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THE SYNTHESIS OF VALINE-2-C14

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UNIVERSITY OF CALIFORNIA

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THE SYNTHESIS OF VALINE-2-C<sup>14</sup>

P. T. Adams and B. M. Tolbert

June 11, 1952

Berkeley, California

THE SYNTHESIS OF VALINE-2-C<sup>14</sup>

by

P. T. Adams and B. M. Tolbert

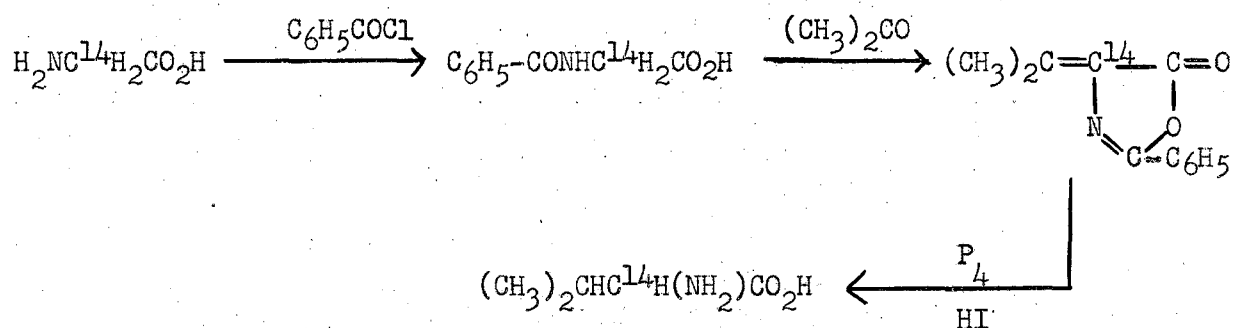
The synthesis of amino acids from carbon-14 labeled hippuric acid has been used satisfactorily in the case of the aromatic amino acids (1), but has not previously

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(1) S. C. Wang, J. P. Hummel & T. Winnick,  
J. Am. Chem. Soc., 74, 2445 (1952).

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been reported in the preparation of aliphatic compounds. Using procedures similar to those employed in the aromatic series, a novel preparation of valine has been developed. By means of the following reactions, we have prepared valine-2-C<sup>14</sup> in 65% yield from glycine-2-C<sup>14</sup>. The method is also readily adaptable to a synthesis of valine-1-C<sup>14</sup>.




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Experimental2-Phenyl-4-isopropylidene-(oxazalone-4-C<sup>14</sup>)-5

Glycine-2-C<sup>14</sup> (120  $\mu$ c, 0.76 g.) was converted to hippuric-2-C<sup>14</sup> acid by reaction with benzoyl chloride (2). The product was ground to a fine powder with 0.6 g. of

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(2) Organic Synthesis, Coll. Vol. II,  
John Wiley & Sons, New York (1943) p. 328

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freshly fused sodium acetate and dissolved in 35 ml. of dry acetone. Acetic anhydride (5 ml.) was added dropwise and the mixture heated under reflux for 15 hours (3). The hot solution was poured onto crushed ice and diluted to 250 ml. with water.

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(3) G. R. Ramage and J. L. Simonsen,  
J. Chem. Soc., 1935, 534-35.

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The precipitate was collected by filtration and dried in vacuo. The yield of oxazalone (m.p. 98-100°) was 1.16 g. (71  $\mu$ c) or 59% from glycine-2-C<sup>14</sup>. The filtrate contained 36  $\mu$ c (30%), of which 28  $\mu$ c (23%) could be recovered as pure glycine.

Valine-2-C<sup>14</sup> - The oxazalone was mixed with 2.0 g. of red phosphorus and 12.5 ml. of acetic anhydride. Hydrogen iodide (12.5 ml., specific gravity 1.7) was added and the solution was heated under reflux for 20 hours. The reaction mixture was filtered and the filtrate evaporated to dryness in vacuo. The residue, dissolved in 100 ml. of 70% ethanol, was poured through a glass column containing 60 ml. of Dowex-50 cation exchange resin (50-100 mesh) in the acid form. The resin was washed free of anions by rinsing with 100 ml. of 70% ethanol followed by 250 ml. of water.

The amino acid was eluted from the resin with 250 ml. of 2 N ammonium hydroxide followed by 250 ml. of water. The eluate was evaporated to dryness on a steam bath and dried in vacuo. The resulting valine-2-C<sup>14</sup> (50  $\mu$ c) was shown to be free from radioactive or amino acid contaminants by two dimensional paper chromatography and radioautography (4). The yield was 83% from the oxazalone, or 65% from unrecovered glycine-2-C<sup>14</sup>.

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A. A. Benson, et.al.,  
J. Am. Chem. Soc., 72, 1710 (1950)

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