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Preparation of Certified Working Reference Material Sources for the National TRU Waste Performance Demonstration Program

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Introduction

Traceable non-destructive assay (NDA) standards containing a variety of radionuclides including uranium, americium, and plutonium oxides mixed with an inert matrix were prepared and certified for use in the U.S. Department of Energy's National TRU Waste Program (NTWP). The NTWP requires traceable nuclear material standards of the Working Reference Material (WRM) class for qualification of NDA instrumentation that is used to quantify nuclear material in DOE-generated waste before the waste is shipped for final disposition at the Waste Isolation Pilot Plant (WIPP) in Carlsbad, New Mexico. Qualification and approval of measurement systems is accomplished in part through successful participation in the Non-Destructive Assay (NDA) Performance Demonstration Program (PDP) and is required for DOE and EPA regulatory compliance. An overview of the PDP program highlighting the role of the certified WRMs fabricated at LANL is presented, as well as a summary of the WRM fabrication process and an overview of the inventory of over 175 WRMs fabricated and deployed to DOE measurement facilities to date.

Results

In addition to traceable nuclear material standards, the other critical component of the PDP are the PDP matrix drums, which are surrogate waste drums that have been manufactured to simulate various waste streams. Matrixes simulated include combustible, metal, glass, and sludge drums. PDP Test Cycles are conducted in which selected PDP standards are inserted into PDP matrix drums in a specified manner to form a PDP test sample. The test sample is then presented to NDA analysts who measure the drum in a blind test and report their results for scoring. Satisfactory performance contributes to qualification of the NDA measurement system, permitting the NDA system to be used to quantify waste bound for WIPP.

Preparation of the required certified, traceable, working reference material standards is a multistep process that begins with specifications established in a Statement of Work. Criteria are designed to span a range of activities specified in WIPP requirements.[1,2] Seven PDP source fabrication campaigns were conducted, producing seven Phases of certified, traceable nuclear material sources, each with distinct properties (see Table 1). The majority of the standards fabricated contained plutonium or uranium in the form of powdered oxides (PuO_2 or U_3O_8), and in these cases the oxide was thoroughly characterized by analytical chemistry before being homogeneously blended with diatomaceous earth (an inert matrix material). The blended material was then packed into a stainless steel cylinder. For the Phase II.B standards, macroscopic plutonium oxide granules were embedded in graphite felt disks; the disks were stacked in layers in the stainless steel cylinder. All cylinder end caps were welded in place using a glovebox-mounted welding stand and TIG welder, after which the cylinders were decontaminated, helium leak-checked, and welded into a second, outer stainless steel cylinder using a second welding stand outside the glovebox to assure that the outer cylinder remained cold and uncontaminated. After final dimensional and radiological inspection, the nuclear material content of the completed WRMs was verified by gamma-ray spectroscopy as a quality assurance measure, validating the certified gram loading and material distribution. Holdup measurements on emptied blend bottles were carried out in order to correctly designate the net weight of nuclear material contained in each WRM. When fabrication, final inspections and verifications were complete, uncertainties were propagated in detail, and Certificates of Content and Traceability were prepared and transmitted to the DOE sites holding the PDP WRM sets.

Stainless steel cylinders were used for double encapsulation of the majority of the standards, although one set was encapsulated in zirconium cylinders to take advantage of the superior transmissibility of neutrons. Both stainless steel and zirconium containers were certified for containment of Radioactive Sealed Sources according to the ANSI Standard N43.7-1997.

Table 1. Summary of NDA PDP Production Phases

Phase	Content	Total Number of WRMs	Number of Sets	Number per Set	Approximate Mass Range
I, IIA	WG Plutonium (^{239}Pu)	104	8	13	0.02-75 g
IIB	WG Plutonium, Increased Particle Size (^{239}Pu)	32	8	4	0.3-30 g
IIC	High-Enriched Uranium (^{235}U)	20	4	5	1-75 g
IID	Depleted Uranium (^{238}U)	6	1	6	0.5-1 kg
IIIA	Increased Am/Pu Ratio (^{241}Am/^{239}Pu)	15	5	3	5-250 mg Am, 0.10-10 g Pu
IIIB	Heat Source Plutonium (^{238}Pu)	10	2	5	0.5-350 mg

Discussion

Required traceability to national standards of critical standard properties such as nuclear material mass, assay, and isotopic composition was established during analytical characterization of the nuclear material feedstock used in each set of standards. The analytical procedures used to establish the critical quantities are calibrated with primary Standard Reference Materials (SRMs) or Certified Reference Materials (CRMs). In addition, all weighing was performed on certified balances whose performance was verified using certified check weights.

The critical quantities must also be bound by uncertainty limits. Careful consideration is given to factors contributing to uncertainty. Standard methods for propagation of uncertainty were employed to arrive at final bounds for all quantities given on the WRM Certificate of Content and Traceability.

The PDP evaluates method capability and performance data submitted by the measurement facility and performs quality assurance audits. The NDA PDP uses prescribed test hardware, measurement routines and data analyses in the overall evaluation of TRU waste NDA system performance per established criteria. The NDA PDP also provides a platform useful for ascertaining inter-laboratory comparability of waste NDA techniques employed at the various sites across the DOE complex.

References

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