formate over a three-day period. The animal was sacrificed fourteen hours after the last injection. Of the $8.22 \times 10^6$ counts per minute injected as formate, $4.87 \times 10^6$ counts per minute

(1) While these experiments were underway, it was announced by Professor H. G. Wood in a Harvey Lecture, February 16, 1950, that Dr. W. Sakami and Professor A. D. Welch have been able to demonstrate the synthesis of "biologically labile" methyl groups from formate *in vivo* in the rat and by rat tissue *in vitro*.

(2) The authors wish to express their appreciation to the Lederle Laboratories Division, American Cyanamid Company, for a research grant which has aided greatly in this work.

(3) du Vigneaud and Verly, THIS JOURNAL, **72**, 1049 (1950).

were recovered in the expired carbon dioxide. The choline chloroplatinate was isolated (Found: Pt, 31.69) and converted to trimethylamine chloroplatinate (Found: Pt, 36.93). The specific activities are given in the table.

| Rat no. | Compound | Spec. activity counts/min./millimole |
|---|---|---|
| 804 ♂ | $C^{14}$-formaldehyde injected | $2.33 \times 10^6$ |
| wt., 195 g. | Choline chloroplatinate | $2.37 \times 10^4$ |
| | Trimethylamine chloroplatinate | $2.19 \times 10^4$ |
| 808 ♂ | Sodium $C^{14}$-formate injected | $1.44 \times 10^7$ |
| wt., 194 g. | Choline chloroplatinate | $1.55 \times 10^5$ |
| | Trimethylamine chloroplatinate | $1.42 \times 10^5$ |

These results have been confirmed in similar experiments.

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RECEIVED APRIL 3, 1950

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## VITAMIN $B_{12}$. XI. DEGRADATION OF VITAMIN $B_{12}$ TO $D_g$-1-AMINO-2-PROPANOL

*Sir:*

Degradation of vitamin $B_{12}$ by acid hydrolysis has yielded $D_g$-1-amino-2-propanol[1] which was characterized by structure examination and by synthesis.

The acid hydrolysis of vitamin $B_{12}$ gives a product reacting with ninhydrin.[2] This product was thought to be 2-aminopropanol[3] on the basis of paper chromatographic evidence, but this conclusion has been withdrawn[4] in view of more recent results.

Vitamin $B_{12}$ has been hydrolyzed in hydrochloric acid solution at 100°, and the dibenzoate of $D_g$-1-amino-2-propanol has been isolated from the products by the following sequence of purification steps: butanol–water partition, benzoylation of the water-soluble fraction, partition of the benzoates between petroleum ether (b. p. 90–100°) and water, countercurrent distribution using a mixture of petroleum ether (b. p. 90–100°) and aqueous methanol to remove benzamide and other materials, vacuum sublimation and recrystallization. The crystals melted at 73–74°; $[\alpha]^{24}D$ $-72 \pm 1°$ (*c*, 0.83 in ethanol). *Anal.* Calcd. for $C_{17}H_{17}NO_3$: C, 72.06; H, 6.05; N, 4.95. Found: C, 72.55; H, 5.88; N, 4.96.

This dibenzoate was not identical with a synthetic specimen of the dibenzoate of $L_s$-2-aminopropanol (m. p. 104–105°).

The free amine was regenerated by acid hydrolysis of the degradative dibenzoate, and then oxidized by addition of sodium metaperiodate. The addition of dimedon gave the corresponding derivatives of acetaldehyde and formaldehyde which were separated and identified.[5] Thus, the structure of the amine is shown to be that of 1-amino-2-propanol, a known compound.[6]

A synthesis of 1-amino-2-propanol from optically active lactic acid would confirm the identification and also give the configuration of the degradation product. Consequently, $D_g$-lactamide[7] was prepared from ethyl $D_g$-lactate,[8] which was obtained from $D_g$-lactic acid made by resolution of the morphine salts from DL-lactic acid.[9] Reduction of $D_g$-lactamide by lithium aluminum hydride by a modification of other amide reductions[10] followed by benzoylation of the product gave the dibenzoate of $D_g$-1-amino-2-propanol, m. p. and "mixed m. p.," 74–75°; $[\alpha]^{24}D$ $-72 \pm 1°$ (*c*, 0.82 in ethanol). *Anal.* Calcd. for $C_{17}H_{17}NO_3$: C, 72.06; H, 6.05; N, 4.95. Found: C, 72.01; H, 5.85; N, 4.92.

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(1) The subscript g refers to glyceric aldehyde, the fundamental substance to which the configuration of the carbohydrates can be related. The subscript s refers to serine, the fundamental substance to which the configuration of the amino acids can be related: Vickery, *J. Biol. Chem.*, **169**, 237 (1947).

(2) Ellis, Petrow and Snook, *J. Pharm. and Pharmacol.*, **1**, 60 (1949).

(3) Ellis, Petrow and Snook, *ibid.*, **1**, 735 (1949); **1**, 950 (1949).

(4) Cooley, Ellis and Petrow, *ibid.*, **2**, 128 (1950).

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(7) Bean, Kenyon and Phillips, *J. Chem. Soc.*, 303 (1936).

(8) Purdie and Williamson, *ibid.*, **69**, 818 (1896).

(9) Patterson and Forsyth, *ibid.*, **103**, 2263 (1913).

(10) Uffer and Schlittler, *Helv. Chim. Acta.*, **31**, 1397 (1948).