Search for low-yield products in the neutron-induced highly asymmetric fission of uranium

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The results of investigations aimed at defining the nature of mass distribution in the reactor neutron-induced highly asymmetric binary fission of natural, depleted, and enriched uranium are presented. Fission yields of mass chains 66, 67, 72, 77, 161, 172, 175, 177 were determined relative to ^{99}Mo using radiochemical methods. Upper limits for the yields of ^{183}Ta and ^{199}Au were also set to see the trend of mass distribution beyond $A = 177$. As expected the yields of highly asymmetric products in the region $A < 70$ and $A > 161$ are sensitive to the excitation energy of the fissioning nucleus, a trend observed in symmetric fission. Computer extrapolation of the yields up to $A = 180$ on the heavier side and $A = 60$ on the lighter side indicates that both wings of the mass-yield curve cannot be represented by a single Gaussian, thereby disturbing the complementarity of the mass-yield curve in the highly asymmetric region. The relatively higher yields for mass regions around $A = 67$ and 177, together with the upper limits for $A = 183$ and 199 in the reactor neutron fission of ^{238}U, tend to suggest the possibility of shoulders in the low-yield wings of the mass-yield curve.

INTRODUCTION

The mass-yield curves for neutron-induced binary fission of ^{235}U, ^{238}U, and other heavy-element isotopes have been well characterized in the region of high fission yields. However, not much experimental data are available along the low mass and high mass branches of the mass yield curves (especially for $A<70$ and $A>161$), except for the recent results of Nethaway *et al.*[1,2] on the 14.8-MeV neutron-induced fission of some heavy-element isotopes. Thus, it was of interest to measure some additional yields in order to define precisely the nature and trend of mass distribution in the highly asymmetric region in the thermal and fast-neutron-induced fission of heavy elements. It was also of interest to extend the yield data in low-energy fission in view of the recent discovery of ternary fission and the consequent reckoning of three distinct modes of mass distributions, viz., asymmetric ternary, asymmetric binary, and symmetric binary modes in the fission of heavy elements at moderate excitation energies.[3-5] Since the fission yields of the products in this region were expected to be extremely low, it was recognized that such an investigation would be possible only by using stringent radiochemical techniques. In this paper, we report the results and observations on the determination of cumulative yields of mass chains 66, 67, 72, 77, 161, 172, 175, 177, 183, and 199 formed in the reactor neutron-induced fission of natural, enriched, and depleted uranium.

EXPERIMENTAL

Three different samples of uranium were used in these experiments: nuclear-pure uranyl nitrate obtained from the uranium metal plant, Babha Atomic Research Center (BARC); enriched ^{235}U (93.4%) obtained from Oak Ridge National Laboratory; and depleted uranium (0.217% ^{235}U) metal obtained from the Health Physics Division, BARC. The ^{235}U content of the depleted uranium sample was estimated by fission track technique.[6]

Prior to irradiation all three uranium samples were extensively purified by extracting uranium from 2 M HNO_3 solution using 30% TBP in xylene followed by anion exchange purification, using a Dowex 2×8 (50–100 mesh) column, from 8 M HCl medium. The solvent extraction-with-anion exchange cycle was repeated 4 times. All the reagents used were of high purity.

The targets consisted of about 500 mg to 1 g of the material in the form of uranyl nitrate hexahydrate in the case of natural and depleted uranium samples, and 2–3 mg in the case of enriched uranium. They were triply sealed in thin polythene bags to avoid rupture and contamination problems. Some of the natural and all the depleted uranium targets were wrapped in 0.3-mm-thick Cd metal foil to reduce the thermal-neutron-fission contribution from ^{235}U.

The irradiations were of 24–48-h duration in the light-water-moderated enriched-uranium-fuelled swimming pool reactor APSARA at Trombay, Bombay at a flux of the order of 1.2×10^{12} n/cm^2 sec. The neutron flux above ^{232}Th(n,f) threshold in the irradiation position was estimated by us to be ~15–20% of the thermal flux. The neutron-flux values above ^{32}S$(n,p)^{32}$P threshold (2.9 MeV), above ^{54}Fe$(n,p)^{54}$Mn threshold (4.3 MeV) and above ^{54}Fe$(n,\alpha)^{51}$Cr threshold (9.1 MeV) in the reactor have been estimated to be ~10%, ~1.3%, and ~10^{-4}%, respectively, of the thermal flux.[7]

TABLE I. Fission product yield data (%).

Sl No.	Nuclide	Enriched Uranium: ^{235}U (93.4%) Our data	Enriched Uranium: ^{235}U (93.4%) Reported data (Refs. 16–18)	Natural uranium without Cd wrap[a] Our data	Natural uranium without Cd wrap[a] Reported data (Ref. 17)	Natural uranium with Cd wrap Our data	Natural uranium with Cd wrap Reported data	Depleted uranium with Cd wrap: ^{235}U (0.217%) Our data	Depleted uranium with Cd wrap: ^{235}U (0.217%) Reported data (Ref. 16)
				Low-yield products					
1	^{66}Ni	$\leq 4.60\times10^{-9}$	2×10^{-8}	$(2.00\pm0.83)\times10^{-7}$	2.9×10^{-8}	...	...	$(4.01\pm1.60)\times10^{-6}$	...
2	^{67}Cu	$\leq 1.60\times10^{-8}$	5.8×10^{-8}	$(1.66\pm0.66)\times10^{-6}$	...	$(4.20\pm1.68)\times10^{-6}$	...	$(2.32\pm0.93)\times10^{-5}$	...
3	^{72}Zn	$(5.85\pm2.34)\times10^{-6}$	1.6×10^{-5}	...	...	...	...	$(6.54\pm2.51)\times10^{-5}$	...
4	^{77}As	...	...	...	...	...	...	$(1.51\pm0.64)\times10^{-3}$	4.0×10^{-3}
5	^{161}Tb	$(1.32\pm0.52)\times10^{-4}$	4.6×10^{-4} [b] 8.2×10^{-5}	...	...	...	...	$(6.45\pm2.58)\times10^{-4}$	1.8×10^{-3}
6	^{172}Er	...	...	$(8.80\pm3.52)\times10^{-7}$	...	...	...	$(9.40\pm3.76)\times10^{-6}$	...
7	^{175}Yb	$\leq 2.4\times10^{-8}$	...	$(6.10\pm2.44)\times10^{-7}$	...	...	...	$(8.00\pm3.20)\times10^{-6}$	...
8	^{177}Lu	$\leq 1.8\times10^{-8}$	...	$(3.70\pm1.48)\times10^{-7}$	...	$(1.40\pm0.56)\times10^{-6}$	...	$(6.60\pm2.64)\times10^{-6}$	...
9	^{183}Ta	...	...	...	...	...	...	$\leq 1.13\times10^{-8}$	...
10	^{199}Au	...	...	...	...	...	...	$\leq 4.66\times10^{-9}$	...
				High-yield products					
11	^{89}Sr	(4.88 ± 0.24)	4.79	(4.66 ± 0.23)	...	(4.47 ± 0.22)	...	(3.13 ± 0.16)	[2.7;3.3]
12	^{99}Mo	...	6.20 (Ref. Std.)	...	6.20 (Ref. Std.)	...	6.20 (Ref. Std.)	...	6.20 (Ref. Std.)
13	^{111}Ag	(0.020 ± 0.001)	0.019	(0.021 ± 0.001)	...	(0.038 ± 0.002)	...	(0.0485 ± 0.0024)	0.064
14	^{140}Ba	(6.6 ± 0.33)	6.43	(5.2 ± 0.26)	...	...	...	(4.80 ± 0.24)	...

[a] The data reported earlier by us [Ref. 9 and V. K. Rao, V. K. Bhargava, S. G. Marathe, S. M. Sahakundu, and R. H. Iyer, Nucl. Phys. Solid State Phys. (India) 14B, 53 (1972)] have been revised by least-squares analysis of the decay curves by computer programs.

[b] For reactor neutron fission.

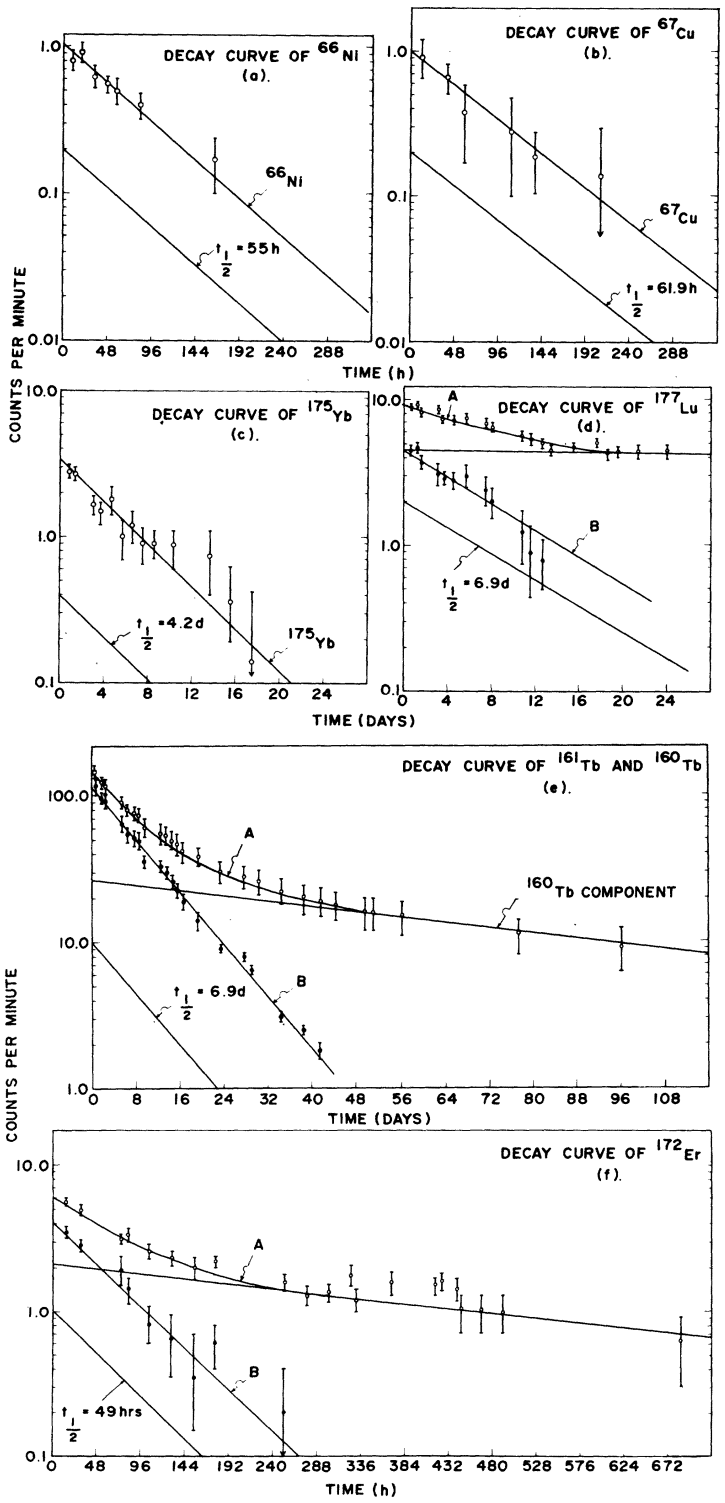

FIG. 1. β-decay curves: 1(a), 1(b), 1(c), 1(d), 1(e), and 1(f) represent the decay curves of ^{66}Ni, ^{67}Cu, ^{175}Yb, ^{177}Lu, ^{161}Tb, and ^{172}Er, respectively. Curves A and B represent the composite decay curve and that due to the pure nuclide (after subtracting the longer-lived component), respectively.

Dissolution and radiochemical processing of the targets

The following activities were isolated from the targets from different irradiations using standard radiochemical carrier techniques[8]: ^{66}Ni, ^{67}Cu, ^{72}Zn, ^{77}As, ^{161}Tb, ^{172}Er, ^{175}Yb, ^{177}Lu, ^{183}Ta, ^{199}Au, ^{99}Mo, and a few other high-yield binary products like ^{89}Sr, ^{111}Ag, ^{140}Ba.

The irradiated targets were dissolved in dilute HCl in the presence of 10–15 mg each of standard carriers of Ni, Cu, Zn, As, Tb, Tm, Er, Yb, and Lu. The target solution was made up to 50 ml in a standard flask. 1 ml from this solution was withdrawn and 10–15 mg each of standard carriers of Mo, Sr, Ag, and Ba were added for the separation of these high-yield products. From the rest of the solution, the activities of interest were separated and purified. A separate irradiation was performed for isolating ^{183}Ta and ^{199}Au acti-

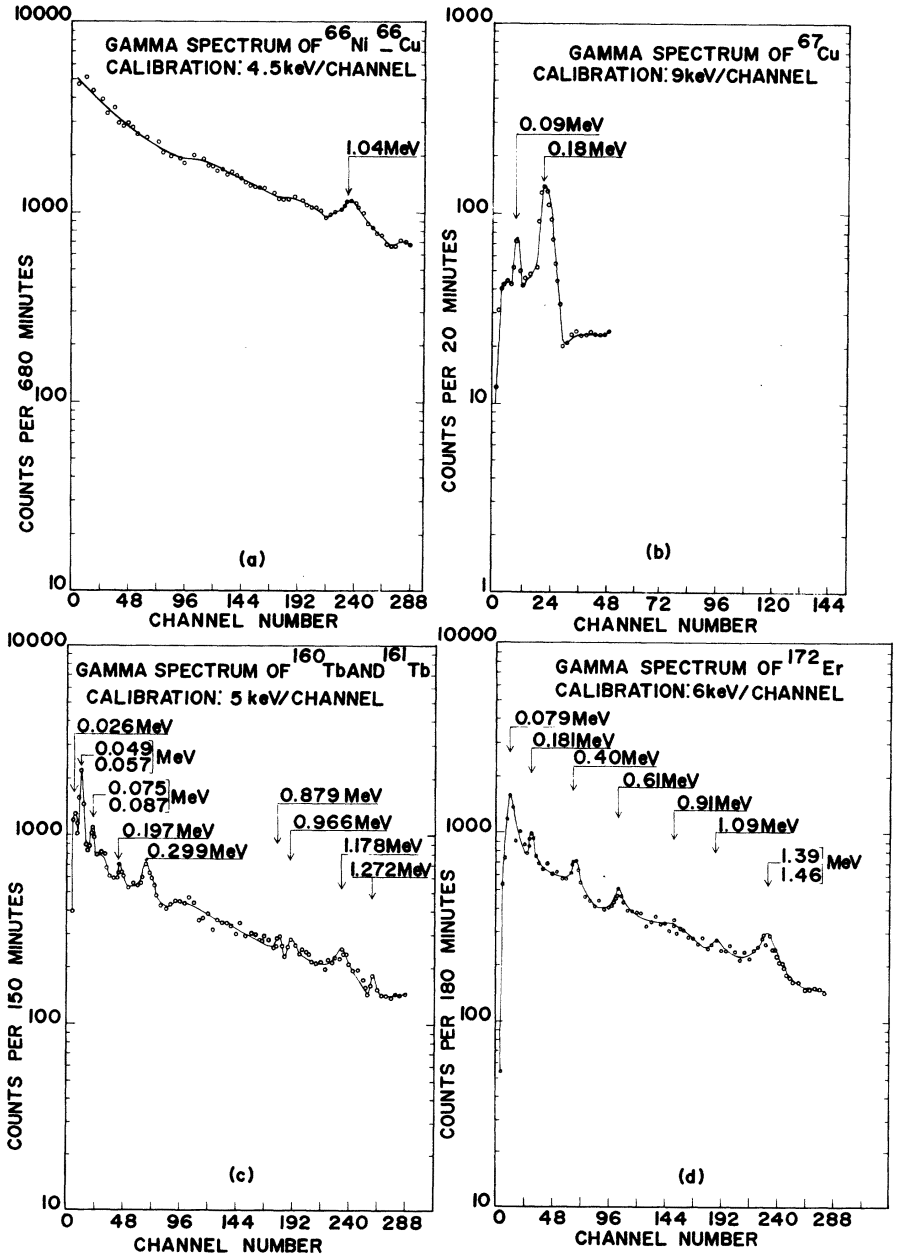

FIG. 2. γ spectra of low-yield fission products: 2(a), 2(b), 2(c), and 2(d) represent the γ spectra of the ^{66}Ni-^{66}Cu, ^{67}Cu, ^{160,161}Tb, and ^{172}Er-^{172}Tm decay sequences, respectively.

vities from the depleted uranium target. 15 mg each of standard carriers of Ta and Au were used for their separation. The existing procedures were suitably modified[9,10] to meet the stringent requirements of separating radiochemically pure products having activities of the order of a few counts per minute (~1–5 cpm) from tens of millicuries of high-yield binary-fission products and other activation products such as ^{239}Np. The activities were mounted in the form of precipitates of known stoichiometry and counted in a low-background β counter with anticoincidence shielding (background 0.9–1 cpm). Since the activities of the samples were low and since the nuclides of interest were of fairly long half-life, all the samples were counted each time long enough (for periods not less than 5000 sec) to accumulate enough counts to reduce the statistical uncertainty in the counting to a minimum. The statistical deviation of counting in each case was computed using the standard procedure.[11] The radiochemical purity was ascertained by the characteristic decay curves [Figs. 1(a)–1(f)], and in the case of ^{66}Ni, ^{67}Cu, ^{161}Tb, and ^{172}Er also by their specific γ-ray energies[12]—determined using a NaI(Tl) detector coupled to a 400-channel analyzer [Figs. 2(a)–2(d)]. In most cases the decay could be followed through at least three to four half-lives. γ counting also was done for a sufficiently long duration with the sample in the form of mounted precipitates or in the form of a solution in a well-type detector. The decay curves were analyzed by a least-squares-fitting program using a Honeywell 400 computer.

Evaluation of fission yields

The observed activities were corrected for chemical yield, decay, efficiency of the counting system, and volume of target solution. The efficiency of the low background counter was determined relative to the β proportional counters for which efficiencies were known.[13] Fission yields were calculated relative to ^{99}Mo taken as internal standard using the relation

$$Y_i(\%) = Y_{99_{Mo}}(\%)\,\frac{N_i}{N_{99_{Mo}}}\,, \qquad (1)$$

where Y_i and N_i are the fission yield and number of atoms, respectively, of a particular nuclide i and $Y_{99_{Mo}}$ and $N_{99_{Mo}}$ are the corresponding yield and number of atoms of ^{99}Mo, respectively. The number of atoms N_i were calculated using the relation

$$N_i = \frac{A_i t}{[1-\exp(-\lambda_i t)]}\,, \qquad (2)$$

where A_i is the activity in dpm associated with nuclide i at the end of the irradiation, λ_i the decay constant, and t the duration of irradiation. The yield of ^{99}Mo was assumed to be 6.2% in all three cases. The observed isotopic yields were corrected for independent yields to get the total isobaric yields. The charge-distribution correction suggested by Wahl *et al.*[14] was used.

RESULTS AND DISCUSSION

The results of yield determinations for the mass chains 66, 67, 72, 77, 161, 172, 175, and 177 are given in Table I. In one irradiation with Cd-wrapped depleted uranium target, we isolated ^{183}Ta and ^{199}Au activities in order to see the trend of the yields beyond $A=177$. The yields of some representative high-yield binary-fission products, e.g. ^{89}Sr, ^{140}Ba, and ^{111}Ag, were also determined to fix the light and heavy peaks and the trough regions of the mass-yield curve. The yield values reported in this work (Table I) for the high-yield and low-yield products are based on at least two (in a majority of the cases three to four) determinations. The yields are in the range 10^{-5}–10^{-7}% for the mass chains in the highly asymmetric region. The yields obtained for mass chains 183 and 199 are only upper limits since the activities isolated could not be unambiguously identified as ^{183}Ta and ^{199}Au.

Since the highly asymmetric binary products are low fission cross-section events, the effect of target impurities that may give rise to products of interest by neutron activation, was seriously considered. Impurity fractions resulting from the purification of the targets and reagent blank were subjected to emission spectrographic as well as activation analyses. Impurity fractions equivalent to 5 g of uranyl nitrate were used for these analyses, while the uranium targets used for irradiation were 5–10 times less in all cases. Spectrographic analysis did not reveal the presence of any of the three rare earths, i.e., Lu, Yb, and Er. Activation analysis showed the presence of some unidentified long-lived activity in the impurity fraction as well as in the reagent blank in the case of lutetium (probably due to natural lutetium activity). Similar long-lived tails were observed in the decay curves of ^{177}Lu separated from the uranium targets (natural lutetium shows ~0.7 cpm/mg due to ^{176}Lu in the counter used). It may be noted in this context that the presence of about 0.02 ppm of lutetium in the target may give rise to about 10^4 dpm of ^{177}Lu by ^{176}Lu$(n,\gamma)^{177}$Lu reaction under the conditions of irradiation. The absence of such abnormal ^{177}Lu activity in the irradiated uranium targets coupled with the results

of spectrographic and activation analyses are taken as evidence for the purity of the targets employed. The presence of Zn impurity (~0.7 ppm) was detected in depleted uranium samples. The formation of ^{66}Ni by ^{70}Zn$(n, n\alpha)^{66}$Ni may be regarded as negligible, as the abundance of ^{70}Zn and the cross section for the above reaction are small. The correction for the fission yield of ^{67}Cu due to ^{67}Zn$(n,p)^{67}$Cu was found to be <1% on the basis of available data[15] on the reaction cross section and threshold energy. All other possible nuclear reactions[15] leading to the formation of ^{66}Ni, ^{67}Cu, ^{172}Er, ^{175}Yb, and ^{177}Lu, etc., were considered while calculating the yields and their effect was found to be negligible. It may be observed that the formation of ^{172}Er by nonfission nuclear reactions under reactor neutron irradiations is extremely improbable.

The use of larger amounts of the target material undoubtedly would give better statistics of the observed data. However, in view of the restrictions imposed on the amount of fissile material to be used in the reactor for irradiation and on the amount of cadmium to be employed for wrapping the targets, the size of the targets had to be restricted in the present work. Further, higher amounts of activities (high-yield binary-fission products and activation product, e.g. ^{239}Np) associated with larger targets are likely to complicate the separation procedures in obtaining radiochemically pure products of interest within a reasonable time (considering the half-lives of nuclides). It may be noted, however, that in spite of the restrictions on target size and irradiation time, we were able to identify the low-yield products unambiguously (see Figs. 1 and 2) and estimate their yields with a reasonable degree of confidence.

In view of the low activities involved, uncertainties in the counter efficiencies, analysis of decay curves, etc., an uncertainty of ±40% is assumed for the measured yields of the highly asymmetric products. Figure 3 shows the plot of the yields in the mass range 72–161 available in literature[16] for the thermal neutron fission of ^{235}U as well as for reactor neutron fission of ^{238}U. The data were extended by computer extrapolation up to $A=180$ on the heavier side and up to $A=60$ on the lighter side by assuming a Gaussian distribution[1,2] of the yields and calculating the yield, Y, as

$$Y = \frac{F}{\sqrt{2\pi}\,\sigma} \exp\left[-\frac{1}{2}\left(\frac{A-\bar{A}}{\sigma}\right)^2\right] , \qquad (3)$$

where σ is the width of the Gaussian, F is the normalizing factor, and $\bar{A}$ =116.78 and 118.18 for ^{235}U and ^{238}U, respectively[19] (2.44 and 2.64 prompt neutron emissions were assumed for the two nuclides). Data from present work are also shown in Fig. 3 for comparison. While trying to extrapolate the available data, we found that a single Gaussian curve does not represent the lighter as well as the heavier side of the mass-yield curve thereby disturbing the complementarity of the mass distribution, particularly in the highly asymmetric region. Even for one side of the mass-yield curve, the width of the Gaussian was found to vary, depending critically on the mass numbers employed in the calculation of the yields. The extrapolations in Fig. 3 were made using the mass-yield data from $A=155$ to 161 on the heavier side and $A=83$ to 72 on the lighter side. Since the present yields are of the order of 10^{-6}–10^{-7}% and lie closer to the ^{238}U mass-yield curve and since we could not detect these products (^{66}Ni, ^{67}Cu, ^{172}Er, ^{175}Yb, and ^{177}Lu) unambiguously in the reactor neutron fission of ^{235}U, we believe that these products are arising most likely from reactor neutron-induced fission of ^{238}U rather than from ^{235}U fission. The only other data available for comparison with the present work in the highly

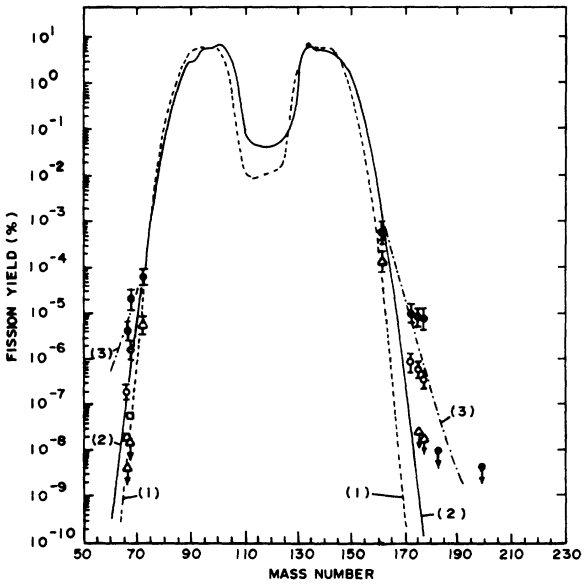

FIG. 3. Mass distribution in the fission of ^{235}U by thermal neutrons and ^{238}U by reactor neutrons. Curves 1 and 2 are drawn using the available data (Ref. 16) on thermal neutron fission of ^{235}U and reactor neutron fission of ^{238}U, respectively. Both these curves have been extrapolated beyond $A=70$ on the lighter side and $A=161$ on the heavier side using Eq. (3) referred to in the text. The triangles, the open circles, and the closed circles represent data from the present work on ^{235}U, natural uranium, and Cd-wrapped depleted uranium, respectively. The squares represent reported data (Refs. 17 and 18) on ^{235}U. Line 3 represents graphical interpolation of the available data on ^{238}U (Ref. 16) and the present data on Cd-wrapped depleted uranium (closed circles).

asymmetric region are those for ^{66}Ni and ^{67}Cu [17, 18] in the thermal neutron fission of ^{235}U. The upper limits obtained in the present work are lower than the reported values and this may be due to the highly purified target material and improved radiochemical and counting techniques employed in the present work.

The data available on yields beyond $A=161$ are those reported by Nethaway, Mendoza, and Voss[1] and these have been used along with other available data[16, 20, 21] up to $A=161$ to illustrate the energy dependence of the yields of very asymmetric products in the fission of ^{235}U and ^{238}U by neutrons of different energies (Figs. 4 and 5). The dotted portions in these figures are computed up to $A=180$. It is evident from these figures that the yields of very asymmetric products are sensitive to the excitation energy of the compound nucleus, a trend well established in the case of symmetric fission. It can also be seen from these figures that this energy dependence increases with increases in asymmetry. The yields obtained in the present work for mass chains 172, 175, and 177 in the reactor neutron fission of ^{238}U follow this general trend although they are a few orders of magnitude higher than the corresponding computed values, as can be seen from Fig. 5. This discrepancy is perhaps due to the difference in the neutron energy spectra[7] used in the present work as well as due to the critical dependence of the shape of the extrapolated curve on the width of the Gaussian, as already outlined earlier. Another possi-

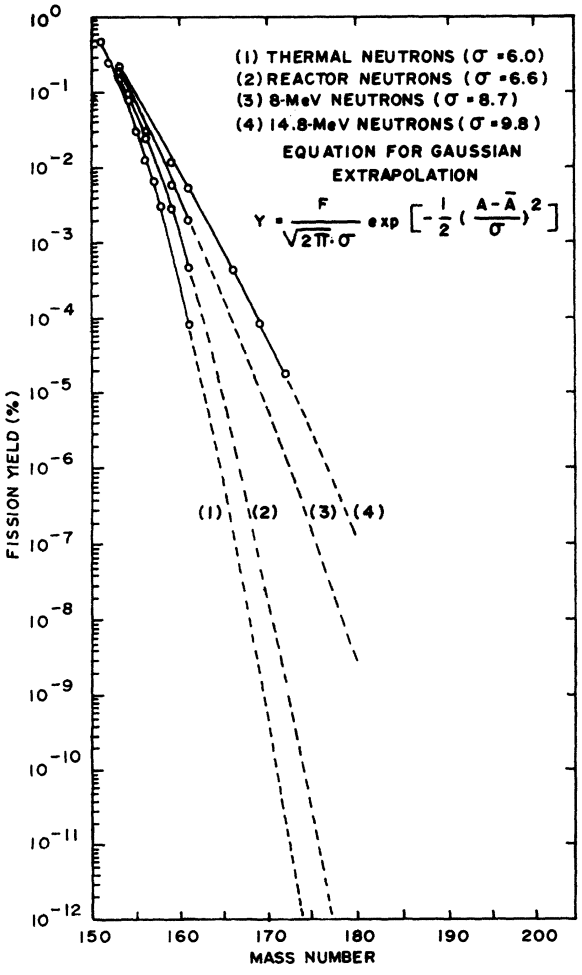

FIG. 4. Low-yield products in the heavier mass region from the fission of ^{235}U with neutrons of different energies. Curves 1, 2, 3, and 4 are drawn using the available data (Refs. 1, 16, 20, and 21) indicated by open circles. The dotted portions of the curve are computer extrapolations using Eq. (3) in the text.

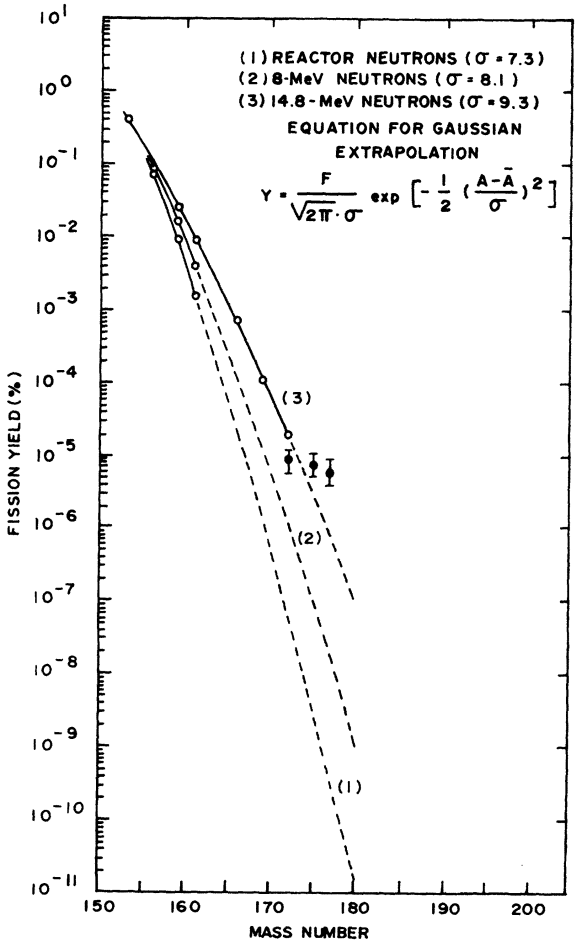

FIG. 5. Low-yield products in the heavier mass region from the fission of ^{238}U with neutrons of different energies. Curves 1, 2, and 3 are drawn using the available data (Refs. 1, 16, and 20) indicated by open circles. The yields of mass chains 172, 175, and 177 from this work in the reactor neutron fission of Cd-wrapped ^{238}U are indicated by closed circles. The dotted portions of the curves are computer extrapolations using Eq. (3) in the text.

bility is the broadening of the wing portions of the mass-yield curve in this region (and therefore the measured yields may not follow the computed curve). However, the upper limits for $A = 183$ and 199 suggest that this broadening tendency does not continue much beyond $A = 177$ in the present system.

A perusal of the data in Table I shows that the yields of highly asymmetric products in the different cases studied have the following trend: Cd-wrapped depleted uranium >Cd- wrapped natural uranium > natural uranium > enriched uranium. This order can be explained on the basis of the difference in the total number of fissions occurring in the different targets resulting from the slow-neutron-induced fission of ^{235}U present in different amounts. For example, the yields of representative products ^{66}Ni and ^{177}Lu in the fission of Cd-wrapped depleted uranium are higher by a factor of about 5 than those in the fission of Cd-wrapped natural uranium (see columns 7 and 9 in Table I). In view of the uncertainties in the yield measurements, this ratio may be considered to be in fair agreement with the ratio of ^{235}U content present (~3.5) as well as the ratio of ^{99}Mo activities (~5) obtained in these two cases when normalized to unit weight of target and unit time of irradiation.

The fact that the yields of the products ^{172}Er, ^{175}Yb, and ^{177}Lu in ^{238}U fission are nearly equal and are of the order $10^{-6}\%$ and lie much above the expected values (extrapolated for the fast fission of ^{238}U) together with the low limits for ^{183}Ta and ^{199}Au tend to suggest the possibility of a shoulder in this region of the mass-yield curve (see Fig. 3). A similar trend also appears to be seen around mass number 67, as is evident from the higher yield than the computed value obtained for ^{67}Cu. It may be observed that there is a 28-proton core in the low mass branch of the mass-yield curve (in the ^{66}Ni-^{67}Cu region) and it is not known at present whether the small indication of shoulders in the low-yield wings of the mass-yield curve is in some way connected with it or not. It will be interesting to determine the yields of other nuclides around $A = 67$ and 177 to settle this point.

For a clearer understanding of these observations, detailed studies of mass distribution around $A > 161$ and $A < 70$ employing different fissile materials and different neutron energies are highly desirable. At present, efforts are under way to extend these studies to ^{232}Th, ^{233}U, and ^{239}Pu.

SUMMARY

We have measured the cumulative yields of mass chains 66, 67, 72, 77, 161, 172, 175, and 177 in the reactor neutron-induced fission of enriched, natural, and depleted uranium using radiochemical techniques. The yields range from 10^{-5} to $10^{-7}\%$ in this region. Upper limits were also set for the yields of ^{183}Ta and ^{199}Au in the reactor neutron-induced fission of ^{238}U. The yields of mass chains 66, 67, 175, 177 in the fission of enriched uranium are also upper limits since activities due to ^{66}Ni, ^{67}Cu, ^{175}Yb, and ^{177}Lu were not identified unambiguously. Comparison of the present data with other similar data from the literature brings out the dependence of yields of very asymmetric products on the excitation energy of the fissioning nucleus, the dependence being more pronounced with increasing asymmetry. Computer extrapolation of the yields in the regions $A > 161$ and $A < 70$ indicates that both wings of the mass-yield curve cannot be represented by a single Gaussian. The present data on ^{238}U indicate a general broadening of the wings of the mass-yield curve in the highly asymmetric region. The relatively higher yields for products in the mass region $A = 67$ and 177 together with the low limits for 183 and 199 tend to suggest the possibility of shoulders in the low-yield wings of the mass-yield curve.

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