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L'ÉNERGIE ATOMIQUE DU CANADA LIMITÉE

A TECHNIQUE FOR THE PREPARATION OF CARBON-14 TARGETS FOR NUCLEAR PHYSICS EXPERIMENTS

Technique permettant de préparer des cibles de carbone 14 pour les expériences en physique nucléaire

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Résumé

On a développé une technique permettant de préparer de l'acétylène à partir du carbonate de baryum (^{14}C) et on a mis au point une méthode pour craquer l'acétylène afin d'obtenir des films de carbone 14 de grande pureté.

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ABSTRACT

A technique has been developed for the preparation of acetylene from barium carbonate (^{14}C). A method of cracking the acetylene to produce high purity carbon-14 films has also been developed.

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INTRODUCTION

The radioisotope carbon-14 can be used in nuclear physics experiments as the projectile (accelerated ion)[1,2] or as the target, in nuclear physics experiments. Recently, several laboratories have shown a renewed interest in backed or self-supporting carbon-14 films suitable for targets in nuclear reaction studies.

Conventional methods of preparing carbon isotopic foils consist of thermal[3] and radiofrequency[4] cracking of methyl iodide. These methods produce targets with substantial quantities of iodine contamination. From the method described in this paper, based on acetylene, which is prepared directly from barium carbonate, one can produce uncontaminated carbon-14 films having greater isotopic purity. The radioisotope carbon-14 can be obtained commercially* as the chemical compound $Ba^{14}CO_3$ with an abundance of > 99%. One could fabricate films by preparing elemental carbon from the carbonate and evaporating the carbon by electron bombardment. However, this could entail working with gram quantities of the radioactive isotope. In our method small quantities (50 mg) of the barium carbonate are used to prepare the acetylene and the carbon films.

PREPARATION OF THE ACETYLENE

Acetylene can be prepared from barium carbonate by heating the compound with lithium metal to approximately 600°C to form lithium carbide[5] and then reacting the carbide with water. We have found that, for our purpose, reacting the barium carbonate with magnesium[6,7] to form barium carbide and then reacting the carbide with water gives a better yield of acetylene. A quantity of 50 mg of $Ba^{14}CO_3$ (with an activity of 0.297 mCi/mg (2.9×10^3 Bq), representing a total of 14.85 mCi (1.49×10^5 Bq)) is weighed and transferred to a stainless steel reaction vessel (See Figure 1). Magnesium metal filings are cleaned by immersion in dilute nitric acid followed by several rinses in distilled water and in alcohol. The filings are then dried in an oven. Magnesium (300 mg) is weighed and transferred to the reaction vessel. The vessel is evacuated

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* Atomic Energy of Canada Limited, Commercial Products, P.O. Box 6300, Ottawa, Ontario, Canada.

to a pressure of <0.1 Pa, and argon is introduced to a pressure of ~50 kPa. The carbonate and magnesium mixture is heated to approximately 800°C for five minutes. It is cooled to room temperature and water is leaked into the vessel (approximately 5 cm^3) by opening valve 3. The vessel is cooled to liquid nitrogen temperature to trap the acetylene, evacuated to remove hydrogen and then isolated by closing valve 2. The acetylene collector vessel (B) is evacuated through valves 1 and 4 and cooled to liquid nitrogen temperature. After closing valve 4, the reaction vessel is warmed to -50°C (in a mixture of methanol and dry ice). The reaction vessel valve 2 is opened and the acetylene in the collector vessel is transferred to the cracking apparatus shown schematically in Figure 2.

PREPARATION OF CARBON-14 FILMS

The radiofrequency cracking of acetylene is of interest in producing carbon-14 foils because small volumes of gas are used and the carbon-14 deposits predominantly on the two parallel surfaces of the electrodes.

The cracking apparatus consists of two electrodes set in Lexan* with only the 24.5 mm diameter faces of the electrodes exposed in vacuum. The vacuum seal is accomplished by an O-ring at the back of the electrode face as shown in Figure 2. Sodium chloride is evaporated to a thickness of 50 $\mu g/cm^2$ on two discs 25 mm in diameter fabricated from chrome-plated brass plates. The discs are placed on the electrode faces and the two sections containing the electrodes are fitted into a glass cylinder with an O-ring at each end to form the vacuum, or reaction, chamber. The system is evacuated by opening valve 4, to a pressure of < 0.1 Pa. The acetylene collector vessel (B) is cooled to -50°C with an alcohol and dry ice mixture. Acetylene is introduced into the system at a pressure of 133 Pa by opening valve 1 and a radiofrequency discharge is struck between the electrodes by connecting a Tesla coil to them. The pressure decreases to approximately 80 Pa and stabilizes when all the acetylene has been cracked. The system is evacuated and the operation repeated until the desired thickness of film is obtained. One cracking cycle will produce two films of 4 $\mu g/cm^2$ total thickness, the disc connected to the radiofrequency voltage (the upper disc in Figure 2) producing a film slightly thicker than the

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* polycarbonate

grounded one. The system was calibrated by weighing the two discs before and after five cracking cycles. The areal density of the deposit was found to increase linearly with the number of cracking cycles.

RESULTS

Acetylene Yield

An estimate of the efficiency of transferring Ba $^{14}CO_3$ into $^{14}C_2H_2$ was obtained in the following manner. The $^{14}C_2H_2$ yield was estimated by measuring the volume of the Pirani gauge and the reactor vessel and the acetylene pressure registered by the Pirani type gauge. It was deduced that 50 mg of Ba $^{14}CO_3$ yielded 2.86 mg of $^{14}C_2H_2$ and therefore 76% of the original ^{14}C is converted to acetylene. The remaining ^{14}C deposits in the apparatus as other carbon compounds.

Yield of ^{14}C from Cracking Acetylene

From 2.86 mg of $^{14}C_2H_2$ a total of 0.784 mg of ^{14}C was cracked on two discs, indicating that 29% of the ^{14}C in the acetylene was deposited on the target discs. Thus the efficiency of the total process is 22%. The remaining ^{14}C is deposited on the walls of the cracking apparatus.

CONCLUSION

With this method we are able to produce carbon-14 targets with an isotopic purity of > 95%, probably the highest purity achieved. The isotopic purity was determined[8] on two tantalum-backed carbon-14 targets prepared for a collaborative experiment with the A.W. Wright Nuclear Structure Laboratory, Yale University, and the Brookhaven National Laboratory.

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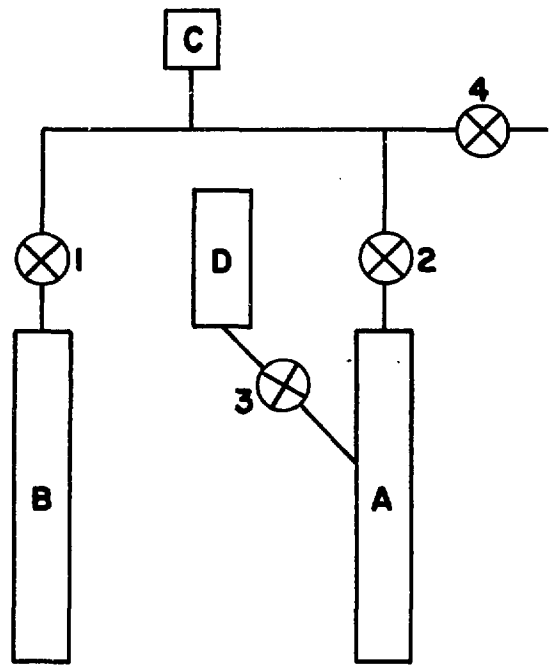

FIGURE 1
A- REACTION VESSEL
B- ACETYLENE COLLECTOR VESSEL
C- PIRANNI TYPE GAUGE
D- WATER VESSEL
1,2,3,4- VALVES

The apparatus for producing acetylene from barium carbonate.

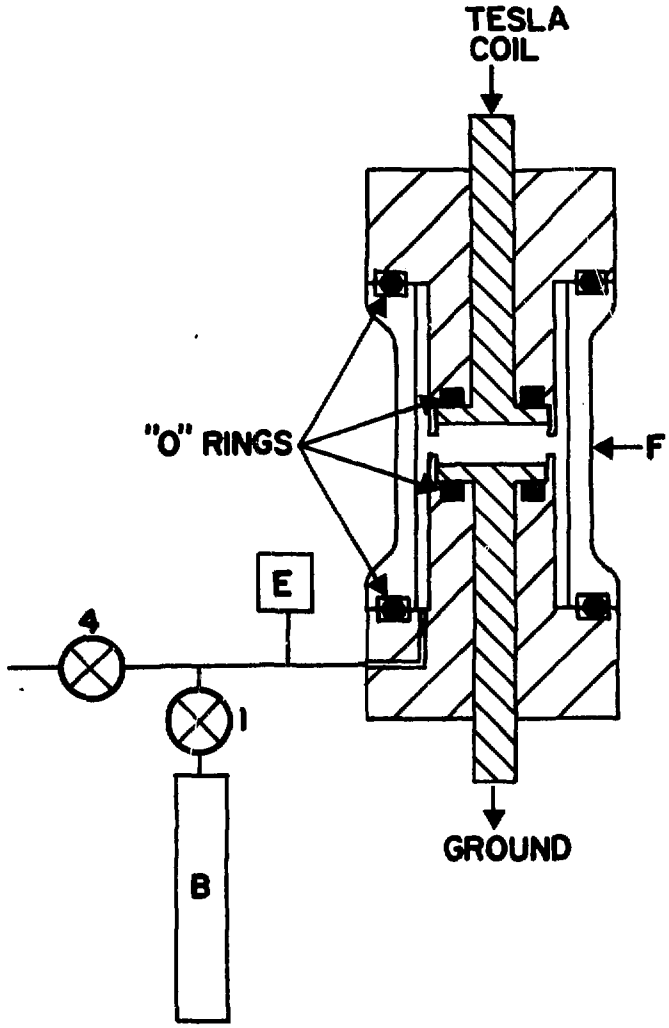

FIGURE 2
B- ACETYLENE COLLECTOR VESSEL
E- PIRANNI TYPE GAUGE
F- GLASS VESSEL
4- VALVE TO VACUUM SYSTEM
1- VALVE TO ACETYLENE COLLECTOR VESSEL

The radiofrequency cracking apparatus with the acetylene collector vessel attached.

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