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AGE DETERMINATION OF LOW-ENRICHED URANIUM IN VARIOUS PHYSICAL FORMS USING GAMMA SPECTROMETRY METHODS

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This paper presents the research results on the age determination of low-enriched uranium in various physical forms using gamma spectrometry methods. The objects of study were uranium working reference materials (uranium oxide powders, uranium alloys, fuel pellets, and microspherical fuel) with ^{235}U enrichment levels ranging from 0.47% to 19.75%, developed at the NSC KIPT. Two analytical approaches for uranium age dating were proposed and tested: Approach 1 is based on a broad-energy germanium detector (BEGe3830) and the ^{214}Bi/^{234}U chronometer; this method demonstrated efficiency across a wide range of enrichments, from natural to enriched uranium. Approach 2 utilizes a low-energy detector (GL1015R) and the ^{223}Ra/^{235}U chronometer; it has been found that this approach is limited to enriched uranium only. The results for the calculated age and model production dates obtained by both approaches are in good agreement. The average measurement uncertainty was approximately 10% with an exposure time of 400 000 s.

Keywords: *Uranium age dating; Isotope chronometer; Gamma-spectrometer; Enriched uranium*

1. INTRODUCTION

In nuclear forensics, the "age" of uranium is defined as the time elapsed since its last chemical purification - the point at which all decay products were removed. In other words, it represents the material's production date or the date of the last technological processing, such as enrichment. The determination of uranium age is a fundamental tool for the attribution of nuclear materials. This parameter allows for the reconstruction of a sample's processing history, the establishment of its technological links to specific stages of the nuclear fuel cycle, and the performance of comparative analyses to identify whether different samples share a common source of origin. Modern research in nuclear forensics highlights several key approaches to dating uranium-containing materials, all based on measuring radioactive disequilibrium. The primary challenge for gamma spectrometry methods remains the necessity of detecting low activities of decay products against the background of high activity from the parent uranium nuclides.

Chronometer ^{214}Bi/^{234}U: The use of short-lived decay products, such as bismuth-214 (^{214}Bi) [1], is considered an effective non-destructive analysis method for the rapid determination of the time elapsed since the last material processing. As stated in IAEA guidelines, high-resolution gamma spectrometry (HRGS) allows such measurements to be performed without opening the containers. However, the accuracy of this chronometer critically depends on the airtightness of the packaging and the retention of radon-222 (^{222}Rn), which is an intermediate product in the decay chain [2]. In a simplified form, the age of uranium is defined as: $t \approx \frac{1}{\lambda^{\text{Ra-226}}} \cdot \ln\left(1 + \frac{A^{\text{Ra-226}}}{A^{\text{U-234}}}\right)$, where λ^{Ra-226} – decay constant of ^{226}Ra; A^{Bi-214} and A^{U-234} – activities of ^{214}Bi and ^{234}U, respectively.

Chronometer ^{230}Th/^{234}U: This ratio is considered the most reliable 'nuclear clock' for uranium age dating. Classic studies [3] have extensively investigated the validity of this chronometer and established that it provides high accuracy for a wide range of materials - from fuel pellets to uranium ore concentrate. Although mass spectrometry is traditionally used for this purpose [4], modern gamma-spectrum processing methods allow for the isolation of thorium-230 (^{230}Th) analytical lines, even in the presence of intense radiation from uranium isotopes [5]. Mathematically, the age of uranium in this case is expressed via the activity ratio as follows: $t \approx \frac{1}{\lambda^{\text{Th-230}}} \cdot \ln\left(1 + \frac{A^{\text{Th-230}}}{A^{\text{U-234}}}\right)$ where $\lambda^{\text{Th-230}}$ is the decay constant of ^{230}Th, and $A^{\text{Th-230}}$ and $A^{\text{U-234}}$ are the activities of ^{230}Th and ^{234}U, respectively.

Chronometer ^{231}Pa/^{235}U: The use of the ^{231}Pa/^{235}U chronometer is considered one of the most complex yet informative methods in nuclear chronometry. The technical value of this method lies in its ability to verify data obtained from the primary thorium cycle. Since the half-life of the intermediate ^{231}Th is only 25.5 hours, the equilibrium between ^{235}U and ^{231}Th is established very rapidly. In contrast, ^{231}Pa has a half-life of 32 760 years, making it ideal for measuring the age of anthropogenic uranium over timescales ranging from several years to centuries.

To enhance the reliability of the results, the cross-dating method is commonly employed. Comparing the results obtained from the ^{230}Th/^{234}U and ^{231}Pa/^{235}U pairs allows for confirmation that the system has remained closed since the moment of chemical separation [6]. Such a comprehensive approach forms the basis of the attribution methodology for unknown nuclear materials, enabling the identification of the sample's source of origin with a high degree of probability.

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The objective of this study was to determine the age of low-enriched uranium in various physical forms. Based on these uranium, working reference materials were developed at the NSC KIPT in 2014 to ensure the traceability of gamma spectrometric measurements within the nuclear material accounting and control (NMAC) system at Ukraine's nuclear facilities. Obtaining additional analytical characteristics (signatures) of these materials is a topical task that will enable their application in nuclear forensics both for ensuring measurement traceability and for conducting inter-laboratory comparisons of gamma spectrometric research results among the national forensic laboratories of the GUAM countries.

2. MATERIAL AND METHODS

For uranium age dating we used working reference materials (WRM) with ^{235}U enrichment levels ranging from 0.47% to 19.75% produced at NSC KIPT. The characteristics of the WRM are given in Table 1 below. Each uranium material sample was placed in an airtight aluminum container with a side wall thickness of 5 mm and an end face thickness of 2 mm.

Table 1. The characteristics of the working reference materials.

#/#	Sample ID	Enrichment, %	Mass of U, g	Description
1	Upo #01	0.47	39.42	Powder UO_2
2	Upo #02	0.72	37.89	Powder U_3O_8
3	Ume #03	0.72	40.72	Alloy U-NbZr
4	Ume #04	0.70	49.22	Alloy U-Ti
5	Upo #05	2.05	39.42	Powder UO_2
6	Upo #06	4.08	39.42	Powder UO_2
7	Upr #06	6.14	39.68	Uncoated microspheres based on UO_2
8	Upr #06	6.25	37.05	Coated microspheres based on UO_2
9	Ume #06	10.00	40.35	Alloy U-Mo-Nb
10	Upo #06	10.00	39.41	Powder UO_2
11	Upo #06	16.46	31.19	Fuel pellets based on UO_2
12	Upo #06	19.75	39.38	Powder UO_2

A broad energy HPGe detector type BEGe 6530 (Canberra, USA) with a diameter of 70 mm and thickness of 30 mm, and a planar HPGe detector type GL 1015 (Canberra, USA) with a diameter of 36 mm and thickness of 15 mm were used for gamma spectra acquisition. Genie-2000 software was used for gamma spectra acquisition and analysis, FRAM and MGAU codes for uranium isotopic composition analysis, and FRAM code for calculation of detection efficiency.

The uranium age determination was carried out according to the following approaches:

Approach 1. Gamma Detector - broad energy HPGe detector type BEGe 3830; Gamma Ray Energies Range - from 50 to 2600 keV; Chronometer - activity ratio ^{214}Bi/^{234}U; Analyzed ^{214}Bi Gamma Lines - 609.32 and 2204.21 keV; Analyzed ^{234m}Pa Gamma Lines - 766.36, 1001.02, 1193.77, 1510.10, 1737.80, and 1831.70 keV; Detection Efficiency: the intrinsic/relative efficiency curve, which is constructed using the intensities of the ^{234m}Pa gamma quanta and the known activity of the ^{238}U isotope.

Approach 2. Gamma Detector - planar HPGe detector type GL 1015; Gamma Ray Energies Range - from 50 to 1100 keV; Chronometer - activity ratio ^{214}Bi/^{234}U and ^{223}Ra/^{235}U; Analyzed ^{223}Ra Gamma Lines: 269.46 keV; Analyzed ^{235}U Gamma Lines: 275.35 keV; Detection Efficiency: the relative efficiency based on FRAM's efficiency approximation (see Fig.1).

3. RESULTS AND DISCUSSION

Fig. 2 shows the results of the experimental gamma-spectrum processing obtained using the Interactive Peak Fit software, which is part of the Genie-2000 software package.

The ^{214}Bi peak at 609.31 keV slightly interferes with the ^{234}Pa peak at 612.00 keV, but these peaks are fairly easily resolved. The ^{214}Bi peak with an energy of 2204.21 keV is an interference-free monopeak. In the energy region of 265–290 keV there are many interfering peaks of ^{235}U, ^{223}Ra, ^{219}Rn, ^{234}Pa, ^{227}Th, ^{231}Pa. Despite the fact that the software "Interactive Peak Fit" allows to calculate the areas of all peaks, the uncertainty of calculations increases and for ^{223}Ra peak with energy 269.46 keV it increases to 4–6%.

In order to calculate the model date of the WRMs according to Approach 1, we calculated the activity ratios ^{234}U/^{235}U and ^{235}U/^{238}U using the peak areas of ^{235}U at energies 143.76, 163.33, 185.72, and 205.31 keV and ^{234}U at energy 120.90 keV, as well as the values of ^{235}U and ^{238}U contents in the sample calculated with the FRAM software.

The next step involved calculating the ^{214}Bi/^{238}U, ^{214}Bi/^{234}U activity ratio and model date of WRMs using the areas of ^{238}U peaks at energies 766.36, 1001.02, 1193.77, 1510.10, 1737.80, 1831.70 keV and ^{214}Bi at energies 609.31 and 2204.21 keV. The combination of activity ratios allows for obtaining the target value $A^{\text{Bi-214}}/A^{\text{U-234}}$ by means the expression given below:

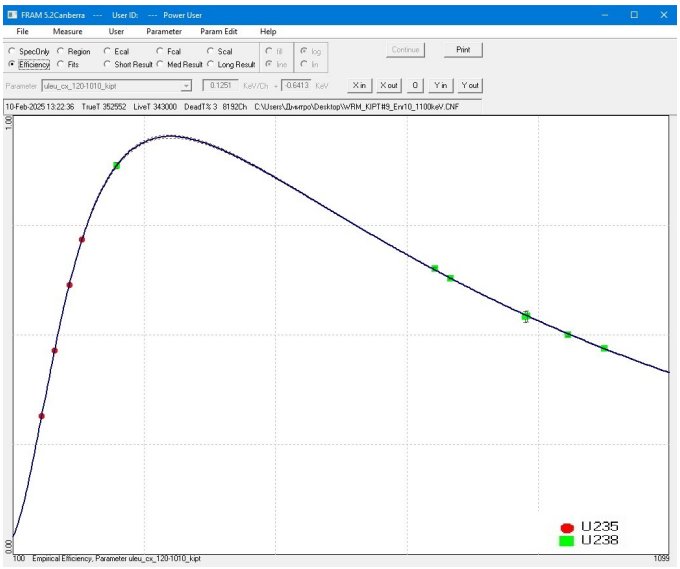

Figure 1. The relative gamma-ray detection efficiency curve based on the results of the FRAM program code approximation.

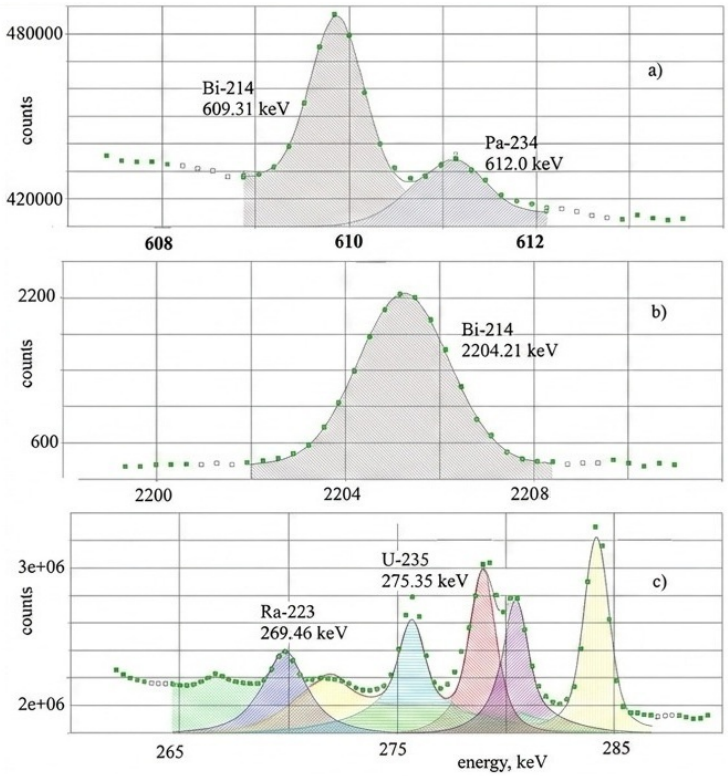

Figure 2. The results of experimental gamma-spectrum processing using the Interactive Peak Fit software. Intervals corresponding to the lines of the uranium chronometer: a) E = 607 - 614 keV, b) 2202 - 2010 keV, c) 260 - 290 keV. See explanations in the text.

$$\frac{A^{\text{Bi-214}}}{A^{\text{U-234}}} = \frac{A^{\text{Bi-214}}}{A^{\text{U-238}}} \cdot \left(\frac{A^{\text{U-235}}}{A^{\text{U-234}}} \cdot \frac{A^{\text{U-238}}}{A^{\text{U-235}}} \right)^{-1} \tag{1}$$

To estimate the uranium age in the working reference materials using Approach 2, the activity ratio $A^{\text{Pa-231}}/A^{\text{U-235}}$ was directly calculated using the detection efficiency values obtained via the FRAM software code. The results are

presented in Table 2 and Fig. 3.

Table 2. Results of the age calculation and the model date of production for the uranium working reference materials.

	Uranium Age (years) / Model Date of Production		
	Approach 1		Approach 2
Sample ID	**609.31 keV, ^{214}Bi**	**2204.21 keV, ^{214}Bi**	**269.46 keV, ^{223}Ra**
Upo #01	45 ± 7 09.02.1979	49 ± 10 15.04.1974	- -
Upo #02	79 ± 8 20.05.1945	78 ± 8 15.06.1945	- -
Ume #03	74 ± 8 28.02.1950	69 ± 8 30.06.1954	- -
Ume #04	59 ± 7 09.12.1964	58 ± 10 28.06.1965	- -
Upo #05	25 ± 3 28.08.1998	29 ± 4 07.04.1994	25 ± 28 03.10.1999
Upo #06	20 ± 2 26.12.2003	21 ± 3 26.10.2002	23 ± 17 25.01.2002
Upr #07	53 ± 5 13.03.1971	56 ± 5 28.05.1967	55 ± 8 17.11.1969
Upr #08	54 ± 5 21.02.1970	56 ± 5 25.05.1967	61 ± 7 15.03.1964
Ume #09	36 ± 3 18.04.1987	39 ± 4 24.09.1984	41 ± 17 12.04.1984
Upo #10	18 ± 2 05.12.2005	23 ± 3 25.11.2000	20 ± 6 29.06.2005
Upr #11	42 ± 4 18.06.1981	50 ± 5 11.07.1967	38 ± 4 20.09.1986
Upo #12	15 ± 2 29.03.2010	- -	18 ± 3 24.02.2007

In general, the results of the uranium age dating for the working reference materials, obtained using different analytical approaches, show good agreement with each other. It is worth noting that Approach 1 can be applied to determine the uranium age across a wide range of enrichments, from natural uranium to enriched material (up to 20%). By contrast, the Approach 2 is limited to the analysis of enriched uranium samples due to the necessity of determining the activity of isotopes within the ^{235}U decay chain.

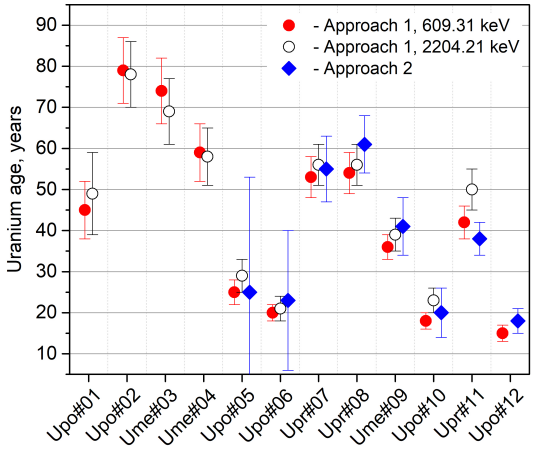

Figure 3. Age calculation results and measurement uncertainties for uranium reference materials.

The use of the 609.31 keV gamma ray of ^{214}Bi for dating relatively "young" uranium samples is preferable to the 2204.21 keV gamma ray, as the intensity of the last one in such samples is comparable to the background intensity of ^{214}Bi.

The average measurement uncertainty was approximately 10% for a measurement time of 400 000 s. The primary contribution to the measurement uncertainty stems from the uncertainty in calculating the areas of certain peaks and the

uncertainty of gamma-ray emission probabilities for specific isotopes. For instance, the uncertainty of the gamma-ray emission probability at 120.90 keV for the ^{234}U isotope is approximately 8%.

4. CONCLUSION

This work presents studies on the age determination of low-enriched uranium in various physical forms using gamma spectrometry. The research was conducted using uranium working reference materials developed at the NSC KIPT, which included uranium oxide powders, uranium alloys, fuel pellets, and microspherical fuel.

Various analytical approaches have been proposed for uranium age calculation using two types of semiconductor detectors (a BEGe3830 broad-energy detector and a GL1015R low-energy detector) and two chronometers based on the determination of the ^{214}Bi/^{234}U and ^{223}Ra/^{235}U activity ratios. The age and model production dates for the uranium working reference materials were calculated.

It was demonstrated that the results of the uranium age calculations obtained through various analytical approaches are in good agreement with each other. The analytical approach based on the ^{214}Bi/^{234}U chronometer can be applied to determine the uranium age across a wide range of enrichments, from natural to enriched uranium (up to 20%). In contrast, the approach based on the ^{223}Ra/^{235}U chronometer is limited to the analysis of enriched uranium samples.

The average measurement uncertainty was estimated to be approximately 10% for an exposure time of 400 000 s. Ways to improve the measurement uncertainty were proposed, such as increasing the exposure time or refining the gamma-ray emission probabilities for certain isotopes.

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ВИЗНАЧЕННЯ ВІКУ НИЗЬКОЗБАГАЧЕНОГО УРАНУ У РІЗНИХ ФІЗИЧНИХ ФОРМАХ МЕТОДАМИ ГАММА-СПЕКТРОМЕТРІЇ

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У роботі представлено результати дослідження з визначення віку низькозбагаченого урану у різних фізичних формах методами гамма-спектрометрії. Об'єктами дослідження стали робочі еталони уранових матеріалів (порошки оксидів, металеві сплави, паливні таблетки та мікросферичне паливо) зі збагаченням за ^{235}U від 0.47% до 19.75%, що створені в ННЦ «ХФТІ». Запропоновано та апробовано аналітичні підходи для визначення віку урану: Підхід 1 – базується на використанні широкодіапазонного детектора BEGe3830 та хронометра ^{214}Bi/^{234}U; цей метод продемонстрував ефективність для широкого інтервалу збагачень, від природного до збагаченого урану; Підхід 2: використовує низькоенергетичний детектор типу GL1015R та хронометр ^{223}Ra/^{235}U; встановлено, що даний підхід обмежений лише збагаченийм ураном. Отримані результати розрахунку віку та модельних дат виробництва за обома підходами демонструють добру узгодженість між собою. Середня невизначеність вимірювань склала приблизно 10% при експозиції 400 000 с.

Ключові слова: *радіоізотопне датування урану; ізотопний хронометр; гамма-спектрометр; збагачений уран*