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A SIMPLE TECHNIQUE FOR CONVERTING CO_2 TO AMS TARGET GRAPHITE

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ABSTRACT. I describe a simple, rapid and inexpensive method for converting CO_2 samples into AMS target graphite. The technique is applicable for both small- and large-scale production facilities. With some modification, the method is suitable for use with very small samples.

INTRODUCTION

The advent of the new generation of high-current, 60-sample sources for accelerator mass spectrometry (AMS) ^{14}C dating necessitates a simple method for converting CO_2 samples into graphite to produce AMS targets. I describe such a technique, suitable for large-scale target production and for preparing very small (a few tens of μg) samples. I developed this method to make graphite from very small samples of CO_2 obtained from ice cores.

Currently, ^{14}C dating using β counting provides higher precision in the >35 ka age range than does AMS ^{14}C dating, opposite to theoretical expectations. The AMS method can count 0.1% of the ^{14}C atoms in a sample in a few minutes, whereas a hypothetical β counter would take eight years. Thus, one would expect AMS to be the method of choice for samples of low ^{14}C content. However, whereas the background/chemical blank is known to better than 1% in most β-counting laboratories, AMS facilities have a high chemical blank (relative to sample size), and rarely can reproduce it to better than 25%. These factors combine to limit the precision with which a very small or very old sample can be dated; it is clearly difficult to date a moderately old 10 μg sample if the uncertainty in the blank is 1 μg (modern).

I describe here research in developing a simpler graphitization system, with a lower, more reproducible blank, capable of mass production and compatible with automation.

METHODOLOGY

The basic difference between this and earlier methods is that the present operation is performed in a sealed Vycor or quartz tube. I use no expensive pressure gauges nor O-ring connectors, which have the potential for leaks over the long term. Figure 1 shows the design of the apparatus. A piece of 6-mm Vycor tube 30 cm long is sealed at the end; a 3.5-cm long piece of 3-mm Vycor tube sealed at one end is loaded with 12 mg Zn powder and dropped to the bottom. With a glassblowing torch, a dimple is made in the side of the 6-mm tube, 7 cm from the bottom. Another similar 3-mm Vycor tube, sealed at the end, is loaded with 0.5-mg Fe powder and slid down the inside of the 6-mm tube to the dimple. The loaded 6-mm tube is then connected to a vacuum line with a Cajon connector and evacuated. The sample of CO_2 (containing 1 mg C – for the amount of reagents given in this example) is frozen into the bottom of the tube with liquid nitrogen. The tube is evacuated and sealed 14 cm from the bottom with a glassblowing torch.

For a small-scale facility that occasionally produces a few samples of target graphite for AMS counting, the tube can be placed overnight in a small electric tube furnace, at 700°C, and the graphite will appear in the bottom of the Fe-containing tube. A simple test for complete reaction, and thus, good vacuum in the tube, is to place the tube back in the tube furnace with its end protruding *ca.* 1 cm. In a good vacuum, excess Zn will rapidly distill to the cold end of the tube and form a mirrored surface.

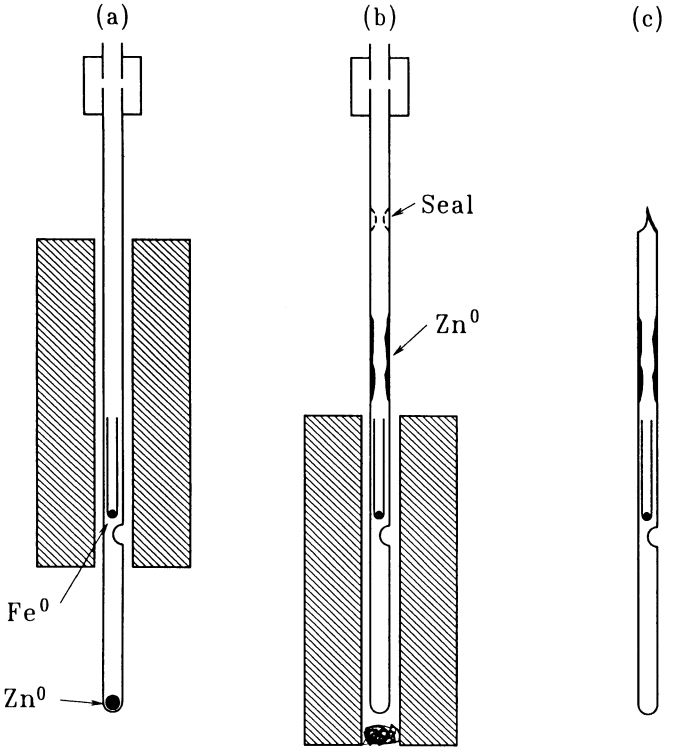

Fig. 1. Apparatus for the conversion of small (4 μmole) CO_2 samples to AMS-target graphite. A. 6-mm Vycor tube is loaded with 0.5 mg Fe and 12 mg Zn and connected to a vacuum system. Upper part of the tube and Fe are baked out in a 725°C tube furnace. B. The lower part of the tube is baked out and the Zn is redistilled to the upper part of the tube to form a high-surface-area Zn mirror, which purifies the Zn; in particular, it decomposes $ZnCO_3$. C. The CO_2 sample is transferred to the tube with liquid nitrogen and the tube sealed. The tube is then placed in a 700°C furnace overnight. The product graphite appears in the bottom of the tube.

For a large-scale operation that processes hundreds of samples per day, one can use an electric furnace into which layers of ceramic tubes have been packed. The sealed, 6-mm Vycor tubes are placed inside the ceramic tubes (with a record kept of exact sample locations) and heated overnight.

Considerable purification can be achieved for "dirty" samples, *e.g.*, bones or sediments, by placing the "Zn end" of the tube in a tube furnace for 1 h, so that the CO_2 (which rapidly reduces to CO) is exposed to hot Zn, which will destroy most impurities before they can react with the Fe.

Development of the Method to Obtain Low Chemical Blanks

The simple, rapid and inexpensive procedure described above produces AMS target graphite using less time, equipment and space than other methods. Because of its simplicity and all-Vycor construction, it lends itself to vacuum line bake-out techniques to reduce the chemical blank, which is important for dating very small or very old samples.

The most important source of chemical blank in this graphite preparation system is Zn powder, which is probably contaminated over its high surface area with $ZnCO_3$ that contains "contemporary carbon", incorporated in the manufacturing process. One can decompose $ZnCO_3$ by heating to >300°C, but if melting occurs (at 420°C), surface area is lost and the reaction proceeds at a much slower rate. For these reasons, I developed the following graphitization procedure:

Zn shot or turnings are used in place of the lower Vycor tube containing the Zn powder. The 6-mm Vycor tube is loaded and evacuated as before. The system is baked out by placing a tube furnace (725°C) over the upper part of the 6-mm tube for 15 min while pumping. The tube furnace is then lowered to bake the Fe and the lower part of the tube (not including the Zn) for a similar amount

of time. The furnace is then removed and the 6-mm tube is allowed to cool. The furnace is then placed over the Zn, which is distilled repeatedly up the tube to form a high surface-area metallic mirror. The furnace is again removed and the 6-mm tube allowed to cool. The CO_2 sample is then transferred to the bottom of the 6-mm tube using liquid nitrogen, and the tube sealed. The upper end of the tube (not including the Fe) is placed in a tube furnace (625°C) for 2 h, so that the hot Zn can react with impurities in the CO_2. The whole tube is then placed in a furnace at 700°C overnight.

The graphite produced using this procedure yields a beam current comparable to that achieved in routine AMS operation, and has a long beam-life. A 1-mg target has, at least, a 4-h life producing a 10 μA current. This is an important consideration for old samples with low ^{14}C content. The chemical blank for a 1-mg sample is 1.7 ± 0.5 μg "modern" equivalent. The uncertainty of the blank (30%) is still surprisingly high, and is under investigation.

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