

CHAPTER III

EXPERIMENTAL INVESTIGATION

3.1 Sample Preparation

All of the neptunium and americium samples were prepared by electrodeposition⁷. Aluminum foils of 2.5 mg/cm^2 thickness were used as the backing. Figure 3.1 shows the components of the electrolysis cell used in the experiment. The cell consisted of a short glass tube having its bottom edge smoothed off in order that the contact between the glass tube and the aluminum backing which served as cathode be complete. The top of the glass tube was connected to a four-way glass joint by plasticine. The upper vertical section of the glass joint had a platinum wire sealed through the top. The platinum wire which extended into the solution to within 1-2 mm above the cathode, served as the anode. The volume of the electrolytic solution used was very small, ranging from 0.05-0.1 ml depending on the diameter of the glass tube. One of the horizontal arms of the glass joint was connected to a syringe, while the other arm was connected to a U-tube filled with water. It was necessary

⁷ J.E. Evans, R.W. Longheed, M.S. Coops, R.W. Hoff and E.K. Hulet, Nucl. Instr. and Meth., 102 (1972), 398.

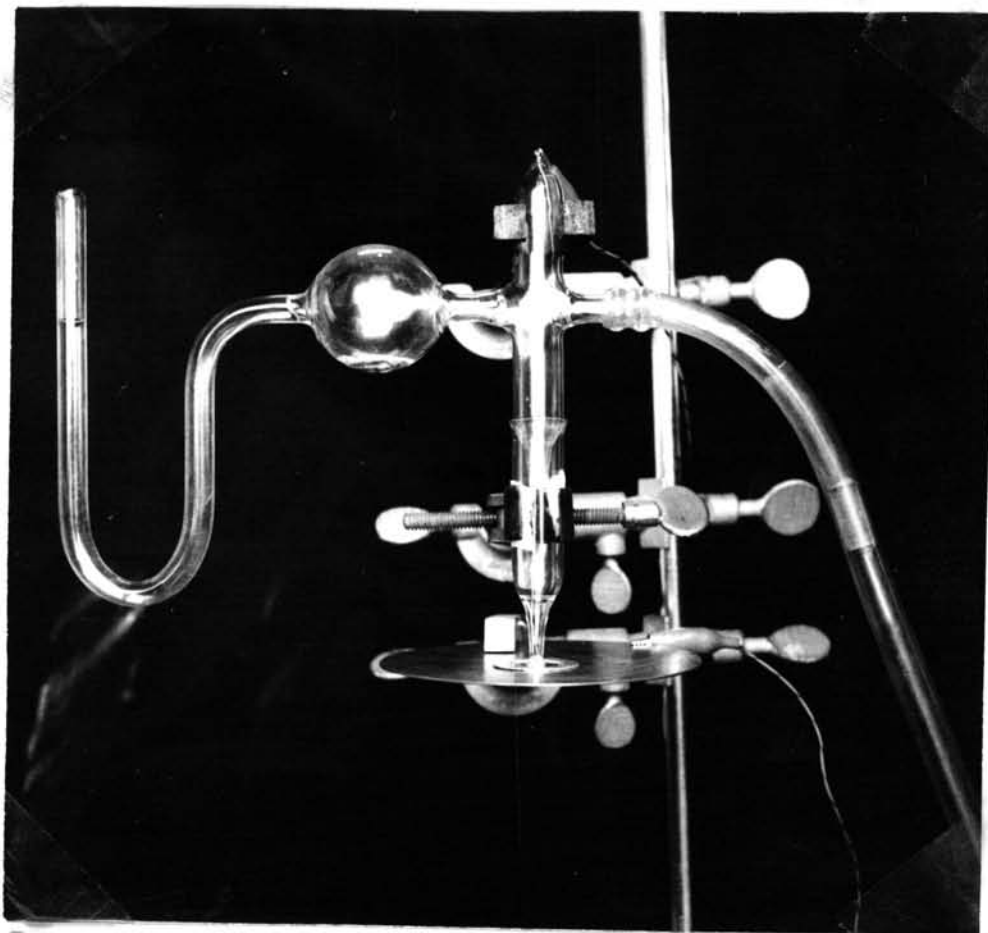


Fig 3.1 Electrolysis cell

that the pressure above the electrolyte be controlled since the gas evolved during the electrolysis of the water could force the electrolyte out of the cell. This was done by drawing out the plunger of the syringe. This arrangement for controlling the liquid level was quite convenient to remove the aluminum backing after the electrodeposition. In this experiment, the samples were prepared by varying the time of deposition and / or the strength of the current due to the varying of the potential across the cell. When the current was turned off, the electrodeposition ceased. The remaining electrolyte was then removed with a 1 cc. syringe. The samples prepared for our investigation are listed in Table 3.1

Table 3.1 Electrodeposition from dilute HNO_3 solution.

Sample number	Material deposited ^a	Current density ^b (mA/cm ²)	Diameter (mm)	Time of deposition (min)
1	^{237}Np - ^{241}Am	2000 - 1000	2.5	5
2	^{237}Np - ^{241}Am	2000 - 400	2.5	10
3	^{237}Np - ^{241}Am	500 - 100	5	15
4	^{237}Np - ^{241}Am	500 - 100	5	30

^a 300 μg of pure ^{241}Am 0.1 mci per 5 ml of solution was used in preparation as known - activity sample.

^b Current was supplied to the disks from a 6 volt storage battery.

3.2 Counting Technique

As mentioned in the Introduction, the activities of all the radioactive sources were measured by a surface barrier detector made from silicon. The detector used in this investigation had an active area of 200 mm^2 and a depletion depth of 422μ at 140 volts bias. The detector was connected in series with a preamplifier, a linear amplifier and a 1024-channel pulse-height analyzer. The operating conditions of the various electronic devices were obtained by adjusting the amplifier and the voltages so that the energy spectrum of the alpha emission from the standard ^{241}Am source was of the desired shape. The arrangement of the detection system is shown in Figure 3.2

For measuring the activities, each radioactive source was placed on the support inside an vacuum chamber at a distance of one cm from the detector. (see Fig. 3.3). The chamber was pumped down to 0.15 torrs in order that there was almost no absorption of the α -particles as they traveled the one cm. between the sources and the detector. A bias voltage of 130 volts was slowly applied to the detector. This was done in order to avoid microplasma breakdown in the detector. The alpha-particle spectrum was then recorded with the multi-channel analyzer. The times required to obtain a fairly accurate counting statistics was five hours for the ^{237}Np samples and one minute for the ^{241}Am standard sources. One reading for each of the ^{237}Np samples was taken while four different readings were taken with the ^{241}Am source.

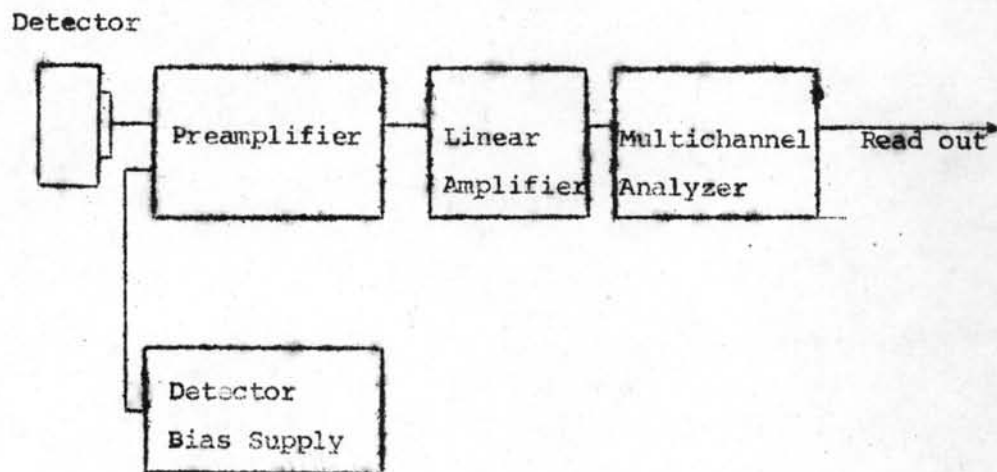


Fig. 3.2 Typical system for alpha particles spectroscopy.

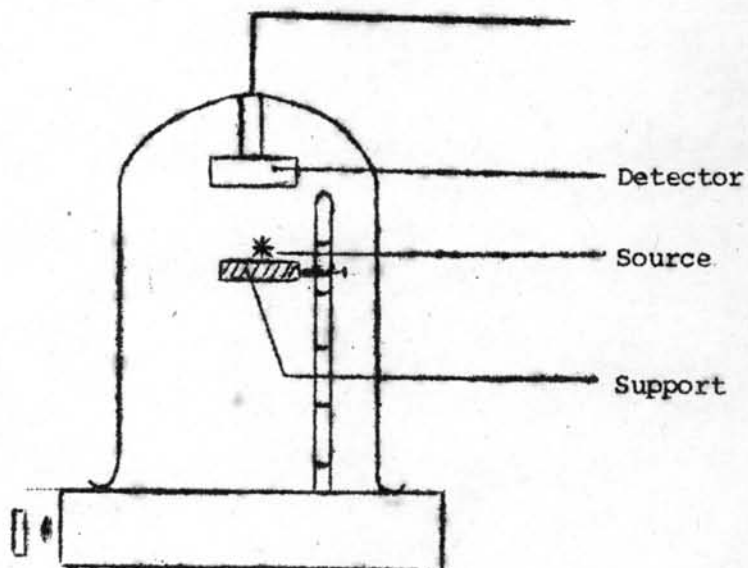


Fig. 3.3 Cross-Section of a vacuum chamber.

The average value of the four readings for the ^{241}Am sources was used in the calculations.

Since equation (2.6.1) was to be used to calculate the specific activities of the ^{237}Np sources, extreme care had to be taken to insure that none of the counting parameters changed. Therefore, each pair of ^{237}Np and ^{241}Am sources having the same geometries were counted immediately after each other. The background radiation was measured each time by replacing the radioactive sources by a dummy plate. The ^{241}Am standard source was always counted to check the performance of the detector.

3.3 Determination of the Mass of Samples

Because the amount of ^{237}Np and ^{241}Am on the various samples were negligible, the mass could not be determined by weighing them on a micro-balance. Instead, the amount of radioactive isotopes on each sample was determined by comparing its activity with the standard curve for the counting rate as a function of the amount of ^{237}Np or ^{241}Am present. The standard curves, Figures 4.6 and 4.7 were obtained by taking solutions of 1000 μg of pure neptunium or 300 μg of pure americium in 5 ml. of liquid, and drawing out various amounts of the solutions with micropipets. The various amounts of solutions were then transferred to the centers of some aluminum foils and then carefully dried out. The activity of each evaporated source of a known amount of radioactive isotope on it was measured.

The activities of six known amounts of ^{237}Np and of six known amounts of ^{241}Am were used to construct the standard curve, Figures 4.6 and 4.7. These six known amounts of radioactive sources and the deposited samples had to be counted in continuous series.