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Down-Blending and Immobilization Methods for Uranium-Graphite and Uranium-Zirconium Fuel of NNC Research Reactors

Technical Meeting on Operating Experience and Lessons Learned on Managing Non-Standard Legacy Spent Fuels from Power and Research Reactors

Yuliya Baklanova, Head of Materials Testing Department

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IMPULSE GRAPHITE REACTOR (IGR)



Reactor Hall

Maximum neutron flux density - $7 \times 10^{16} \text{ n/cm}^2 \cdot \text{s}$ Maximum thermal neutron fluence - $3.7 \times 10^{16} \text{ n/cm}^2$ Pulse half-width is minimum - 0.12 s Geometric dimensions of the core - $1800 \times 1000 \times 1000$ mm Operation of the reactor: since 1960 Core modernization: 1966–1967 Core weight \approx 2.6 t Charge with ²³⁵U: First core (1960 – 1966) – 7.3 kg

Second core (1968 to present) – 9.1 kg



Core

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RESEARCH WATER-COOLED HETEROGENEOUS REACTOR IVG.1M



Reactor Hall

Thermal power72 MWEffective core diameter548 mmCore height800 mmThermal neutron flux density

 $3.5 \times 10^{14} \text{ n/cm}^2 \cdot \text{s}$



WCTC-HEU scheme



Core

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PURPOSES AND TASKS OF RESEARCH

Purposes:

- 1. Reduce enrichment
- 2. Produce a reliable matrix for fuel immobilization
- 3. Exemption of fuel from IAEA safeguards

Tasks :

- 1. Develop a concept for down-blending irradiated HEU fuel from research reactors to LEU fuel parameters (IGR, IVG.1M)
- 2. Develop a concept for immobilizing downblended irradiated fuel from research reactors (IGR, IVG.1M) for subsequent exemption of fuel from IAEA safeguards
- 3. Demonstrate the possibility of implementing the proposed technology within the R&D

Down-Blending and Immobilization Criteria for Irradiated HEU Fuel

Criteria	Reference
Exposure of personnel to γ -irradiation: MED \leq 20 mSv/year	SERRS
Fuel enrichment in 235 U: \leq 20 wt.%	RRRFR, RERTR
Uniform distribution of uranium in the matrix volume	IAEA
Impossibility of extracting ²³⁵ U at any stage of the technological process	IAEA
²³⁵ U concentration in matrix: \leq 50 ppm	IAEA
Fuel particle size: ≈ 200 µm	IAEA
Proportion of graphite in the cement mixture: \leq 15 wt.%	Papers
Requirements for the stability of the matrix with LEU fuel:	
- mechanical strength: ≥10 MPa	
- leaching rate < 10^{-3} g/(cm ^{2day})	
- frost resistance:	GOSTs
strength ≥10 MPa, strength reduction < 25%	
- resistance to immersion in water:	
strength ≥ 10 MPa, strength reduction< 25%	

CONCEPTUAL SOLUTIONS FOR HANDLING IGR HEU FUEL

Fuel – uranium-graphite blocks and rods Fuel enrichment (HEU): 90%Core weight ≈ 2.6 t Mass concentration of uranium in fuel : 0.3%



Radionuclide composition of IGR fuel irradiated with HEU

#	Nuclide	No. (number of nuclei of nuclides)	Q (nuclide activity), s ⁻¹
1	¹³⁷ Cs	9.65·10 ²⁰	7.03·10 ¹¹
2	⁹⁰ Sr	8·10 ²⁰	6.15·10 ¹¹
3	¹⁵¹ Sm	9.03·10 ¹⁹	2.2·10 ¹⁰
4	⁹⁹ Tc	2.71·10 ²¹	2.21·10 ⁸
5	¹⁵⁵ Eu	1.36·10 ¹⁶	6.04·10 ⁷
6	⁹³ Zr	3.11·10 ²¹	4.38·10 ⁷
7	¹³⁵ Cs	3 ·10 ²¹	2.86·10 ⁷
8	¹²⁹	5.93·10 ²⁰	8.3·10 ⁵
9	¹⁰⁷ Pd	1.3·10 ²⁰	4.2·10 ⁵



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TESTING THE DRY MIXING METHOD



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DETERMINATION OF MATRIX COMPOSITION AND COMPONENT RATIOS



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TESTING THE MATRIX FOR COMPLIANCE WITH DURABILITY REQUIREMENTS



Mechanical strength

Strength measurement (not less than 10 MPa)



Immersion in water for 90 days, strength measurement (not less than 10 MPa, and strength reduction not more than 25%)

Moisture resistance

The matrices of the compositions passed the tests in full W/C = 0.40 ± 0.02 , C/FA = 1 / 2.5; W/C = 0.42, C/FA = 1 / 1.77Work with the selected compositions continued in full-scale experiments.



Frost resistance:

Thermal cycling in the range of -40 to +40, strength measurement (not less than 10 MPa, and strength reduction by no more than 25%)





Determination of 137 Cs in solutions (leaching rate of 137 Cs - no more than $1 \cdot 10^{-3}$ g/(cm²×day)

Leaching rate:

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FULL-SCALE TESTS

Tasks to be completed in full-scale experiments

- ✓ Obtaining optimal mixing conditions:
 - Sequence of unloading components;
 - Component loading speed;
 - Mixer operating modes during unloading and mixing of components;
 - Duration of mixing components;
- ✓ Measuring parameters:
 - Water bleeding
 - ➢ Heat release
 - Uniformity of uranium distribution throughout the matrix volume
 - > Enrichment

Mixing unit project by INL and AVANTech

Experimental mixing unit for full-scale experiments by NNC RK



55-gallon drum from Skolnik CQ5508 Built-in mixer from NNC RK MATERIALS SCIENCE TESTING DEPARTMEN

DEFINITION OF WATER BLEEDING AND HEAT RELEASE



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EVALUATION OF THE URANIUM DISTRIBUTION UNIFORMITY IN THE MATRICES AND ENRICHMENT MEASUREMENT

Test 1-LEU





		Tes	t 2-	HE	J		
Re	2	(3)	4)	1.		14	-4
. 5	6.	7	3				
0	(10.)	11.	(12)				18
(3)	(14)	15	(16.)				
	(8.)	19	20.		1.		
	22	23)	29				
20	20	27.	28				
2	30	3	32			-	-
Ť						-	

	Sample #
三川	IS-1
	16 10

UO_2 0,10 0,09 0,11 0,09 0,09 0,10 0,09 0,10 0,09 0,09 0,11 0,09 0,09 0,11 0,10 0,11 0,08 0,10 0,10 0,14 0,10 0,10 0,09 0,09

Results of determination of 235U enrichment in samples (MGAU)

Comple #	Isotope content, wt. %					
Sample #	²³⁴ U	²³⁵ U	²³⁸ U			
	$0.167 \pm$	$\textbf{4.996}\pm$	94.837 ±			
12-1	0.037	0.076	0.076			
10 10	$\textbf{0.157}\pm$	$\textbf{4.898}\pm$	94.946 ±			
15-10	0.037	0.110	0.110			
10 10	$\textbf{0.173}\pm$	$\textbf{5.200}\pm$	94.627 ±			
12-18	0.038	0.081	0.081			
	$\textbf{0.254}\pm$	$5.167\pm$	94.579 ±			
13-20	0.059	0.113	0.113			
10.00	$0.113\pm$	4.976 ±	94.911 ±			
19-30	0.028	0.105	0.105			

	UO ₂							
0,10					0,11			0,13
	0,12						0,13	
					0,12			0,11
	0,13			0,12			0,10	
								0,10
						0,10		
	0,12			0,11				
								0,12
0,11								
				0,11			0,11	
		0,04		0,13				0,15

 UO_2 0,10 0,11 0,11 0,00 0.09 0,12 0,11 0,11 0,09 0,10 0,11 0,10 0,10 0,10 0,11 0,10 0,10 0,11 0,11 0,11 0,10 0,10 0,24

0,01

		0.10		0,05	0,11	0,05	0,05		
								1	
Μ	IC E	ENERG	Y BRANC						

0,10 0,11 0,10 0,10

0.00 0.11 0.00 0.00

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EVALUATION OF MATRIX COMPLIANCE WITH WAC

Conditions for Down-blending and immobilization of irradiated HEU fuel from the IGR reactor

- Packaging: Type A drum with a capacity of \approx 208 l (55 gallons) with a built-in mixer
- Matrix: 58÷76 kg of Portland cement, 147÷134 kg of fly ash, 84÷90 l of water
- Fuel: 1 drum contains 4 kg of fuel (12 r²³⁵U) and 300 g of UO₂ (0,27%);
- Total number of drums 640 pcs.
- Productivity 2 drums/day

Expected results for compliance with the criteria

- MED on the cementing line: up to 0.02 mSv/h;
 MED at the stage of fuel extraction from the daily container: up to 0.3 mSv/h;
- \checkmark Targeted enrichment through dry mixing : 5±0.17 %;
- **\checkmark** Concentration of ²³⁵U in a concrete block : 40 ÷ 45 ppm;
- ✓ Proportion of graphite in the cement mixture: 1.6÷2 %;
- ✓ Strength: not less than 14 MPa;
- ✓ Leaching rate of ¹³⁷Cs in water medium: $\leq 5.1 \times 10^{-5}$ g/(cm²×day);
- ✓ Resistance to long-term exposure to water (90 days): increase in strength up to \approx 29 MPa;
- ✓ Uniformity of uranium distribution throughout the matrix volume: more than 81%;
- Frost resistance (30 cycles, temperature range from -40 to +40, cycle duration – 4.5 hours) : 13.7 ± 2.2 MPa, decrease in strength by no more than 20%

IMPLEMENTATION OF THE IMMOBILIZATION TECHNOLOGY FOR HEU FUEL FROM THE

IGR REACTOR



CONCEPTS FOR HANDLING HEU FUEL FROM THE IVG.1M REACTOR











Fuel – uranium-zirconium fuel elements Fuel enrichment (HEU): 90% Number of SFA– 33 pcs; Mass concentration of uranium in fuel: ≈3-5%

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EXPERIMENTAL BENCH FOR TESTING THE METHOD FOR REMELTING IVG.1M HEU FUEL



VCG-135 Test-bench

The stand allows for controlled heating of any smallsized samples to a high temperature (3000°C) with subsequent cooling due to heat leaks into a watercooled inductor when the generator is switched off.

Specifications:

- Generator frequency, kHz: 66;
- Inner diameter of the working chamber, mm: 600;
- Working chamber height, mm: 700;
- Inductor dimensions, mm: Ø80x150, Ø120x150;
- Temperature sensors: thermocouples, pyrometers;
- Gas environment in the working chamber: vacuum, argon;
- Mass of the loaded batch, g: 100-500



Experimental

assembly



Double graphite crucible

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CONDITIONS FOR DOWN-BLENDING OF IVG.1M HEU FUEL BY REMELTING METHOD

Purpose of the experiment:

• to obtain data on the phase state of uranium during remelting of zirconium alloy E110 (fuel model) and UO₂ powder (model of "diluent") enriched to 0.27 wt.% in ²³⁵U)



Schematic diagram of the phase diagram of the U-Zr-O system at 2000°C

[P.J. Hayward, I.M. George/Journal of Nuclear Materials 232 (1996) 13-22] Main conclusion :

Weight parameters of burden loading for RSNF-1 experiment

Name	Weight (g)
W1 (cup-1)	104.08
W2 (cup-2)	99.71
Chip 1(UO ₂ <200>100)	6.07
Zr 1 (E110 plates)	63.93
Chip 2(UO ₂ <200>100)	23.63
Zr 2 (E110 plates)	46.37



 $\begin{array}{l} \mbox{Loading tungsten cups:} \\ \mbox{cup 1} - 19.75 \mbox{ wt.\% in } ^{235}\mbox{U} \\ \mbox{cup 2} - 5,00 \mbox{ wt.\% in } ^{235}\mbox{U} \end{array}$





• The red marker indicates the composition of the burden when simulating fuel enrichment up to 5%, yellow – up to 19.75%. As diagram shows, both compositions confidently lie in the region of existence of a homogeneous melt upon reaching 2000°C

RESULTS OF TESTING THE REMELTING METHOD FOR DOWN-BLENDING HEU FUEL



Main conclusions:

- During remelting, the uranium in the fuel rod of the high-explosive uranium fuel and the uranium in the "diluent" (depleted UO₂ pellet) predominantly crystallizes into an intermetallic phase with zirconium.
- Simulating enrichment reduction to \approx 5%, non-uniform uranium distribution was revealed.

CONDITIONS OF IMMOBILIZATION OF IVG.1M FUEL BY REMELTING IN AN AL MATRIX

Purpose of the experiment:

• to obtain a uniform distribution of uranium and zirconium in the aluminum matrix









Main conclusions:

UO₂-Al phase diagram not found

U-Al phase diagram shows that:

- Above the melting point of AI, a liquid phase of U is formed with an atomic concentration of about 2-2.5% and the concentration of dissolved uranium will increase
- Theoretically it is possible to dissolve all uranium (~0.4% at.) in molten aluminum

The AI-Zr diagram shows that:

- Above the melting point of AI, a liquid phase with a very low atomic concentration of zirconium and a solid phase AI₃Zr with a melting point above 1500°C are formed.
- At a temperature of about 900 °C, all zirconium (fuel element) at its target concentration of ~0.7% at can be dissolved in the liquid phase of Al.

Phase diagram of the AI-Zr system

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TESTING THE METHOD OF REMELTING HEU FUEL FOR IMMOBILIZATION IN AN AL MATRIX



Experimental assembly heating diagram

Weight parameters of burden loading for MMI-1 experiment

Name	Weight (m), g
G (cup-1) MPG-8	19.97
G (cup-2) MPG-8	20.44
Chip 1(U0 ₂ <400>200)	0.282
Al rods	23.91
Al rods	23.91
Zr (E110 bar)	0.566

Weight parameters of burden loading for MMI-2 experiment

Name	Weight (m), g
G (cup-1) MPG-8	20.11
G (cup-2) MPG-8	20.29
UO_2 chip 1	0.205
UO2 chip 2	0.210
Al rods 1	17.56
Al rods 2	17.68
Zr1 (E110 bar)	0.429
Zr1 (E110 bar)	0.431



UO₂ Chip

Al rods



Zr bar





ESULTS OF TESTING THE REMELTING METHOD FOR IMMOBILIZATION OF IVG.1M FUEL IN AN AL MATRIX



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RESULTS OF TESTING THE REMELTING METHOD FOR IMMOBILIZATION OF IVG.1M FUEL IN AN AL MATRIX



t1_5
t1_4
t1_3
<i>t1_2</i>

t1_1

t2_4(н,в)

t2_3(н,в)



Samples for elemental analysis

MMI-1, ingot 2 (AI – UO_2 composition)					
Sector	AI	U			
t1_5	99,938±0,007	0,062±0,007			
t1_4	99,953±0,018	0,047±0,018			
t1_3	99,785±0,020	0,215±0,020			
t1_2	99,33	0,67±0,10			
t1_1	98,32	1,68±0,13			

Results of elemental analysis

	MMI-2, ingot 1 (AI – E110 - UO_2 composition)								
Sector	AI	Zr	Nb	U					
t2_4	99,81±0,08	0,19±0,08	-	-					
t2_3	97,5±0,6	2,5±0,6	0,030±0,012	_					
t2_2	97,9±0,6	2,1±0,6	0,026±0,011	-					
t2_1	91,5±1,8	8,2±1,5	0,091±0,015	0,24±0,26					

	MMI-2, ingot 1 (AI – E110 - UO ₂ composition)							
t2_2(н,в)	Sector	AI	Zr	Nb	U			
	t2_4в	99,79±0,05	0,21±0,05	-	-			
t2_16	t2_4н	99,68±0,04	0,32±0,04	-	_			
	t2_3в	99,04±0,19	0,94±0,19	0,014±0,005	-			
	t2_3н	98,68±0,02	1,30±0,02	0,021±0,008	_			
	t2_2в	99,35±0,12	0,63±0,12	0,01±0,02	0,02±0,07			
	t2_2н	95,2±0,4	4,7±0,4	0,053±0,005				
	t2_1в	93,3±0,4	6,6±0,4	0,069±0,005				

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CONDITIONS FOR IMMOBILIZATION OF IVG.1M FUEL BY REMELTING IN A Zr MATRIX

Weight parameters of burden loading for

Purpose of the experiment:

• to obtain a uniform distribution of uranium throughout the metal matrix



Experimental assembly heating diagram

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RESULTS OF TESTING THE REMELTING METHOD FOR IMMOBILIZATION OF IVG.1M FUEL IN A ZR MATRIX



Distribution of phases in the microstructure of the MMI-3 ingot

Distribution of elements by height in a diametrical section

		Zr			Nb		U		
		Aver. value		Ave	er. val. Ave		er. val.		
	I-3_t2_4	97,94		1	,07	0	,99		
	I-3_t2_3	98,0	0	1	,06	0	,93		
	I-3_t2_2	97,9	7	1	,08	0	,95		
	I-3_t2_1	97,9	9	1	,06	0	,95		
U	Aver. value	Δ _{0,95} Ave. Val.	Rel. <i>L</i>	۱ _{0,95}	Δ _{0,95} u	.val	Dabs	;	Drel
	0,96	0,04	4%	6	0,0	5	0,10		10%

Distribution of elements by height in cross sections

[Zr	Nb			U	
[Aver. val.	Aver. val. A		Ave	r. val.	
	I3_4_в	98,05	1	,04		0,91	
	I3_4_н	98,04	1	,05		0,91	
	I3_3_в	98,00	1	,06		0,93	
	I3_3_н	97,97	1	,08		0,95	
	I3_2_в	97,99	1	,06		0,95	
	I3_2_н	98,02	1	,07		0,91	
L	I3_1_в	98,00	1	,07		0,93	
U	Aver. val.	Δ _{0,95} Aver. val.	Rel. Δ _{0,95}	Δ u	0,95 .val	Dabs	Drel
	0,927	0,016	2%	0	,04	0,07	8%

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CONCLUSIONS ON TESTING THE REMELTING METHOD FOR DOWN-BLENDING AND IMMOBILIZATION OF IVG. 1M FUEL

Main conclusions:

- When down-blending the HEU fuel of the IVG.1M reactor with depleted UO₂ pellets at T=2000 °C in an inert environment, the uranium of the fuel and the uranium of the diluent (UO₂) are converted into a metallic phase. Thus, the identity of the uranium phases of the HEU and LEU is ensured, which makes the dilution process possible
- In terms of structural homogeneity and uniform distribution of uranium-containing phases, the zirconium matrix is more promising.
- Remelting can be carried out in a graphite crucible of an induction heating unit. Remelting is possible using both "cold" and "hot" crucible methods.

Preliminary data for the technological process:

1) When diluting one SFA with UO_2 (enrichment of 0.27% in ²³⁵U)

- ingots weighing from 7.5÷10 kg will be formed in the amount of 33 pcs (according to the number of SFAs);
- the volume of the ingot will be 1.5÷2 l, crucible V ~3-5 l.

2) To immobilize all the fuel, with a total mass of SFAs of \approx 289 kg, it will be necessary:

- ≈120÷135 kg of pellets of depleted UO₂ (the data is also relevant for dilution by oxidation);
- \approx 24.55 t of aluminum (\$47,500) or \approx 10.2 t of zirconium (\$316,200);

3) If the immobilized fuel is not exempted from IAEA safeguards, the following options are possible for expanding SILO (V \approx 39):

- Up to 99 cells, S=600-700 m² (Al matrix, immobilization without crucible)
- Up to 180 cells, S=1300-1500 m² (Al matrix, crucible immobilization);
- Up to 43 cells, S=250-300 m² (Zr matrix, without crucible);
- Up to 75 cells, S=500-600 m² (Zr matrix, with crucible)

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RESULTS OF TESTING THE OXIDATION METHOD FOR DOWN-BLENDING THE IVG.1M HEU FUEL

Change in specific weight gain after oxidation in the range of 900+1200 °C for 0.5 h



● Zr (99,99%) ● Zr-1%Nb (fuel w/o U) ● Zr-2,5%Nb ● Zr-1%Nb

Temperature dependence of oxidation rate





Grinding



Main conclusions:

- When oxidizing a fuel rod under T=900 °C for 3 hours, the oxidation degree is 100%
- During oxidation of a fuel rod under T=1000 °C for 3 hours, the oxidation degree is 98%, the main phase of uranium is α-U₃O₈
- After oxidation, the fuel rod is easily crushed.

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RESULTS OF OXIDATION OF DEPLETED UO2 PELLETS





Main conclusions:

- In the sample annealed at 500 degrees for 1 hour there are two phases: α -U₃O₈ (\approx 66 wt.%) and β -U₃O₈ (\approx 34 wt.%)
- In the sample annealed at 1000 degrees for 3 hours: α -U₃O₈ (\approx 94%) and β -U₃O₈ (\approx 6 wt.%)
- When U₃O₈ is annealed at a temperature above 1000°C, the uranium oxide is reduced to UO₂, but when cooled in air, the resulting uranium dioxide again turns into U₃O₈

RESULTS OF COMBINED OXIDATION OF FUEL ROD AND DEPLETED UO2 PELLET



The result of the combined oxidation of a model fuel rod (E110 alloy) and a depleted UO_2 pellet at T=1000°C C and conditioning for 3 h

The result of the combined oxidation of a model fuel rod (E110 alloy) and a depleted UO_2 pellet at T=500°C and conditioning for 0.5 h

MAIN CONCLUSIONS ON TESTING THE OXIDATION METHOD FOR DOWN-BLENDING THE IVG.1M HEU FUEL

Main conclusions:

- At T=1000°C in an air environment and holding for about 3÷6 h, h, the fuel uranium and the diluent uranium (UO₂) transfer into the α-phase U₃O₈, i.e. the identity of the uranium phases of HEU and LEU is ensured. Thus, when they are mixed, the dilution process can be considered feasible.
- For more complete oxidation of fuel, it is necessary to dismantle SFAs and fragment fuel rods.
- When fuel and diluent are oxidized, the fuel becomes brittle and is easily crushed even by hand, and the UO_2 pellet is transformed into powder. To obtain a single fraction, the components after oxidation are simultaneously crushed in a disk mill to a specified size ($\approx 200 \ \mu m$).
- Simultaneous oxidation of fuel rods of the IVG1.M fuel with depleted UO₂ pellets should be performed with mixing of the components, for which it is proposed to use a rotating and tilting tubular furnace.

Preliminary data for the technological process:

- 1) When down-blending IVG.1M SNF with $\rm UO_2$ pellets (enrichment 0.27% in 235U) using the oxidation method
 - SFA cutting and fuel rod fragmentation (up to 200 mm);
 - the daily batch of fuel rod fragments (I=200 mm) will be 99 pcs (554 g)
 - the mass of oxidized (LEU) fuel will be 1017 g
- 2) To immobilize all the fuel, with a total mass of SFAs of \approx 289 kg, it will be necessary :
 - Number of oxidation batches 524 pcs.
 - ≈120÷135 kg of depleted UO₂ pellets (data also relevant for dilution by oxidation method);
 - ≈70.2 t of fly ash (\$1,404)
 - ≈39,6 t of Portland cement (\$3,165);

3) If the immobilized fuel is not removed from IAEA safeguards, then options for expanding the arch storage facility by 80% are possible.



Nabertherm single-zone rotary and tilting tube furnace model RSRC 80/500/11

CONCLUSION

- 1. As the technology for down-blending irradiated HEU fuel from the IGR reactor, it is proposed to use dry mixing of HEU fuel powders and depleted UO_2 (0.27%);
- 2. It is proposed to use the cementation method to immobilize IGR fuel;
- 3. For handling highly enriched spent nuclear fuel from the IVG.1M research reactor, it is proposed to use:
 - Oxidation method for fuel down-blending stage (up to enrichment of 5 wt.% in ²³⁵U);
 - Cementation method for the immobilization stage (developed earlier for irradiated IGR fuel).
- 4. For a more detailed study of the oxidation method of uranium-zirconium fuel, several issues need to be addressed as part of research work.



Thank you for attention!



IAE NNC RK

071100, Kazakhstan, EKR, Kurchatov 10, Beybit Atom Str., <u>iae@nnc.kz</u>