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PREPARATION OF VALINE-1-C¹⁴

R. E. Self and B. M. Tolbert

May 17, 1951

Berkeley, California

PREPARATION OF VALINE-1-C¹⁴

R. E. Selff and B. M. Tolbert

Radiation Laboratory and Department of Chemistry

University of California, Berkeley^(*)

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ABSTRACT

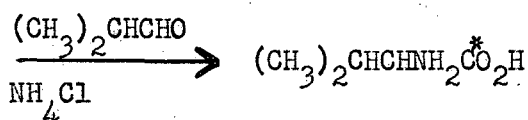
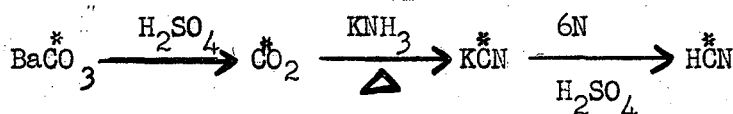
Valine-1-C¹⁴ has been prepared on a 20 mmole scale by the Strecker reaction. The yield was 51% based on potassium cyanide used to begin the synthesis.

(*) The work described in this paper was sponsored by the U. S. Atomic Energy Commission.

PREPARATION OF VALINE-1-C¹⁴

R. E. Selff and B. M. Tolbert

Radiation Laboratory and Department of Chemistry

University of California, Berkeley^(*)Valine-1-C¹⁴ was prepared via the Strecker reaction as follows:

The product was analyzed by two dimensional paper chromatography using butanol-propionic acid-water and phenol-water as the two developing agents. ⁽¹⁾ Radioautographs and ninhydrin sprays of the paper showed only one radioactive ninhydrin sensitive compound.

EXPERIMENTAL

Hydrogen Cyanide-C¹⁴: - One mmole barium carbonate-C¹⁴ was converted to CO₂ and reduced to KC¹⁴N by a modification of the procedure of Loftfield. ^(2,3) Hydrogen cyanide was prepared from KCN by treatment with 6 N sulfuric acid as described.

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Valine-1-C¹⁴: - This HC¹⁴N (approximately 0.8 mmoles) was distilled in vacuo into a 100 ml. round bottom flask containing 1.28 g. potassium cyanide (97%). 1.8 ml. freshly distilled isobutyraldehyde, 40 ml. water, 0.8 ml. of 1 N potassium hydroxide and 1.3 g. ammonium chloride. The flask was closed, removed from the vacuum line and placed in an ice bath for 20 minutes where it was shaken several times.

A reflux condenser was attached to the flask and the reaction mixture heated for 24 hours at 70-80°. The reaction mixture was then extracted with four 25 ml. portions of methylene chloride and the methylene chloride evaporated on a steam bath. The flask containing the residue was fitted with a reflux condenser, 50 ml. concentrated hydrochloric acid were added and the mixture heated on a steam bath for 24 hours. The hydrochloric acid followed by four 60 ml. portions of water were then distilled off under reduced pressure.

The residue was dissolved in a few ml. water and equal portions added to two ion exchange columns (20 cm. x 15 mm. I.D.) containing 60 ml. packed Dowex 50 cation exchange resin each in the acid form. The columns were then washed with 300 ml. water. The purified valine was eluted with 300 ml. 2 N ammonium hydroxide and washed with 800 ml. water. The combined eluate and wash was evaporated to dryness. Two crops of valine were crystallized from water-ethanol. The mother liquor was evaporated to dryness and a third crop obtained by sublimation in vacuo.

The yield of pure material was 1.196 g. or 51.4% based on the cyanide used to begin the synthesis. The specific activity of the valine was 1.6 μ C/mg.

SUMMARY

Valine-1-C¹⁴ has been prepared on a 20 mmole scale by the Strecker reaction. The yield was 51% based on potassium cyanide used to begin the synthesis.

REFERENCES

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 - (2) R. E. Selff and B. M. Tolbert, UCRL-1299.
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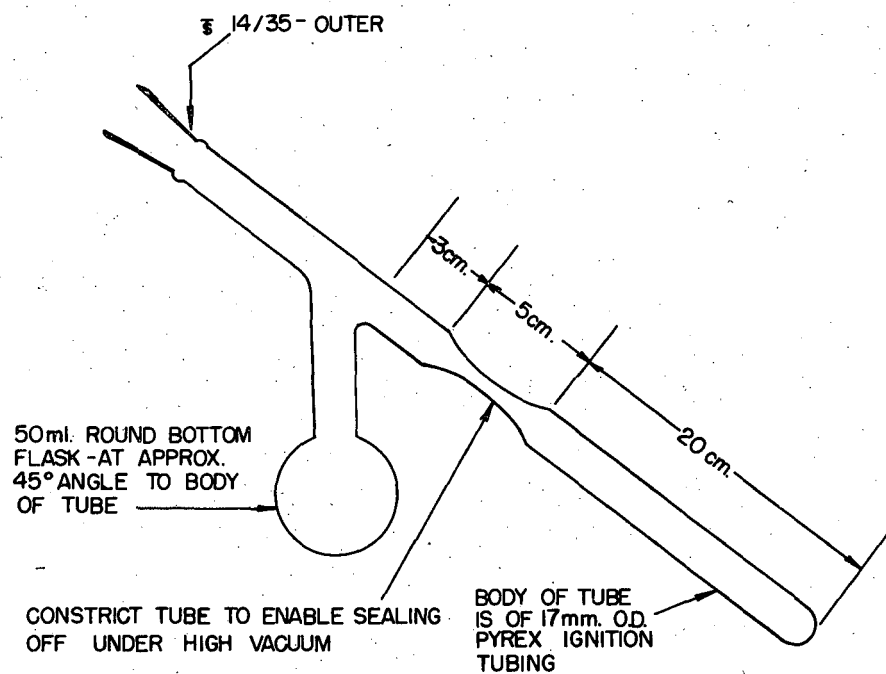


FIGURE 1 - REACTION TUBE FOR SYNTHESIS OF POTASSIUM CYANIDE (2 MMOLE SCALE)

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