NOTES

SYNTHESIS OF ACETYLENE-$^{13}C_2$

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SUMMARY

The preparation of acetylene-$^{13}C_2$ from calcium and amorphous carbon-13 by way of the intermediate calcium carbide-$^{13}C_2$ is described.

Key Words: Acetylene-$^{13}C_2$, calcium carbide-$^{13}C_2$, carbon-^{13}C

An efficient procedure for the preparation of acetylene-$^{13}C_2$ has been developed. This procedure is based on the historical method for the synthesis of calcium carbide from amorphous carbon,[1] rather than from barium carbonate.[2] Advantages of this method are that it is efficient, simple, and produces good yields. The cost for preparing enriched acetylene is currently at least a factor of five less than purchasing it directly. Since a wide variety of carbon-13 enrichments is commercially available for amorphous carbon, a variety of acetylene enrichments may be prepared.

The preparation of $Ca^{13}C_2$ from calcium and amorphous carbon-13 utilizes a steel tube, three inches long and three-eighths of an inch in diameter, threaded at both ends and fitted with caps. After 0.15 grams of amorphous carbon-13 (Prochem) are placed into the tube, it is packed tightly with approximately one gram of reagent grade calcium metal in the form of one-eighth inch shot. The tube is then capped and shaken to insure mixture of carbon-13 and calcium metal. The reaction vessel is then heated with a torch, with the tube maintaining a bright orange color for approximately five minutes. The tube containing calcium carbide-$^{13}C_2$ is then allowed to cool.

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Preparation of the acetylene from the calcium carbide is accomplished utilizing a 500 ml 3-neck round bottom flask equipped with a magnetic stirring bar, a ground glass stopper, an addition funnel containing water, and an adapter connected to a high vacuum system equipped with two adjacent U traps. The first U trap is immersed in an ethanol bath cooled to -100°C with liquid nitrogen and is used to separate the water from the generated acetylene. The second trap is immersed in a liquid nitrogen bath for the collection of the acetylene-$^{13}C_2$.

The tube containing $Ca^{13}C_2$ is placed inside the flask after the caps are removed. The tube is positioned such that one of the open ends of the tube is directly below the outlet of the addition funnel, thus enabling the water to drip directly onto the calcium carbide. A small portion of glass wool aids in maintaining the correct position of the tube in the flask. Water is slowly added from the addition funnel to the reaction vessel until the steel tube is completely submerged. The mixture is briefly stirred until the generation of acetylene-$^{13}C_2$ is complete. The acetylene-$^{13}C_2$ is collected in the second U trap of the vacuum system.

The yield, based on the acetylene produced, is usually well over seventy-five percent. Overall yields above ninety percent have been obtained. This procedure has been routinely used for the preparation of small amounts of enriched acetylene.

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[1]Brauer, Georg, Ed., Handbook of Preparative Inorganic Chemistry, volume 1, Academic Press (London, 1967), pages 943-946. See also references cited therein.

[2]Cox, J.D. and R.J. Warne, J. Chem. Soc., 1893 (1951).