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Radiochemical and Elemental Analysis of Mixed Uranium-Plutonium Fuel Irradiated in the BN-600 Reactor

V. N. Momotov[a,*], E. A. Erin[a], A. Yu. Volkov[a], V. N. Kupriyanov[a], M. I. Khamdeev[a], D. E. Tikhonova[a], A. Yu. Shadrin[b,], and Yu. S. Khomyakov[b]**

[a] *Research Institute of Nuclear Reactors, Dimitrovgrad, Ul'yanovsk oblast, 433510 Russia*
[b] *JSC "Proryv," Moscow, 107140 Russia*
**e-mail: momotov@niiar.ru*
***e-mail: anyshadrin@rosatom.ru*

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Abstract—The results of destructive radiochemical and elemental analysis of samples of experimental mixed nitride uranium-plutonium (MNUP) fuel irradiated in the BN-600 reactor are presented. Data on the nuclide composition and weight content of U, Pu, Am, Cm, Nd were obtained, the specific activity of ^{14}C, ^{3}H, ^{106}Ru, the weight content of Zr, Mo, Pd, Rh in solution and in the undissolved residue of MNUP SNF were determined. The burn-up depth of MNUP fuel is calculated from the ratio of the number of fission product atoms, burn-up monitor, to the number of heavy atoms. The total accumulation of ^{145}Nd and ^{146}Nd isotopes, as well as ^{148}Nd, was used as burn-up monitor.

Keywords: mixed nitride uranium-plutonium irradiated nuclear fuel, destructive radiochemical analysis, α-spectrometry, γ-spectrometry, liquid scintillation spectrometry, mass spectrometry, isotope dilution, atomic emission spectrometry, ion exchange, extraction chromatography

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INTRODUCTION

The strategy for the development of nuclear energy in Russia involves the creation of a closed nuclear fuel cycle with fast neutron reactors, for which mixed nitride uranium-plutonium (MNUP) fuel is considered as a fuel composition [1]. Appropriate experimental studies are required to substantiate the safe use of nitride fuel, verify calculation codes, establish a balance of fissile materials and fission products, and accumulate potentially radiation-hazardous nuclides.

At present, experimental data on the MNUP SNF composition are limited. The literature reports radiochemical analysis data on MNUP fuel irradiated in the BOR-60 reactor [2]. In [2] the isotopic composition and weight content of U, Pu, Ce, Cs, Am, and Nd nuclides were detected and in these data the fuel burn-up was determined, 2.57% of heavy atoms (h.a.).

The work [3] presents data on the ^{14}C specific activity. In studies [4, 5], a method was developed for determining the ^{3}H specific activity in the SNF MNUP. The authors of [6] report data on determining the weight content of ^{232}U and ^{236}Pu nuclides in the SNF MNUP by the α-spectrometry method.

This study presents the results of comprehensive studies of samples of experimental MNUP fuel irradiated in the BN-600 fast breeder reactor.

EXPERIMENTAL

We studied two samples of MNUP SNF irradiated in the BN-600 reactor. Data on the studied samples are presented in Table 1.

The nuclide composition and weight content of fissile materials and fission products in the MNUP SNF were studied in the following sequence:

– determination of the MNUP SNF weight;

– sample dissolution using a dissolution apparatus equipped with a reflux condenser and a system of traps;

– replacement of the reflux condenser with a refrigerator and partial distillation of the SNF solution with subsequent determination of the tritium specific activity in the condensate;

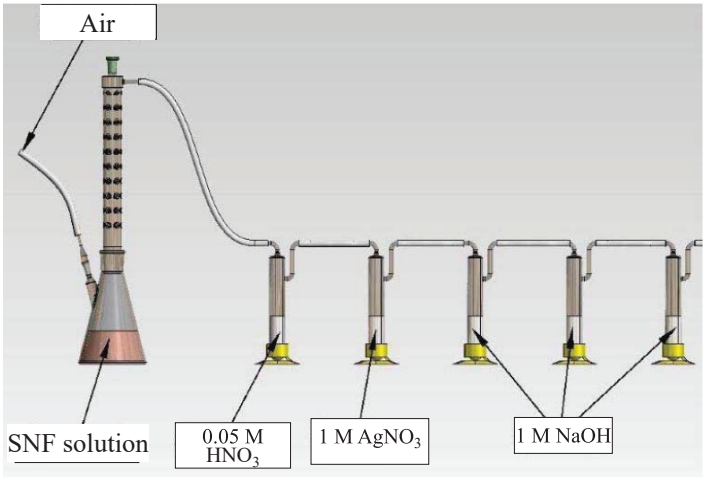

Fig. 1. Scheme of the apparatus for dissolving the SNF sample irradiated as part of KETVS-1 [2].

– determination of the ^{14}C specific activity in alkaline traps;

– filtration of the initial solution through a double cellulose filter and analysis of the undissolved residue for the content of Zr, Mo, Pd, Rh, U, and Pu;

– sampling an aliquot of the initial solution and analysis for the content of Zr, Mo, Pd, Rh;

– dilution of the initial solution;

– determination of the ^{106}Ru specific activity in the SNF working solution, on the walls of the reflux condenser, in trap solutions, and in undissolved residue;

– α,γ-spectrometric analysis of the SNF working solution to estimate the volume of the aliquot required for the chromatographic separation of fractions;

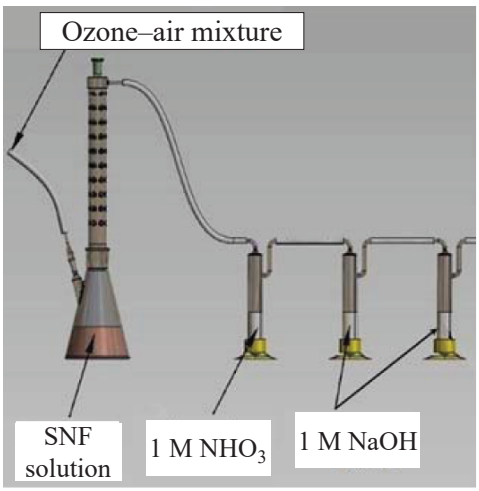

Fig. 2. Scheme of the apparatus for dissolving the SNF sample irradiated as part of KETVS-7.

– carrying out sorption separation of nuclides U, Pu, Am-Cm-rare-earth fraction for mass-spectrometric measurements of the isotopic composition;

– repetition of the sorption separation of nuclides U, Pu, Am-Cm-rare-earth fraction in the presence of a complex label to measure their weight content.

To determine the fuel weight at the first stage, the sample with the fuel element cladding was weighed. Upon completion of the fuel dissolution, the cladding was removed, washed, dried, and weighed. The fuel weight content in the sample was found from the difference between the weight of the sample with the fuel element cladding and the cladding weight.

The weight of the sample and fuel cladding after dissolution was found on an analytical balance AX 205 (Mettler Toledo) with a weighing error of ±0.25 mg.

The dissolution of the fuel sample irradiated as part of KETVS-1 was carried out in an apparatus (Fig. 1) [4].

The first trap is designed to capture ruthenium released during the dissolution of the SNF sample, the second is to absorb iodine isotopes, and three alkaline absorbers are to trap ^{14}C released in the form of carbon dioxide. The purging rate of the apparatus volume with air was 1.2 L h^{-1}.

The dissolution of the fuel sample irradiated as part of combined experimental fuel assembly KETVS-7 was carried out in a dissolution apparatus (Fig. 2). The purging rate of the apparatus volume with an ozone–air mixture was 1.2 L h^{-1}, which corresponds to an ozone supply rate of 2.5×10^{-2} g h^{-1}. The change in the device design is due to the results of radiochemical studies of

Table 1. Data on the studied samples of SNUP SNF irradiated in the BN-600 reactor

Assembly no.	Cladding no.	Coordinate of the cut from the fuel rod bottom, mm	Operating time of the assembly in the reactor, effective days	Fuel exposure time at the time of analysis, years
KETVS-1	64	1380–1430	433.1	2.5
KETVS-7	5	1540–1552	589.2	2.5

Table 2. Sample weighing results

Sample no.	Cladding sample weight (m_1), g	Cladding weight (m_2), g	Fuel weight (m), г
KETVS-1	3.3285	0.5115	2.8170
KETVS-7	4.6823	0.7024	3.9799

the MNUP SNF irradiated as part of KETVS-1, and a series of model experiments to prove the completeness of the oxidation of various carbon forms to CO_2 and the capture of the resulting carbon dioxide by alkaline traps [3], as well as the results of testing KETVS-1 for the content of iodine isotopes.

After cooling the MNUP SNF solution to room temperature, the reflux condenser was replaced with a refrigerator, and part of the SNF solution was distilled off at a temperature of 110–120°C. The condensate was collected in a 50 mL volumetric flask. The tritium content was determined in view of a preset proportionality coefficient (K_{pr}), which takes into account the tritium fraction that passed from solution to condensate. At a volume of the MNUP SNF initial nitric acid solution of 250 mL and a condensate volume of 50 mL, $K_{pr} = 0.24 \pm 0.02$ [4].

The activity of ^{3}H in the condensate solution and ^{14}C in alkaline solutions of Petri absorbers was determined by liquid scintillation spectrometry.

The part of the SNF solution remaining after distillation was filtered through a double cellulose "blue ribbon" filter 11.0 cm in diameter with a pore size of 1–3 μm to separate the undissolved residue. The flask with the initial solution remains was washed three times with 1 M HNO_3 (25 mL). The washing solutions were used to rinse the filters and combined with the basic SNF solution remaining after distillation.

The double cellulose filter with the undissolved residue of the SNUP SNF irradiated as part of KETVS-1 was separated. Each filter was dissolved, and the contents of U, Pu, Zr, Mo, Pd, and Rh were determined in the resulting solutions.

Uranium was determined by the spectrophotometric measurement of the light absorption of the uranium complex with arsenazo III; plutonium, by α-spectrometry; and fission products, by the atomic emission spectral method. The testing procedure is described in detail in [2].

At the final stage, 8 M HNO_3 solution (200 mL) was added to a flask with an undissolved fuel cladding, and control dissolution was carried out at a temperature of 95–100°C for 5 h. Upon dissolution process completion the α- and γ-spectrometric analyzes of the solutions were performed after the main and control dissolutions.

The analytical line energies of radionuclides, their yields, as well as the half-lives for calculating the specific activities of radionuclides when calculating their volumetric activity in term of concentration, were taken from [7, 8].

The isolation and purification of elements was carried out using a combination of cation-, anion-exchange, and extraction chromatograpy. The techniques developed at RIAR were taken as the basis for the separation of SNF components. The isotopic composition of the nuclides U, Pu, Nd, Am, and Cm was determined in the isolated fractions. The analysis procedure is described in detail in [2–6, 9–11].

Quantitative determination of the U, Pu, Nd content was carried out by repeating the procedure for their isolation in the presence of a complex label by isotope dilution with mass spectrometric ending.

To find the fuel burn-up depth, the value of ^{148}Nd accumulation and the total ^{145}Nd and ^{146}Nd accumulation were used as monitors.

When analyzing a sample of SNF MNUP irradiated as part of KETVS-1, aliquots were taken from the

Table 3. Results of radiochemical analysis of MNUP SNF fragments

Parameter	BOR-60 [2]	BN-600	
Assembly no.	OETVS 160E	KETVS-1	KETVS-1
Fuel burnup, % h.a.	2.57	5.57	6.84
Fuel exposure time at the time of analysis, years	8	2.5	2.5
^{3}H Specific activity, Bq/g of SNUP SNF	$(8.5 \pm 1.1) \times 10^{6}$	$(2.6 \pm 0.4) \times 10^{6}$	$(1.8 \pm 0.3) \times 10^{6}$
^{14}C Specific activity, Bq/g of SNUP SNF	–	$(1.2 \pm 0.3) \times 10^{7}$	$(1.5 \pm 0.3) \times 10^{7}$
^{106}Ru Specific activity, Bq/g of SNUP SNF T	$(3.8 \pm 0.4) \times 10^{8}$	$(4.0 \pm 0.4) \times 10^{9}$	$(6.1 \pm 0.6) \times 10^{9}$
^{3}H Specific activity in the fuel cladding structural material, Bq/g	–	2.5×10^{5}	–

initial SNF solution to determine the Zr, Mo, Pd, and Rh content by atomic emission spectrometry. The analytical lines of the elements being determined, free from superposition of the U and Pu spectral lines were used: Mo 317.043; Pd 342.124, 340.458; Rh 343.489, 339.682; Ru 342.832; Zr 339.198, 327.926, 327.305 nm. The weight content of Mo, Zr, Rh, Pd, and Ru was calculated with the calibration curves constructed from reference samples. The analysis procedure is described in detail in [2].

The isotopic and elemental composition of the MNUP SNF was determined using the analytical equipment described in [2–6, 12].

RESULTS AND DISCUSSION

The data on weighing the MNUP SNF samples are listed in Table 2.

The control dissolution of the fuel cladding was carried out upon completion of the dissolution process and separation of the undissolved residue. According to the data of α- and γ-spectrometric measurements of solutions, the content of α-emitters in the control solution is $(3 \pm 0.3) \times 10^{-3}$% of their amount in the initial solution, content of γ-emitters is 0.1 ± 0.01%.

The results of determining the burn-up depth, ^{3}H, ^{14}C, ^{106}Ru specific activities in MNUP SNF irradiated in the BN-600 reactor, in comparison with the results, which we obtained earlier for MNUP fuel irradiated in the BOR-60 reactor, are presented in Table 3.

The discrepancy between the results of determining the tritium specific activity is due to different types of reactor plants and fuel composition irradiation modes, as well as the degree of ^{3}H diffusion through the fuel cladding.

Studies [13, 14] state that one of the main factors determining the tritium specific activity in SNF is the fuel temperature at the irradiation time. As the temperature rises, the tritium content in the fuel composition diminishes. It was shown in [14–16] that tritium formed during SNF irradiation diffuses through the fuel element cladding by 95–99%. Our data on the tritium specific activity in the fuel cladding agree with the results of [17], where the tritium specific activity in the fuel cladding with uranium oxide fuel and burn-up of 6.6–7.1% was 1.2×10^{5} Bq/g.

At the same time, it was found in [18] that the tritium content in the fuel element cladding of the VVER-440 reactor has a significant spread and is in the range from 3.9×10^{6} to 3.4×10^{7} Bq/g. The authors note that the uneven distribution of tritium is observed both along the height and along the circumference of the fuel-element cladding. They conclude that the main part of the tritium enters the cladding through areas of a damaged protective zirconium oxide film.

From data presented in Table 3, it can be seen that the burn-up values of the MNUP SNF samples and the ^{14}C specific activity in them change symbatically. With increasing in the burn-up of the analyzed SNF sample by 23%, the ^{14}C specific activity increased by 25%.

In the course of SNF dissolution, ruthenium is distributed over all elements of the dissolution apparatus: it partially remains in the SNF solution, condenses on the inner walls of the dephlegmator and connecting hoses, water cooler, enters the solution of the first Petri absorber containing the HNO_3 solution, passes into the condensate solution produced at the tritium specific activity determination stage, remains in the undissolved residue of MNUP SNF. Table 3 shows the total content

Table 4. Isotopic composition and weight content of nuclides in MNUP SNF

Isotope	Isotopic composition, wt %			Weight content, kg/t of $(U + Pu)_{ref}$		
	BOR-60, OETVS-1 160E [2]	BN-600		BOR-60	BN-600	
		KETVS-1-1	KETVS-1-7		KETVS-1-1	KETVS-1-7
^{3}H	2.4×10^{-5}	–	–	–	7.4×10^{-6}	5.1×10^{-6}
^{14}C	–	–	–	–	0.07	0.09
^{232}U	–	1.37×10^{-7}	–	–	1.1×10^{-6}	–
^{234}U	0.004(2)	1.97×10^{-3}(1)	<0.01	0.03(1)	0.016(2)	–
^{235}U	0.414(3)	0.216(3)	0.113(6)	3.16(7)	1.77(2)	0.89(5)
^{236}U	0.026(1)	0.024(3)	0.037(8)	0.20(1)	0.20(2)	0.29(6)
^{238}U	95.555(4)	99.758(5)	99.85(3)	771.3(16)	818.94(5)	796.3(2)
ΣU				774.69	820.93	797.48
^{236}Pu	–	1.60×10^{-6}	–	–	2.50×10^{-6}	–
^{238}Pu	0.035(6)	0.056(1)	0.09(2)	0.05(1)	0.077(2)	0.12(3)
^{239}Pu	93.81(1)	89.450(12)	86.66(3)	137.0(29)	123.60(17)	116.12(4)
^{240}Pu	6.029(1)	9.998(10)	12.42(2)	8.87(18)	13.78(13)	16.71(3)
^{241}Pu	0.114(2)	0.470(2)	0.72(2)	0.17(1)	0.660(3)	0.97(3)
^{242}Pu	0.012(1)	0.026(1)	0.11(2)	0.014(1)	0.037(2)	0.15(3)
ΣPu				146.10	138.15	134.19
^{142}Nd	0.89(1)	0.24(1)	0.464(3)	0.028(1)	0.014(1)	0.031(1)
^{143}Nd	25.27(3)	26.41(2)	25.86(24)	0.086(2)	1.360(2)	1.749(2)
^{144}Nd	22.87(5)	21.52(1)	22.27(30)	0.78(2)	1.089(1)	1.506(2)
^{145}Nd	18.31(3)	18.19(1)	18.04(20)	0.63(1)	0.979(1)	1.221(1)
^{146}Nd	15.92(3)	16.44(1)	16.43(24)	0.55(1)	0.873(1)	1.111(2)
^{148}Nd	10.38(3)	10.52(1)	10.38(23)	0.37(1)	0.578(1)	0.702(2)
^{150}Nd	6.36(2)	6.28(1)	6.55(18)	0.23(1)	0.367(1)	0.443(1)
ΣNd				2.67	5.26	6.76
^{241}Am	–	92.59(11)	95.26(2)	5.7×10^{-2}(4)	0.22(1)	0.30(2)
^{242m}Am	–	1.52(8)	1.01(1)	–	3.6×10^{-3}(3)	3.0×10^{-3}(2)
^{243}Am	–	5.89(7)	3.73(1)	–	1.4×10^{-2}(2)	1.2×10^{-2}(8)
ΣAm				0.057	0.238	0.315
^{242}Cm	–	37.5(15)	2.04(2)	9.7×10^{-7}(8)	2.8×10^{-4}(2)	4.0×10^{-5}(4)
^{243}Cm	–	42.4(85)	66.33(4)	–	3.2×10^{-4}(7)	1.3×10^{-3}(1)
^{244}Cm	–	20.1(75)	31.63(3)	1.0×10^{-4}(1)	1.5×10^{-4}(6)	6.2×10^{-4}(6)
ΣCm				1.0×10^{-4}	7.5×10^{-4}	1.4×10^{-3}

of ^{106}Ru detected from the results of γ-spectrometric analysis of the listed solutions and washings from the inner walls of the elements of the dissolution and distillation apparatuses.

It was established that the increase in the ^{106}Ru specific activity in the MNUP SNF samples is ahead of the growth in the fuel burn-up depth. With increasing the burn-up depth by 23%, the ^{106}Ru specific activity increased by 52%. This experimental fact requires additional verification.

Iodine isotopes were not detected in any of the dissolution apparatus traps, nor in the initial solution, nor in the condensate obtained to determine the tritium specific activity. According to the calculations performed using the SCALE 4.4A package, the ^{129}I specific activity in the MNUP SNF irradiated in the BN-600 reactor with a burn-up of 70 MW day/kg (U_{ref}) and holding after irradiation for 2 years should be 2.93×10^{9} Bq/t, the remaining iodine isotopes either are absent or are contained in negligibly small amounts, for example, the ^{131}I content is 6.5×10^{-11} Bq/t.

Taking into account the weight of the analyzed SNF sample irradiated as part of KETVS-1, in the 200 mL initial solution, the calculated content of ^{129}I should

Table 5. Mo, Pd, Rh, Zr content in MNUP STF

Element	Mo	Pd	Rh	Zr
The content in the solution of MNUP SNF, kg/t	4.70	2.35	0.87	4.18
Content in undissolved residue of MNUP SNF, kg/t	0.22	0.21	0.23	0.01

Table 6. Weight content of U and Pu on cellulose filters

Filter	Upper	Lower
Content of U, mg	8.5 ± 0.4	6.0 ± 0.3
Content of Pu, mg	0.6 ± 0.06	0.8 ± 0.08
Fraction of U in the filter, % of the total	0.3	0.2
Fraction of Pu in the filter, % of the total	0.02	0.03

be 8.2×10^3 Bq, and the ^{129}I specific activity will be 41 Bq/mL. For radiochemical studies of SNF samples, a hundredfold dilution of the initial solution is utilizsed. We failed to determine the ^{129}I content in a dilute solution against the background of other SNF components. For detecting iodine, it is necessary to develop special procedures for its isolation from a SNF sample.

The results of determining the isotopic composition and weight content of the nuclides U, Pu, Nd, Am, Cm in the MNUP SNF are given in Table 4. In parentheses are the values of the total absolute error in units of the last digits of the result for a confidence level of 0.95.

When filtering the MNUP SNF solution through a double cellulose filter, a light gray coating was observed on the upper filter. The sediment weight could not be fixed, since the initial weight of the cellulose filter turned out to be greater than the weight of the filter with sediment. This experimental fact is probably associated with the destruction of cellulose under the action of α- and γ-emitting nuclides. According to the data of [19–21], the sediment weight upon uranium oxide SNF dissolution in nitric acid is no more than 0.6% of the dissolved fuel weight. It was found in [22] that, upon dissolution of irradiated mixed-oxide fuel with a burn-up of 31.7 to 54.7 GW/t in 3 HNO_3 at 95°C, the the undissolved sediment weight does not exceed 1.3% of the SNF weight. When studying the dissolution of model nitride SNF [23], the volumes of 7.8 and 9.44 M HNO_3 required for dissolution were calculated based on the final concentration of metals in the solution: 280 and 400 g/L, respectively. It is shown that the weight of the undissolved residue is 1.8 and 2.26%, respectively.

The results of detecting the content of Mo, Pd, Rh, Zr in the solution and the undissolved residue of the MNUP fuel irradiated in the BN-600 reactor as part of KETVS-1 are presented in Table 5.

The presence of Mo, Pd, Rh, Zr was registered only on the upper cellulose filter; there were no these elements on the lower filter. The content of these elements in the solution obtained after the dissolution of the upper filter was equivalent to their content in the undissolved residue of MNUP SNF. The total weight of Mo, Pd, Rh, Zr in the undissolved residue of MNUP SNF irradiated in the BN-600 reactor as part of KETVS-1 was 0.0019 g, which is 0.07% of the dissolved SNF weight.

The results of determining the uranium and plutonium content in the undissolved residue of the MNUP fuel irradiated in the BN-600 reactor as part of KETVS-1 are presented in Table 6.

The data in Table 6 indicate that the U and Pu contents in the upper and lower filters are comparable. The close values of these quantities evidence that most of the U and Pu we found in solutions after the dissolution of the filters were sorbed by cellulose.

The excess of the U content in the upper filter compared to the lower filter may indicate the presence of undissolved uranium-containing compounds. The weight of undissolved U estimated from the difference between the U weight on the upper and lower filters, is 2.5 mg, or 0.09% of the total uranium content in the analyzed SNF sample.

CONCLUSIONS

For the first time, destructive radiochemical studies were carried out and experimental data were obtained on the composition of mixed nitride uranium-plutonium spent fuel irradiated in a fast breeder reactor. A complex technique for conducting destructive radiochemical studies, including dissolution, fractionation of SNF components, and measurements using nuclear-physical methods of analysis: α-, γ-, liquid scintillation, isotope dilution mass spectrometry, emission spectral analysis, was proposed and tested with real SNF. The isotopic composition and weight content of U, Pu, Am, Cm, Nd nuclides were measured, the ^{106}Ru, ^{14}C, ^{3}H specific activities, and the mass contents of Mo, Pd, Rh, and Zr in the solution and in the undissolved residue of MNUP SNF were found.

The data obtained make it possible to carry out a computational analysis and verification of the computational codes in order to justify the radiation safety in the handling of MNUP SNF, and to optimize the methods of its processing.

CONFLICT OF INTERESTS

The authors declare no conflict of interest.

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