

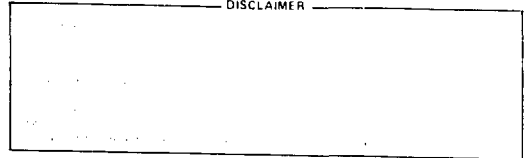
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METHODS TO REDUCE CONTAMINATION IN TARGETS
PREPARED BY VACUUM DEPOSITION

by

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METHODS TO REDUCE CONTAMINATION IN TARGETS

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For some time, both the experimenters and we as target makers have been concerned about the source of impurities which were found in the experimental data obtained with the targets prepared by vacuum vapor deposition. These impurities may have come from the process of producing the target, the separated isotope used in the evaporation, or the contaminants introduced during the experiment. Some impurities such as carbon, oxygen and nitrogen are likely to be introduced into the target during evaporation, due to the residual gases and contaminants such as diffusion pump oil present in the vacuum system. The present study was initiated to determine the source of impurities found in our targets. We would like to mention some of the experimenters who were kind enough to cooperate with us by bringing to our attention these impurities whenever they found them and in addition letting us use some of their data — D. F. Geesaman, W. Henning, M. S. Kaminsky, D. G. Kovar, W. Kutschera, G. C. Morrison, M. Paul and S. J. Sanders.

Our evaporation system, a Varian NRC,¹ is shown schematically in Fig. 1. It uses an 18 inch glass bell jar, a NRC VHS6 diffusion pump having a 2000 liters per second maximum pumping speed, a 400 liters per minute Welch 1397B roughing pump and it uses a large-capacity liquid nitrogen cold trap. The liquid nitrogen cold trap improves the vacuum by about a factor of ten over a refrigeration type trap. A Kronos crystal thickness monitor² was used in all evaporations.

From Deutschmann,³ we find that the number of atoms striking the surface per second is given by the following equation:

$$\gamma = 1/4 n V_a$$

$$\gamma = 3.513 \cdot 10^{22} \frac{P}{(MT)^{1/2}} \text{ cm}^{-2} \text{ sec}^{-1}$$

where M = mass (AMU), T = degrees Kelvin, and P = pressure in mm Hg. For a typical evaporation system at 10^{-6} torr pressure, the number of oxygen atoms striking a given substrate would be 1×10^{14} per square cm per second. A monolayer of oxygen consists of $\sim 8 \times 10^{14}$ molecules per square cm per second. In other words, there is a monolayer of O_2 striking the substrate about every 2 seconds. For a vacuum of 10^{-6} torr, 0.02 micrograms per square cm of oxygen would potentially be deposited on the surface of the substrate per second, if the oxygen sticking probability was unity. It should be pointed out that this amount of oxygen will not all be deposited on the substrate but the above estimate does indicate that there is a possibility of considerable oxidation of the target prepared by vapor deposition in a vacuum of 10^{-6} torr, which is usually considered to be suitable for an evaporation. When an evaporation is made in a vacuum system evacuated with an oil diffusion pump, dissociation of hydrocarbons (from the foil vapor) may increase the carbon contamination problem.

Next are some examples of experimental data obtained with the targets which we have prepared. This gives some idea of both the kind and magnitude of the impurities which we have found. Boron-11 targets were prepared for Morrison et al.⁴ for their experiments of elastic scattering studies of ${}^6\text{Li}$ from ${}^{11}\text{B}$ at the Argonne FN tandem accelerator. It was found that the first set of targets, made some time ago, contained many impurities,

as can be seen in the data shown in Fig. 2. In addition to carbon and oxygen, other impurities were found. A second set of targets were then evaporated in a vacuum of 10^{-6} torr with a high purity sample of ^{11}B , purchased from Eagle Picher.⁵ The data obtained with this new set of targets, shown in Fig. 2, clearly indicate that the impurities other than carbon and oxygen disappear. This observation is attributed to the improved purity of the ^{11}B sample used in the evaporation. The oxygen and carbon contaminants are in part due to the residual gases and oil vapor present in the vacuum chamber during the evaporation. For the new target, the ratio of the intensity of oxygen line to that of the boron line is somewhat reduced while the ratio of the carbon intensity to boron intensity is larger. These results suggest that the new target was produced in both a better and cleaner vacuum.

Next, data taken by S. J. Sanders et al.⁶ is shown in Fig. 3. Again, this experiment was performed at the tandem accelerator using 51 MeV oxygen-16 particles with the Compton spectrometer. The targets were 70 microgram per cm^2 magnesium-24 foils produced jointly by our facility and that of F. Karasek's.⁷ These were the targets which first started us thinking seriously about the source of oxygen and other impurities. The oxygen impurity found in these targets made it difficult for the experimenters to analyze their data. Occasionally foils would be produced which had less oxygen impurity than others but we were not sure of the cause. It was not certain if the decrease in oxygen impurity was a result of the rolling or some other phenomena as the rolled targets were not always the lowest in oxygen content.

Recently, some vanadium targets were produced for Kaminsky's⁸ group for their experiments at the 2 MeV Van de Graaff at Argonne. These targets were prepared using an electron gun to evaporate 50 monolayers thick

vanadium on a $50 \mu\text{g}/\text{cm}^2$ aluminum backing. Figure 4 shows some of the Rutherford back scattering spectra of the targets, obtained with their back scattering spectrometer. In addition to the vanadium and aluminum peaks which are characteristics of the target and substrate, peaks of oxygen, nitrogen and carbon are clearly observable. These impurities were undesirable as the oxygen and carbon peaks appeared in the region of their experimental interest. Furthermore, vanadium targets having the highest possible purity were a necessity because these targets were to be used as standards to calibrate other targets obtained by means other than vacuum vapor deposition. It is believed that the oxygen, nitrogen and carbon impurities were due to the presence of residual gases in the vacuum chambers during the evaporation of the aluminum substrate and on the vanadium target.

The data obtained with the aluminum targets on carbon backing that we prepared for Kaminsky's group⁹ further illustrate the problem of oxygen contamination due to residual gases present in the vacuum system. Three aluminum targets were made in a single evaporation by placing three carbon substrates at distances 7.0, 22.1, and 70.0 cm, respectively, from the source of evaporation. Figure 5 shows the aluminum to oxygen concentration ratio vs. the thickness of the aluminum target. It is seen that this ratio is directly proportional to the aluminum thickness. This result can be explained by the difference in the aluminum arrival rate and a constant arrival rate of oxygen at the substrates. It should be noted that a constant arrival rate of oxygen is merely an indicator of the residual oxygen partial pressure in the evaporator vacuum. It is unlikely, based on Deutschmann's,³ effusion equation that the evaporated aluminum would pick up significant amounts of oxygen as it travelled to the substrate in the vacuum used ($\sim 10^{-6}$ torr). The oxygen contamination must have occurred on the target

surface during evaporation. As aluminum hits the substrate, oxygen is also hitting the substrate, forming an oxide as it is being deposited or the oxygen may just be embedded in the aluminum.

Further studies were made by preparing various aluminum foils at different evaporation rates as well as under different vacuum conditions. It is realized that by having a fast evaporation, one may decrease the uniformity of the evaporated surface. It would be nice to have a vacuum of 10^{-8} or 10^{-9} torr in a system in which there was no back streaming from the diffusion pump. Under these conditions, one could make long time evaporations with many less impurities but we feel that most of us still have less than ideal vacuum systems and the evaporation time is often a determining factor. The aluminum targets were all $50 \mu\text{g}/\text{cm}^2$ thick and were analyzed by the Rutherford back scattering technique using 1.25 MeV protons from the 2 MeV Van de Graaff. In the first group of data shown in Fig. 6, the evaporation was made at 10^{-5} torr, with the evaporation time being 2 minutes and in the second group, the evaporation time was 12 seconds. For the two minute evaporation, one can see the oxygen peak is rather strong while for the 12 second evaporation, it is virtually non-existent. The boron contamination was due to the contamination from a boron evaporation made in the previous run. It is rather significant, we feel, that by having a fast evaporation rate one can eliminate the oxygen background. If one evaporates the targets at 10^{-6} torr pressure, it should be noted that both the carbon and oxygen peak intensities are reduced in comparison with those evaporated at 10^{-5} torr. For the two minute evaporation, the carbon peak is enhanced probably due to the back streaming from the diffusion pump. As could be expected, evaporating for 12 seconds at 2×10^{-7} torr greatly reduces the intensity of both the carbon and oxygen contamination.

Table I points out some significant differences of various evaporation times and pressures. For a constant evaporation time, the carbon to aluminum ratio gets larger as one has a better vacuum. Likewise, at low vacuum pressure and for a short evaporation time, the oxygen contaminant as well as the carbon contaminant becomes much smaller. By having a better vacuum, one increases the carbon contaminant but reduces the oxygen.

The conclusions of the present work are the following. In particular, for targets such as magnesium, aluminum, calcium and other readily oxidized materials, the following points are important. One needs a clean system, the best possible vacuum attainable, and as short an evaporation time as the desired uniformity of a target will permit. One must decide what is important for a particular experiment. A word about the mentioned e-gun type evaporation — normally it takes a relatively long time for this type of evaporation. For such a long evaporation time, a good vacuum is even more of a necessity. The oxygen contamination will become more severe as fewer atoms of the evaporated material strike the substrate per unit time. A very good vacuum will certainly reduce the oxygen contamination. Relatively little developmental work has been done on the cryopump system but it has potential for reducing many contaminants, particularly carbon. There are several problems associated with these systems, but they are not insurmountable. Again, higher purity targets are going to become increasingly more important to the experimenter. More time must be devoted to high purity material, type of evaporation produced and conditions under which we do this evaporation.

1. Varian/NRC 3117 vacuum coating system manufactured by Varian, Palo Alto Vacuum Division, 611 Hansen Way, Palo Alto, CA 94303.
2. Kronos thickness monitor, Model QM321. Distributed by Veeco Instruments Inc., Terminal Drive, Plainview, NY 11803.
3. S. Deutschmann, Scientific Foundations of Vacuum Technique, second edition, edited by J. M. Lafferty (John Wiley and Sons, Inc., NY, 1962), p. 14.
4. G. C. Morrison, D. F. Geesaman, W. Henning, and D. G. Kovar, private communication.
5. Eagle Picher Industries, Inc., Electronics Division, Miami, OK 74354.
6. S. J. Sanders, M. Paul, J. Cseh, D. F. Geesaman, W. Henning, D. G. Kovar, C. Olmer, and J. P. Schiffer, Resonant Behavior of the $^{24}\text{Mg}(^{16}\text{O}, ^{12}\text{C})^{28}\text{Si}$ Reaction, to be published.
7. F. J. Karasek, Rolling of Evaporated Magnesium Isotopes, published in these proceedings.
8. M. S. Kaminsky and S. K. Lam, private communication.
9. S. K. Lam and M. S. Kaminsky, private communication.

Table I. Summary of RBS results on contamination analysis for $50 \mu\text{g}/\text{cm}^2$ Al foils prepared under different conditions.

P (torr)	Time of Evaporation (secs)	$\frac{n_C}{n_{Al}}$ (%)	$\frac{n_N}{n_{Al}}$ (%)	$\frac{n_O}{n_{Al}}$ (%)
1×10^{-5}	12	3	3	4
1×10^{-5}	120	3	12	30
1×10^{-6}	12	6	2	2
1×10^{-6}	120	7	3	3
2×10^{-7}	12	7	2	2

EVAPORATION SYSTEM

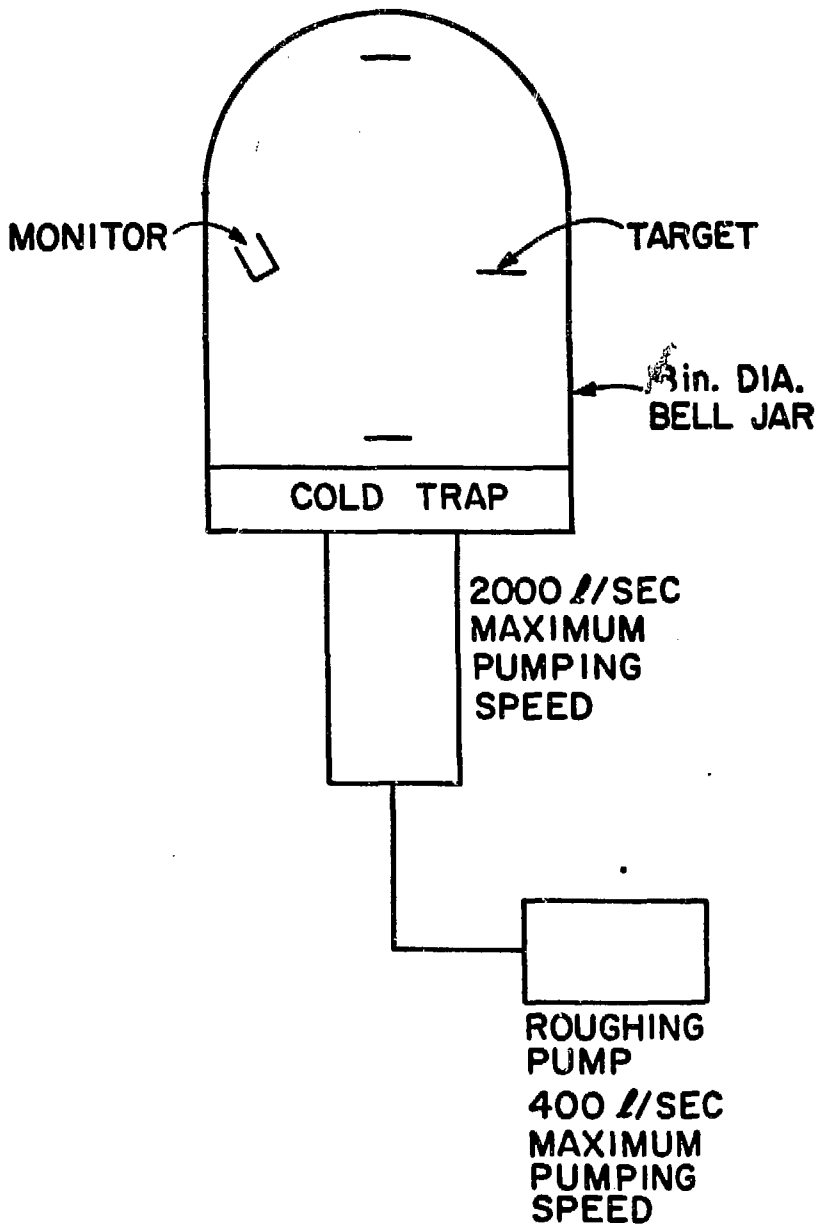


Figure 1

${}^6\text{Li} + {}^{11}\text{B} \rightarrow \text{ELASTIC SCATTERING}$

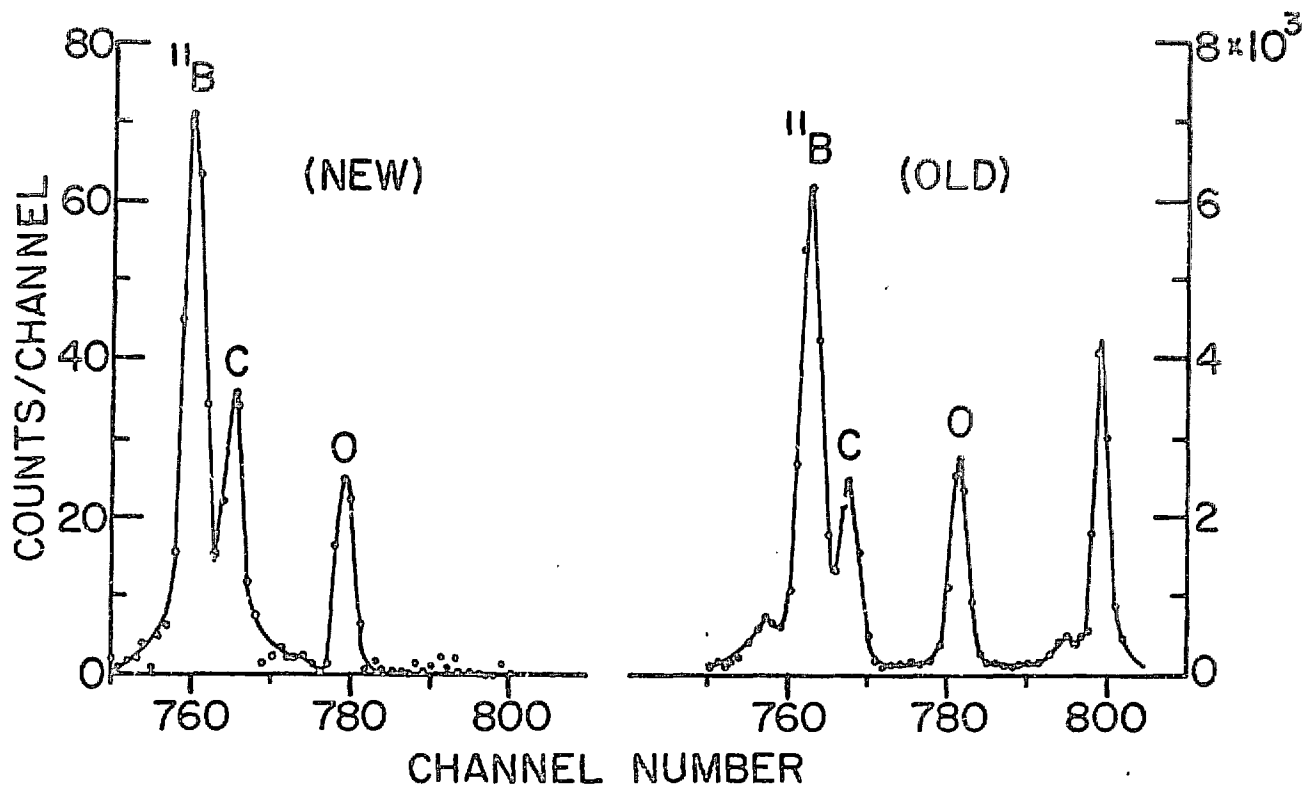


Figure 2

<u>RATIO</u>	<u>OLD</u>	<u>NEW</u>
$\frac{C}{B}$	0.40	0.51
$\frac{O_2}{B}$	0.45	0.36

11/18/75
309-79-539

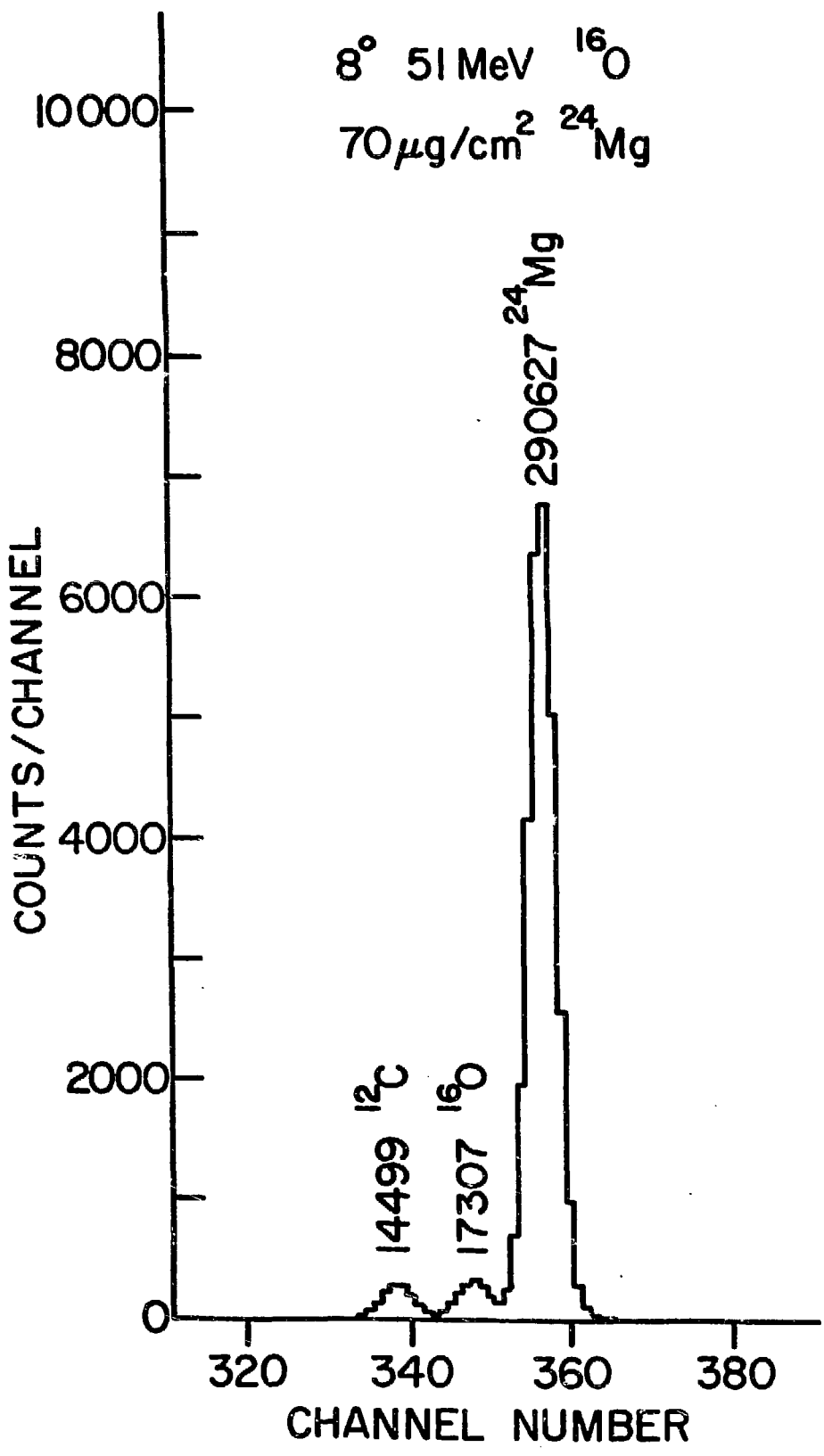


Figure 3

phy. 15.317
309-79-540

2-MeV Van de Graaff, 1.25 MeV PROTONS
50 monolayer VANADIUM on $50 \mu\text{g}/\text{cm}^2$ Al
ELECTRON GUN EVAPORATION

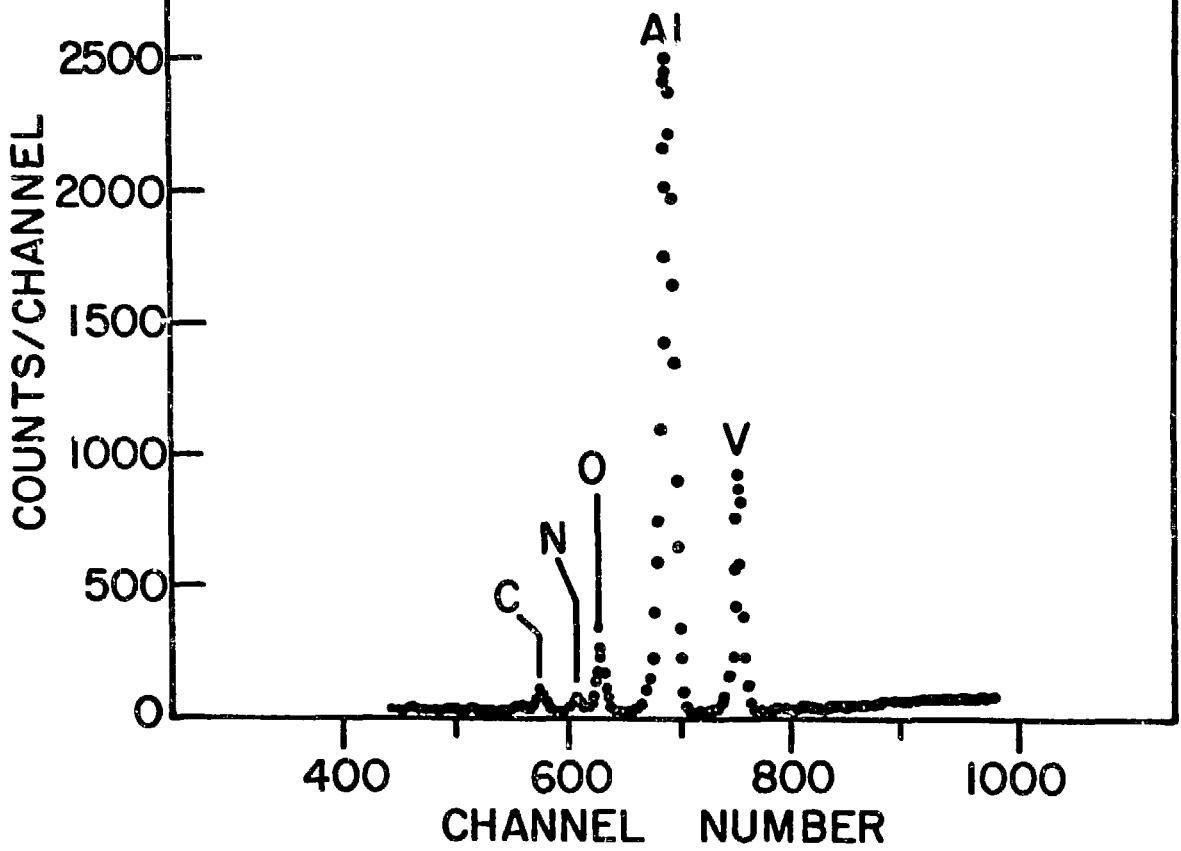


Figure 4

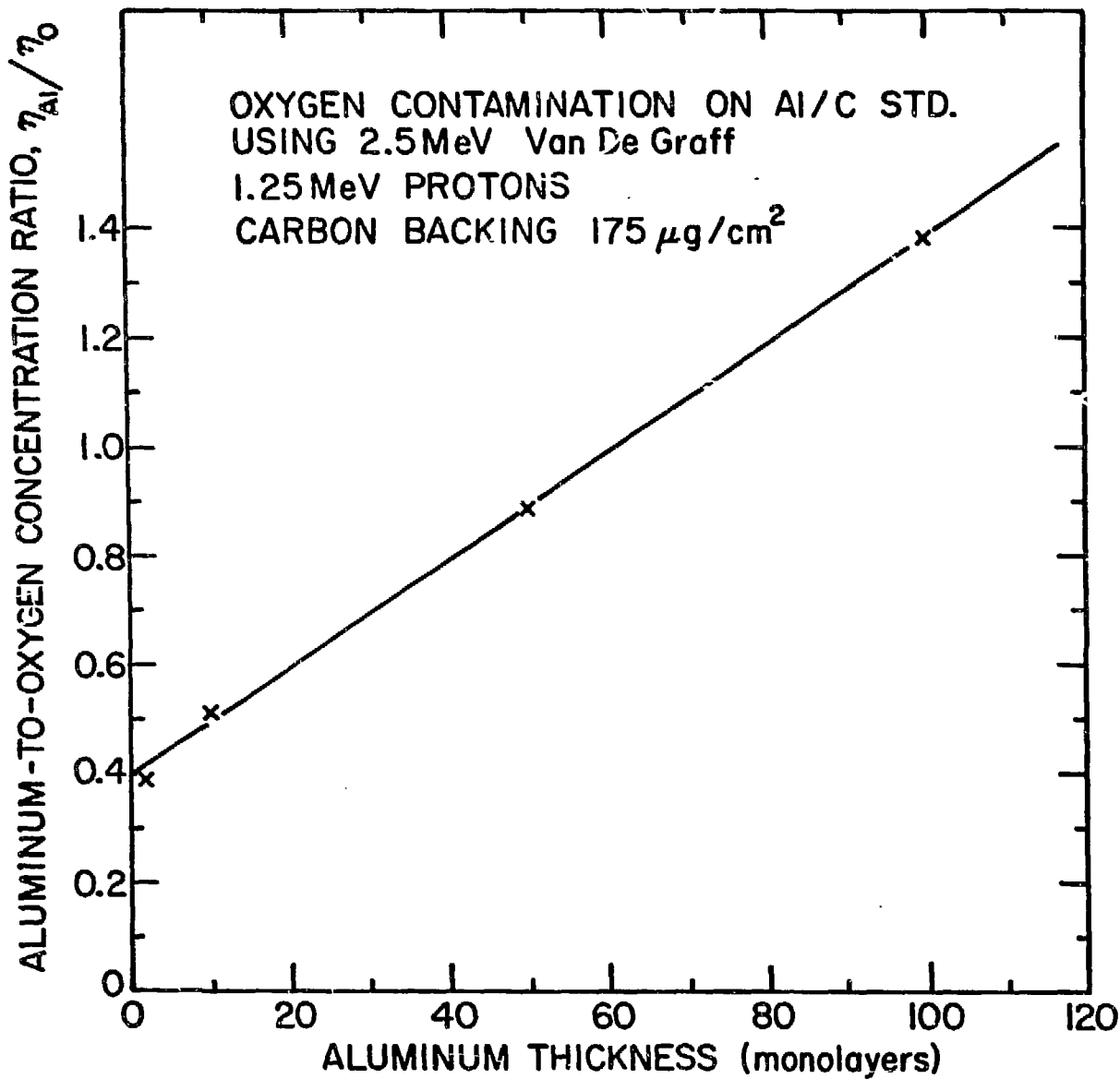


Figure 5

2MeV Van De Graff, 1.25 MeV PROTONS, ALUMINUM $50 \mu\text{g}/\text{cm}^2$

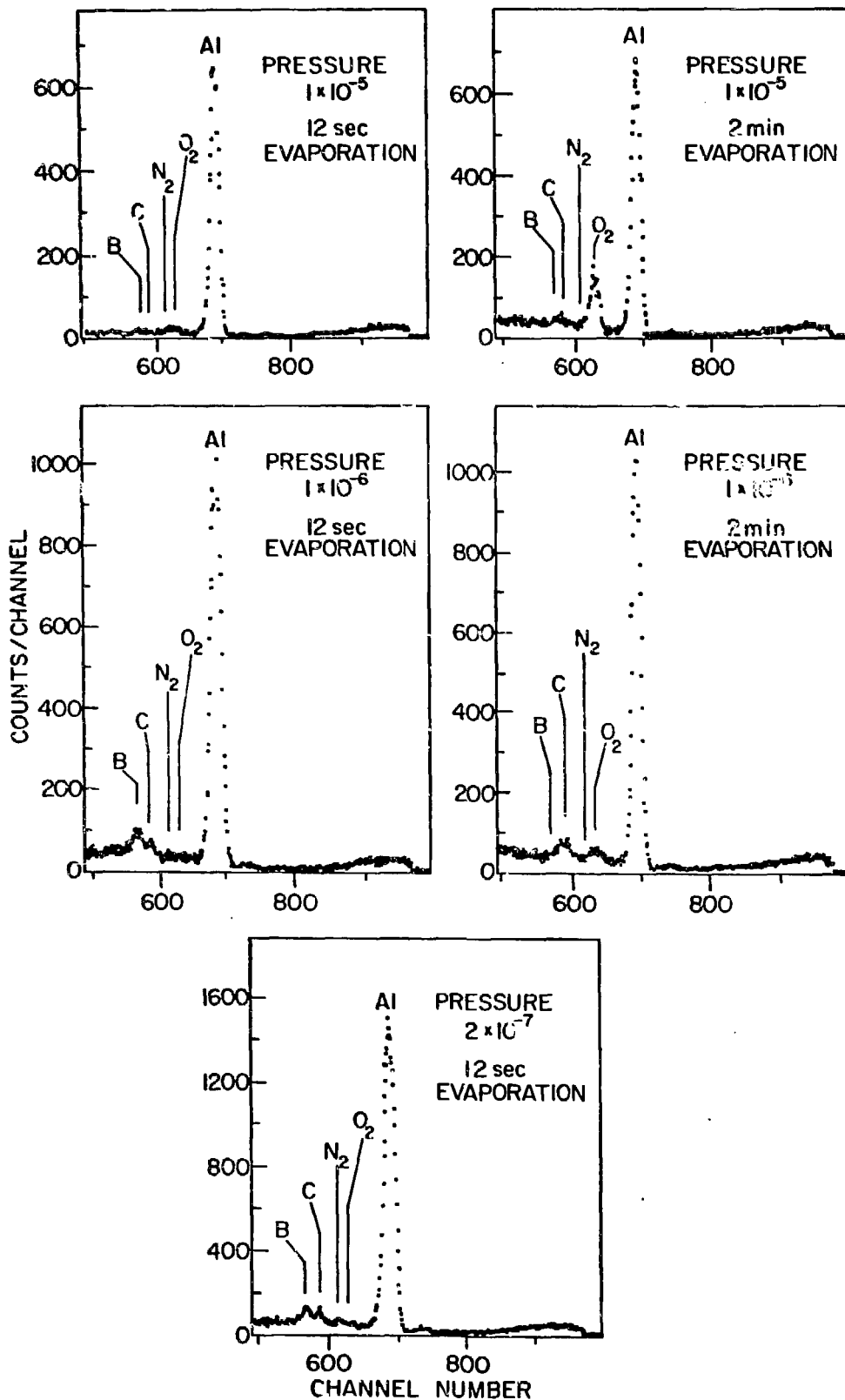


Figure 6

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