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Title: FABRICATION OF 12% 240PU CALORIMETRY STANDARDS

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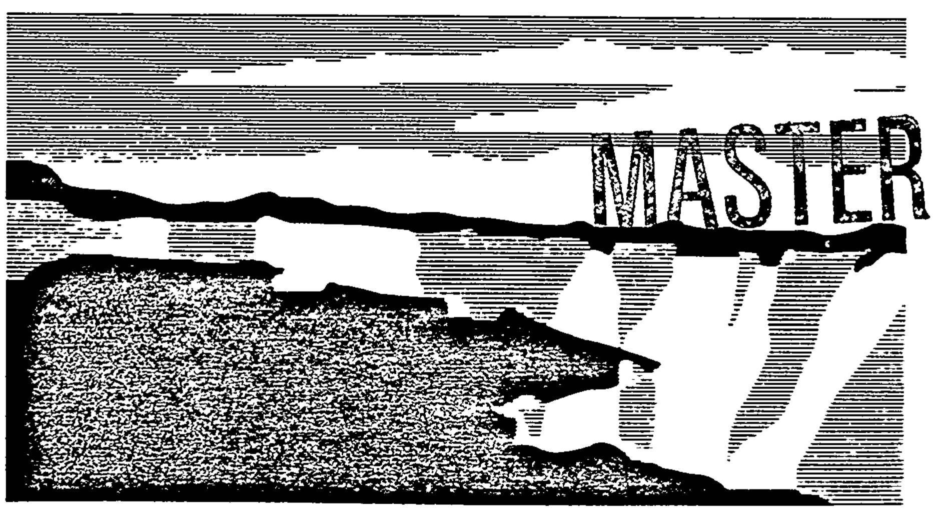

Los Alamos
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DISCLAIMER

FABRICATION OF 12% ^{240}PU CALORIMETRY STANDARDS*

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ABSTRACT

Throughout the DOE complex, laboratories are performing calorimetric assays on items containing high burn-up plutonium. These materials contain higher isotopic range and higher wattages than materials previously encountered in vault holdings. Currently, measurement control standards have been limited to utilizing 6% ^{240}Pu standards. The lower isotopic and wattage value standards do not compliment the measurement of the higher burn-up material. Participants of the Calorimetry Exchange (CALEX) Program have identified the need for new calorimetric assay standards with a higher wattage and isotopic range. This paper will describe the fabrication and verification measurements of the new CALEX standard containing 12% ^{240}Pu oxide with a wattage of approximately six to eight watts.

HISTORY

Since 1979, an annual calorimeter meeting is attended by representatives of the various DOE laboratories. In 1981 each laboratory was given a well characterized 6% ^{240}Pu oxide to intercompare numerous measurements on calorimeters and various isotopic codes (GRPAUT, FR^M, TRIFID, and MGA). The results are compiled and published in quarterly and annual reports.

In the past three years, the CALEX participants recognized the need for high wattage standards due to an increase in the measurements of higher burn-up plutonium material. Contributing factors include the consolidation of large amounts of high burn-up plutonium material in vault holdings. There is also an increase in shipper/receiver measurements of the same type of material. High burn-up material was needed for calorimeter standards to compliment the one watt 6% ^{240}Pu standards and encompass the wide range of wattages being encountered in the field.[1] The Calorimetry Exchange Group (the Exchange) identified 12% ^{240}Pu oxide material to fabricate the high wattage standards. In addition to alleviating the previous problems, the oxide standards would also provide information on the performance of the calorimeters and gamma-ray isotopic measurements in higher wattage standards.

INTRODUCTION

The Exchange decided ten standards were needed to distribute to the various laboratories. The net weight of each oxide standard would be two kilograms in order to achieve the necessary higher wattage. A total of 24 kilograms of material was needed for destructive analysis sampling, completing the standards, and archive material for historical purposes. Each laboratory was given the task of locating the needed oxide at their respective sites. The ideal oxide needed for the project was found at Westinghouse Hanford Laboratory. The oxide was 12% enriched and came from a large homogenized lot with a sufficient quantity for fabricating the standards. This would alleviate the need to blend non-related batches to accommodate the 24 kilograms.

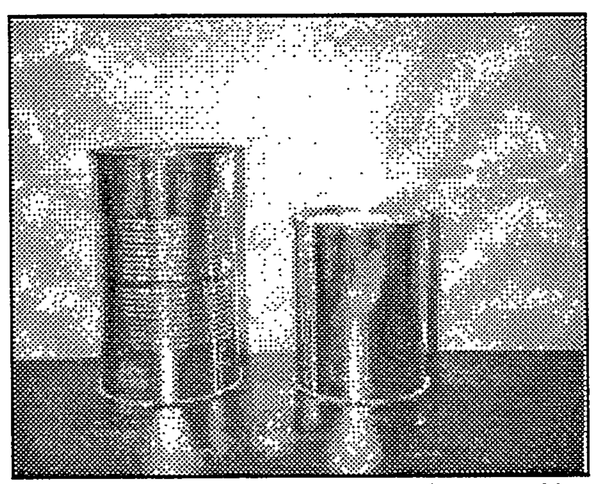

Figure 1 Inner and outer container used in the fabrication of the 12% CALEX standards.

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STANDARD PREPARATION

Containers

The standards were packaged in two thin-walled, .025 gauge, stainless steel cans. The inner can is a 401 x 411 ($4\ ^{1}/_{16}$ x $4\ ^{11}/_{16}$). The outer can is a 404 x 615 ($4\ ^{1}/_{4}$ x $6\ ^{15}/_{16}$).

The inner can was filled with the desired amount of oxide and hermetically sealed in the glovebox. It was extracted from the glovebox line and placed into a plastic bag, taped, and placed into a secondary plastic bag as a safety precaution against contamination. The doubly plastic wrapped can was then placed into the outer container and also hermetically sealed. This procedure will be discussed later.

Oxide Preparation

The total batch of oxide from Hanford Laboratory consisted of 17 cans. Each can was confirmed by calorimetry for shipper/receiver differences. They were introduced into the glovebox, verified by weight, and opened. The 24 kilograms were combined, then split into three equal batches. Each batch was high fired at 950° C for approximately eight hours using a ramp and soak technique. This consisted of raising the temperature at slow rates over a long period of time until a maximum temperature of 950°C was achieved. After the calcination was complete, the oxide was reweighed. A >0.5% weight loss was noted for each batch. The oxide was sieved through a 100 mesh screen (150um) to eliminate the large clumps or other debris that might appear in the oxide during unpackaging. Due to the limited volume in the blender, each batch was again split into three lots, A', B',and C'. The three A' lots were combined into a large mixing jar and blended in a Turbula blender for approximately forty minutes. The same was done for the B' and C' lots. The lots were then combined into one large lot for sampling and loading the inner standard containers.

Sampling of the Oxide

The homogeneity of the oxide was verified by destructive analysis. Fifteen, five gram samples were taken at random locations throughout the oxide, packaged, and sent for analysis at Los Alamos National Laboratory, New Brunswick Laboratory, and Westinghouse Hanford Laboratory. The multi-laboratory analysis will minimize any bias within one laboratory's chemical assay.[2] All three laboratories used calibration standards certified through the National Institute of Science and Technology (NIST). Results have not been obtained from any of the participating laboratories and thus will not be included in this paper.

Loading/Sealing of the Inner Containers

Once the samples were taken from the total batch, the ten inner containers were loaded. Each inner container was tared and filled with two kilograms net weight of oxide. The loaded inner container was placed on a mechanical canner and sealed. The sealed containers were weighed and recorded.

Removing the Standards from the Glovebox

The ten inner contaminated cans were taken out of the glovebox using a procedure called a can-out. This procedure is a two person operation in which one individual will hand the contaminated can to the other through a port. The can is placed into a thin layer plastic bag and taped closed. A thicker layered plastic bag is placed over the thin bag to ensure contamination is encased within the bags and does not pose a threat to the personnel.

A radiological health physician checks the doubly wrapped container before it is placed into the outer container. The outer container is placed onto the "cold" (non-contaminated) canner and sealed. The total package is checked for contamination, weighed, recorded and labeled.

Verification Measurements

The standards were taken to the Non-Destructive Assay laboratory for verification on wattage, isotopics, and plutonium loadings. The standards were loaded on ROBOCAL (an automated calorimeter and isotopics robot) and measured in 5" and 7" calorimeters. The calorimeters were calibrated with two heat source standards encompassing the wattage window of the 12% oxide standards. Initial results show all ten standards measured at 6.2 watts. Initial results show all ten CALEX standards measuring at 6.2 watts. The standards showed no significant differences in wattage between the two different chambers. See Table 1.0 for wattage results. The oxide standards verified in homogeneity by isotopic specific power measurements. The measurements taken on the FRAM and TRIFID isotopic codes show little variance between the two codes. See Table 2.0 for the results. Further comparison measurements will also be on done on GRPAUT and MGA. Wattage measurements will be done by EG&G Mound Laboratory before the standards are shipped to the representative laboratories.

WATTAGES FOR 12% CALEX STANDARDS	
STANDARD ID	WATTAGES CALORIMETERS 3 or 4
CAL12%-1	6.234241 (4)
CAL12%-2	6.232959 (3)
CAL12%-3	6.232213 (3)
CAL12%-4	6.235318 (3)
CAL12%-5	6.238634 (3)
CAL12%-6	6.240940 (3)
CAL12%-7	6.234773 (4)
CAL12%-8	6.244066 (4)
CAL12%-9	6.241826 (4)
CAL12%-10	6.243504 (4)
AVERAGE	6.2378474
STANDARD DEVIATION	0.003947

Table 1.0 Wattage measurements on the individual 12% CALEX standards.

SPECIFIC POWER FOR THE 12% CALEX STANDARDS		
STANDARD ID	FRAM WATTS/ GRAM PU	TRIFID WATTS/ GRAM PU
CAL12%-1	3.55207 +/- 0.01464 E-03	3.54160 +/- .029 E-03
CAL12%-2	3.57910 +/- 0.01455 E-03	3.51670 +/- .033 E-03
CAL12%-3	3.55440 +/- 0.01326 E-03	3.55740 +/- .030 E-03
CAL12%-4	3.56830 +/- 0.01454 E-03	3.52700 +/- .029 E-03
CAL12%-5	3.57620 +/- 0.01490 E-03	3.53800 +/- .031 E-03
CAL12%-6	3.55901 +/- 0.01546 E-03	3.52470 +/- .025 E-03
CAL12%-7	3.55739 +/- 0.01128 E-03	3.53960 +/- .030 E-03
CAL12%-8	3.57222 +/- 0.01214 E-03	3.52380 +/- .031 E-03
CAL12%-9	3.55619 +/- 0.01296 E-03	3.54140 +/- .029 E-03
CAL12%-10	3.56364 +/- 0.01413 E-03	3.52770 +/- .028 E-03
AVERAGE	3.563852 +/- 0.013786 E-03	3.53379 +/- .0295 E-03
STANDARD DEVIATION	0.009585 +/- 0.001333 E-03	0.002179+/- .002179

Table 2.0 Specific Power comparisons using FRAM and TRIFID isotopic codes on the ten standards.

Conclusion

Initial measurements used in determining the homogeneity and wattage consistency of the CALEX standards indicate that the oxide is well mixed and performing well to the specified fabrication requirements. Since the standards were just recently fabricated, the awaiting analytical results will determine the true homogeneity, isotopics, plutonium loadings, and the moisture content of the oxide. Intercomparisons will be done with the calorimeter/isotopic and the analytical results.

References

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