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Title: Reference Material Manufacture and Certification for the AVNG

~~AVNG stands for "Attribute Verification Systems with Information Barriers for Plutonium with Classified Characteristics utilizing Neutron Multiplicity Counting and High-Resolution Gamma-ray Spectrometry"~~

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Reference Material Manufacture and Certification for the AVNG

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ABSTRACT

Testing and demonstration of any radiation measurement system requires the use of appropriate radioactive sources. An attribute measurement system (AVNG) was developed and fabricated at the Russian Federal Nuclear Center, VNIIEF, Russia, under contract with LANL, USA. The AVNG detects neutron and gamma radiation signatures and compares the data analysis results with the specified threshold values for three unclassified attributes; plutonium is present or absent, plutonium mass is greater than or less than the specified threshold value and plutonium isotopic ratio (^{240}Pu to ^{239}Pu) is greater than or less then the threshold value.

A set of reference materials (RMs) was specially manufactured for the AVNG with masses and isotopic ratios above and below the selected thresholds. The set of RMs was certified in compliance with the Russian metrological requirements. The RMs were used to debug and test the AVNG and to demonstrate the AVNG operation to an American delegation in June 2009. In this presentation, we will describe the various steps in the manufacture and certification of these RM sources.

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INTRODUCTION

- **Goal: to manufacture plutonium dioxide standard reference materials (SRMs) for the testing, calibration and demonstration of the AVNG.**
- **Certified by the Russian Federal Agency on Technical Regulation and Metrology (FATRM).**
- **The AVNG was successfully tested and demonstrated in June 2009 using the SRMs.**

A REQUIREMENTS SPECIFICATION ON MANUFACTURE AND CERTIFICATION OF STATE REFERNCE MATERIALS OF ISOTOPIC COMPOSITION AND MASS OF PLUTONIUM IN PLUTONIUM DIOXIDE

The main requirements to the reference materials are specified. The requirements are divided into two categories: basic metrological characteristics and supplementary characteristics. The supplementary characteristics determined the requirements to impurity content and container parameters for the reference materials. In the TO specified also were the requirements on security and safety, transportation and storage, labeling.

REFERENCE MATERIAL CERTIFICATION PROGRAMS

In the program the requirements are specified to the basic and supplementary characteristics determination accuracy, an order is established for certified values determining. In particular, a list of preparatory jobs is specified, the sampling procedure, the number of samples, techniques to be used for certified values determining.

JOB LIST ON SRM MANUFACTURE

- Manufacture of the initial material for RM
- Samples taking for MS determining and archive samples taking
- Manufacture of package
- RM material packing into containers

MEASUREMENT TECHNIQUES

- Mass-spectrometric method of isotopic composition determining.
- Measurement technique for plutonium mass fraction determining in plutonium dioxide with the coulometric method

REFERENCE MATERIAL MANUFACTURE

$^{240}Pu/^{239}Pu$ ratio - 0.25

The batches were gathered which included 6 VVER-440 assemblies and 2 BN-600 assemblies.

assembly cutting→ dissolution → filtration of the solutions→ extractive purification of U, Pu, and Np to remove fission fragments →plutonium separation from uranium → plutonium separation from neptunium → second extractive purification of plutonium to remove fission fragments → deposition of plutonium oxalate → annealing with plutonium packing into containers

REFERENCE MATERIAL MANUFACTURE

$^{240}Pu/^{239}Pu$ ratio - 0.07, 0.1

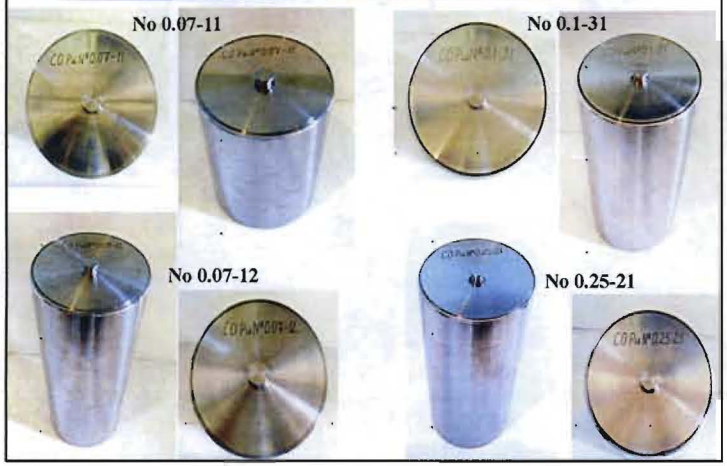
When after VVER-440 spent fuel element processing there takes place transition to BN-600 spent fuel element processing the isotopic composition changes due to plutonium from already processed fuel depositing in the apparatus and service lines. From the massif of plutonium built up during the transition period containers were sampled with $^{240}Pu/^{239}Pu$ ratios of 0.07 and 0.1.

Container number	Isotopic composition					$^{240}Pu/^{239}Pu$ ratio
	Pu-238	Pu-239	Pu-240	Pu-241	Pu-242	
1	0.74	90.41	6.27	1.71	0.87	0.07
2	0.91	91.01	6.43	1.16	0.41	0.07
3	0.91	91.11	6.45	1.13	0.40	0.07
4	0.19	89.99	8.63	0.76	0.43	0.10
5	0.35	88.97	8.61	1.26	0.81	0.10
6	0.41	88.64	8.56	1.43	0.91	0.10

MANUFACTURE

- ✓ Load PuO_2 powder into the containers;
- ✓ Weigh the containers;
- ✓ Weld the lids on the containers;
- ✓ Air leak test the containers; and
- ✓ Perform acceptance testing and inspection of the containers.

REFERENCE STANDARDS



ANALYTIC INVESTIGATIONS TO DETERMINE METROLOGICAL CHARACTERISTICS OF THE REFERENCE MATERIALS

Parameter \ RM identification		Pu №0.07-11	Pu №0.07-12	Pu №0.25-21	Pu №0.1-31
Pu^{240}/Pu^{239} ratio		0,072		0,254	0,107
Plutonium isotope mass fraction in plutonium, %	Pu-238	0,954		1,876	3,679
	Pu-239	*90,952*		*70,639*	*84,312*
	Pu-240	*6,522*		*17,970*	*9,022*
	Pu-241	1,139		5,698	2,012
	Pu-242	0,434		3,817	0,975

ANALYTIC INVESTIGATIONS TO DETERMINE METROLOGICAL CHARACTERISTICS OF THE REFERENCE MATERIALS

Parameter	RM identification in the AVNG set			
	Pu № 0.07 – 11	Pu № 0.07 – 12	Pu № 0.25- 21	Pu № 0.1- 31
Plutonium mass in RM, g	993,7	3477,0	999,0	2006,3

Parameter	RM identification in the AVNG set			
	Pu № 0.07 - 11	Pu № 0.07 - 12	Pu № 0.25- 21	Pu № 0.1- 31
Plutonium mass fraction in plutonium dioxide, %	86,02		86,03	86,04

REFERENCE MATERIAL CERTIFICATION

Several documents were required as part of the certification process.

1. Pattern approval certificate for the type of a single exemplar of the set of standards of plutonium mass and isotopic composition in plutonium dioxide.
2. Specification of the type of the set of standards of plutonium mass and isotopic composition in plutonium dioxide.
3. Testing program and technique for certification of the set of standards of plutonium mass and isotopic composition in plutonium dioxide.
4. Conformance sheet of the results of tests of the set of standards of plutonium mass and isotopic composition in plutonium dioxide.

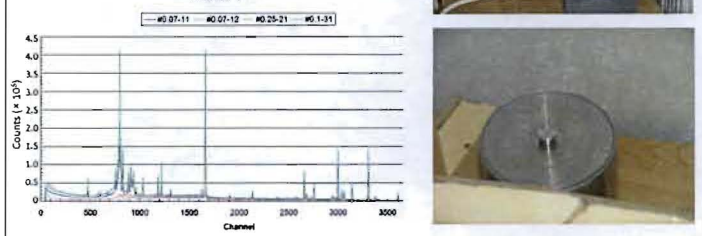
ACCEPTANCE TESTING

Acceptance testing performed at the Russian Federal Institute of Physics and Power Engineering (IPPE)

- **Gamma Spectrometry** (plutonium isotope fractions)
- **Calorimetry** (mass of plutonium)
- **Neutron multiplicity counting** (mass of plutonium)

GAMMA SPECTROMETRY

1. Two high-resolution gamma-spectrometric systems based on InSpector 2000 analyzers and coaxial detectors GC2518, GC2018, named conventionally FRAM-1, FRAM-2.
2. Two high-resolution gamma-spectrometric systems based on InSpector analyzers with planar detectors CL0515R, named conventionally MGA-1, MGA-2.



CALORIMETRY

Calorimetric measurements of the four SRMs were performed with the use of the calorimeter Antech, Series 200, model 263, 0804/03-/P1626323.

Only SRMs No 0.07-11 and No 0.25-21 were analyzed with calorimeter due to the high power of the remaining standards

Reference No	Pu mass (kg)	$^{240}Pu/^{239}Pu$	Expected thermal power
Pu No 0.07-11	.994	0.72	7.72
Pu No 0.25-21	.999	0.254	13.96
Pu No 0.07-12	2.003	0.107	27.03
Pu No 0.1-31	3.477	0.072	47.99

NEUTRON MULTIPLICITY COUNTING

- AWCC, JSR-14 shift register in the mode 2150, control and data processing program INCC 4.05;
- HLNCC-1, AMSR-150 shift register, control and data processing program INCC 4.05;
- HLNCC-2, JSR-14 shift register in the mode 2150, control and data processing program INCC 4.05;
- NMCS, Canberra 2150 shift register, control and data processing program INCC 4.03

ACCEPTANCE TESTING RESULTS

GAMMA SPECTROMETRY

Isotope	Deviation from the certificate values, %		
	CO Pu № 0.07 – 11,12	CO Pu № 0.25- 21	CO Pu № 0.1- 31
^{238}Pu	1.3	1.1	1.6
^{239}Pu	0.05	0.3	0.24
^{240}Pu	1.0	2.6	1.9
^{241}Pu	4.0	4.3	2.1

CALORIMETRY

Deviation from the certificate data for the mass from the calorimetric measurements for SRM № 0.07-12 and № 0.1-31 is less than 0,5%.

NEUTRON MULTIPLICITY COUNTING

Deviation from the certificate data of the masses measured by the neutron multiplicity counting is:

№ 0.07	– 11,12	0.7 %
№ 0.07	– 12	1.9 %
№ 0.25	– 21	0.6 %
№ 0.1	– 31	4.5 %.

Questions?

РФЯЦ ВНИИЭФ

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