FACULTY RESEARCH EDITION

of

The Savannah State College Bulletin

Published by

The Savannah State College

Volume 21, No. 2 Savannah, Georgia December, 1967

HOWARD JORDAN, JR., *President*

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Synthesis of Kaempferol-2-C^{14}

By

Kamalakar B. Raut

This paper describes a synthesis of chromatographically pure kaempferol-2-C^{14} on a semi-micro scale, using the readily available potassium cyanide-C^{14} as starting radioactive compound. The various steps in the synthesis are adapted to suit requirements of the radioactive compounds formed at the various stages. All the steps of the labelled synthesis were first worked out in trial runs, using non-labeled materials.

Experimental

4-Iodanisole. (I). A solution of 28 ml (0.2 mol) of redistilled anisole in 75 ml. of 95% ethanol was heated to 60° and while stirring, was treated with 50 g. of iodine and 30 g. of mercuric oxide. The iodine in 5 gram portions, and the mercuric oxide in 3 gram portions was added alternately over a period of one hour. After the addition was completed, the solution was filtered, and the alcohol was distilled from the filtrate. The residue, a dark red oil was dissolved in ethyl ether and washed with solutions of sodium thiosulfate and sodium hydroxide, and finally with water. After drying over anhydrous magnesium sulfate, the ether was evaported and the residue distilled under reduced pressure.

Cuprous Cyanide-C^{14}. This was obtained by heating cuprous iodide and potassium cyanide (C^{14}).

Anisonitrile (Nitrile-C^{14}). Cuprous cyanide-C^{14}(2.4 g. specific activity 0.033 mc/mM) and *I* (7.02 grams, 0.03 mole) were heated on an oil bath at 230°C with stirring for two hours. The cooled product was purified by crystallization from ethanol.

Anisic acid (carbonyl-C^{14}) (III). *II* was dissolved in 120 ml. of a 15% potassium hydroxide solution and 40 ml. of methanol. After boiling under reflux for a total of 30 hours the methanol was distilled and the resulting aqueous solution was extracted twice with 15 ml. portions of ethyl ether to remove I and *II*. To the aqueous solution, concentrated hydrochloric acid was added at 70°C. The resulting precipitate was crystallized from ethanol m. p. 184-5°C.

Anisoyl Chloride (Carbonyl-C^{14}) (IV). Thionyl chloride (25 grams) and III (3.04 grams) were refluxed on a water bath for 2 hours. After removing thionyl chloride by vacuum distillation, a low melting solid (IV) was obtained.

Anisaldehyde (Carbonyl^1-C^{14}) (V). The anisoyl chloride was reduced to the corresponding aldehyde by Rosenmund reaction. Anisaldehyde obtained was a pale yellow oil.

The above compound was condensed with 2-hydroxy, 4, 6-dimethoxy acetophenone and the chalcone obtained was converted to Kaempferol-2C^{14} by the usual procedure. The final product obtained was about 0.29 grams. The overall conversion of labeled potassium cyanide into Kaempferol was 3.4%. Products at all stages were purified chromatographically using Magnesol.