THE PREPARATION OF SODIUM ACETATE LABELED WITH RADIOACTIVE CARBON IN THE METHYL GROUP*

BY B. M. TOLBERT

(From the Radiation Laboratory and the Department of Chemistry, University of California, Berkeley)

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The purpose of this paper is to describe the preparation of sodium acetate from methyl iodide1 on a 15 to 25 mm scale by carbonating methyl magnesium iodide.

The several steps of the preparation and yields are as follows:

(1) \[ ^{14} \text{CH}_3 \text{I} + \text{Mg} \rightarrow ^{14} \text{CH}_3 \text{MgI} \quad 100\% \]

(2) \[ ^{14} \text{CH}_3 \text{MgI} + \text{CO}_2 \rightarrow ^{14} \text{CH}_3 \text{CO}_2 \text{MgI} \quad 70-75\% \]

(3) \[ ^{14} \text{CH}_3 \text{CO}_2 \text{MgI} \rightarrow ^{14} \text{CH}_3 \text{CO}_2 \text{H} \quad \]

(4) \[ ^{14} \text{CH}_3 \text{CO}_2 \text{H} \rightarrow ^{14} \text{CH}_3 \text{CO}_2 \text{Na} \quad 100\% \]

Steps (1) and (2) were carried out in an evacuated closed system (Fig. 1). The 150 ml. conical reaction flask, D, containing 50 ml. of dry ether and 0.5 gm. of magnesium turnings, was chilled with liquid nitrogen and 1 ml. (2.28 gm.) of methyl iodide distilled in from storage vessel B. The reaction vessel was closed off and the ether refluxed 1 hour. The reaction flask was then cooled to -20° and carbon dioxide, that had been dried by passing through a spiral immersed in dry ice-acetone and freed of oxygen and nitrogen by condensing with liquid air and evacuating at low pressures, was added from bulb J until a pressure of about 30 cm. was maintained in the system. Stirring was then continued for 10 minutes. The reaction vessel was removed from the line and opened in the hood and the cold (-20° to -50°) Grignard complex decomposed with 15 ml. of 6 N sulfuric acid. After decomposition, an additional 35 ml. of water were added. 5 gm. of silver sulfate were added to precipitate the iodide present. The ether was distilled off and the acetic acid was steam-distilled from the reaction mixture with about 300 ml. of water. This distillate was exactly neutralized with 1 N sodium hydroxide solution with a glass electrode, evaporated to a small volume, filtered, evaporated to dryness, and dried in vacuo at 10^{-3}

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The yield of white anhydrous sodium acetate was 70 to 75 per cent, the titration and weighings agreeing within a few tenths per cent.

In several test runs with inactive methyl iodide, titration of the Grignard solution showed the yield to be 98 to 100 per cent in this step. The silver sulfate is necessary to precipitate the iodide from the reaction mixture; if it is not added, free iodine will distil over and the product will be contaminated with iodine compounds.

**SUMMARY**

Sodium acetate labeled with $^{14}C$ in the methyl group has been prepared on a 15 to 25 mm scale in 70 to 75 per cent yield by carbonating labeled methyl magnesium iodide in a vacuum system.
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