

with radioactive carbon-14. Jeanes²⁾ has recently reported a convenient synthesis of sodium cyanide directly from barium carbonate. In his synthesis, barium carbonate, zinc dust and sodium were heated at 650° in a stream of ammonia which had been passed over hot iron; and it was claimed that the yield of sodium cyanide was quantitative. This method appeared very attractive, involving only a simple apparatus and eliminating the need for intermediate preparation of carbon dioxide or alkali carbonate. In our hands, however, it gave yields ranging only from 63 to 65%, in spite of repeated trials closely following Jeanes's procedure. We have now found that the use of potassium instead of sodium gives invariably 90 to 100% yields of potassium cyanide.

Our procedure, which is essentially similar to that of Jeanes²⁾, is as follows: An intimate mixture of 200 mg. of barium carbonate and 1 g. of zinc dust is placed in a porcelain boat together with 0.40 g. of potassium cut in small pieces. The boat is inserted into a quartz tube (700 mm. in length and 17 mm. inside diameter) until it touches a loosely wound prolate ball of iron wire (10 g., 0.3 mm. in diameter), which has been placed beforehand at the middle of the tube. Anhydrous ammonia is introduced into the tube at the end nearest the iron wire, and the tube is heated at 650° for four hours. It is then allowed to cool while ammonia is being passed through it. Subsequently, the product is worked up according to the usual procedure: hydrogen cyanide is distilled by heating the reaction mixture with dilute sulfuric acid and is absorbed in 1N alkali hydroxide. Yields were estimated by titration with a standard silver nitrate solution.

Typical results are given in the accompanying table, which also includes the results of experiments without zinc dust. Zinc does not seem to be essential for the preparation, but in its presence the highest yield of cyanide is secured.

A Note on the Preparation of Alkali Cyanide from Barium Carbonate

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Numerous methods¹⁾ have hitherto been described of the preparation of alkali cyanide- C^{14} from radioactive carbon dioxide or carbonate, because that cyanide is one of the most important materials for the synthesis of organic compounds labelled with carbon-14. No available methods are, however, satisfactory in every respect, for it is desirable that the apparatus and operation should be simple and the yield high on a micro or small-scale preparation suitable for work

EXPERIMENTAL CONDITIONS AND YIELDS
OF ALKALI CYANIDE

No.	BaCO ₃ (mg.)	Zn (g.)	Alkali metal (g.)	Yield based on BaCO ₃ (%)
1	200	1.0	Na 0.29	63.3
2	202	1.0	Na 0.28	64.5
3	201	1.0	K 0.40	100.0
4	200	1.0	K 0.35	ca. 93
5	201	0	K 0.41	83.2
6	200	0	K 0.38	82.2
7	199	0	Na 0.24	61.7

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1) See, e.g., F. L. J. Sixma, H. Hendriks, K. Helle, U. Hollstein and R. Van Ling, *Rec. trav. chim.*, **73**, 161 (1954), in which are summarized the available methods.

2) J. K. Jeanes, *Science*, **118**, 719 (1953).

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