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UCRL-99

Isotopes of the New Element, Curium (Atomic Number 96)

By

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B. S. (University of California) 1942

DISSERTATION

Submitted in partial satisfaction
of the requirements for the degree of

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of the

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INTRODUCTION

In 1934 Fermi[1] bombarded uranium with slow neutrons and found that a number of radioactive isotopes were produced which, on the basis of previous experience, led him to believe that elements 93 to 97 inclusive had been produced. It was shown by Hahn and Strassmann[2], however, that these activities were not due to transuranium elements but rather to elements of approximately one-half the weight of uranium. Thus the early search for the transuranium elements may be considered to be responsible for the discovery of nuclear fission and the numerous consequences of that discovery.

After the explanation of Hahn and Strassmann for the activities which Fermi had found, the question of what the properties of the transuranium elements were remained unanswered. The first part of the answer was found by McMillan and Abelson[3] in 1940 when they discovered element 93, which they named neptunium after the planet Neptune.

The number of true transuranium elements was extended one more upon the discovery of element 94 (named plutonium) by Seaborg, McMillan, Kennedy, and Wahl in 1940[4]. The isotopes Pu^{238} and Pu^{239} were produced and the very important observation made that Pu^{239}, like U^{235}, undergoes nuclear fission with slow neutrons[5].

Following the discovery of plutonium and the

development of the chain reacting piles by which plutonium could be made in large quantities it became feasible to start a search for the next higher elements. Fortunately it was at about this same time that Dr. J. G. Hamilton and his group at the Crocker Radiation Laboratory improved the 60-inch cyclotron so that larger currents of higher energy bombarding particles could be obtained.

In 1944, a series of experiments was carried out by Seaborg, James, and Ghiorso[6] which resulted in the first production and identification of isotopes of the element with atomic number 96. In the same year, but at a somewhat later date, the element of atomic number 95 was found by Seaborg, James and Morgan[7]. Element 95 was named americium (symbol Am) after the Americas because of its analogy to the rare earth europium. The name curium was chosen for element 96 because the evidence indicates that element 96 contains seven 5f electrons and is thus analogous to the element gadolinium, with its seven 4f electrons in the regular rare earth series. On this basis element 96 was named after the Curies in a manner analogous to the naming of gadolinium, in which the chemist Gadolin was honored. These names and symbols will be used throughout the remainder of this dissertation in which only the isotopes of curium and such other closely related nuclei as parents and daughters will be considered.

A consideration of the methods by which isotopes of

curium might be produced led to the use of two methods. The bombardment of plutonium with high energy helium ions should produce isotopes of this element. The neutron irradiation of Am^{241} should lead to capture of neutrons and formation of Am^{242} which would decay by negative beta-particle emission to Cm^{242}. Both of these methods were used in the investigations reported here.

It was expected of course that only minute (tracer) amounts of the new element would be formed and that identification would depend upon showing by radiochemical techniques that the radioactivity resided in an element with unique chemical properties, different from all other known elements.

It was essential to have at hand from the beginning some hypothesis or set of hypotheses as to what the chemical properties of element 96 might be. This was necessary because of the large number of radioactive fission products produced in such a bombardment and because of the high level of alpha-radioactivity of the target Pu^{239}. For a reasonable half-life of an isotope of curium one could not expect that the amount produced in a reasonable time by cyclotron bombardment would emit more than about one alpha-particle for every 10^6 alpha-particles from the Pu^{239}. The chemical investigation of the heaviest known elements (atomic numbers 89-92 inclusive) and the more recently discovered neptunium and plutonium, had led to the view[8] that the heavy elements constitute a new "rare earth" series in which the 5f electron shell is

being filled and which formally begins with thorium. On this basis it was expected that element 96 would have a very stable III oxidation state with higher states being formed with great difficulty, if at all. Thus the anticipated chemical properties to be utilized for the isolation of detectable amounts of this element were to be those of elements with the typical III oxidation state such as the rare earths. Using this hypothesis, one would expect that curium could be separated from the target plutonium and from all of the fission product elements except the rare earths by oxidation of the plutonium to its VI state and coprecipitation of the curium with lanthanum fluoride. The first detectable quantities of curium were isolated using just this procedure and it was not until considerably later that a method of separating the rare earth elements from curium was found.

GENERAL EXPERIMENTAL METHODS

At first the plutonium targets for bombardment in the 60-inch cyclotron were prepared by the evaporation of plutonium nitrate solutions on grooved platinum plates, followed by mild ignition to form plutonium oxide. Later it was found to be more satisfactory to prepare the targets by precipitation of $(NH_4)_2PuF_6$ and transferring this compound with a small amount of water to the platinum target and igniting after the water had been slowly evaporated. The targets were then bombarded directly in the target chamber of the cyclotron. Several such bombardments have been made using the 60-inch cyclotron of the University of California at Berkeley, the first one employing helium ions of 32-Mev energy, and all later ones, helium ions with ca. 38 Mev of energy.

Following the bombardments, the plutonium oxide was dissolved by the use of sulfuric acid, heating until extensive fumes of sulfur trioxide appeared, and then heating further to dryness. This was followed by the dissolution of the plutonium sulfate in dilute nitric acid and the remaining undissolved oxide was dissolved by heating with nitric acid, together with a small amount of added hydrofluoric acid.

On the assumption that the III oxidation state of curium

would be present and would have an insoluble fluoride, lanthanum fluoride was precipitated from the solution after oxidizing the plutonium to the soluble VI oxidation state. Several different oxidizing agents have been used for this purpose with almost equal success; 0.1 N potassium dichromate and 0.1 N potassium permanganate are examples. The precipitate contained the beta-active rare earth fission product elements and presumably also contained any curium present in the III oxidation state. The precipitate was dissolved and the operation was repeated until all of the plutonium was eliminated. Although this procedure of necessity led to a concentration of a large amount of beta-activity due to the fission products along with the curium, it was still possible to examine the alpha-activity which remained in this fraction. Later it became possible to separate the rare earth fission products and to examine also the Geiger counter radiations of curium isotopes.

At the beginning of the investigation the alpha-particles were identified as to range by means of absorption in very thin mica sheets placed immediately over the sample in an ordinary parallel plate ionization chamber. Later a multichannel pulse analyzer was constructed and all subsequent energy measurements were made by employing it[9]. With this instrument a thin sample is placed in an ionization chamber in which the total ionization of an alpha-particle can be measured as a voltage pulse. Individual pulses are

sorted electronically and recorded on a number of fast mechanical registers in such a way as to separate the individual alpha-particle energies in a mixture of alpha-emitters.

The neutron irradiation of the americium samples was made in the uranium graphite chain reacting piles at the Clinton Laboratories and the Hanford Engineer Works.

In the experiments in which the plutonium daughters were chemically separated after their growth as decay products from the isotopes of curium, essentially the same method of chemical separation of plutonium from curium was used. In this case, of course, it was the oxidized plutonium which was recovered following the successive separation of lanthanum fluoride precipitates which removed the curium.

The separation of curium from the rare earths may be accomplished by partially precipitating a rare earth fluoride such as LaF_3 by accurate control of the fluoride ion concentration. The control of this fluoride ion concentration is best attained by means of fluosilicic or fluoboric acid. These acids hydrolyze to produce a small fluoride ion concentration which can be accurately controlled by varying the

Start

here

hydrogen ion concentration. The same results can be attained by the use of very low concentrations of hydrofluoric acid, but this is much less convenient because of the precautions which must be taken to maintain the very low fluoride ion concentration at the desired value. A suitable set of conditions using fluosilicic acid involves the precipitation of 2-5 mg/ml of lanthanum in a solution 3 M in nitric acid and 1 M in fluosilicic acid. Under these conditions, which incidentally give approximately optimum separation of the rare earths from curium, 90% of all the rare earths are precipitated while only 30% of the curium is found in the precipitate. The curium which remains in solution can be precipitated with the small amount of lanthanum remaining by the addition of a large excess of hydrofluoric acid and the procedure repeated as many times as is necessary to achieve the desired degree of separation.

Curium and americium are isolated together by the above procedure with no detectable separation. These two elements can be separated from each other by selective elution from columns of cation exchange resins[10]. A suitable procedure for this separation is elution from Nalcite Resin (Dowex 50) columns 10 to 50 cm in length using as eluting solution 0.25 M ammonium citrate solution adjusted to pH 3.0-3.1 with hydrochloric acid.

DISCUSSION OF RESULTS

The Isotope Cm^{242}. The helium-ion bombardment of plutonium led to the first definite identification of an isotope of curium[6]. In the first helium-ion bombardment about 10 mg. of Pu^{239} was bombarded with helium ions of 32-Mev energy for a total of about 37 microampere-hours in the Berkeley 60-inch cyclotron. After the chemical separations described above, the rare earth fraction was found to contain 500 disintegrations per minute of an alpha-activity with a range of 4.75 ± 0.1 cm in air at 15°C and 760 mm of mercury pressure. This activity decayed with a half-life of five months (5.0 ± 0.1 months). The first isotopic assignment of this activity was to the isotope Cm^{242}, formed in the reaction $Pu^{239}(\alpha,n)Cm^{242}$. That this has now been confirmed to be the correct isotopic assignment will be made clear from the discussion of the remainder of the results of this investigation.

This same radioactivity was produced later in a series of neutron bombardments of americium in which both bombardment time and total neutron exposure varied widely as a result of the use of the chain reacting piles at both Clinton and Hanford. In all of these neutron irradiations it was found that the ratio of the total intensity of the 4.75 cm alpha particles to that of the 4.05 cm alpha particles of

Am^{241}, after correction for the five month half-life of the 4.75 cm particles, varied as the first power of the total neutron irradiation. The following sequence of nuclear reactions accounts for these observations:

$$Am^{241}(n,\gamma)Am^{242} \qquad (1)$$

$$Am^{242} \xrightarrow{\beta^-} Cm^{242} \qquad (2)$$

The 4.75 cm alpha-particles are due to the isotope Cm^{242}. The decay rate here again corresponds to a half-life of five months so that this radioactivity agrees both in half-life and alpha-particle range with the Cm^{242} formed from the helium-ion bombardment of Pu^{239}.

The chemical evidence which allowed all previously known elements to be eliminated is as follows:

(1) The activity is carried quantitatively by lanthanum fluoride from solutions previously treated with various reducing and oxidizing agents ranging from zinc amalgam to argentic ion. The carrying is not influenced by the use of ammonium fluoride instead of hydrofluoric acid. This evidence alone eliminates all previously known elements except indium, lanthanum, and all of the rare earths, yttrium, actinium, thorium, and possibly protactinium.

(2) The activity is not precipitated with indium sulfide from acetic acid solution, thus eliminating indium.

(3) Under the same conditions of acidity and concentration of reagents, the activity is not coprecipitated with ceric or zirconium iodates to as great an extent as are

actinium, protactinium, and thorium, thus eliminating these last three elements and cerium.

(4) The activity can be partially separated from the rare earth yttrium subgroup by any one of three standard procedures: precipitation of the activity with lanthanum or praseodymium carrier from neutral to alkaline carbonate, oxalate, or formate solutions with yttrium, gadolinium, or lutecium holdback carrier.

(5) Europium can be separated by reduction to the II oxidation state with zinc amalgam and precipitation of $EuSO_4$. The activity is not coprecipitated with europium under these conditions.

(6) Fractional separation from the remaining elements (lanthanum, praseodymium, neodymium, element 61, and samarium) can be achieved by the use of fluosilicate ion in solution during a partial precipitation of rare earth fluoride as carrier.

Confirmation of the isotopic assignment of the five month alpha-activity of 4.75 cm range came from a study of its decay product. A sample containing about 2×10^6 disintegrations per minute of Cm^{242} (produced by neutron irradiation of Am^{241}) was allowed to decay for about a month and at the end of this time the plutonium fraction was isolated and was found to consist of an alpha-activity with 4.08 cm. range which can be ascribed to Pu^{238}(4). The amount of Pu^{238} which grew was quantitatively determined in another experiment

by addition of Pu^{239} tracer to establish the chemical loss during separation of the plutonium. The details of this experiment were as follows: A sample containing 1.293×10^6 counts per minute of Cm^{242} was very carefully purified of all plutonium, 1102 counts/minute of Pu^{239} was then added and the sample allowed to stand for 70 days. At the end of that time the plutonium was separated from the curium and the ratio of Pu^{238} to Pu^{239} determined by pulse analysis (Figure 1). The average of several such pulse analyses gave the value 1.45 for the ratio of Pu^{238} to Pu^{239}. From these data and taking the half-life of Cm^{242} as 150 days, the half-life of Pu^{238} is calculated to be 92 $\pm$ 2 years, within the limit of error of the value obtained by Jaffey[11] in direct decay measurements.

In addition to its alpha-particles Cm^{242} also emits a small amount of soft electromagnetic radiation and some electrons. Absorption curves of the radiations emitted by samples of Cm^{242} produced by neutron irradiation of Am^{241} are shown in Figures 2, 3, 4 and 5. The Cm^{242} in these amples was decontaminated from rare earth fission products means of the fluosilicate procedure mentioned above and parated from americium by selective elution from a resin wex 50) absorption column[10]. Samples from two differ- neutron irradiations of Am^{241} differing by a factor of oximately 3 in total number of neutrons were measured he absorption curves were found to be identical in all

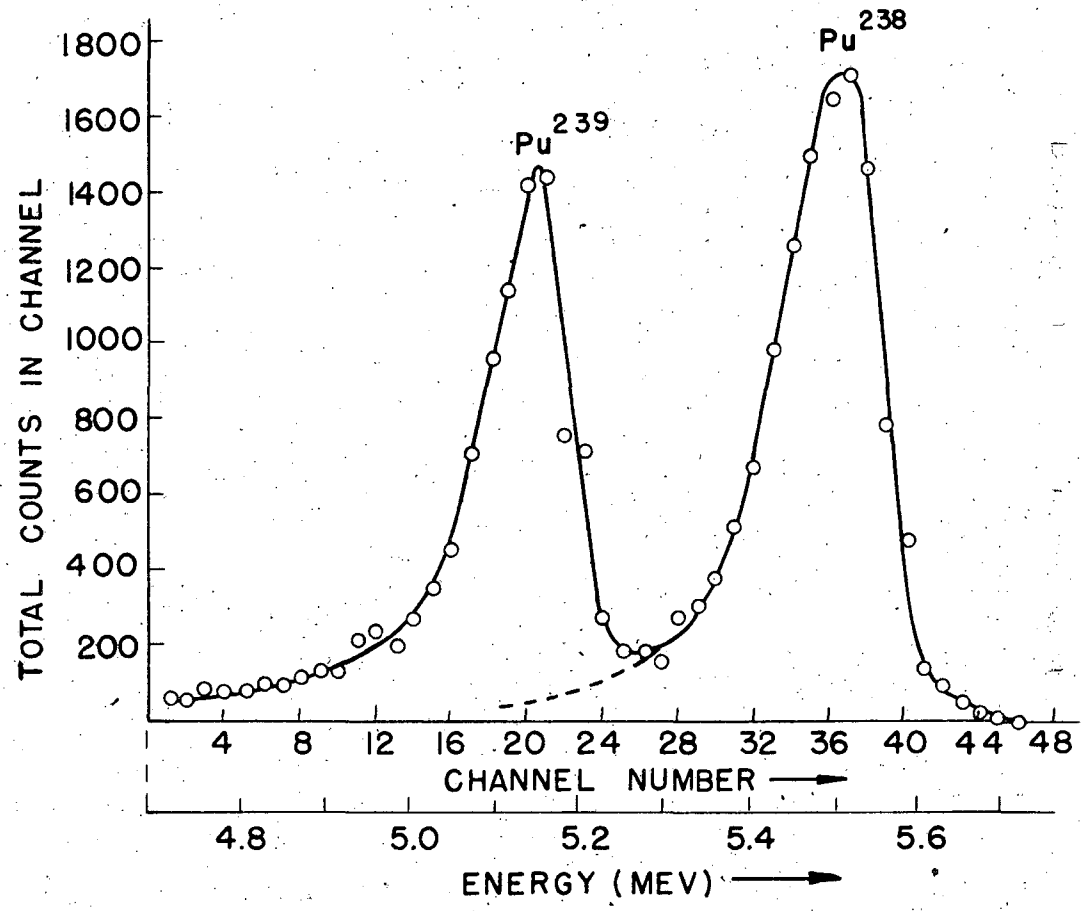


Figure 1: Alpha-particle pulse analysis curve of plutonium which grew from Cm^{242} (with added Pu^{239}).

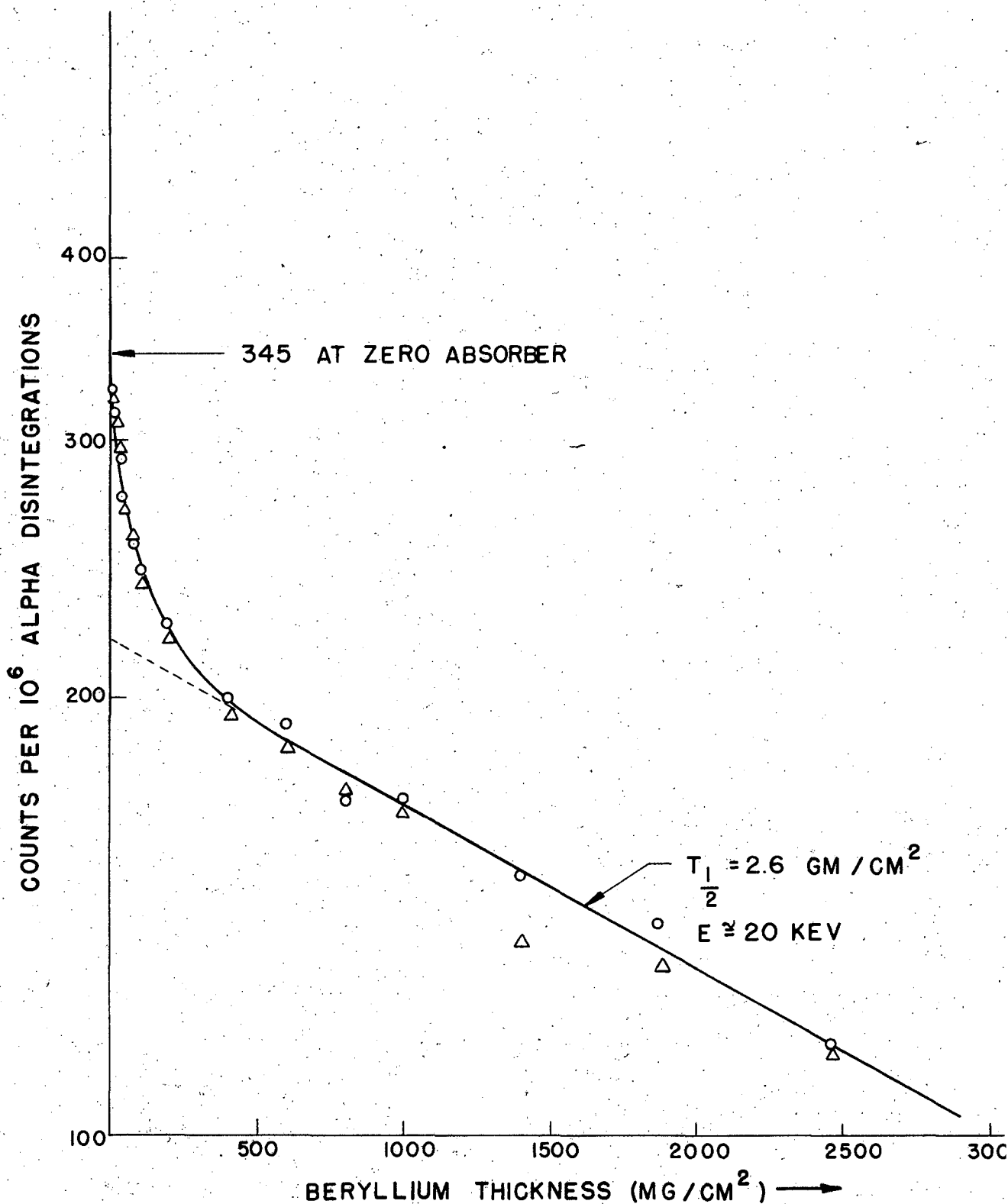


Figure 2: Geiger counter beryllium absorption curve of Cm^{242} radiations. The geometrical efficiency was about 10% and the curve has been normalized to 10^6 disintegrations per minute.

O points for Cm^{242} produced by neutron irradiation of Am^{241} of 6.6×10^{19} neutrons/cm^2.

Δ points for Cm^{242} produced by neutron irradiation of Am^{241} of 2.0×10^{20} neutrons/cm^2.

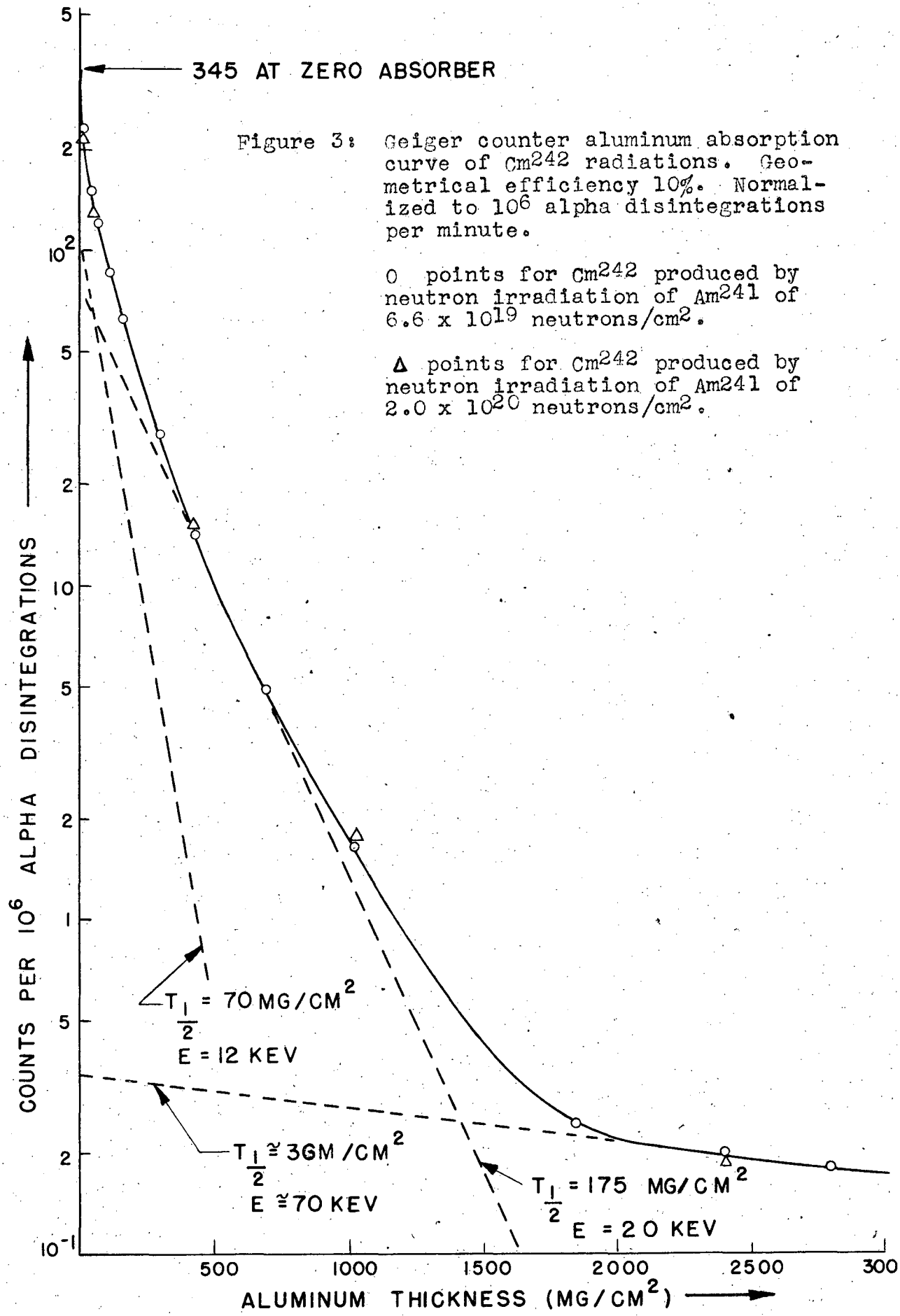


Figure 3: Geiger counter aluminum absorption curve of Cm^{242} radiations. Geometrical efficiency 10%. Normalized to 10^6 alpha disintegrations per minute.

O points for Cm^{242} produced by neutron irradiation of Am^{241} of 6.6×10^{19} neutrons/cm^2.

Δ points for Cm^{242} produced by neutron irradiation of Am^{241} of 2.0×10^{20} neutrons/cm^2.

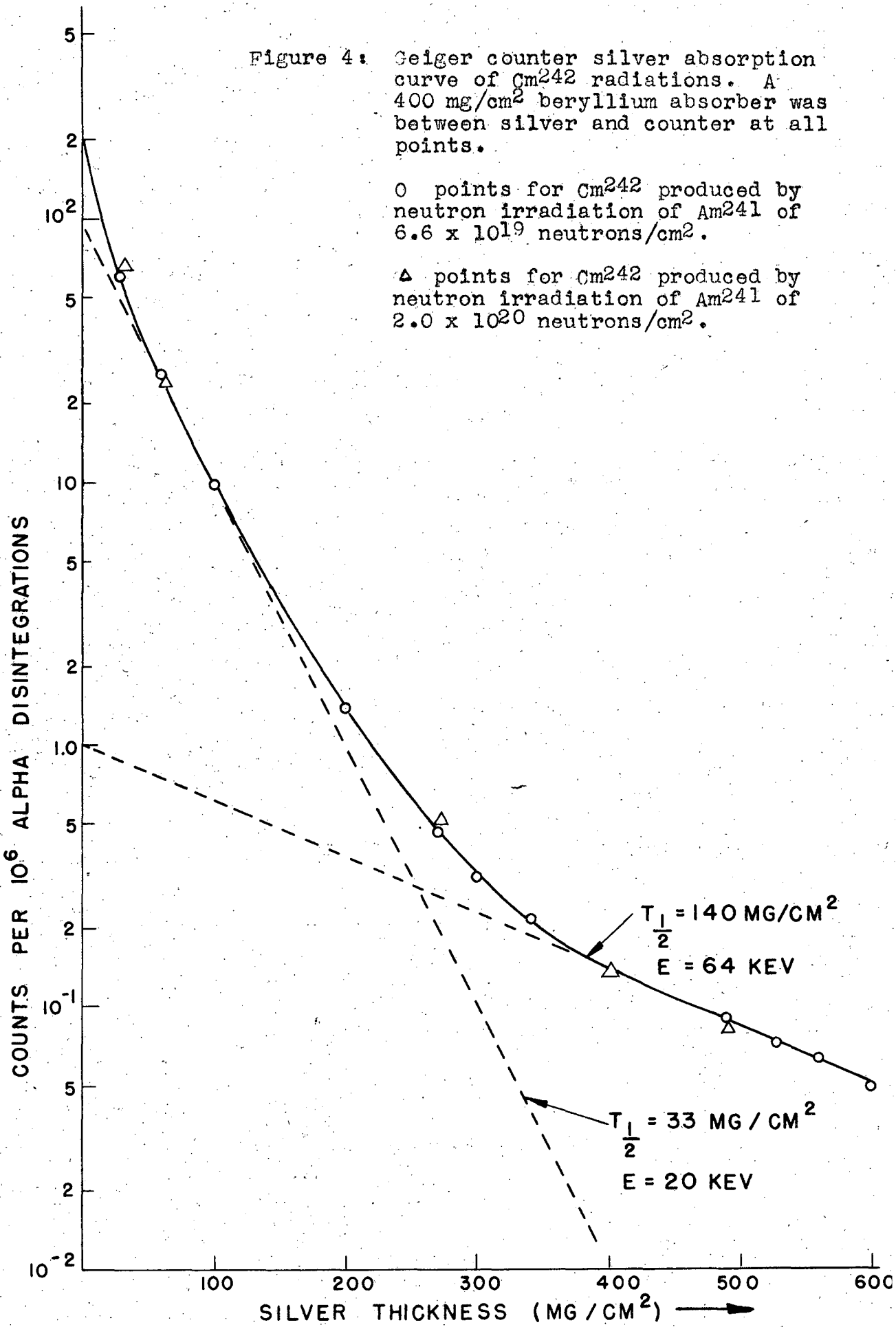


Figure 4: Geiger counter silver absorption curve of Cm^{242} radiations. A 400 mg/cm^2 beryllium absorber was between silver and counter at all points.

O points for Cm^{242} produced by neutron irradiation of Am^{241} of 6.6×10^{19} neutrons/cm^2.

△ points for Cm^{242} produced by neutron irradiation of Am^{241} of 2.0×10^{20} neutrons/cm^2.

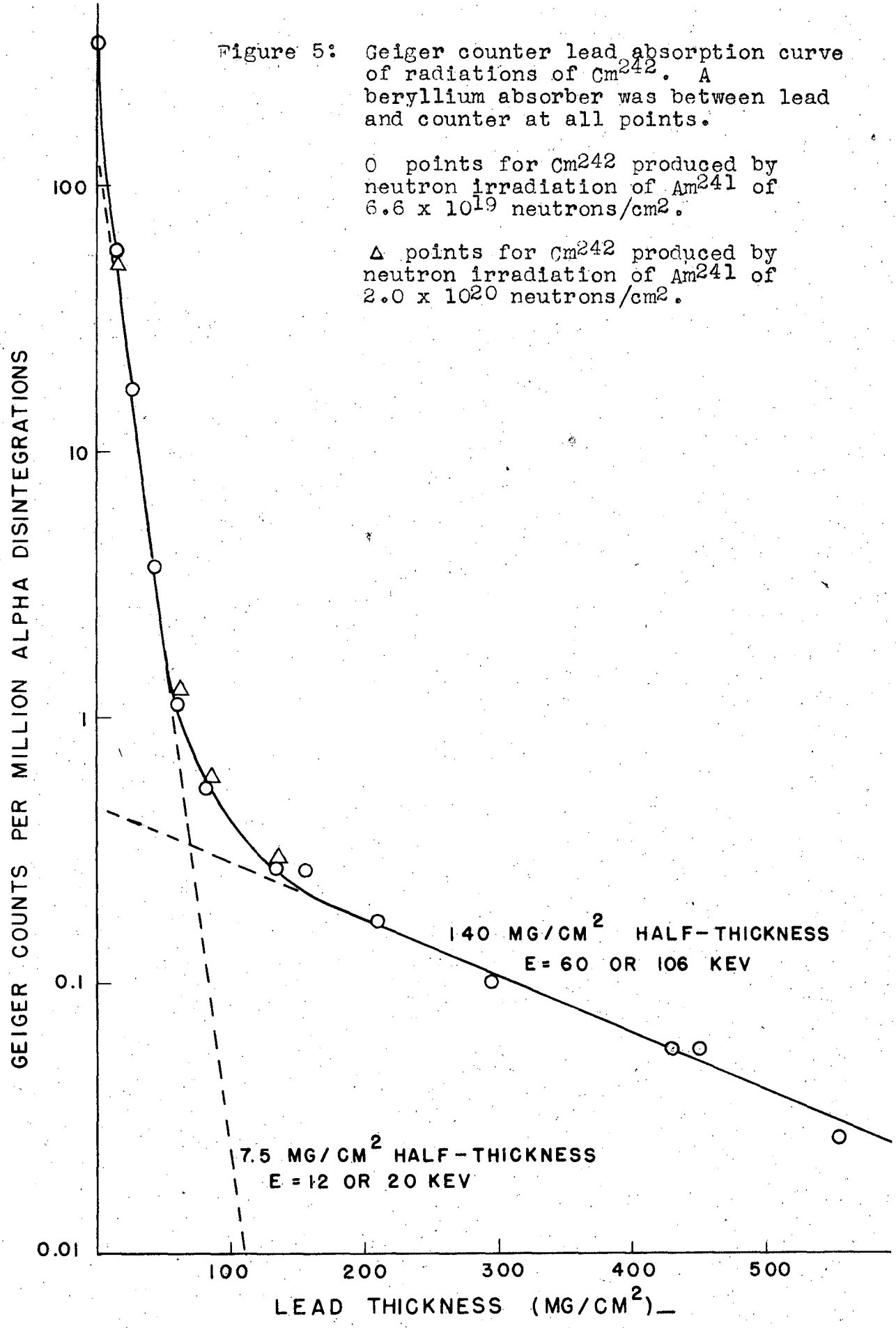


Figure 5: Geiger counter lead absorption curve of radiations of Cm^{242}. A beryllium absorber was between lead and counter at all points.

O points for Cm^{242} produced by neutron irradiation of Am^{241} of 6.6×10^{19} neutrons/cm^2.

Δ points for Cm^{242} produced by neutron irradiation of Am^{241} of 2.0×10^{20} neutrons/cm^2.

respects showing that (1) the radiations are not due to fission-product contamination since the two samples had radically different chemical histories and times of decay between removal from the pile and measurement of the radiations; and (2) that the radiations are actually due to Cm^{242} and not Cm^{243} or a higher mass isotope since, if they were due to Cm^{243}, the level of activity would have been much higher in the sample which received longer irradiation. In addition to these curves it was found by means of bending the particles in a magnetic field that approximately 12% of the counts at zero absorber are due to low energy negative electrons and the remainder to electromagnetic radiation. These radiations and their approximate abundances are summarized in Table 1.

TABLE 1

Electromagnetic and Electron Radiations of Cm^{242}

	Counts per 10^6 alpha disintegrations at 10% geometrical efficiency	Assumed counting efficiency	Number of events per disintegration
65 Kev gamma-ray	0.5	1%	1.8×10^{-3}
L x-rays	200	1.5%	0.13
Softer x-rays	100	--	--
Electrons	40	100%	4×10^{-4}

The Isotope Cm^{240}. In the second bombardment of Pu^{239} and in all subsequent bombardments employing the

60-inch cyclotron helium ions of energy ca. 38 Mev were used. In the first bombardment employing these higher energy particles 100 mg of Pu^{239} was bombarded for 63 microampere-hours. A sample was treated by the same chemical procedure as already described and the final curium fraction contained about 2 x 10^5 alpha-particle disintegrations per minute.

It was found that 20% of this activity was the Cm^{242} activity described above while the remaining 80% was due to another alpha-emitter which emits alpha-particles with a range of 4.95 ± 0.1 cm in air at 15°C and 760 mm of mercury pressure. The initial over-all rate of decay of the alpha-particles in this sample gave a half-life of about one month indicating that the half-life of the 4.95 cm range activity was somewhat less than one month and later resolution (Fig.6) gave the value 26.8 days. This activity was thought to be due to either Cm^{241} or Cm^{240} produced by the reaction $Pu^{239}(\alpha,2n)Cm^{241}$ or $Pu^{239}(\alpha,3n)Cm^{240}$.

In a later bombardment a sample of this activity was set aside and allowed to decay from the second to the fourth day after shut-down of the cyclotron. The plutonium fraction was then isolated and the pulse analysis curve (Fig. 7a) as well as the alpha decay curve (Fig. 7b) of this plutonium sample gave definite evidence of Pu^{236} whose radioactive properties had been definitely established[12]. It was thus definitely shown that the new isotope was Cm^{240}. The curium fraction was then allowed to grow plutonium again (77 days of

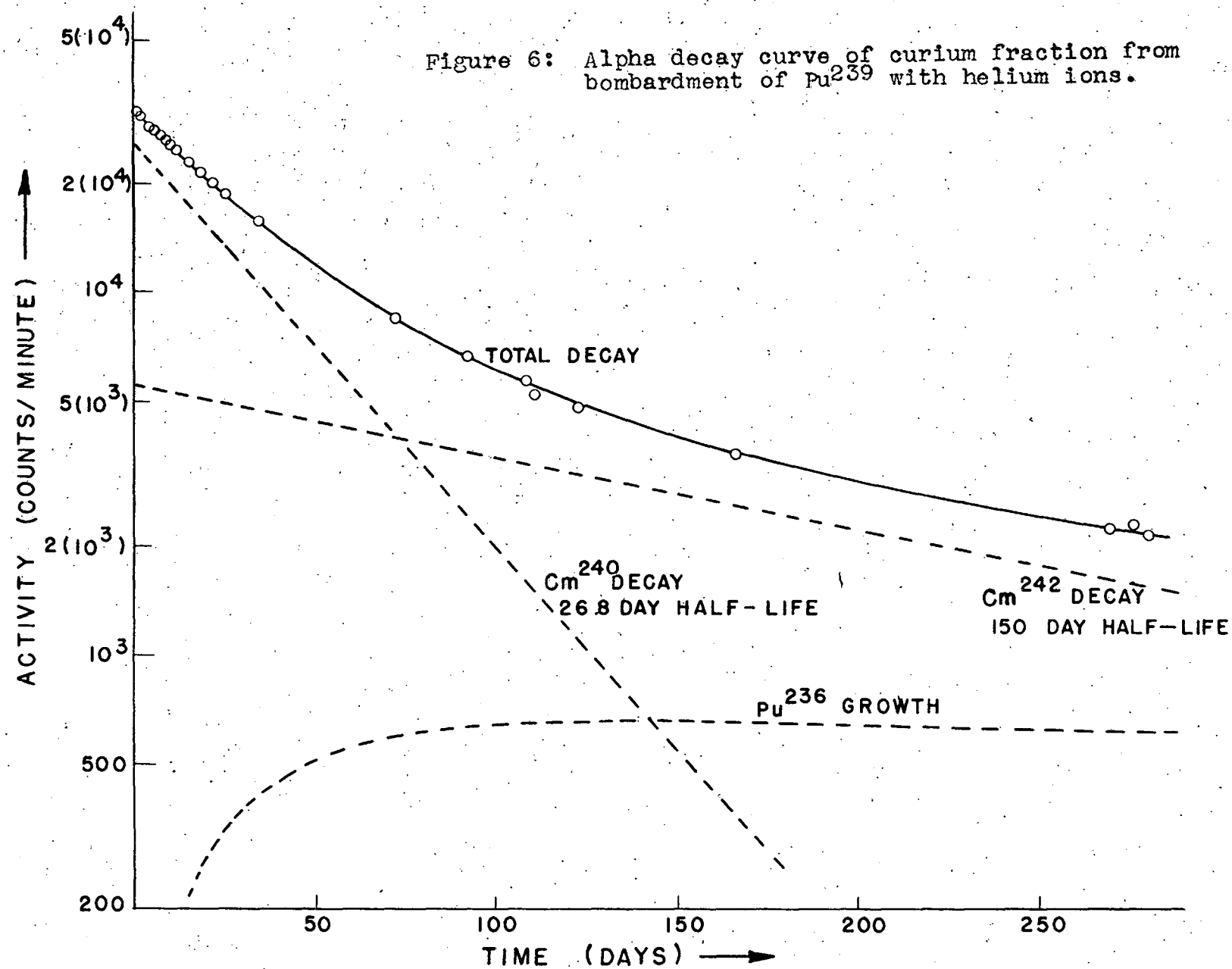
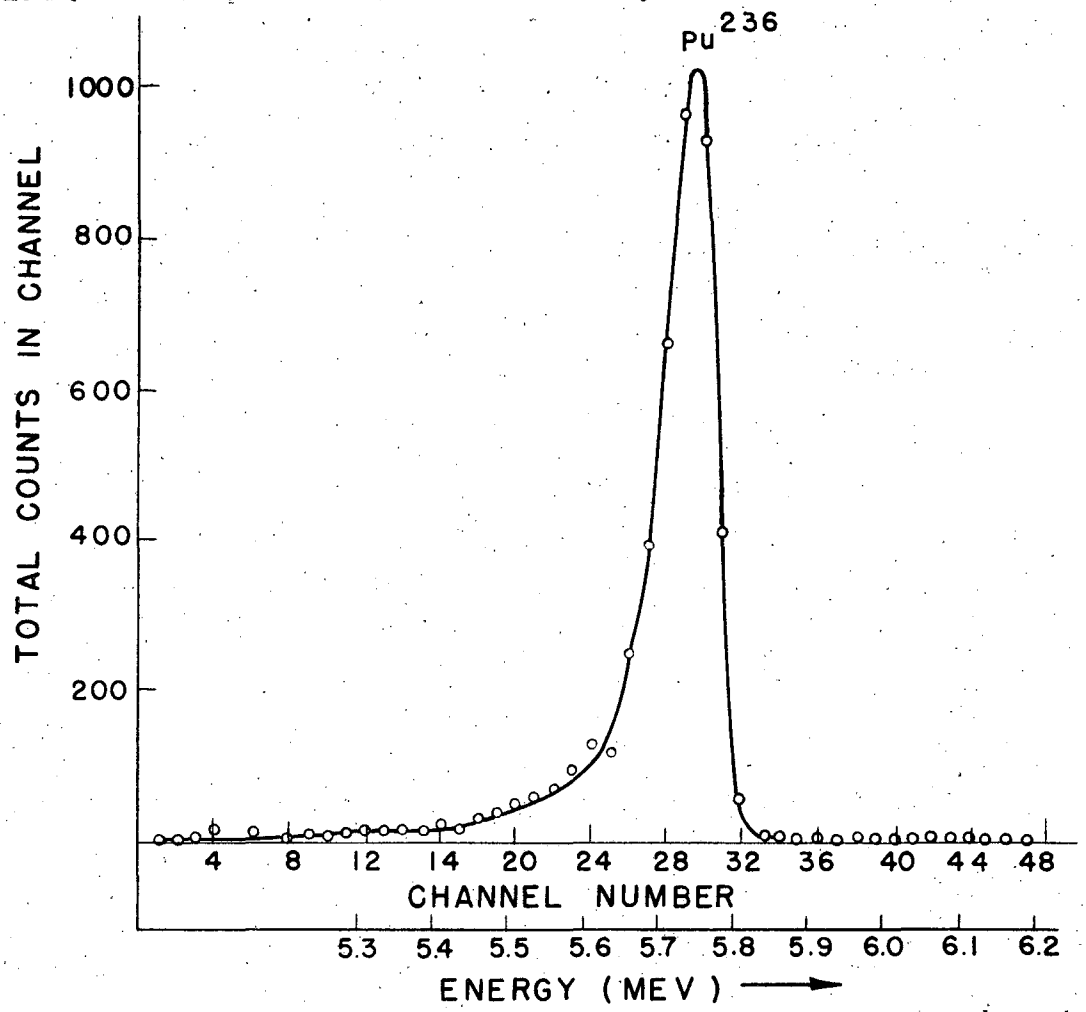


Figure 6: Alpha decay curve of curium fraction from bombardment of Pu^{239} with helium ions.

Figure 7a: Alpha-particle pulse analysis curve of activity in plutonium fraction removed from curium fraction of a Pu^{239} target bombarded with helium ions. Growth period-second to fourth day after end of bombardment.



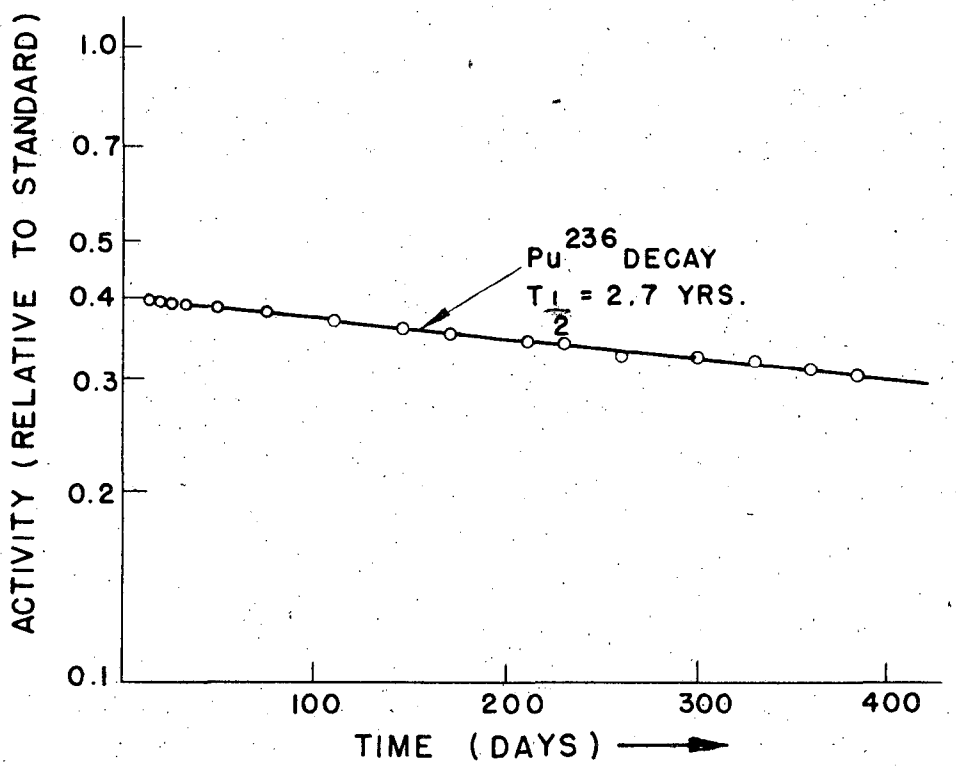


Figure 7b: Decay of alpha activity in plutonium fraction removed from curium produced by bombarding Pu^{239} with helium ions. Growth period-second to fourth day after end of bombardment.

growth) and the amount of Pu^{236} which had grown was quantitatively determined by the addition of Pu^{239} tracer to establish the chemical loss in the process of separation of the plutonium from the curium. The additional details of this experiment which allowed a calculation of the half-life of the curium parent were as follows: 1.38×10^5 alpha counts/minute of the Cm^{242}-Cm^{240} mixture of which initially 70.2% were due to Cm^{240} decayed for 77 days. At the end of that time the plutonium fraction was isolated after addition of 1935 counts per minute of Pu^{239} and the resulting plutonium sample pulse analyzed. The average of several such pulse analyses (see Fig. 8) gave 1.14 as the value of the ratio of Pu^{236} to Pu^{239}. Using the value 983 days (obtained from Fig. 7b) for the half-life of Pu^{236} one calculates 26.7 days as the half-life of Cm^{240} which is in excellent agreement with the value 26.8 days obtained by resolution of the alpha decay curve (Fig. 6).

The Isotope Cm^{241}. In addition to the isotopes Cm^{240} and Cm^{242} one other activity has been observed which is very probably due to Cm^{241}.

When the Geiger counter activity of the combined americium-curium fraction of plutonium targets bombarded with helium ions is followed with various absorbers, the decay curves shown in Figs. 9, 10, and 11 are obtained. From the percentage of the alpha-activity in the sample which is due to Cm^{242} the Geiger counter activity due to this isotope can be obtained from Figs. 2, 3, 4, and 5. If this

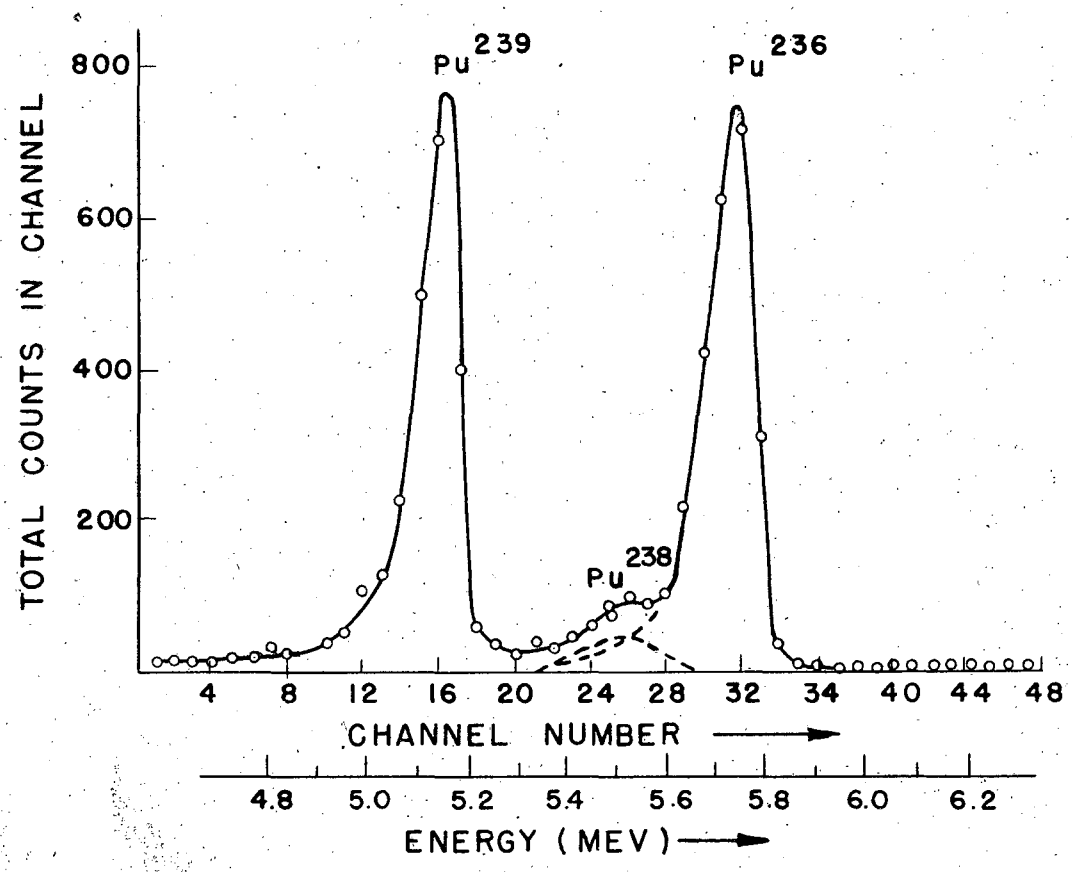
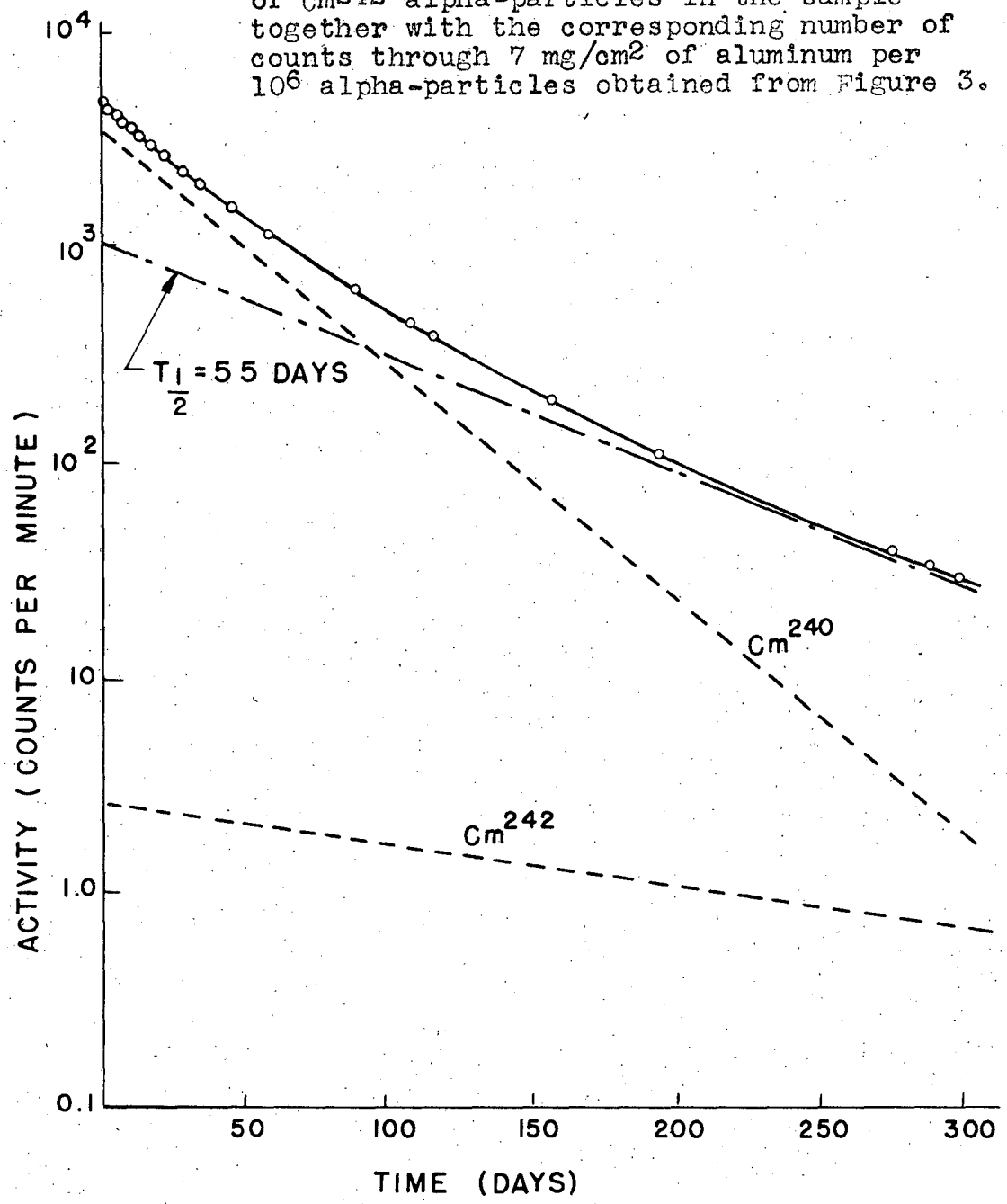


Figure 8: Alpha-particle pulse analysis curve of plutonium which grew in curium produced by helium ion bombardment of Pu^{239}. Growth period - fifth to eighty-second day after end of bombardment. (Pu^{239} tracer added to determine yield)

Figure 9: Geiger counter decay of americium-curium fraction of Pu^{239} target bombarded with helium ions. Decay measured through 7 mg/cm^2 aluminum. The Cm^{242} curve was constructed from a knowledge of the number of Cm^{242} alpha-particles in the sample together with the corresponding number of counts through 7 mg/cm^2 of aluminum per 10^6 alpha-particles obtained from Figure 3.



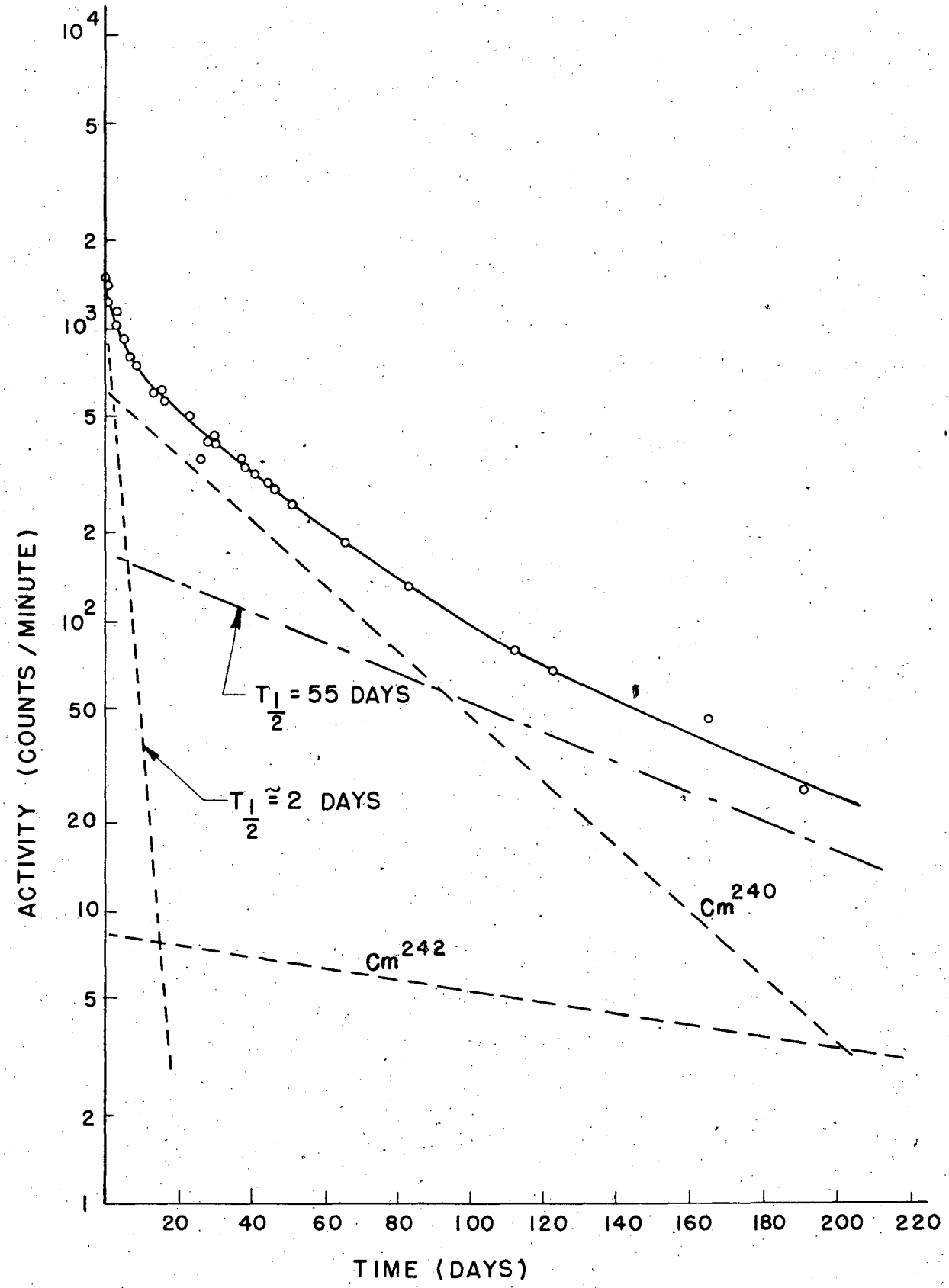


Figure 10: Geiger counter decay of americium-curium fraction of Pu^{239} target bombarded with helium ions (1.85 gm/cm^2 beryllium filter).

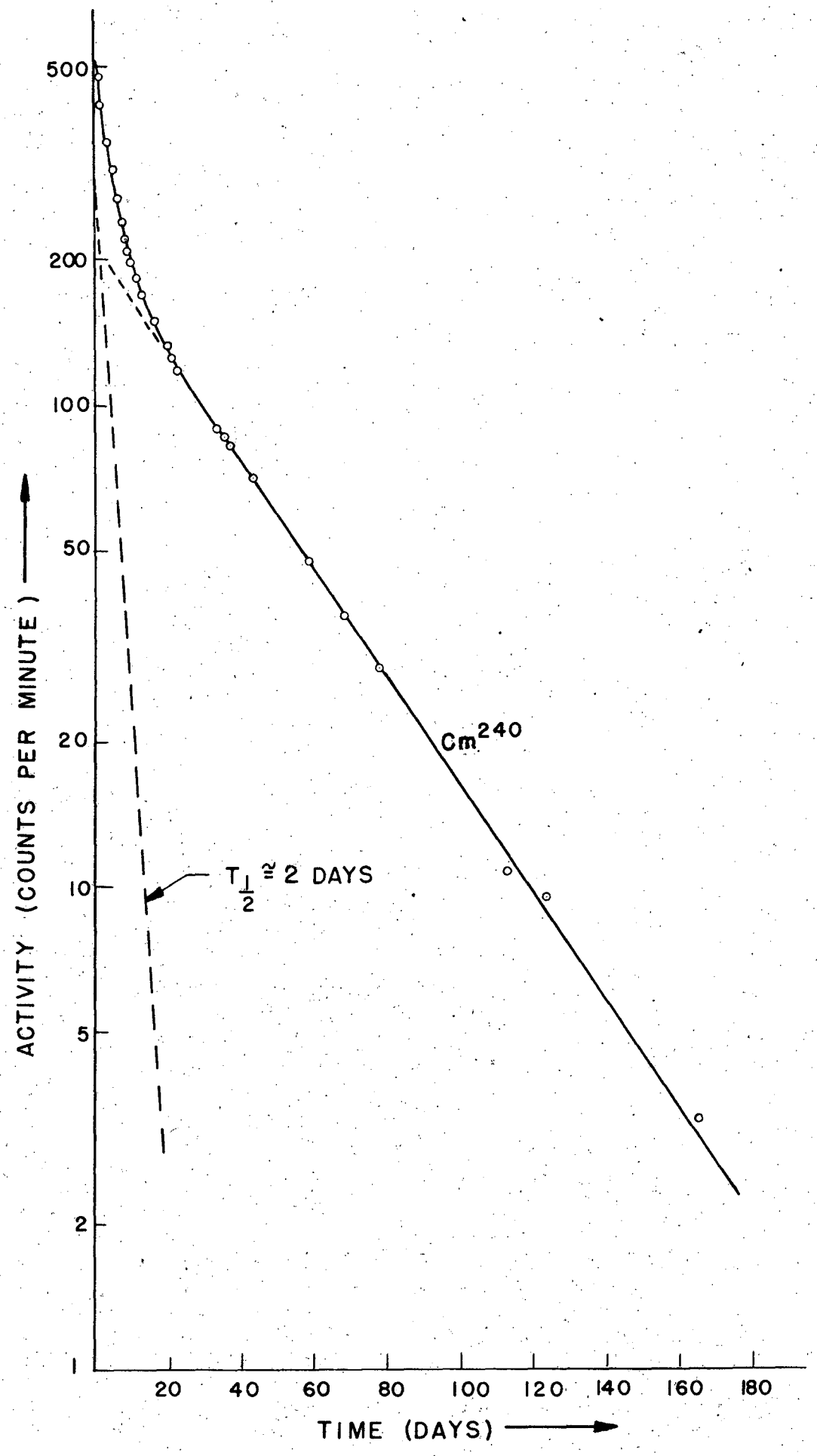


Figure 11: Geiger counter decay of americium-curium fraction of Pu^{239} target bombarded with helium ions (5 gm/cm^2 lead filter)

is subtracted from the total, it is found that there remains, in addition to an activity corresponding to the Cm^{240} half-life, an activity of about 55-day half-life and one of about 2-day half-life. The ca. 2-day activity has been shown to be the 50-hour Am^{238}. The 55-day activity has a large amount of soft-electromagnetic radiation, perhaps some electrons, but no hard gamma rays. It is probable that the activity is due to Cm^{241} decaying by orbital electron capture.

To test for the possibility of alpha branching in Cm^{241} the Geiger counter activity of the plutonium which grew in these samples was examined for the presence of Pu^{237}. Pu^{237} decay by orbital electron capture with a half-life of 40 days(12) so that its characteristics are not very favorable for detection. However, the fact that no Geiger counter activity above the amount expected from the Pu^{236} and Pu^{238} was found in this sample shows that Cm^{241} decays predominantly by orbital electron capture.

Discussion of Lower Mass Isotopes. Bombardment of Pu^{239} with 38-Mev helium ions would be expected to produce Cm^{242}, Cm^{241}, Cm^{240}, and probably Cm^{239}. The first three of these have been produced and are described in the preceding sections. The reason why Cm^{239} has not been found is not clear. The times involved in the separations of the curium fraction from plutonium and fission products have been about five hours at best so that any activity with a half-life of less than an hour or two would have escaped detection. However, it would

be rather surprising in view of the long half-lives of Cm^{240} and Cm^{241} for Cm^{239} to have a half-life for either orbital electron capture or alpha-particle emission as short as an hour. It seems more probable that the (α,4n) reaction on Pu^{239} just does not have a sufficiently large cross section with 38-Mev particles to make detection of the Cm^{239} possible in the presence of the other curium isotopes. One would expect Cm^{239} to have a half-life for alpha-particle emission of about 10 days and an orbital electron capture half-life comparable to this so that branching decay is quite probable.

The production of even lighter mass isotopes is now quite possible using the 184-inch cyclotron, and experiments designed for this purpose are now in progress.

SUMMARY

Three isotopes of the new element with atomic number 96 have been produced by the helium ion bombardment of plutonium. Element 96 has been given the name curium and the symbol Cm because of its similarity to gadolinium, the analogous element in the lanthanide series.

The tracer chemistry of curium has been studied and has revealed only a very stable (III) oxidation state.

The isotopes of curium which have been produced and studied are: (1) Cm^{242} which emits alpha-particles with a range of 4.75 ± 0.1 cm in air (energy 6.1 Mev) and decays with a half-life of 5.0 ± 0.1 months, (2) Cm^{240} which emits alpha-particles with a range of 4.95 ± 0.1 cm in air (energy 6.3 Mev) and decays with a half-life of 26.8 ± 0.3 days, and (3) Cm^{241} which decays by orbital electron capture with a half-life of 55 ± 15 days.

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