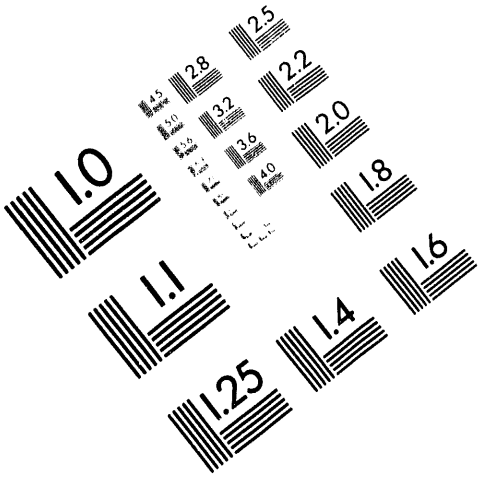

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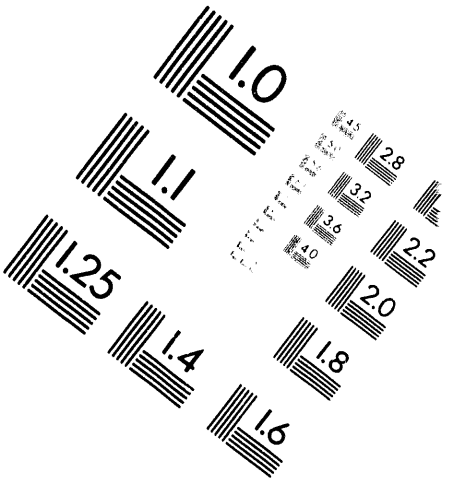

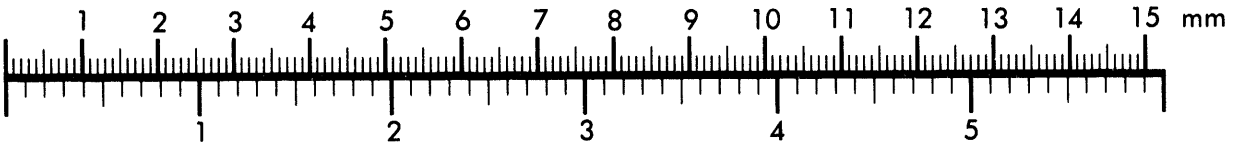

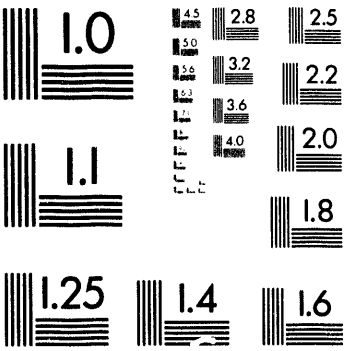

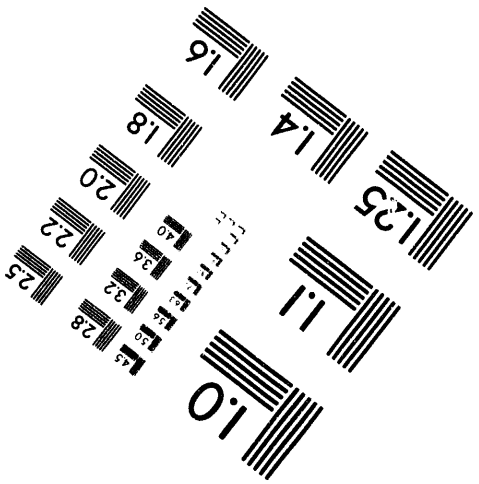

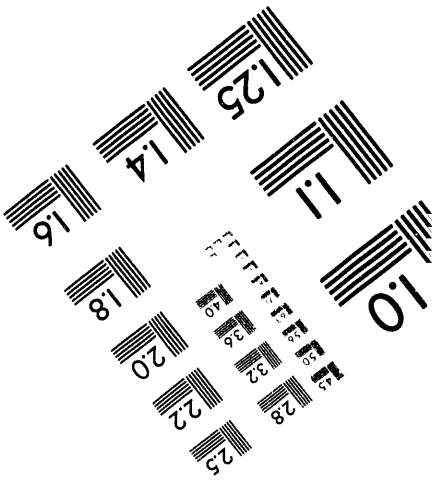

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Title: DESIGN AND FABRICATION OF 55-GALLON DRUM SHUFFLER STANDARDS

Author(s): S. M. Long, F. Hsue, C. W. Hoth, R. A. Fernandez, C. Bjork, and J. K. Sprinkle

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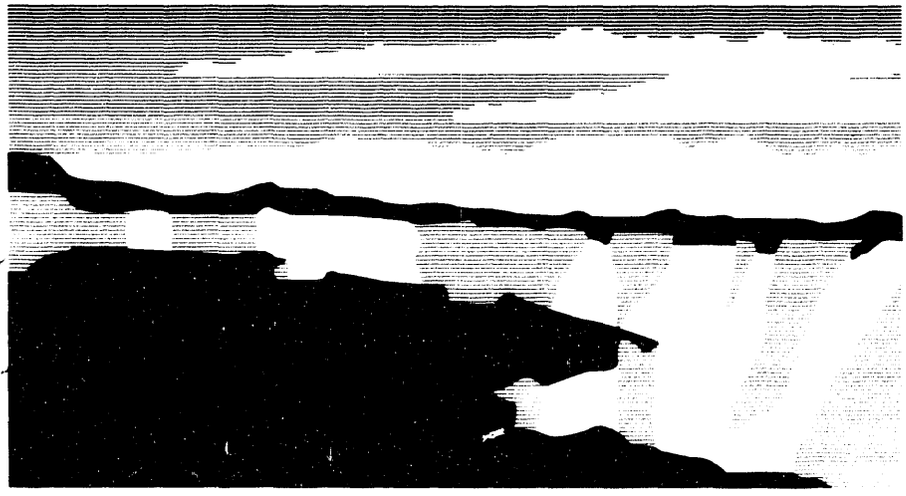

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DESIGN AND FABRICATION OF 55-GALLON DRUM SHUFFLER STANDARDS

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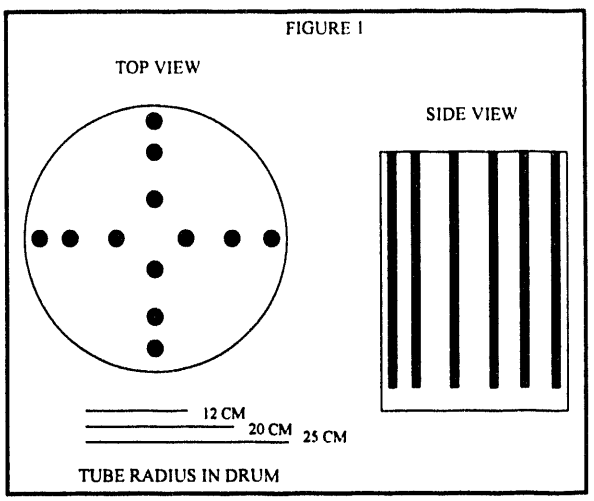
ABSTRACT

To analyze waste with varying levels of nuclear material, suitable standards are needed to calibrate analytical instrumentation. At the Los Alamos Plutonium Facility, we have designed and fabricated a single drum standard for a passive-active neutron counter (shuffler). The standard is modified simply by adding or subtracting plutonium or uranium cylinders to adapt to a range of nuclear material. The plutonium or uranium oxide was placed into small cylindrical containers (1-in. diameter by 5-in. long) and diluted with diatomaceous earth. The cylinders were welded closed and removed from the glove box environment without any external contamination. The containers were leak tested and then placed on a segmented gamma scanner to assure homogeneous distribution of the nuclear material. The cylinders are now placed into the drum to achieve the needed ranges for calibration of the instruments.

INTRODUCTION

The shuffler (passive-active neutron counter) instruments installed at Los Alamos National Laboratory are used to measure waste generated at the Plutonium Facility and the Chemical and Metallurgical Research Building. Assays are based on the use of well-characterized calibration standards. The actinide material must be well analyzed with National Institute of Standards and Technology (NIST) traceability to meet the physics and regulatory requirements of DOE order 5633.3.[1]

A 55-gallon drum standard was needed for the shuffler calibration. Waste material generated at both facilities was being packaged in drums and measured. Currently the instruments being used in the measurements are calibrated with physically small standards. Initial designs for the shuffler standards were of a 55-gallon drum with approximately 64 aluminum tubes symmetrically placed throughout the drum. The final drum design contains 12 aluminum tubes. Figure 1 shows the arrangement of the tubing. The aluminum tubes would then be loaded with vials filled with plutonium or uranium oxide diluted in diatomaceous earth. The vials can now be arranged in any pattern with varying amounts of material to achieve the low and high range needed for calibration.



Previous experiments using Portsmouth vials (alumina soaked with uranyl nitrate) showed neutron self-shielding while being measured. Self-shielding describes the measurement situation where Special Nuclear Materials (SNM) on the outer surface shields the SNM in the center from the interrogating neutrons in the instrument. This effect would result in a lower calibration curve. The response of the standard drum would be less than that of a real waste drum being measured. The question was how to reduce this effect.

STANDARD PREPARATION

Containers

Because of the neutron self-shielding effects found in previous studies, a new design of vials was addressed. It is desirable for the instrument response to be independent of the SNM containers. The objective is for the instrument calibration to depend on the mass of SNM, yet be independent of the number and size of the SNM containers. It is desirable to encapsulate the SNM in a material which is transparent to neutrons. Aluminum and zirconium have cross sections for thermal neutrons ten times smaller than iron. Iron and aluminum are both

susceptible to acid attack, if the containers had to be decontaminated. Stainless steel can be readily decontaminated, but the iron content poses the problem. Zirconium became the preferable choice to stainless steel, aluminum, or iron. The new Zircalloy vials have a 1/16-in., wall thickness to minimize self-absorption. The vials would also be of greater length, five inches as compared to four of the Portsmouth vials. The plutonium vials were doubly encapsulated. The inner vial is 3/4-in. in diameter and a nominal size smaller than the 5-in. vial. The uranium oxide was singly packaged in the 1-in. diameter vial. Vial spacers filled with diatomaceous earth was also fabricated to allow for stacking and separation of the oxide vials.

The 55-gallon drum is filled with layers of cellutex and cored to fit the aluminum tubes (See Figure 1). Vials can then be placed in various positions throughout the drum achieving a variety of patterns for calibration and for experimenting in matrix effects.

Matrix

Diatomaceous earth was used as a diluent in the vials. The particle size of the diatomaceous earth is between 75 μm to 106 μm with the density at 0.26.[2] g/cm^3. Previous studies using graphite as a diluent showed a greater attenuation of the neutrons than that of diatomaceous earth. We favored the use of diatomaceous earth over graphite in other standards because it demonstrated excellent properties in handling, mixing uniformly with the SNM, and staying mixed for long periods of time. The necessary amounts of diluent was estimated form previous experience and calculation.[3]

Oxide Preparation

Both the plutonium and uranium oxide were high-fired at 950°C. The materials were blended for approximately four hours in a V-type blender and sieved through a 100-mesh screen (<150μm). Samples were then taken at various points of the blend and analyzed by CST-1 of the Los Alamos National Laboratory. See Table I for analytical results.

Table I. Plutonium and Uranium Analytical Results

Plutonium Analytical

SAMPLE ID		CST-1#	PU238	PU239	PU240	PU241	PU242	AM241(PPM)	LOI%	PU RECEIVED %	PU CALCINED %
PEOM77S1		75885	0.0125	93.735	5.993	0.2261	0.0336	118	0.11	87.84	87.97
PEOM77S2		75886	0.013	93.742	5.998	0.2235	0.0333	115	0.13	87.93	88.06
PEOM77S3		75887	0.0128	93.732	5.995	0.2244	0.035	117	0.08	87.94	88.05
PEOM77S4		75888	0.0127	93.734	5.993	0.2268	0.0331	117	0.04	87.88	87.86
PEOM77S5		75889	0.0124	93.745	5.984	0.2249	0.0338	117	0.06	87.94	87.93
	AVERAGE		0.01268000	93.73760000	5.99260000	0.22514000	0.03376000	116.80000000	0.08400000	87.90600000	87.97400000
	STDEV		0.00023875	0.00559464	0.00522494	0.00132023	0.00074364	1.09544512	0.03646917	0.04449719	0.08384510
	RSD (%)		1.882860629	0.005968406	0.087189871	0.586402795	2.202724223	0.937881092	43.41567269	0.05061906	0.095306676

Uranium Analytical

Sample ID	Uranium (Wt. %)	^{235}U (At. %)	^{235}U (Wt. %)	LOI (%)
78571	84.34	92.18	92.11	0.01
78572	84.39	92.18	92.11	0.04
78573	84.35	92.18	92.11	0.01
78574	84.29	92.20	92.12	0.08
78575	84.38	92.18	92.11	0.06
Average	84.33	92.18	92.11	
STD	0.039	0.009	0.005	
RSD	0.05	0.01	0.01	

Standard Loading

Before the vials were introduced into the glovebox, the vials were wrapped in a finger condom to minimize the contamination on the outer surface of the vial. This proved to be valuable in the plutonium loading, as it diminished the alpha contamination. The process helped in decontamination of the vial prior to extraction from the glovebox.

Calculated amounts of plutonium or uranium were placed into a tared vial. They were reweighed and verified, then they were filled with diatomaceous earth to an approximately 70% full level. A void was left in the vial to facilitate the blending of the oxide with the diatomaceous earth. The vials were then reweighed and the actual amounts of oxide and diatomaceous earth were calculated.

Welding

The loaded vials were taken to the welding glovebox for sealing of the lids. This was accomplished by automated Tungsten inert gas (TIG) welding. The plastic finger condoms were removed before welding and the vials were swiped before inserting them into the rotary chuck. The glovebox was flooded with helium before and during the welding process. Helium was used so the vials could be helium leak-tested when retrieved from the glovebox line.

Blending

All blending of the vials was performed in a T2C Turbula blender. The blending took approximately 20 minutes per vial. After the blending was complete, homogeneity was checked by Segmented Gamma Scanner (SGS).

VERIFICATIONS

SGS (Segmented Gamma Scanner)

Once the vials were welded, decontaminated, leak checked, and blended, they were tested for homogeneity. By measuring the vial in individual segments and analyzing the segments, it could be determined if the plutonium or uranium oxide had completely mixed with the diatomaceous earth. The segments were taken at 1/4" intervals.

Drum Standard

Many vials were produced to encase the wide measurement range of waste drums. See Table II for the number of vials fabricated and their plutonium and uranium loadings. Since the SGS verification has not yet been completed, the final measurements of the shuffler drum standard are incomplete.

Table II.

Plutonium

#	Unit mass	Total mass
55	4g	220g
4	2g	8g
6	1g	6g
4	0.5g	2g

Uranium Vials

#	Unit mass	Total mass
50	4g	20g
4	2g	8g
4	1g	4g
4	0.5g	2g

CONCLUSIONS

Preliminary reports show that the shuffler standards will furnish the needed ranges for calibration of the instruments. This paper hopefully shows the thought and careful handling considerations needed in producing such standards. Because of unforeseen circumstances at the Plutonium Facility, all necessary data could not be compiled before publishing. Further studies and measurements will require a follow-up report.

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1. Department of Energy Order 5633.3.

2. S.-T. Hsue, S.M. Simmonds, V.L. Longmire, and S.M. Long, "Design and Fabrication of SGS Plutonium Standards, ANS, 4th International Conference on Facility Operations- Safeguards Interface

3. P.M. Rinard, E.L. Adams, H.O.Menlove, J.K. Sprinkle, Jr., LA-12466-MS, "The Nondestructive Assay of 55-Gallon Drums Containing Uranium and Transuranic Waste Using Passive-Active Shufflers," Nov. 92.

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