Title

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ABSTRACT

Experimental details are presented on the preparation of hydrogen cyanide-C^{14} from barium carbonate by reduction with potassium in the presence of ammonia (procedure of Cramer and Kistiakowsky as modified by Loftfield).

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

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Hydrogen cyanide has been routinely prepared from barium carbonate-C^{14} by the reduction of CO_2 with potassium in the presence of ammonia. The procedure described here is essentially the method of Loftfield(1,2) except that dilute sulfuric acid is used to generate the hydrogen cyanide. No difficulties were encountered in the use of this method, as has been reported by other workers, and yields of 80-90% were obtained.

EXPERIMENTAL

Clean potassium (1.8 g.) was cut into small chunks washed with xylene, thiophene free benzene and finally ether and dropped into the bulb of a reaction vessel (see Figure 1). The vessel was evacuate to about 25 microns pressure through a flexible tygon tubing and the potassium distilled to the bottom of the tube. Then by alternately heating and cooling this distilled potassium, a mirror was formed in the ignition tube.

One mmole of barium carbonate (395 mg.) was converted to CO_2 with sulfuric acid(3) and the carbon dried by passing through a Dry Ice-cooled spiral. This CO_2 and 4 mmoles of dry ammonium were distilled into the bomb

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tube which was then sealed off at the constriction. It is important that the constriction is not too thick walled or too big a bore so that excess strains may be avoided in this sealing operation.

The sealed tube was placed in an iron pipe capped at both ends and fitted with a small hole in one end and a thermocouple lead. The thermocouple was wrapped around the glass tube so it was in contact with the glass-metal wall. This unit was placed in a furnace heated to 150-800°. In a few minutes an equilibrium temperature of 590-620°C was established in the bomb and the furnace was held at this temperature. Typical heating data is given in Table I.

TABLE I

Time	Furnace Temperature °C	Bomb Temperature °C
0 min	824	25
1	524	124
2	542	306
3	565	408
4	587	490
5	598	542
6	602	568
7	615	590
8	624	598
9	624	614
10	624	618
11	620	618
12	614	618
13	614	614
14	611	611
15	618	612
16	621	614
17	624	618
18	628	620
19	628	620
20	622	620
21 bomb off		

After a total of 20 minutes of heating, including the warming period, the iron tube was removed and quickly cooled with a fine spray of water. The reaction tube was removed, cooled in liquid nitrogen and opened with a small oxygen-gas flame. The reaction tube was then connected by a short length of tygon tubing with a dropping funnel of the type used in the CO_2 generation containing 6 N sulfuric acid. This unit was connected to the vacuum line and the dilute acid added slowly to decompose the excess potassium and generate the hydrogen cyanide. The $HC^{14}N$ was collected in a trap cooled in liquid nitrogen. One and two millimole runs gave comparable results.

SUMMARY

Experimental details are presented on the preparation of hydrogen cyanide-C^{14} from barium carbonate by reduction with potassium in the presence of ammonia (procedure of Cramer and Kistiakowsky as modified by Loftfield).

REFERENCES

(1) R. D. Cramer and G. B. Kistiakowsky, J. Biol. Chem., 137, 547 (1941).

(2) R. B. Loftfield, Nucleonics, 1, No. 3, 54 (1947).

(3) See Figure 72 "Isotopic Carbon", M. Calvin, et.al., John Wiley & Sons, New York, New York (1949) p. 179.