

$\text{H}_2\text{S}^{35}\text{O}_4$ and then converting to sulfanilic acid using a small scale modification of the baking method of Huber.⁶ The yields were about 95% for 50 mg. and 60 to 70% for 2 to 5 mg. batches. The product contained less than 0.2% of ortho or meta isomers and two recrystallizations from carrier sulfate reduced the free S^{35}O_4 to less than 0.2% of the total S^{35} . Not more than 0.6% of the sulfanilic acid sulfur exchanged with free sulfate in acid, basic, or neutral solution in 55 days at 80°.

Full experimental details of this preparation are available on microfilm.⁷

(5) Obtained from the Isotopes Division of the U. S. Atomic Energy Commission, Oak Ridge, Tennessee.

(6) W. Huber, *Helv. Chim. Acta*, **15**, 1372 (1932).

(7) For full experimental details of this preparation order Document 3489 from American Documentation Institute, 1719 N Street, N. W., Washington 6, D. C., remitting \$1.00 for microfilm (images 1 inch high on standard 35-mm. motion picture film) or \$1.00 for photocopies (6 × 8 inches) readable without optical aid.

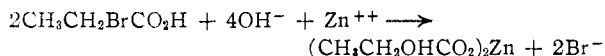
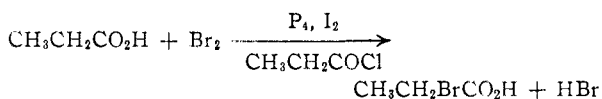
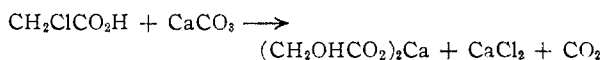
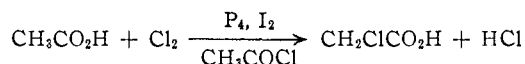
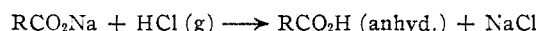
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Small Scale Synthesis of Several Carbon-14 Labeled α -Hydroxy Acids¹

By D. M. HUGHES, R. OSTWALD² AND B. M. TOLBERT

For a series of biological studies the preparation of the two singly labeled glycolates and the three singly labeled lactates was undertaken as follows³⁻⁵



The purity of the calcium glycolate was checked by three methods. In the first, the salt was recrystallized from water and the specific activity remeasured. It was found that the activity did not change, thus confirming the gross purity of the product. A C and H analysis of the product agreed well with the calculated values, although it was noted that sometimes the dihydrated salt crystallized out and sometimes the anhydrous calcium glycolate was obtained. A two-dimensional paper chromatographic separation (butanol-propionic acid-water in the first direction; phenol-water in the second) and radioautographs of the resulting

(1) Details of the chemical procedure are available on microfilm. Order Document 3567 from the American Documentation Institute, 1719 N Street, N. W., Washington 16, D. C., remitting \$1.00 for microfilm or \$1.20 for photocopies readable without optical aid.

(2) Supported by a grant to Prof. D. M. Greenberg, University of California, from the American Cancer Society, Committee on Growth of the National Research Council. The work described in this paper was sponsored by the U. S. Atomic Energy Commission.

(3) A. Hölzer, *Ber.*, **16**, 2955 (1883).

(4) A. Kekulé, *Ann.*, **130**, 18 (1864).

(5) E. Fischer and G. Zemplén, *Ber.*, **42**, 4891 (1909).

paper showed only one radioactive spot, thus confirming the radioactive purity of the salt.⁶

The purity of the zinc lactate was similarly checked by elementary analysis and paper chromatography. Attempts to recrystallize the crude zinc lactate from distilled water failed to give pure products because of partial hydrolysis of the zinc lactate. This was corrected by crystallizing the product from a 0.1 M zinc chloride solution.

In order to produce a stable zinc lactate of uniform hydration, the product was first dried *in vacuo* and then hydrated in the laboratory (relative humidity ~ 50%). Evidence for exactly three waters of hydration was obtained not only from the analytical work (C, H, ash) but also from the weight ratio of the anhydrous to the hydrated material.

For biological experiments these salts can be easily and quantitatively converted to an aqueous solution of the free acid by mixing a solution of the salt with excess Dowex-50 ion exchange resin in the acid form. When the resin is filtered off, a zinc or calcium-free solution of the organic acid is left.

Yields, specific activities and scale of the several reactions are summarized in Table I.

TABLE I

Compound	Scale of reaction, mmoles	Sp. act. of prod., $\mu\text{c}/\text{mg.}$	Yield based on fatty acid, %
Calcium glycolate-1-C ¹⁴	13.3	0.24	65.0
Calcium glycolate-2-C ¹⁴	13.7	0.30	60.5
Zinc lactate-1-C ¹⁴	6.3	3.85	81.8
Zinc lactate-2-C ¹⁴	13.5	6.90	84.0
Zinc lactate-3-C ¹⁴	12.7	3.09	76.3

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(6) A. A. Benson, *et al.*, *This Journal*, **72**, 1710 (1950).

RADIATION LABORATORY AND
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The Synthesis of Bis-(2-hydroxy-3,5,6-trichlorophenyl)-methane-C¹⁴ (Hexachlorophene)¹

By HERBERT M. ISIKOW AND WILLIAM S. GUMP

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In the course of a study of the uptake of bis-(2-hydroxy-3,5,6-trichlorophenyl)-methane² (hexachlorophene) from soap solutions by the skin of experimental animals, it was necessary to prepare this compound labeled with carbon-14. Its preparation was readily effected by condensing 2,4,5-trichlorophenol with formaldehyde-C¹⁴ in the presence of sulfuric acid. Carbon-14 was thus incorporated into the methylene bridge of hexachlorophene as shown by the equation

(1) For detailed paper order Document 3568 from American Documentation Institute, 1719 N Street, N. W., Washington 6, D. C., remitting \$1.00 for microfilm (images 1 inch high on standard 35-mm. motion picture film) or \$1.00 for photocopies (6 × 8 inches) readable without optical aid.

(2) W. S. Gump, U. S. Patent 2,250,480 (July 29, 1941).