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ISOTOPIC IDENTIFICATION OF PHOTOFISSED NUCLEAR MATERIALS IN STAINLESS STEEL CONTAINERS USING DELAYED GAMMA-RAYS

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The results of isotope identification of nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) packed in hermetic stainless steel containers by their stimulated delayed gamma radiation are presented. The ratios of the values of the time dependences of the intensity of gamma radiation of the light and heavy product of their photofission were used for the analysis. Gamma radiation from photofission products ^{89}Rb (1031.9; 1248.1 keV) and ^{138}Cs (1009.8; 1436.8; 2218.0; 2639.6 keV) was used to differentiate nuclear materials. The photofission reaction the samples of nuclear materials was stimulated on an electron accelerator, an M-30 microtron, at maximum bremsstrahlung energy of 12.5 MeV. The possibility of carrying out isotopic identification of nuclear materials using the following sets of ratios of the values of the time dependences of the gamma-line intensity has been demonstrated: X(1031.9)/X(2639.6), X(1248.1)/X(1009.8), X(1248.1)/X(1436.8), X(1248.1)/X(2218.0), X(1248.1)/X(2639.6).

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INTRODUCTION

The control of non-proliferation of shielded (in hermetically sealed stainless steel containers) nuclear materials (fertile – ^{232}Th, ^{238}U and fissile – ^{235}U, ^{239}Pu) is one of the most important tasks of atomic science and technology [1]. The reliable information about their isotopic composition is required to successfully solve this problem. One of the widely used non-destructive methods for the detection and isotopic identification of unshielded and shielded nuclear materials is based on the use of delayed gamma-rays from their fission products [1–10].

The essence of this method is to use experimentally obtained information on the ratio of the intensity of stimulated delayed gamma-rays from light and heavy fission products [1–4]. The yields of light product fragments significantly depend on the mass of fissile nuclei. Their mass distributions shift towards larger masses with increasing mass of fissile nucleus at the fixed excitation energies. At the same time, the mass distribution of heavy fragments is almost constant and does not depend significantly on the mass of the fissile nucleus (Fig. 1) [11].

As a result of the theoretical analysis [11, 12] and experimental [2–9] studies, the possibility of using ^{89}Rb and ^{138}Cs products obtained as a result of fission by neutrons [2–5] and photons [6–10] for isotope identification of unshielded [2–7] and shielded [8–10] nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) has been established.

The numerical values of the ratio of the yields of a light fragment to a heavy fragment (Y(A = 89)/Y(A = 138)) for the photofission of ^{232}Th, ^{235}U, ^{238}U, ^{239}Pu with bremsstrahlung at 12.5 MeV boundary energy are equal to 0.944, 0.691, 0.496 and 0.369, respectively. The differences between the numerical values of the fragments yield ratio (Y(A = 89)/Y(A = 138)) for neighbouring pairs of nuclei are 26.8% for ^{232}Th and ^{235}U, 28.3% for ^{235}U and ^{238}U, 25,6% for ^{238}U and ^{239}Pu, respectively [11,12]. The accuracy of determining the absolute values of the yields of fission products, based on the measurement of their radioactive characteristics (gamma radiation from fission products), is equal to 3…6 % [13, 14].

Stimulation of the fission reaction of shielded nuclear materials by bremsstrahlung [8–10] has an advantage due to the threshold nature of photonuclear reactions [15–17].

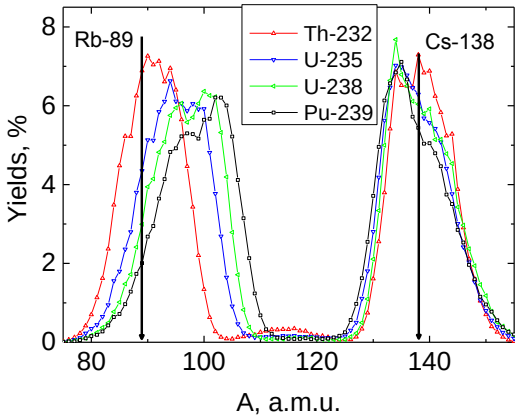

Fig. 1. Products yields of nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) photofission [11]

As a result of the analysis carried out in [18], it was found that the optimal bremsstrahlung energy for stimulating delayed gamma quanta from shielded (in hermetically sealed stainless steel containers) nuclear materials should not exceed 12.5 MeV. That is, the bremsstrahlung energy must be below the thresholds of possible photonuclear reactions for the elements from which the container is made [18,19].

The aim of this work is to experimentally study the possibility of using (applying) delayed gamma rays from photofission products (^{89}Rb and ^{138}Cs) of nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) packed in hermetic stainless steel containers for their isotope identification on the electron accelerator – microtron M-30.

1. MATERIALS AND METHODS

Determination of the ratio of the intensity of delayed gamma-radiations of light (^{89}Rb) to heavy (^{138}Cs) product of photofission of nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) in hermetic stainless steel containers was carried out by semiconductor gamma spectrometry [20, 21]. The investigated quantity in measurements is the count rate in the peaks of total absorption (or peak intensity) of gamma quanta of fission products (^{89}Rb and ^{138}Cs), which depends on the values of their activities, the values of the absolute measurement efficiency, corrections for self-absorption and the intensity of gamma lines, and the values of fission product yields and the number of fission events in the sample [21].

1.1. THEORETICAL BACKGROUND

The experimentally determined quantity in the isotopic analysis of nuclear materials is the count rate in the total absorption peaks (*n(E)*) from the products of their photofission and is given by formula (1) [21]:

$$n(E) = q\varepsilon(E)I_\gamma(E)s(E), \quad (1)$$

where $\varepsilon(E)$ is the spectrometer efficiency for energy E; $I_\gamma(E)$ is the absolute quantum yield of the gamma line with energy E; $s(E)$ is the correction for self-absorption in the source at this energy.

If X_i is given by formula (2)

$$X_i = Y_i\varepsilon(E)I_\gamma(E)s(E)n_f, \quad (2)$$

(Y_i – photofission product yield, n_f – the number of divisions in the sample per unit of time) then relations (1) can be written as:

$$\begin{cases} n_1(t_{ir}, t_c) = X_1\left(1 - e^{-\lambda_1 t_{ir}}\right)e^{-\lambda_1 t_{ir}} \\ n_2(t_{ir}, t_c) = A_1 e^{-\lambda_1 t_c} + A_2 e^{-\lambda_2 t_c} \end{cases}, \quad (3)$$

where

$$A_1 = X_1 \frac{\lambda_2}{\lambda_2 - \lambda_1}\left(1 - e^{-\lambda_1 t_{ir}}\right), \quad (4)$$

$$A_2 = \left(X_2 - X_1 \frac{\lambda_2}{\lambda_2 - \lambda_1}\right)\left(1 - e^{-\lambda_2 t_{ir}}\right). \quad (5)$$

The pulse counting rate at the photopeak, measured over the time interval $[t_i, t_i + \tau]$, is determined by the relation (6):

$$n(t_i, \tau_i) = \frac{1}{\tau_{ilive}} \sum_{j=1}^{2} A_j e^{-\lambda_j \tau_i} \frac{\tau_{ilive}}{\tau_i} \frac{\left(1 - e^{-\lambda_j \tau_i}\right)}{\lambda_j}, \quad (6)$$

where τ_{ilive} is the "live" measurement time.

From the experimental set of measured numerical values of n_{exp} at different cooling times and taking into account the values of their errors $\sigma_n(t_i, \tau_i)$, the least squares method is used to find the values of the coefficients A_j and their errors $\sigma(A_j)$ by minimizing the expression (7):

$$S = \sum_i \left[\sum_{j=1}^{2} A_j e^{-\lambda_j t_i} \frac{\left(1 - e^{-\lambda_j \tau_j}\right)}{\lambda_j \tau_i} - N_E(t_i, \tau_i)\right]^2 [\sigma^2(t_i, \tau_i)]^{-1}. \quad (7)$$

The value of X_i for a particular photofission product depends on the value of Y_i according to (2); the remaining parameters (spectrometer efficiency for energy E; absolute quantum yield of a gamma line with energy E; correction for self-absorption in the source) are constant.

The values of Y_i, depend on the count rate at the peak of total absorption, the time of irradiation, exposure and measurement, as well as the half-lives of the parent and daughter members of the isobaric chain.

The error in determining X_i is mainly related to the count rate at the peak of total absorption (i.e., the area of the peak); other parameters do not introduce significant errors.

Thus, the values of X_i obtained from experimental measurements of the peak counting rate can be used for the isotopic identification of nuclear materials.

1.2. SAMPLES OF NUCLEAR MATERIALS IN HERMETIC CONTAINERS

Samples (targets) with isotopes of actinides nuclei ^{232}Th (weight – 1.0018 g), ^{235}U (0.514 g), ^{238}U (1.119 g), ^{239}Pu (0.400 g), which were packed in hermetic stainless steel containers, were used for experimental studies. All containers had the shape of a cylinder (diameter – 23 mm, height – 30 mm, weight – 70 g). The containers were made from stainless steel grade 12X18H10T. A spectral analysis was carried out to determine the precise number of chemical elements in a steel sample, since the reference book of standards [15] determines only the percentage limits for the presence of chemical elements in various types of steel grades. The percentage of chemical elements in steel were – $_6$C – 0.07 (≤ 0.12), $_{14}$Si – 0.6 (≤ 0.8), $_{15}$P – 0.03 (≤ 0.035), $_{16}$S – 0.02 (≤ 0.02), $_{22}$Ti – 0.8 (~ 0.4…1), $_{24}$Cr – 17.6 (17…19), $_{25}$Mn – 1.6 (≤ 2), $_{26}$Fe – 70.06 (65.73…70.33), $_{28}$Ni – 9.2 (9…11), $_{29}$Cu – 0.02 (≤ 0.3), $_{26}$Fe – 70.06 (65.73…70.33). The values of the limits of the content of chemical elements in steel from the reference book of standards are given in parentheses.

Samples of nuclear materials in hermetic stainless steel containers were manufactured at the V.G. Khlopin Radium Institute (Saint Petersburg, Russia). The samples were not exposed to radiation before the experimental studies.

1.3. SIMULATION OF THE SAMPLES IN HERMETIC CONTAINERS ACTIVATION PROCESS

To simulate the spectra of photons, residual electrons and secondary photoneutrons that hit the screened target, a C++ program was developed using the Geant4 toolkit [22, 23]. The program was developed for the Windows platform using the multi-threading mode.

The modeling implemented the technical characteristics of the M-30 microtron, the design features of the electron output unit [24] and the activation schemes of the samples of nuclear materials, which were packed in hermetic stainless steel containers (Fig. 2).

The simulation considered the geometric dimensions of the original electron beam: the shape – an ellipse, the dimensions of the semi-axes – 11 and 3 mm) and the characteristic of the samples of nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu), which were packed in hermetic stainless steel containers.

Calculations were performed for 10E9 electrons in the initial beam on computers with 6-core Intel(R) Core(TM) processors i7-9750HCPU@2.60GHz and 36 GB RAM.

The influence of the elements of the activation circuit (converter, B_4C filter [25], stainless steel containers) on the final shape of the spectra of photons, residual electrons and secondary photoneutrons, which hit the samples of nuclear materials were established (Fig. 3).

As a result of the simulation, the total number of photons, residual electrons and secondary photoneutrons

normalized per electron hitting the samples of nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) in stainless steel containers was calculated: 2.870E-3 photons (with an energy > 6 MeV, involved in stimulating the photofission reaction – 2.862E-4); 3,892E-5 electrons (with an energy > 6 MeV – 2.454E-6).

When performing calculations according to the indicated activation scheme, photoneutrons that hit samples of nuclear materials in sealed containers made of stainless steel were not detected.

The influence of structural elements (B_4C filter, stainless steel) of the sample's activation scheme on the characteristics of photons and residual electrons that hit on nuclear materials was analyzed. A filter made of B_4C absorbs 15.96% of photons (with an energy > 6 MeV – 8.20%). Filter with stainless steel absorb 42.02% of photons (with energy > 6 MeV – 36.31%) and – 95.46% of electrons (with an energy > 6 MeV – 99.66%). Residual electrons that hit the targets (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) fission assembly did not affect the results of the experiment.

The performed analysis indicates that the product yields of the accompanying photonuclear reactions do not affect the accuracy of the results.

1.4. STIMULATION OF THE DELAYED GAMMA-RADIATIONS FROM NUCLEAR MATERIALS PHOTOFISSION

To stimulate delayed gamma radiation from the samples of nuclear materials in hermetic stainless steel containers, bremsstrahlung with energy of 12.5 MeV was used. Irradiation of the samples of nuclear materials were performed on the M-30 microtron electron accelerator of the Institute of Electron Physics of the National Academy of Sciences of Ukraine (see Fig. 2).

To generate bremsstrahlung, a Ta-converter was used (thickness – 1 mm), located at 12 mm from the output window (Ti, thickness – 50 μm) of the electron output unit. The fission assembly was installed perpendicular to the beam axis at 100 mm from the Ta-converter. The filter of residual electrons and secondary photoneutrons (B_4C) was installed close to the Ta-converter perpendicular to the axis of the beam [25].

The electron extraction efficiency was ~ 95%. The instability of the electron energy during target irradiation did not exceed 0.04 MeV [24].

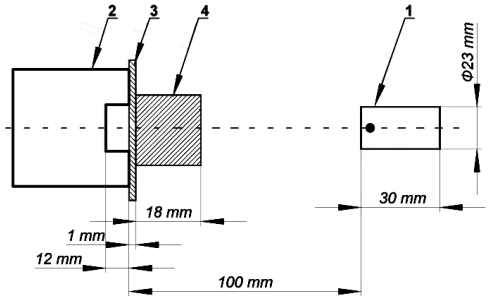

Fig. 2. Scheme of activation of the nuclear material samples on the microtron M-30: 1 – target (actinides in hermetic stainless steel containers); 2 – output node of the microtron M-30; 3 – photons converter (Ta, 52x45x1 mm); 4 – filter B_4C (D = 30 mm, thickness = 19 mm)

Containers with nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) and an empty container were irradiated during the experiment. All the samples were located at 100 mm from the bremsstrahlung target along the beam axis during irradiation. The irradiation time for all samples was 30 min. The integral electron flux upon activation was ~ 3.2E16 electrons for all samples.

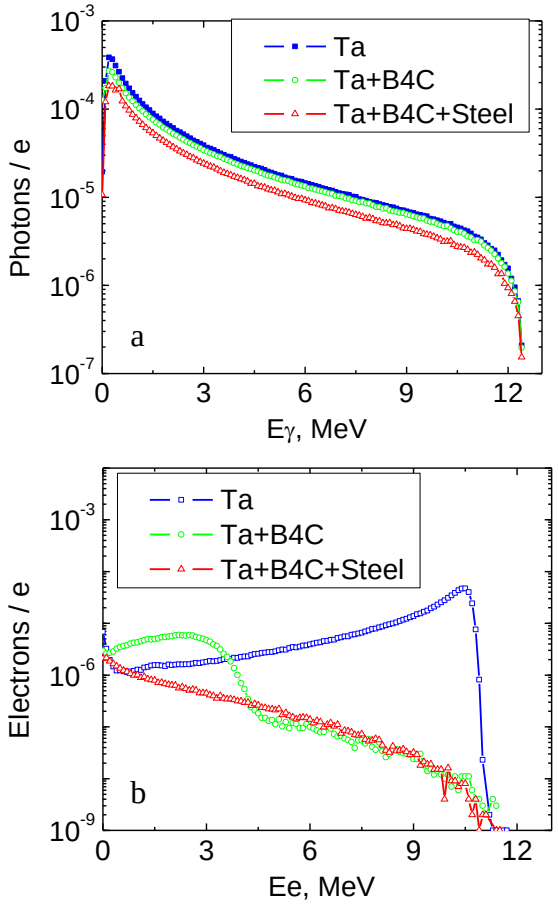

Fig. 3. Spectra of photons (a), residual electrons (b) on the nuclear materials in sealed stainless steel containers: square – Ta-converter – nuclear materials; circle – Ta-converter + B_4C filter – nuclear materials; triangle – Ta-converter + B_4C filter + stainless steel container – nuclear materials

1.5. SPECTROMETRIC STUDIES OF DELAYED GAMMA-RAYS FROM THE SAMPLES

For the research, spectrometric complex based on semiconductor detector was used: HPGe (Ortec), the volumes of which were 150 cm^3 with an energy resolution of ~ 2.45 keV for the line ^{60}Co (1,332.5 keV) [26].

The setup scheme for measurements is shown in Fig. 4.

First step: Background measurements were performed by measuring an empty container and were carried out periodically (at least once a day) in order to verify the stability of background counting rates.

Second step: Measurements of characteristic (background) gamma radiation of nuclear materials (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) samples were carried out before irradiation.

Third step: Measurement of gamma radiation from an empty stainless steel container was carried out after activation.

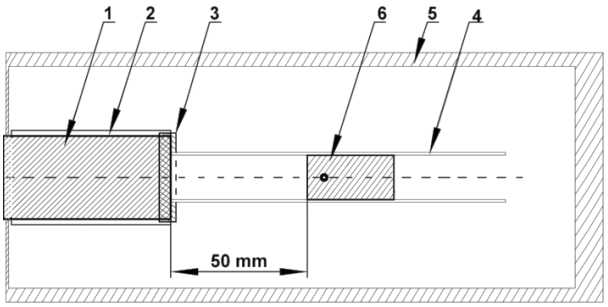

Fig. 4. Scheme of delayed gamma-rays measurement from nuclear materials photofission products: 1 – the detector active volume; 2 – cadmium sheet (0.1 cm); 3 – the tube holder; 4 – tube for fixing the position of the samples: distance – the active surface of the detector; 5 – passive lead shielding (10 cm); 6 – samples

Fourth step: Measurements of the delayed gamma radiation of nuclear materials in containers (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu) have been carried out.

Measurements of all irradiated samples were carried out during 180 min after the end of irradiation. The 10 series of measurements were carried out for each sample. The time for a single measurement was 10 min for all samples.

During the measurements, the drift of the energy scale, resolution and recording efficiency of the spectrometric complex were constantly monitored using point standard gamma-active source ^{60}Co. The drift of these parameters did not exceed 1%.

Fig. 5 shows: 1 – delayed gamma-ray spectra from products of activation of nuclear materials (^{232}Th; ^{235}U; ^{238}U; ^{239}Pu), which were packed in container; 2 – characteristic (background or not irradiated) gamma-rays from isotopes (^{232}Th; ^{235}U; ^{238}U; ^{239}Pu) of nuclear material; 3 – gamma-rays of products of activation of chemical elements from which the container is made. Measurement time for all samples was 10 min. Irradiation time for all samples was 30 min. Cooling time was 60 min.

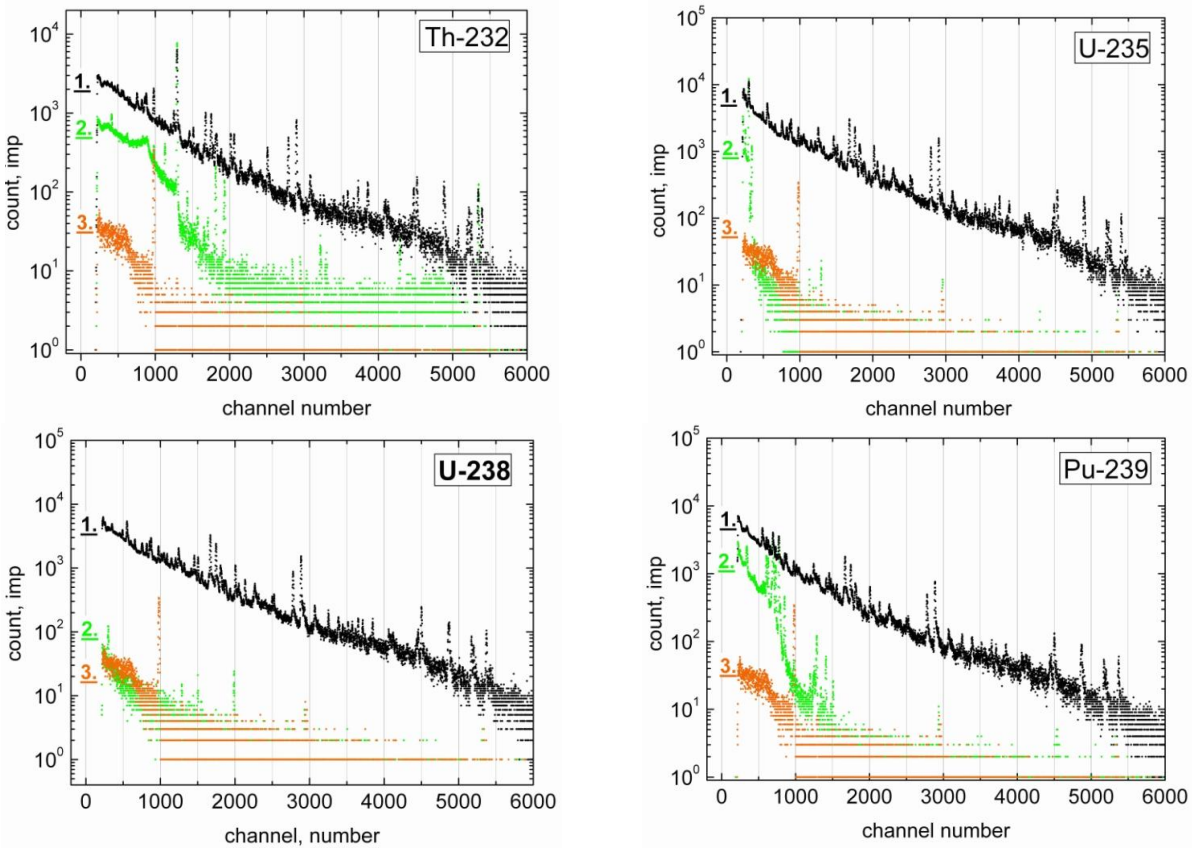

Fig. 5. Gamma spectra of activation of nuclear materials (1), characteristic (background) gamma lines of isotopes of nuclear materials (2), and the products of activation of chemical elements from the container (empty)

Gamma-lines of ^{138}Cs (1009.78; 1435.77; 2218.00; 2639.59 keV) and ^{89}Rb (1031.92; 1248.14 keV) [27] were used in isotopic analysis of nuclear materials. Fission products (gamma-rays emitted) were identified by the energies of their characteristic gamma lines, considering their half-lives and measurement, accumulation, and cooling times. Additionally, the half-lives of their predecessors along isobaric chains were considered.

Gamma-ray spectra of samples were measured in real time. The live time of the spectrometer did not exceed 5…7% during all measurements.

The value of the statistical error in detecting gamma lines from products (which were used for self-calibration and isotopic analysis of nuclear materials) of photofission of actinides did not exceed 5%.

Spectroscopic information was processed using the Winspectrum software package [28].

RESULTS AND DISCUSSION

For the analysis, we used the experimental values of the time dependences of the intensity of the following gamma lines on the products of photofission of nuclear

materials: ^{138}Cs (1009.78; 1435.77; 2218.00; 2639.59 keV) and ^{89}Rb (1031.92; 1248.14 keV).

The gamma lines ^{89}Rb (2570.2; 2707.3 keV) and ^{138}Cs (2639.6 keV) were not used for analysis due to poor counting statistics.

The ^{89}Rb gamma line (2195.92 keV) was not used for analysis, since the spectrum contained an interfering ^{88}Kr gamma line (2195.84 keV) with comparable absolute quantum yield (14.5 and 13.2%, respectively) [27].

The values of *X* were calculated based on the experimental data of n_{exp} at different cooling times according to formula (7) using the MINUIT D506 program [29], which implements the least squares algorithm.

The calculated *X* values are shown in Table.

Prod.	Eγ, MeV	^{232}Th	^{235}U	^{238}U	^{239}Pu
^{89}Rb	1031.92	57.9	115.6	42.1	14.7
^{89}Rb	1248.14	44.2	89.9	44.5	14.5
^{138}Cs	1009.78	56.2	163.2	131.1	70.0
^{138}Cs	1435.77	110.7	308.0	266.7	132.0
^{138}Cs	2218.00	16.6	44.5	43.3	19.1
^{138}Cs	2639.59	6.9	20.2	16.6	8.7

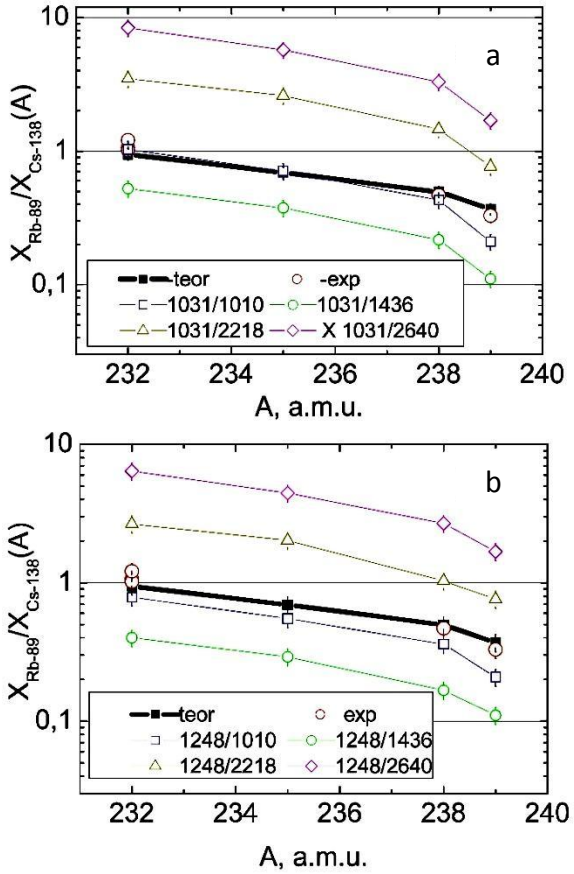

Fig. 6. Dependence of the ratios of the X values of the gamma line of the light product ^{89}Rb to the gamma lines of the heavy product ^{138}Cs on mass of fissile nuclei

Dependence of the ratios of the *X* values of the gamma line of the light product ^{89}Rb (1031 keV) to the gamma lines of the heavy product ^{138}Cs (1009.78 – light square; 1435.77 – light circle; 2218.00 – triangle; 2639.59 keV – rhombus) on mass of fissile nuclei (nuclear materials – ^{232}Th; ^{235}U; ^{238}U; ^{239}Pu) is shown in Fig. 6,a. A similar dependence of the ratios of X values for the gamma line of the light product ^{89}Rb (1248 keV) is shown in Fig. 6,b. The same figure shows the calculated and experimental yields for these products [11].

The values of the dependence of the ratios *X* for the indicated pairs of gamma lines on the mass of fissile nuclei qualitatively reflect the structure of the dependence of the ratios of the yields of the indicated pair of products. These dependencies are almost parallel to each other. The difference in their values (for fixed gamma lines for one fissile nucleus) is due to the absence in formula (7) of normalizations to the absolute quantum yield of gamma lines, their self-absorption and absorption in samples, and the efficiency of the spectrometer according to formula (1).

CONCLUSIONS

As a result of experimental studies, the possibility of isotope identification of nuclear materials in stainless steel containers using delayed gamma radiation has been demonstrated.

The use of calculated *X* values from measured intensity values of gamma lines of nuclear materials photofission products made it possible to expand the number of gamma lines (for a wider energy range) suitable for analysis, as well as to increase its accuracy and reliability.

The ratio of time dependencies of gamma lines intensity of the light and heavy photofission products (X(1031.9)/X(2639.6), X(1248.1)/X(1009.8), X(1248.1) /X(1436.8), X(1248.1)/X(2218.0), X(1248.1)/X(2639.6)) can be used to differentiate nuclear materials. In this case (for the parameters of our experiment), the accuracy of measuring gamma lines intensity should not exceed 5%.

The obtained experimental results are consistent with the results of our previous simulation of the possible use of gamma radiation from the products (^{89}Rb and ^{138}Cs) of the shielded nuclear materials photofission [11,18] for the analysis of their isotopic composition.

Results of characteristics simulation of bremsstrahlung radiation beam which hit the samples for M-30 microtron, with help of the Geant 4 toolkit, allowed to optimize the scheme of stimulation of delayed gamma radiation from photofission of nuclear materials in hermetic stainless steel containers.

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ІЗОТОПНА ІДЕНТИФІКАЦІЯ ФОТОПОДІЛУ ЯДЕРНИХ МАТЕРІАЛІВ У КОНТЕЙНЕРАХ З НЕРЖАВІЮЧОЇ СТАЛІ ЗА ДОПОМОГОЮ ЗАТРИМАНОГО ГАММА-ВИПРОМІНЮВАННЯ

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Представлено результати ізотопної ідентифікації ядерних матеріалів (^{232}Th, ^{235}U, ^{238}U, ^{239}Pu), упакованих у герметичні контейнери з нержавіючої сталі, за їх стимульованим затриманим гамма-випромінюванням. Для аналізу використовувалося відношення значень часових залежностей інтенсивності гамма-випромінювання від легкого та важкого продуктів їхнього фотоподілу. Гамма-випромінювання продуктів фотоподілу ^{89}Rb (1031.9; 1248.1 кеВ) та ^{138}Cs (1009.8; 1436.8; 2218.0; 2639.6 кеВ) використовувалися для диференціації ядерних матеріалів. Стимуляція реакції фотоподілу зразків проводилася на електронному прискорювачі – мікротроні М-30 з максимальною енергією гальмівного випромінювання 12.5 МеВ. Продемонстровано можливість проведення ізотопної ідентифікації ядерних матеріалів за наступними наборами відносних значень часових залежностей інтенсивності гамма-ліній: X(1031.9)/X(2639.6), X(1248.1)/X(1009.8), X(1248.1)/X(1436.8), X(1248.1)/X(2218.0), X(1248.1)/X(2639.6).