Title
NOTES ON THE PREPARATION OF HYDROGEN CYANIDE-C14 FROM BaC14O3 II

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NOTES ON THE PREPARATION OF HYDROGEN CYANIDE-$^{14}$
FROM $\text{BaCl}_{2}$O$_{3}$. II.

R. M. Lemmon

November 21, 1952
NOTES ON THE PREPARATION OF HYDROGEN CYANIDE-$^{14}C$

FROM $^{14}C_{12}O_3$. II.

R. M. Lemmon

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November 21, 1952

ABSTRACT

Experimental details are presented for the preparation of hydrogen cyanide-$^{14}C$ from barium carbonate on a 5-millimole scale and with yields of approximately 90 percent.

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NOTES ON THE PREPARATION OF HYDROGEN CYANIDE-C\(^{14}\)
FROM BaC\(^{14}\)O\(_3\). II.

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An earlier report (UCRL-1299) describes the preparation of hydrogen cyanide-C\(^{14}\) from barium carbonate-C\(^{14}\) by the reduction of CO\(_2\) in a bomb tube in the presence of potassium and ammonia. Since that time we have found it more convenient to use the method of McCarter\(^{(1)}\) to obtain the labeled cyanide. By using a modification of this method, we have found it possible to carry out the reaction on a 5-millimole scale and to obtain yields of 87-93%.

EXPERIMENTAL

Five millimoles of C\(^{14}\)-labeled potassium carbonate was prepared from the same amount of Oak Ridge barium carbonate by absorption of the C\(^{14}\)O\(_2\) in 2 cc of a 5.5 N potassium hydroxide solution, followed by freezing the solution and evaporating it to dryness under high vacuum. The powdered carbonate was thoroughly mixed with 5 g of zinc dust and placed in a Corr's size 6A porcelain boat which was then inserted into a 700 x 15 mm quartz tube and placed next to a loose ball of iron wire in the middle of the tube. Anhydrous ammonia was allowed to flow through the tube so that it passed first over the wire and then over the boat. From the far end of the quartz tube an attached glass tube led under the surface of a N NaOH solution which served to trap any cyanide and to indicate the speed of passage of the ammonia. The quartz tube was then heated by an electric furnace to a temperature of 650\(^{\circ}\) (measured inside the quartz tube by a thermocouple). The tube was maintained at this temperature under a constant stream of ammonia (about 3-4 bubbles per second) for 10-12 hours.
After the quartz tube had cooled, the porcelain boat was transferred to a two-necked round bottom flask used to generate the hydrogen cyanide. The hydroxide solution which was at the end of the quartz tube, as well as washings from the tube itself, were added to the flask. The flask was connected to a dropping funnel and, through a Liebig condenser, to a gas washing bottle filled with sodium hydroxide solution. Excess dilute sulfuric acid was slowly added to the flask from the funnel, and the contents of the flask were boiled for about 10 minutes to insure the evolution of all hydrogen cyanide. The yields of cyanide were determined (1) by titrating a small aliquot portion with standard silver nitrate solution in the presence of iodide ion and a small excess of ammonia and (2) from the specific activity of the labeled cyanide obtained after evaporating the cyanide solution to dryness. The values for the yields were always within 2% of agreement.

SUMMARY

Experimental details are presented for the preparation of hydrogen cyanide-$^{14}C$ from barium carbonate on a 5-millimole scale and with yields of approximately 90 percent.

REFERENCE