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GOVERNMENT OF INDIA
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MOLYBDENUM-99 FISSION YIELD IN THE THERMAL NEUTRON-INDUCED FISSION OF PLUTONIUM-239 AND URANIUM-235

by

H. C. Jain and M. V. Ramaniah
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BHABHA ATOMIC RESEARCH CENTRE
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ABSTRACT

The large discrepancies in the earlier reported values of ^{99}Mo fission yield in the thermal neutron-induced fission of ^{239}Pu and the difficulty in selecting a particular value for the ^{99}Mo yield while determining the fission yields of many other nuclides in the Neutron-induced fission of ^{239}Pu using gamma-ray spectrometry with Ge(Li) detector, led us to a detailed investigation and its absolute measurement. The disintegration rate of ^{99}Mo was determined using the recently published work. The number of fissions was calculated by irradiating accurately known and isotopically analysed amounts of ^{239}Pu and ^{235}U and monitoring the neutron flux using $^{59}Co\ (n, \gamma)^{60}Co$ reaction.

The modified 'comparison method' gave a value of 6.79 ± 0.15 for the ^{99}Mo fission yield in the thermal neutron-induced fission of ^{239}Pu. The 'absolute method' gave a value of 6.66 ± 0.07 and 6.06 ± 0.16 in the case of ^{239}Pu and ^{235}U fission respectively. The value 6.06 ± 0.16 obtained for ^{99}Mo yield in ^{235}U fission by the 'absolute method' in the same series of experiments indicates the accuracy of the present measurements since this value compares reasonably well with 6.16 which is the literature accepted value.

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1. INTRODUCTION

An accurate knowledge of ^{99}Mo fission yield in different fissioning nuclei is essential since quite often it is used as a standard due to its convenient half-life, simple decay scheme and high fission yield. Several investigators measured the fission yield of ^{99}Mo in the thermal neutron-induced fission of plutonium-239[1-5]. However, the values they obtained varied over a wide range as 5.61, 6.02, 6.10, 6.44 and 6.59. This large variation and the difficulty in selecting a particular value for ^{99}Mo yield while determining the fission yields of many other nuclides in the neutron-induced fission of ^{239}Pu using gamma-ray spectrometry with Ge(Li) detector[6-7] inspired us to investigate it in detail. The absolute disintegration rate of ^{99}Mo was determined taking into account the recently published work[8-11]. The number of fissions was calculated by irradiating accurately known and isotopically analysed amounts of ^{239}Pu and ^{235}U and monitoring the neutron flux using $^{59}Co(n,\gamma)^{60}Co$ reaction. The epicadmium neutron flux contribution towards fission or cobalt activation was less than 0.5%. Molybdenum-99 fission yield in ^{235}U fission was determined simultaneously to find out any major discrepancy in the experimental procedure used in determining ^{99}Mo yield in ^{239}Pu fission. Molybdenum-99 yield in ^{235}U fission has been reported by more than ten workers and is well established as 6.16[12]. This report describes the results of experiments carried out in detail on the fission yield of ^{99}Mo in the thermal neutron-induced fission of ^{239}Pu and ^{235}U.

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*A summary of this work was presented at the DAE Chemistry Symposium, Madras (25-28 November, 1970)

2. EXPERIMENTAL

2.1 Target Preparation

Plutonium supplied by the Fuel Reprocessing Division was used for target preparation, after chemical purification. Solution of purified plutonium was analysed for its specific activity and isotopic composition using the standardised radiometric procedure[13]. Subsequently the mass spectrometric analysis was also carried out on the plutonium sample. The ^{239}Pu content determined by the two methods was in good agreement within 0.5%. Natural uranium solution analysed for ^{235}U content by mass spectrometry and estimated gravimetrically was used. Targets from these solutions were prepared by evaporating accurately weighed amounts of solutions on 'superpure' aluminium foil of one mil thickness. These were wrapped in another aluminium foil. About 10 µg of ^{239}Pu or ^{235}U were used to avoid any self-shielding.

The targets for neutron flux monitoring should be very thin to avoid any self-shielding. They are best made from Co-Al alloys containing 0.1 to 1% cobalt known accurately and made from nuclear pure materials. There were a few such foils available from a stock supplied by the IAEA, but it was not possible to afford one for each irradiation. Cobalt solution was made from specpure cobalt metal wire. Targets from this solution were prepared by evaporating accurately weighed amounts of solution on 'superpure' aluminium foil of one mil thickness. These were wrapped in another aluminium foil. About 1 mg of ^{59}Co was used to avoid any self-shielding. Neutron flux monitors supplied by IAEA (Co-Al alloy containing 1% cobalt) were also irradiated in some of the experiments to check the self-shielding effect, impurities etc in the cobalt solution used for flux monitoring.

2.2. Irradiations

Plutonium, uranium and cobalt targets were irradiated simultaneously for 6 to 10 hours in one of the outer positions in the natural uranium fuelled heavy water moderated reactor CIRUS with a thermal neutron flux of about 10^{12} n cm^{-2} sec^{-1}. The cadmium ratio in this position for the contribution to fission products or cobalt activation was found to be greater than 200. One irradiation was carried out with about 10^{9} n cm^{-2} sec^{-1}, which is essentially a thermal neutron position.

2.3 Dissolution and Radiochemical Separations

After irradiation, the plutonium and uranium targets were separately dissolved

along with aluminium cover in acid or alkali with or without carrier. A known amount of molybdenum carrier was added to an aliquot of the solution and allowed to interchange. Molybdenum was separated, purified[14-15] and finally precipitated as lead molybdate which was convenient for mounting. The precipitates were filtered through Whatman No.42 filter paper (2.5 cm diameter) using a perspex filter chimney. They were washed, dried and weighed along with filter paper to determine the chemical yield. The samples were mounted as macro samples of about 20 mg precipitate weight on standard aluminium plates with double-coated cellulose tape and covered with cellophane paper of thickness about 3 mg/cm^2.

The neutron flux monitors were dissolved and samples were prepared for four-pi beta-gamma coincidence counting and gamma counting on NaI(Tl) or Ge(Li) detector.

2.4 Counting

2.4.1 Disintegration rate of Molybdenum-99

Figure 1 gives the decay scheme of ^{99}Mo - ^{99m}Tc. To determine the disintegration rate of this activity one should either count a high specific activity sample every time with negligible residue on VYNS films in the four-pi beta counter and apply corrections for the efficiencies of the two beta energy groups of ^{99}Mo and the ^{99m}Tc contribution or determine the counting efficiency of an end-window methane gas flow beta proportional counter with respect to lead molybdate precipitate weight. The latter was preferred since it is always convenient to process the samples with ^{99}Mo - ^{99m}Tc activity carried on an inactive carrier in a reproducible manner while to count in four-pi beta counter on VYNS films the amount of residue may vary and it is difficult to correct for the self-absorption etc in each sample. High specific activity ^{99}Mo - ^{99m}Tc solution was prepared by irradiating enriched ^{98}Mo. The purity was checked from the gamma-ray spectrum using Ge(Li) detector and following the half-life. Samples from this solution having residues less than 10 μg were prepared on VYNS films coated with gold. These were counted in the four-pi beta and four-pi beta-gamma coincidence counters. The efficiency for the two beta energy groups of ^{99}Mo, viz. 1.23 MeV (about 83%) and 0.45 MeV (about 17%) on these VYNS films was estimated using the method of Crowther and Eldridge[9]. The single channel analyser of the four-pi beta-gamma coincidence unit was gated to accept pulses of energy between 0.74 and 0.78 MeV, which is in coincidence with only the 0.45 MeV betas. From this the ratio of gamma to ccincidence counting rate gave the beta counting efficiency for the 0.45 MeV beta group and this was found to be (87.5 ± 2.5) %. The efficiency of the 1.23 MeV beta group was estimated

to be (98 ± 1) %.

The ^{99m}Tc contribution was determined by three different methods (a) following the growth of ^{99m}Tc in a purified ^{99}Mo sample in the four-pi beta counter (b) counting ^{99m}Tc on Ge(Li) detector with known efficiency for ^{57}Co peak and counting a sample prepared from this solution in four-pi beta counter and (c) by determining ^{99m}Tc in the four-pi beta-gamma coincidence counter according to the procedure of Goodier and Williams[11] and calculating the beta contribution. Different methods were tried to separate pure ^{99m}Tc from ^{99}Mo activity. The simplest method was the extraction of technetium with purified ethyl-methyl ketone from 4-5M NaOH solution of the ^{99}Mo activity. The purity was checked by taking a gamma-ray spectrum using Ge(Li) detector and following the half-life. For purifying ^{99}Mo from ^{99m}Tc, solvent extraction with tetraphenyl arsonium chloride in chloroform was used[16]. The procedure followed in (c) above for estimating the ^{99m}Tc contribution was found to give better accuracy. The decay scheme of ^{99m}Tc is shown in Figure 2. The internal conversion coefficients of the transitions γ_1 and γ_3 are very large and that of γ_2 is 0.1. If coincidences were observed between the transitions γ_1 detected in the beta counter and γ_2 in the gamma counter. The relationship between 'N$_o$' the absolute disintegration rate, and the observed beta count rate N_β and gamma count rate N_γ and the coincidence count rate N_c in a simplified form is given by equation (1)

$$\frac{N_\beta \; N_\gamma}{N_c} = N_o \left[1 + \frac{1-E_{\beta_1}}{E_{\beta_1}} \cdot \frac{E_\beta \, a\alpha_2}{1+\alpha_2} + \frac{(1-a)\alpha_3}{1+\alpha_3} \right] \qquad .. (1)$$

Here E_{β_1} is the efficiency of the beta counter to the 2 keV transition γ_1. If E_{β_1} is varied without altering E_β extrapolation of $E_{\beta_1} = 1$, gives the absolute disintegration rate 'N$_o$'. Using this method sources of ^{99m}Tc having practically no residue were counted in the four-pi beta-gamma coincidence counter. The efficiency of the beta counter to conversion electrons from the γ_1 transition was varied by altering the beta counter voltage. All measurements were carried out within the range of the normal beta plateau, so that E_β was not altered. A plot of the observed quantity $\frac{N_\beta \, N_\gamma}{N_c}$ versus $\frac{1-E_{\beta_1}}{E_{\beta_1}}$ is given in Figure 3. This gave a beta contribution of 8.8 electrons per 100 disintegrations of ^{99m}Tc counted on VYNS films under the same conditions of counting as the high specific activity ^{99}Mo - ^{99m}Tc solution. Three such experiments gave an average value of 9.0 ± 0.5 electrons per 100 disintegration of ^{99m}Tc. The ^{99m}Tc gamma activity was taken to be 0.964 times the ^{99}Mo activity at

equilibrium[9]. From these determinations it was possible to calculate the true disintegration of rate ^{99}Mo alone in the solution containing ^{99}Mo ^{99m}Tc equilibrium activity. Using this solution the efficiency of an end-window methane gas flow beta proportional counter was determined with respect to lead molybdate precipitate weight mounted and counted under standard conditions. Figure 4 gives the efficiency curve for ^{99}Mo activity alone (after subtracting ^{99m}Tc contribution) versus lead molybdate precipitate weight. From this curve the disintegration rate of ^{99}Mo was determined in samples of about 20 mg precipitate weight.

2.4.2 Disintegration rate of Cobalt-60

The decay scheme of ^{60}Co is given in Figure 5. In principle it should be possible to assay the activity by beta or gamma counting. To determine the disintegration rate of ^{60}Co by beta or gamma counting it is necessary to know the counting efficiency accurately and reproduce the sample in the same counting conditions as the standard. The presence of solids interferes in the case of beta counting. For these reasons four-pi beta-gamma coincidence counting was found to be most reliable and gave reproducible results. A standard ^{60}Co solution supplied by IAEA was counted in this counter and the disintegration rate determined was found to be in agreement within 0.3% of the value quoted by the suppliers.

2.4.3 Beta counting

Molybdenum samples from uranium and plutonium were counted under identical conditions in an end window methane gas flow beta proportional counter. All samples were counted each time, long enough to keep statistical errors below 1%. Wherever possible, samples were counted till background level was reached.

3. CALCULATIONS

The activity formed in a neutron irradiation and fission is given by equation (2).

$$A(T) = N\sigma f Y(1-e^{-\lambda t})e^{-\lambda T} \qquad \ldots \qquad (2)$$

If accurately known amounts of ^{239}Pu and ^{235}U are irradiated simultaneously and the ^{99}Mo - ^{99m}Tc activity from the samples is processed and counted under identical conditions then the fission yield is given by equation (3)

$$(Y_{Mo})_{239_{Pu}} = \frac{(A_{Mo})_{239_{Pu}}}{(A_{Mo})_{235_U}} \times \frac{N_{235_U}}{N_{239_{Pu}}} \times \frac{577}{741} \times 6.16 \quad .. \quad (3)$$

Here $(A_{Mo})_{239_{Pu}}$ and $(A_{Mo})_{235_U}$ are simply the counting rates. Equation (3) is a modified form of the 'comparison method' where the internal standard is eliminated by irradiating known amounts of the fissionable materials and assuming fission cross-section values. The thermal neutron fission cross-section values of 741 barns and 577 barns for ^{239}Pu and ^{235}U respectively were taken from the table of isotopes[17]. The half life of ^{99}Mo was experimentally observed to be 66.18 ± 0.12 hours based on the decay of more than 60 samples and a least square calculation using a computer.

Determination of thermal neutron flux using $^{59}Co(n,\gamma)^{60}Co$ reaction is a well tested method and was used as a standard procedure in more than a dozen laboratories[18]. The neutron flux in reactors can be considered under three categories.

(a) thermal neutron flux with Maxwellian energy distribution,

(b) epithermal neutron flux with an energy distribution proportional to the reciprocal of the neutron energy "E" and

(c) fast component in the MeV range.

As mentioned earlier the irradiations were carried out in the reactor in a position where the cadmium ratio was greater than 200. For this reason it is not necessary to discuss here the calculations of the epithermal or fast component of the neutron spectrum. The thermal neutron flux does not show a perfect Maxwellian distribution. These factors have been discussed by different authors, like Westcott[19] who quoted effective cross-sections for well-moderated reactors. Another important requirement is that there should be no self-shielding in the target material as otherwise it can lead to large errors[20]. The reactions in ^{59}Co irradiated with thermal neutrons are shown schematically in Figure 6(a). Due to the short half-life of ^{60m}Co and nearly complete decay to ground state through ^{60}Co, the reaction scheme can be simplified as shown in Figure 6(b) with negligible loss of accuracy.

The burn-out of ^{59}Co and ^{60}Co during the irradiation period of a day and at flux of about 10^{12} n cm^{-2} sec^{-1} is negligible. The half-life of ^{60}Co is much longer than the irradiation period. The activity formed is given by equation (4)

$$A(T) = N_0 \sigma f \lambda t \, e^{-\lambda T} \quad .. \quad (4)$$

where ϕ is the average thermal neutron flux during time t of irradiation, A(T) is the activity of ^{60}Co measured after time T from the end of irradiation, λ the decay constant, σ the neutron absorption cross-section and N_o is the number of ^{59}Co atoms. The following nuclear data assessed and recommended by Kohler[18] were used for calculation of 'ϕ' using equation (4)

(n,γ) cross-section of ^{59}Co σ	= 37.3 barns
Decay constant of ^{60}Co λ	= 4.16×10^{-9} sec^{-1}
Number of ^{59}Co atoms N_o	= 1.0219×10^{19} mg^{-1}

The ^{99}Mo fission yield in ^{239}Pu was calculated using the modified 'comparison method' i.e. equation (3). Absolute determination was made from the irradiations where the neutron flux monitors were also irradiated and all factors could be substituted in equation (2). Data of a typical irradiation and calculations are shown in the appendix.

4. RESULTS AND DISCUSSION

A summary of the fission yields of ^{99}Mo determined in the thermal neutron-induced fission of ^{239}Pu and ^{235}U is given in Table I. An average value of 6.79 ± 0.15 was obtained from ten irradiations for the ^{99}Mo yield in ^{239}Pu fission by the modified 'comparison method' using equation (3) and ^{99}Mo yield in ^{235}U fission as 6.16. The measurement by 'absolute method' using equation (2) gave a value of 6.66 ± 0.07 and 6.06 ± 0.16 in the case of ^{239}Pu and ^{235}U fission respectively. These are about 2% lower when compared to 6.79 and 6.16 in the two cases. The uncertainties even after careful determination of all the different factors in equation (2) are much more than in equation (3) and thus leads to a slightly lower value. Therefore the recommended value for ^{99}Mo fission yield in ^{239}Pu fission based on the present work is 6.79 ± 0.15. Of the parameters used in equation (3) described earlier ^{99}Mo fission yield in ^{235}U fission as 6.16 is well established[12]. The fission cross-section values are accurate within 0.5 per cent. The reliability on the number of atoms of ^{239}Pu and ^{235}U can be placed as 1% and on the ^{99}Mo activity as 2%. The overall estimated error affecting the value of ^{99}Mo yield be a maximum of 5%.

The value 6.06 ± 0.16 obtained for ^{99}Mo yield in ^{235}U fission by the 'absolute method' in the same series of experiments indicates the accuracy of the present measurements since this value compares reasonably well with 6.16 which is the literature accepted value.

The ^{99}Mo yield of the present work which is 6.79 is the highest of all, the next lower values are 6.59 determined using Ge(Li) detector by Idaho Group[5] and 6.44 the interpolated value of the mass spectrometric data of Fickel and Tomlinson[4]. The lowest is 5.61 the radiochemical value of Marsden and Yaffe[1].

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Table I

Fission yield of ^{99}Mo

S.No.	Fission yield %		
	^{239}Pu fission comparison basis	^{239}Pu fission absolute measurement	^{235}U fission absolute measurement
1	6.83		
2	6.80		
3	6.80		
4	6.62		
5	6.75		
6	6.64	6.63	5.52*
7	6.83	6.63	5.89
8	6.85	6.76	6.28
9	6.80	-	-
10	6.96	6.62	6.00
Average value	6.79 ± 0.15	6.66 ± 0.07	6.06 ± 0.16
Accepted literature value	?	?	6.16
Value recommended from the present work	6.79		"6.16"

*Excluded from the average

APPENDIX

Data of a typical Irradiation and Calculations

1. Number of ^{239}Pu atoms irradiated

   a) Weight of plutonium solution evaporated on 'superpure' aluminium foil = 129.35 mg

   b) Concentration of ^{239}Pu in the plutonium solution on the basis of specific activity and isotopic composition = 0.1497 µg of ^{239}Pu/mg of plutonium solution

   c) Therefore number of ^{239}Pu atoms

   $= 129.35 \times 0.1497 \times 10^{-6} \times 6.023 \times 10^{23} / 239 = 4.88 \times 10^{16}$

2. Number of ^{235}U atoms irradiated

   a) Weight of natural uranium solution evaporated on 'superpure' aluminium foil = 146.95 mg

   b) Concentration of ^{235}U in the uranium solution = 0.1371 µg of ^{235}U/mg of uranium solution

   c) Therefore number of ^{235}U atoms

   $= 146.95 \times 0.1371 \times 10^{-6} \times 6.023 \times 10^{23} / 235 = 5.16 \times 10^{16}$

3. Number of ^{59}Co atoms irradiated

   a) Weight of cobalt solution evaporated on 'superpure' aluminium foil = 41.05 mg

   b) Concentration of cobalt in the solution = 0.0209 mg of Co/mg of cobalt solution

   c) Therefore number of ^{59}Co atoms

   $= 41.05 \times 0.0209 \times 1.0219 \times 10^{19} = 8.77 \times 10^{18}$

4. Irradiation time

   00,05 to 10,05 hours on 25.6.70

5. Molybdenum carrier added

   To plutonium and uranium targets, 5 ml each, concentration 9.94 mg of Mo/ml = 190.25 mg of $PbMoO_4$

6. $^{99}Mo - ^{99m}Tc$ activity

From the decay curves of the macro samples followed in the end window methane gas flow beta proportional counter at 00,00 hours on 8.7.70

Pu Mo(1)	=	48100 counts/min
Pu Mo(2)	=	34000 counts/min
U Mo(1)	=	32000 counts/min
U Mo(2)	=	37200 counts/min

7. Chemical yield factors

Pu Mo(1)	=	190.25/27.55
Pu Mo(2)	=	190.25/19.85
U Mo(1)	=	190.25/25.65
U Mo(2)	=	190.25/29.20

8. Counting efficiency factors

Pu Mo(1)	=	100/27.25
Pu Mo(2)	=	100/27.75
U Mo(1)	=	100/27.00
U Mo(2)	=	100/26.75

9. ^{99}Mo disintegration rate

At 00,00 hours on 8.7.70, corrected for chemical yield and counting efficiency

From ^{239}Pu target = 1.99×10^4 dps

From ^{235}U target = 1.49×10^4 dps

10. Disintegration rate of ^{60}Co

Total activity on the basis of four-pi beta-gamma coincidence counting = 9.90×10^4 dps

11. Neutron flux

$$\phi = \frac{9.90 \times 10^4 \times 5.28 \times 365 \times 24}{8.77 \times 10^{18} \times 37.3 \times 10^{-24} \times 0.693 \times 10} = 2.02 \times 10^{12} \text{ n cm}^{-2} \text{ sec}^{-1}$$

12. Formation and decay factor for ^{99}Mo activity

$$(1-e^{-\lambda t}) = (1-e^{-\frac{0.693 \times 10}{66.18}}) = 0.09875$$

$$e^{-\lambda T} = e^{-\frac{0.693 \times 302}{66.18}} = 0.04170$$

13. On the basis of modified 'comparison method'

$$(Y_{^{99}Mo})^{239}Pu = \frac{(A_{Mo})^{239}Pu}{(A_{Mo})^{235}U} \times \frac{N_{235}U}{N_{239}Pu} \times \frac{577}{741} \times 6.16$$

$$(A_{Mo})_{239}Pu = (48100/27.55 + 34000/19.85)/2 = 1729$$

$$(A_{Mo})^{235}U = (32000/25.65 + 37200/29.20)/2 = 1261$$

$$(Y_{^{99}Mo})^{239}Pu = \frac{1729}{1261} \times \frac{5.16 \times 10^{16}}{4.88 \times 10^{16}} \times \frac{577}{741} \times 6.16 = \underline{6.96}$$

14. On the basis of 'Absolute method'

$$(Y_{^{99}Mo}) = A(T) / N\sigma f (1-e^{-\lambda t})e^{-\lambda T}$$

$$(Y_{^{99}Mo})^{239}Pu = 1.99 \times 10^{4} \times 100/4.88 \times 10^{16} \times 741 \times 10^{-24} \times 2.02 \times 10^{12} \times 0.09875 \times 0.04170 = \underline{6.62}$$

$$(Y_{^{99}Mo})^{235}U = 1.49 \times 10^{4} \times 100/5.16 \times 10^{16} \times 577 \times 10^{-24} \times 2.02 \times 10^{12} \times 0.09875 \times 0.04170 = \underline{6.01}$$

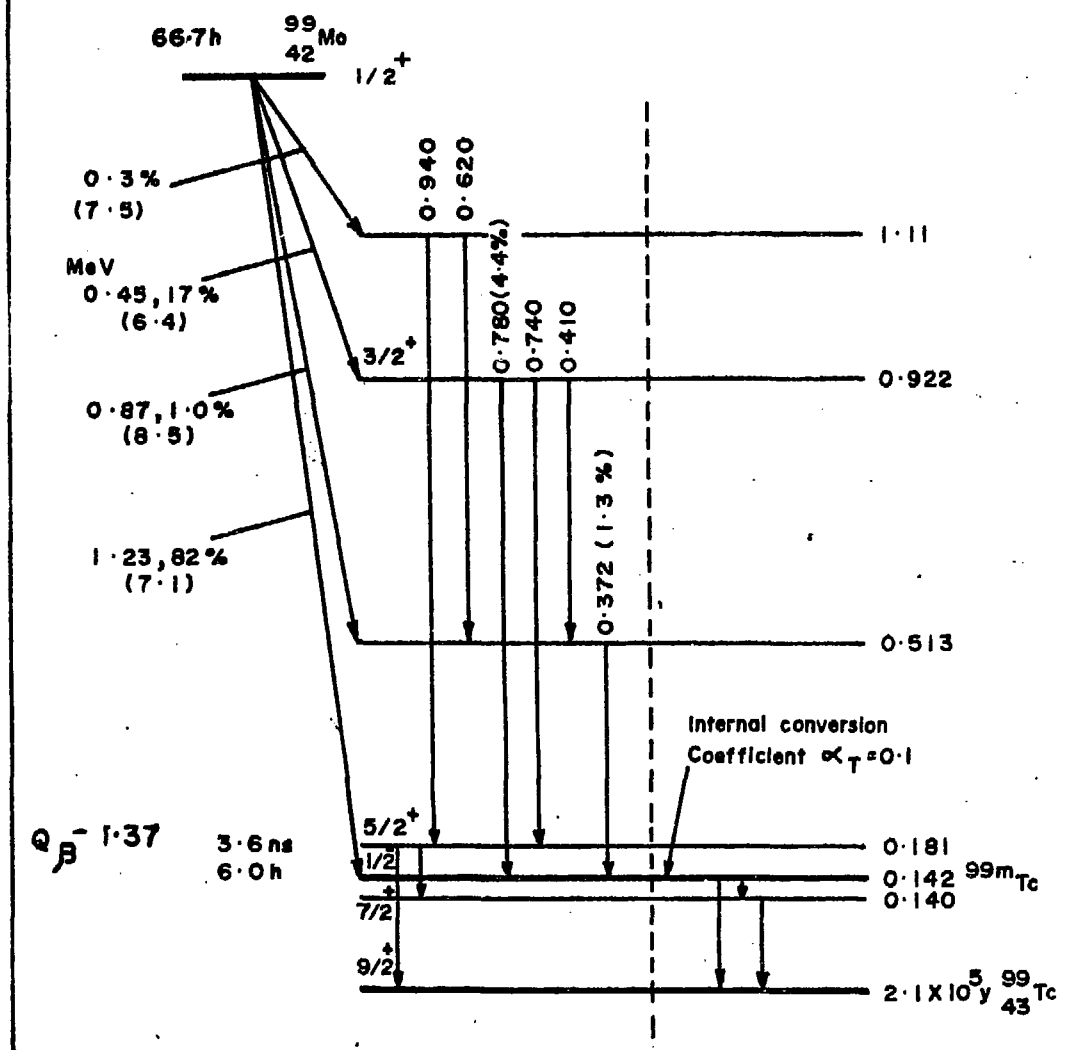

FIGURE-1. DECAY SCHEME OF ^{99}Mo-^{99m}Tc

(Transitions in ^{99m}Tc are shown to the right of vertical dotted line).

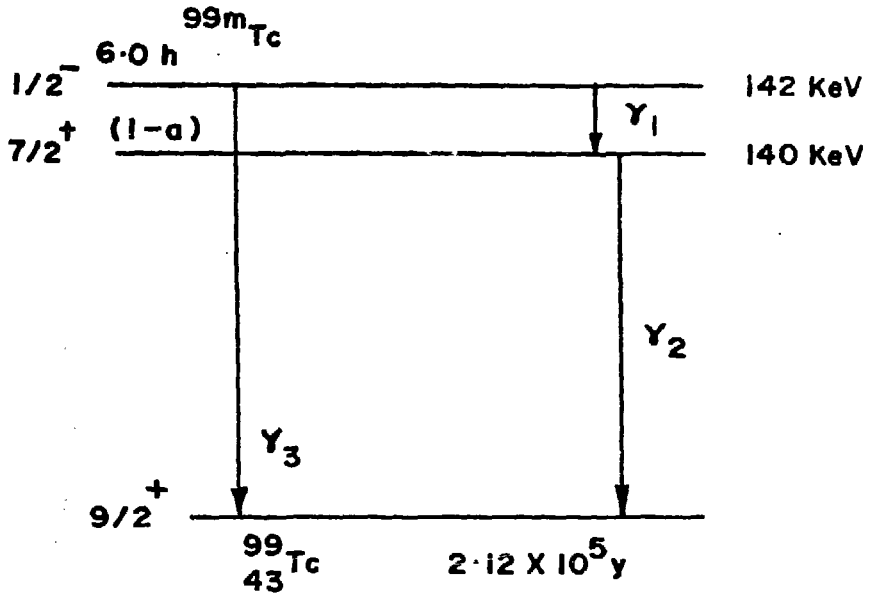

FIGURE - 2 . DECAY SCHEME OF ^{99m}Tc

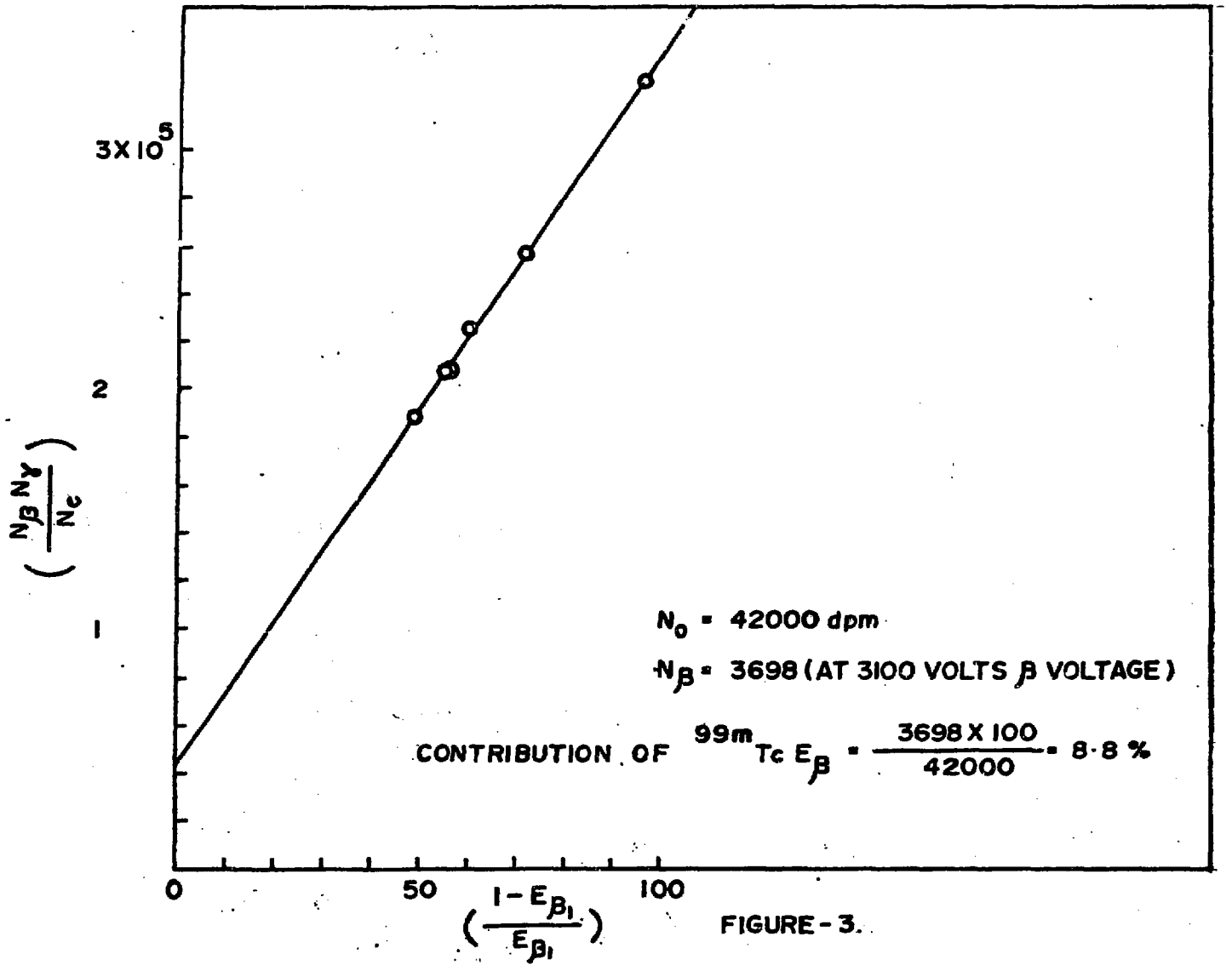
3X10^5
2
1
0
50
100
$\left(\frac{N_\beta N_\gamma}{N_c}\right)$
$\left(\frac{1-E_{\beta_1}}{E_{\beta_1}}\right)$
N_0 = 42000 dpm
N_β = 3698 (AT 3100 VOLTS β VOLTAGE)
CONTRIBUTION OF $^{99m}Tc\ E_\beta = \frac{3698 \times 100}{42000} = 8.8\ \%$

FIGURE - 3.

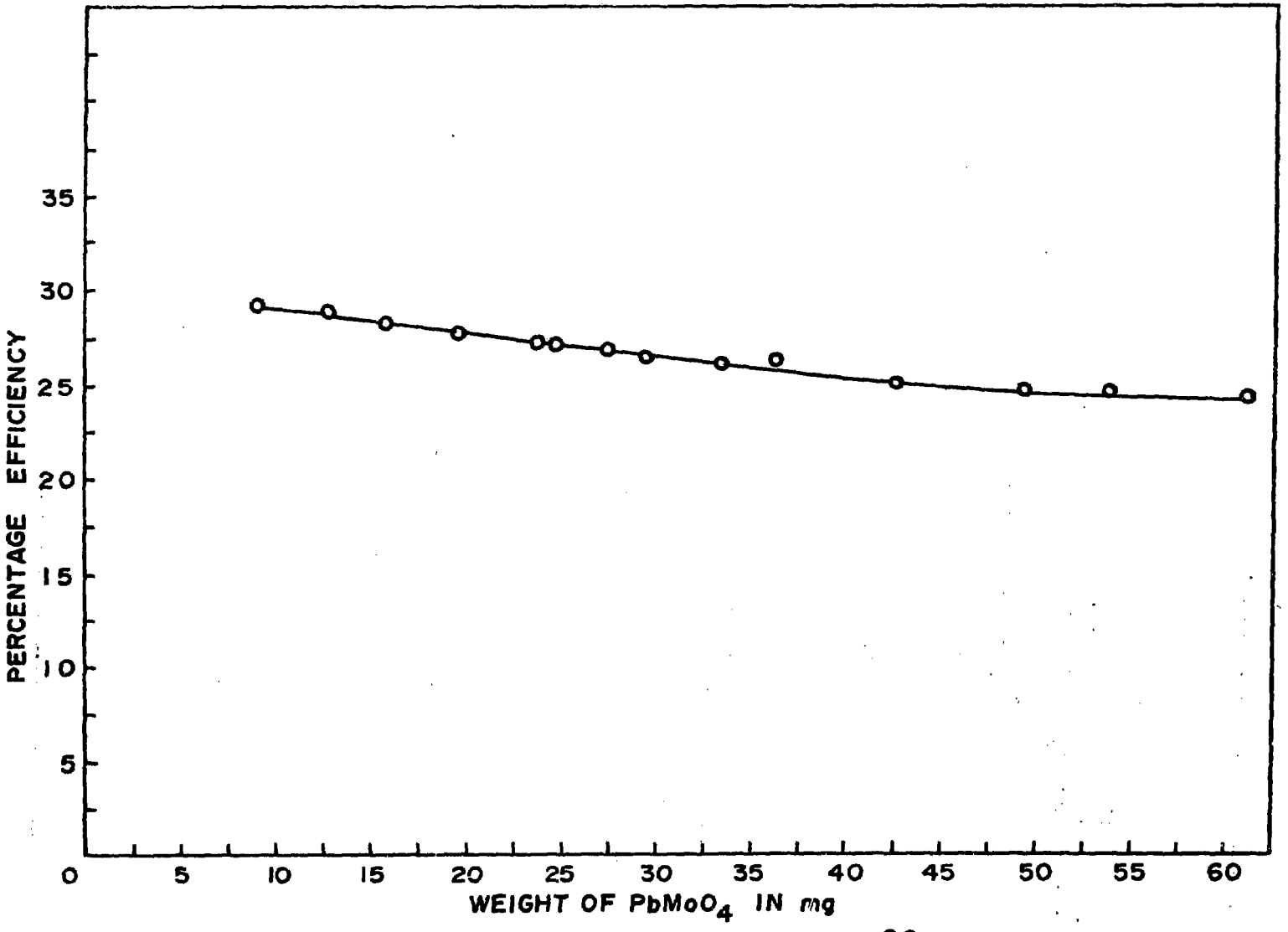

FIGURE-4. EFFICIENCY CURVE FOR ^{99}Mo

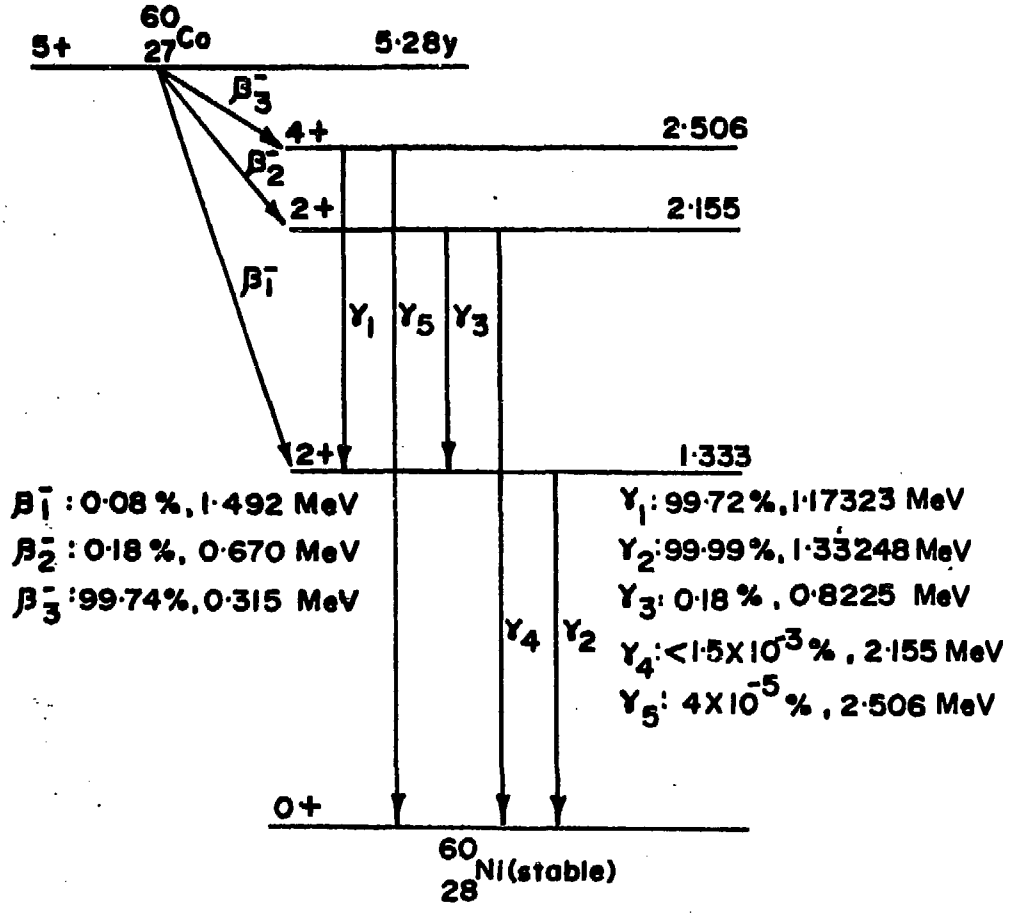

FIGURE – 5 . DECAY SCHEME OF ^{60}Co

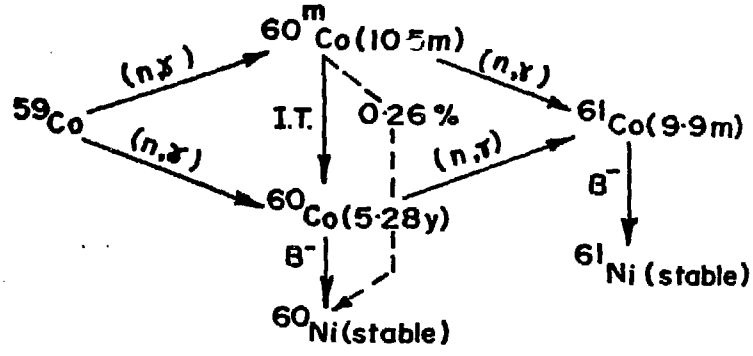

FIGURE – 6 .(a).

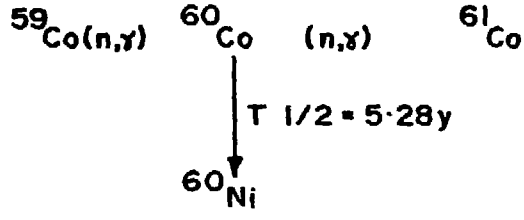

FIGURE – 6 .(b).