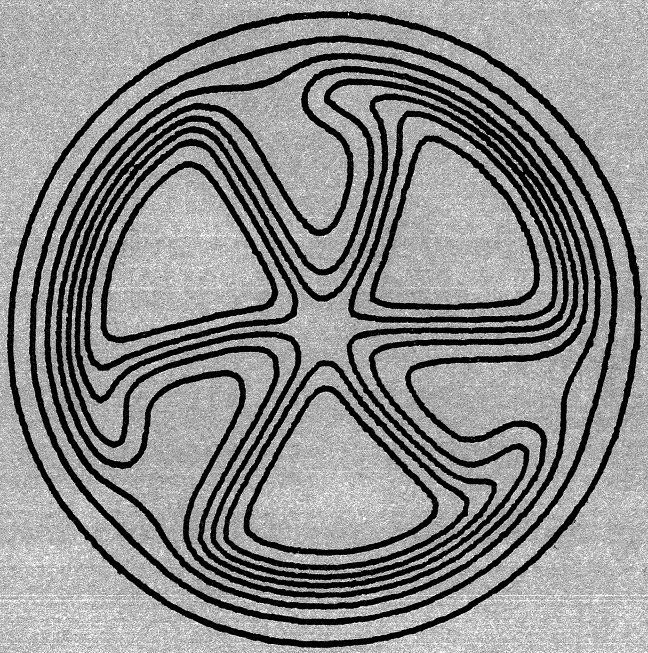
MICHIGAN STATE UNIVERSITY

CYCLOTRON LABORATORY

PREPARATION OF A THICK ^{14}C TARGET

MICHAEL A. CABOT, AARON GALONSKY, R.R. DOERING
and JOSEPH COSTANZO



MSUCL-152 JANUARY 1975

Preparation of a Thick ^{14}C Target*

Michael A. Cabot,+ Aaron Galonsky, R. R. Doering,
Cyclotron Laboratory, Physics Department, Michigan
State University, East Lansing, Michigan 48824

and

Joseph Costanzo
Action Plastic, Charlotte, Michigan 48813

ABSTRACT

A high-temperature epoxy was mixed with ^{14}C powdered graphite and baked at 225°C. The resulting wafer had a ^{14}C thickness of 3.5 mg/cm^2 and was used for $^{14}C(p,n)^{14}N$ measurements with microampere beams from our isochronous cyclotron.

*Supported by the National Science Foundation and the Office of Naval Research.

+Present address: Physics Department, UCLA, Los Angeles, Cal. 90024.

I. INTRODUCTION

Cracking of methyl iodide has been used for some time in the production of thin carbon targets.[1] Other carbon compounds have also been used, and when the carbon was deposited on thin nickel foils, subsequent etching left an unbacked region.[2] The technique has been improved by cracking onto a barium chloride-coated surface from which the carbon film was then floated.[3] With this improvement, unbacked ^{14}C targets were made with 30% efficiency; they were 0.1 to 0.2 mg/cm^2 thick and were small--they were mounted on frames with a 7/32-inch opening.[3]

Targets such as these are too thin and too small for our purpose, which was to measure the angular distribution of the $^{14}C(p,n)^{14}N$ reaction to the three lowest states of ^{14}N with incident protons at several energies between 25 and 50 MeV.

Our neutron time-of-flight system[4] has an unavoidably low efficiency of a few percent. With this efficiency and with many of the cross sections expected to be of order 10 μb/sr, we could not tolerate a target which was unnecessarily thin. In this case "unnecessarily thin" means "much less than the energy spacing of the final states." A 0.2 mg/cm^2 target is 2.5 keV thick to 35 MeV protons, whereas the relevant states of ^{14}N are at 0, 2.31, 3.95, and 4.91 MeV. Clearly, 0.2 mg/cm^2 is unnecessarily thin by a factor of about 100.

For reasons of counting rate, it is also desirable to use a beam of high-intensity. Because neutrons lose no energy and are little attenuated in a target frame, the frame opening must

be large enough to conveniently pass an entire beam of high intensity; otherwise a continuous background will result from proton interactions in the frame. A target backing, such as nickel, even ^{58}Ni, will result in neutron background when bombarded with 25-50 MeV protons, and is to be avoided.

The key to our solution of the problem is the very low Q-value, -18.1 MeV, of the (p,n) reaction on ^{12}C. This suggested that we embed elemental ^{14}C in some type of binder. Of the carbon in a binder, the 1.1% of ^{13}C, with its (p,n) Q-value of -3.00 MeV, would be the main source of background. For our purposes, ^{12}C, H, and ^{16}O (Q(p,n)=-16.8 MeV) would be completely inert.

II. TARGET PREPARATION

Were it not for its low melting point, polystyrene would be an ideal binder in which to embed ^{14}C graphite powder. As it is, however, polystyrene flows at 75°C. This would limit beam intensities to values below 0.1 μA. Instead we chose a high-temperature epoxy, one that begins to deform at approximately 350°C, thus allowing a beam intensity about 20 times that allowed by polystyrene. In order to keep neutron background to a minimum, we chose an epoxy which is free of nitrogen. (Without this restriction, more suitable epoxies would be available.) It is a mixture of a cycloaliphatic epoxy, vinyl cyclohexene dioxide (VCD), and two rather obscure anhydrides, methyl nadic anhydride (MNA) and a pyromellitic dianhydride (PMDA). It is hard (Rockwell Hardness M 110-120) and brittle (rod impact $\gtrsim$0.3 ft-lbs.) and has a coefficient of thermal expansion $\cong 5\times10^{-5}$/°C. In units of molecular weights the epoxy mixture was VCD($C_8H_{12}O_2$) 2 parts, MNA($C_{10}H_{10}O_3$) 1 part, and PMDA ($C_{10}H_2O_6$) 1 part. The atomic percentages of carbon, hydrogen,

and oxygen were 42%, 42%, and 15%, respectively. The completed target contained 2.5 mg ^{14}C in 11 mg epoxy; the ratio of ^{13}C atoms to ^{14}C atoms was 0.0366.

Pure ^{14}C, 4.2 C/g, in powder form was obtained from New England Nuclear. Because of the radiation damage that could result from a spill of the highly mobile powder, the powder was handled under a hood until it was mixed with the epoxy. Experience showed that the proper use of the hood was with its fan off, but with someone ready to turn it on in the event of a spill. That event did not occur. After one bad experience caused by the hood fan we operated with no detectable dispersal of ^{14}C powder. As additional protection against inhaling potentially lethal quantities of the powder, we wore porous paper masks commercially manufactured for this purpose.

Although we practiced with ordinary graphite powder, our first attempt with ^{14}C was a failure; the target was visually non-uniform, having many cracks, holes, and clumps. The ^{14}C grain size was too great. Henceforth, after the powder was weighed out in a small beaker, it was mixed with a solvent (we used ethylene dichloride) and ground in the beaker with a thin glass rod. From a large batch of reasonably-fresh epoxy, the desired amount, as estimated, was added and dissolved into the solvent. The thoroughly-mixed contents of ^{14}C powder, solvent, and epoxy were transferred by eye dropper to a mold consisting of a polished steel ring approximately 1 cm in diameter and several mm high on a 0.001-inch teflon sheet which was on a small flat plate. After

the solvent evaporated the steel ring was carefully removed; the remainder being transferred to an oven and baked at 225°C for one hour. At this point, it was deemed safe to have the oven outside of the hood. A target frame with a hole larger than the ^{14}C-epoxy wafer was placed on the teflon, centered around the wafer. Several fine threads wetted with epoxy were placed between the frame and points on the very edge of the wafer. After another bake at 225°C the frame with thread-mounted target was removed without difficulty from the teflon sheet.

III. RESULTS

Targets of ^{12}C, ^{13}C, and ^{14}C made as described above are shown in Fig. 1. The cracks in the ^{14}C target developed during baking. Presumably, grinding to a finer powder is indicated. The targets in Fig. 1 were all used in a (p,n) experiment before the photograph was taken. With currents generally between 0.2 and 0.5 μ A of protons at 25, 35, and 45 MeV, the ^{14}C target had sustained about 12 μA-hrs, the ^{13}C target about 2 μA-hrs, and the ^{12}C target about 1 μA-hr. In these bombardments, the beam diameter was intentionally expanded to 6 mm in order to reduce the chance of overheating any part of the target.

There was no evidence of beam hitting the aluminum target frame, although the difference in ^{27}Al and ^{12}C (p,n) Q-values would have made neutrons from ^{27}Al prominent in spectra taken with the ^{12}C target. The string mount is certainly an asset in achieving a minimal background. Alternatively, it can be looked upon as a means of conserving an expensive isotope.

The diameters of the ^{14}C target are 9.3 mm and 9.8 mm parallel and perpendicular to the cracks, giving an area of 0.72 cm^2, an average ^{14}C thickness of 3.5 mg/cm^2, and an average ^{14}C-plus-epoxy thickness of 18.8 mg/cm^2. Although the target has a gross appearance of being smooth and uniform, except for the cracks it is relevant to know the ^{14}C thickness over the 6 mm central region actually used in the (p,n) experiment. Perhaps edge or surface effects in the mold did not result in uniform sedimentation of the ^{14}C powder in the epoxy. It should be noted that cross sections are determined only by ^{14}C density; epoxy only adds to energy loss of the proton beam. In order to measure the distribution of ^{14}C only, we scanned the target with a 1/2 mm collimator and a silicon surface-barrier detector, counting the ^{14}C beta-rays. Were it not for self absorption of the beta rays, at each point in the scan the activity would be in direct proportion to the ^{14}C areal mass density. In fact, the full target thickness, 18.8 mg/cm^2, is about 10 mg/cm^2 below the range of a beta ray with the maximum energy, 156 keV. Hence, many electrons from the back regions of the target cannot be counted; the front regions are emphasized. One can imagine unusual areal mass distributions which, through self absorption, produce vastly different radioactivity distributions. However, we counted each side of the target and found a difference of only 5%. We conclude that it is a good approximation to take ^{14}C areal density as proportional to areal radioactivity. With this assumption the result of scans parallel and perpendicular to the cracks is shown in Fig. 2. Clearly the target is thicker in the center, where it reaches a

value of ∿5 mg/cm^2. Over the central 6 mm we used, the average thickness is 4.4 mg/cm^2.

The final assessment of the target is the data produced with it. A sample of these data is in Fig. 3, a time-of-flight spectrum of neutrons produced at 60° to a beam of 25-MeV protons. The current and run time were 0.3 μ A and 14 min. The three final states of interest, the 0-, 2.31-, and 3.95-MeV states of ^{14}N, are clearly resolved.

REFERENCES

1. R. A. Douglas, B. R. Gasten, and Ambuj Mukerji, Can. J. Phys., 34(1956)1097.
2. E. Kashy, R. R. Perry, and J. R. Risser, Nucl. Instr. and Meth. 4(1959)167,
3. J. L. Gallant, Nucl. Instr. and Meth., 102(1972)477.
4. R. K. Jolly, T. M. Amos, A. Galonsky, R. Hinrichs, and R. St. Onge, Phys. Rev. C7(1973)1903.

FIGURE CAPTIONS

Fig. 1.--Photograph of the ^{14}C, ^{13}C, and ^{12}C targets (top to bottom) after use in a $^{14}C(p,n)$ experiment. Each target is about 9.5 mm in diameter and is attached to its frame with epoxy via several fine threads.

Fig. 2.--Mass distributions, as determined by beta-ray counting, of ^{14}C along diameters parallel to (open points) and perpendicular to (solid points) cracks in the target seen topmost in Fig. 1.

Fig. 3.--Neutron time-of-flight spectrum obtained with 25-MeV protons on the ^{14}C target.

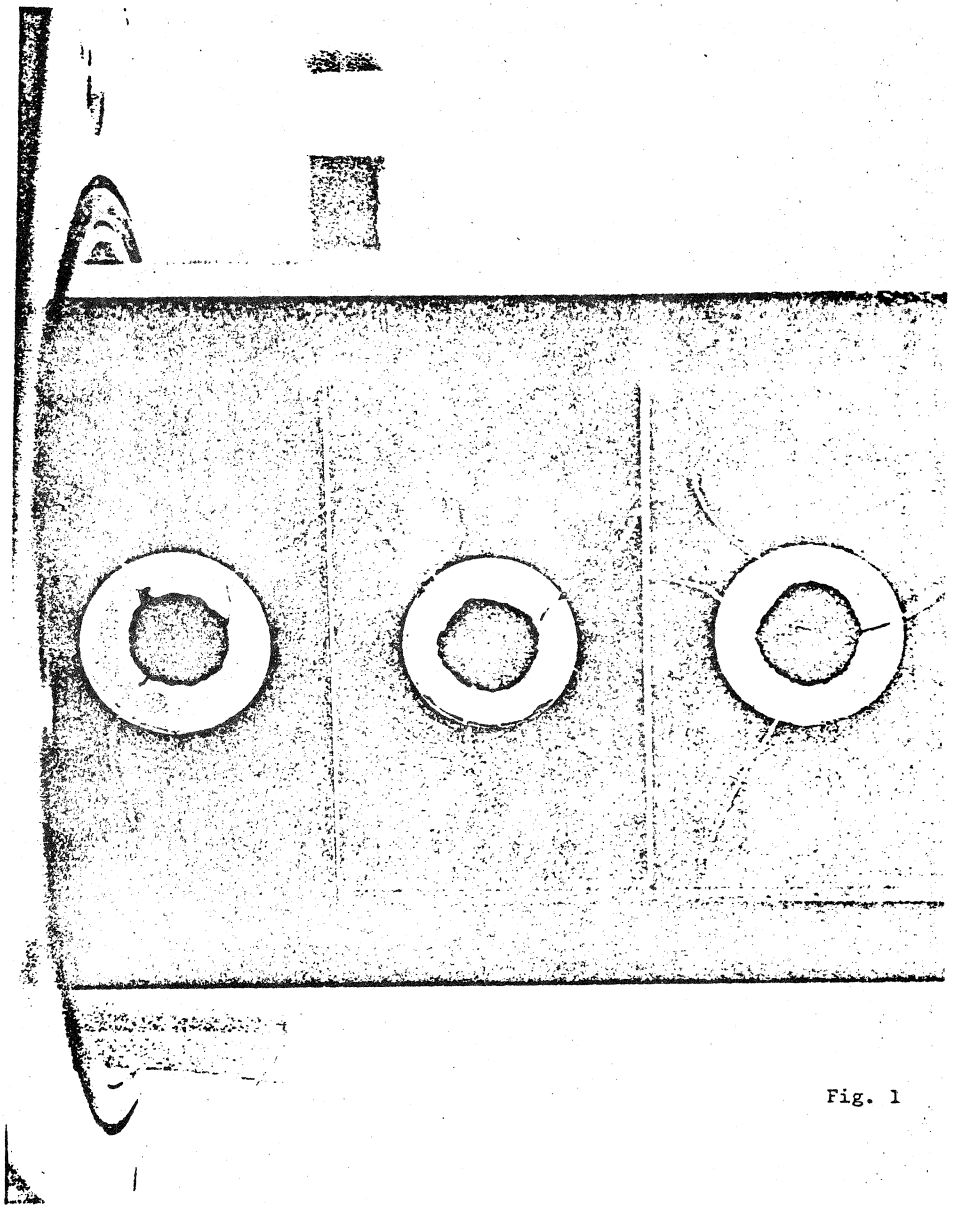


Fig. 1

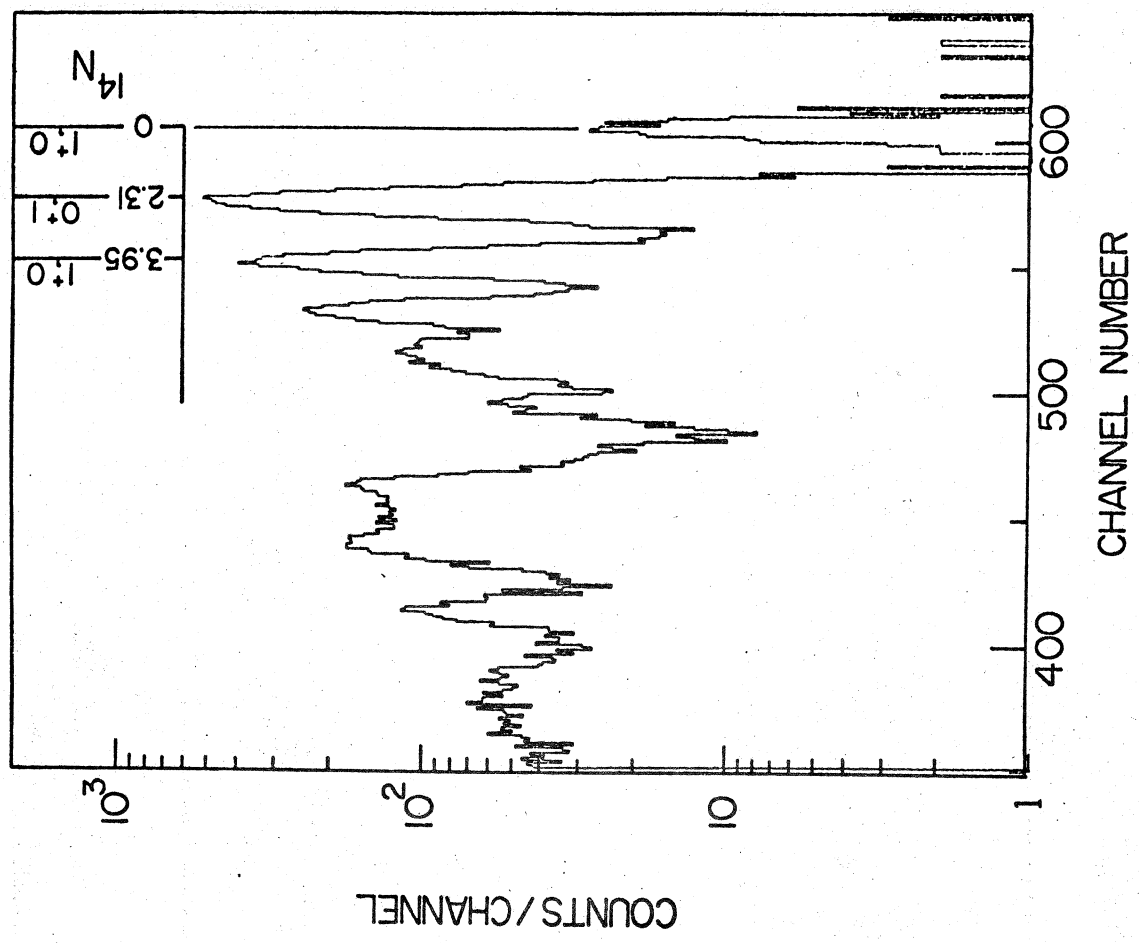

^{14}N
0
0+1
2.31
0+1
3.95
1+0
1+0
600
500
400
CHANNEL NUMBER
10^3
10^2
10
1
COUNTS / CHANNEL

Fig. 3

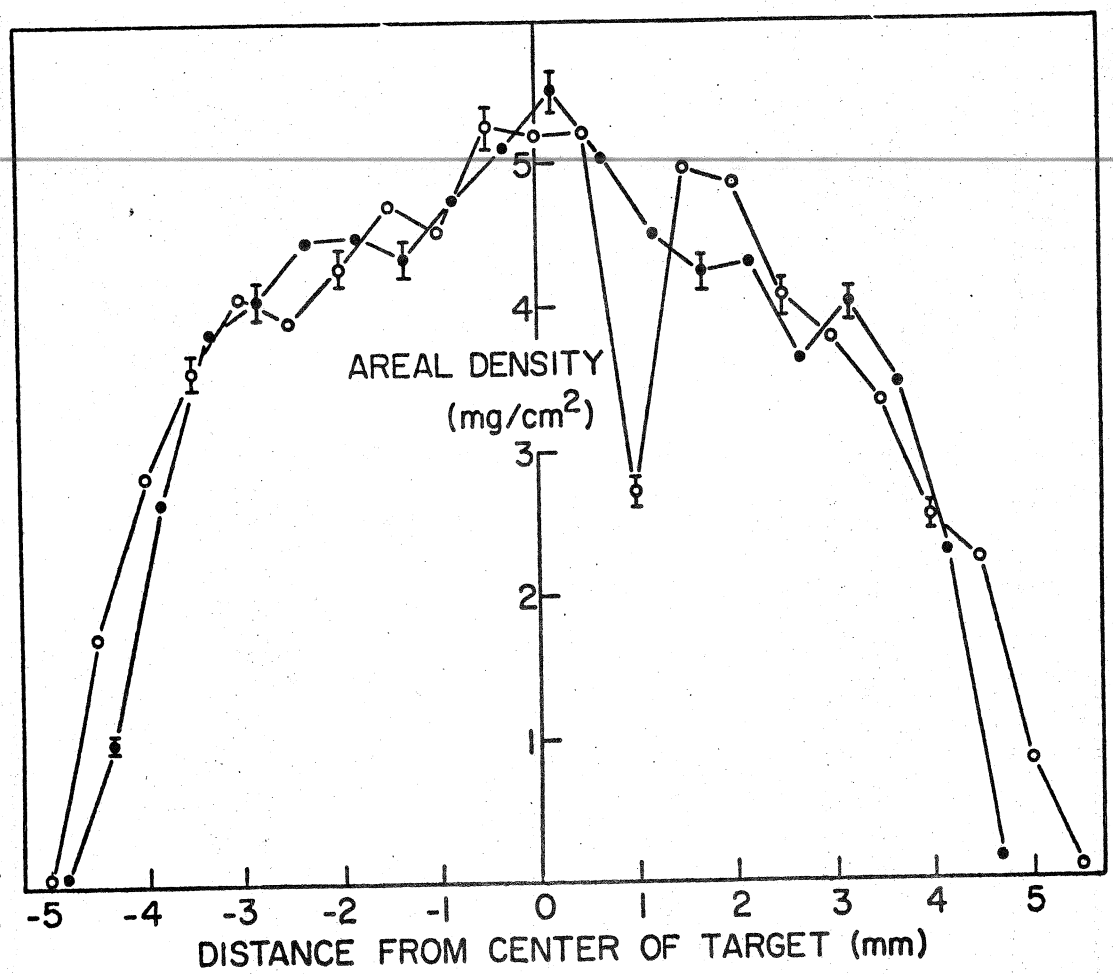

5
4
AREAL DENSITY
(mg/cm^2)
3
2
1
-5
-4
-3
-2
-1
0
1
2
3
4
5
DISTANCE FROM CENTER OF TARGET (mm)

Fig. 2