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EXECUTIVE SUMMARY

The Department of Energy (DOE) has identified the Dilute and Dispose[a] approach as the preferred method for the disposition of surplus plutonium materials. Dilute and Dispose requires surplus plutonium to be converted to an oxide product and then dry blended with a multi-component adulterant mixture to reduce the Safeguards and Security attractiveness level of the materials. The resulting product will be discarded in a geologic repository at the Waste Isolation Pilot Plant (WIPP).

A recent Nuclear Criticality Safety Assessment (NCSA) concluded that a nominal amount of neutron absorber (boron carbide) included as part of the multi-component adulterant would ensure subcriticality is maintained for all scenarios for credible configurations that could occur at WIPP during the post-closure disposal time period (10,000 years). Boron, like other light elements, interacts with alpha particles to create neutrons. Thus, adding boron carbide to plutonium as a neutron poison during the downblend process may increase the neutron exposure for workers producing and handling the dilute materials for discard.

To evaluate the effect of boron carbide on the neutron dose rate, measurements were performed on representative materials. Multiple batches of diluted plutonium material were created with approximately one tenth the total material of an actual dilute and dispose container. These batches were measured prior to dilution, after addition of the multi-component diluent without boron carbide, and with diluent containing boron carbide.

Recognizing the potential for increased neutron radiation dose rates, two forms of boron carbide have been identified for use in the multi-component adulterant. One form was plain boron carbide particles, and the other form was boron carbide particles coated with metallic nickel to block the alpha particles from reaching the boron carbide. Both uncoated and nickel-coated boron carbide particles were added to separate batches of adulterated material for neutron measurements. The thickness of the nickel coating was chosen to block a high percentage of the alpha particles emitted by plutonium or americium. It was expected that the nickel coating would reduce or eliminate the potential increase in neutron dose associated with use of boron in the multi-component adulterant mixture.

Six aliquots were made for each round of measurements, with the final batch containing three samples with uncoated boron carbide and three samples with nickel-coated boron carbide. As shown in the summary table, the addition of the uncoated boron carbide increased the neutron rate by almost 33% over the material with diluent only. In comparison, the nickel-coated boron carbide increased the neutron rate by only 4%.

Evaluation of the neutron dose rate for the multi-component adulterant mixture containing boron carbide also provided an opportunity to assess potential operational impacts associated with non-destructive assay (NDA) for the downblended plutonium mission. While the addition of boron carbide to the weapons grade plutonium used in these experiments was observed to increase the neutron rate, this increase is not large enough to substantially impact the precision of neutron-

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[a] The terms dilute and downblending are used interchangeably by the U.S. Department of Energy (DOE) and National Nuclear Security Administration (NNSA) to describe the process for mixing surplus plutonium with an adulterant to ensure plutonium is not recoverable without extensive processing. DOE Office of Environmental Management (DOE-EM) uses the term downblending to describe this process. The term dilution is the international nomenclature for using an adulterant to provide proliferation resistance and is in no way intended to avoid any applicable regulatory requirements.

based NDA. It is expected that fuel-grade plutonium materials, which contain a higher quantity of alpha-emitters, will display a similar effect when mixed with boron carbide.

Summary Table. Measured neutron rates for pure, diluted, and poisoned samples

	total neutron rate (n/s/g)	compared to diluted oxide
pure oxide	113.3 ±0.49	84.9%
diluent added	133.5 ±0.47	100.0%
diluent + nickel coated B_4C	138.7 ±0.26	103.9%
diluent + uncoated B_4C	177.3 ±1.15	132.8%

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ASTAR	Program to calculate stopping power and range tables for helium ions in various materials
AWCC	Active Well Coincidence Counter
CCO	Criticality Control Overpack
CSDA	Continuous-Slowing Down Approximation
DOE	Department of Energy
EM	Environmental Management
FRAM	Gamma ray isotopic analysis software from Los Alamos National Laboratory
HPGe	High-Purity Germanium
ICRP	International Commission on Radiological Protection
IDMS	Isotope Dilution Mass Spectrometry
INCC	International Neutron Coincidence Counting
KAC	K Area Complex
LANMAS	Local Area Network Material Accounting System A computerized application used for tracking nuclear inventories, documenting material transactions, and issuing periodic reports.
NCSA	Nuclear Criticality Safety Assessment
NDA	Non-Destruction Assays
NIST	National Institute of Standards and Technology
NMC	Neutron Multiplicity Counter
NNSA	National Nuclear Security Administration
NPT	Neutron Poison Test or Identifier of the adulterating recipe used
NPT1	Item identifier for the plutonium oxide used in this work
ORNL	Oak Ridge National Laboratory
PSR-B	Portable Shift Register
RCRA	Resource Conservation and Recovery Act
SEM	Scanning Electron Microscope
SNM	Special Nuclear Material
SRNL	Savannah River National Laboratory
SRS Stardust	Dilution recipe from SRNS-N2000-2011-00161
TCLP	Toxicity Characteristic Leaching Procedure
WIPP	Waste Isolation Pilot Plant
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

1.0 Introduction

The dilute and dispose approach (or downblending) is an ongoing activity at the K-Area Complex (KAC) for dispositioning plutonium material under the Department of Energy (DOE) office of Environmental Management (EM). Dilute and dispose is also the preferred approach for dispositioning additional plutonium held by the National Nuclear Security Administration (NNSA). The dilute and dispose materials would ultimately be discarded in the geologic repository at the Waste Isolation Pilot Plant (WIPP).

A recent Nuclear Criticality Safety Assessment (NCSA) concluded that, although the dilute and dispose material would be packaged in criticality control overpacks (CCO), shifting during the geological storage at WIPP might produce a configuration that could potentially achieve criticality during the post-closure disposal time period (10,000 years).[1] Several additional scenarios were considered where natural-abundance boron was added to the mixture as a neutron absorber to reduce the nuclear reactivity of the final configuration. The boron was added as boron carbide as part of the multi-component adulterant. The assessment concluded that, even under bounding post-closure scenarios, a small amount of boron carbide, well mixed with the dilute and dispose material, would eliminate the possibility of a criticality.

Natural boron contains about 20 wt % ^{10}B and 80 wt % ^{11}B. ^{10}B has a large cross section for thermal neutron absorption, making it useful for reducing nuclear reactivity. However, both ^{10}B and ^{11}B have significant cross sections for (α,n) reactions, which produce a neutron when the nucleus is struck by an alpha particle. When mixed with an alpha emitter such as plutonium, boron can produce large quantities of neutrons and thereby increase the neutron dose rate from the material.

One key consideration of any nuclear materials processing is radiation exposure to operations personnel. For the dilute and dispose mission, radiation exposure is important during all phases of the process, beginning with the blending operation itself, continuing with storage in KAC, and concluding with final disposition at WIPP. Since the addition of boron carbide will increase the radiation dose to operations personnel, it was proposed that the boron carbide particles be coated with nickel to prevent the alpha particles from reaching the boron carbide to reduce the neutron dose from the (α,n) reaction.

The Savannah River National Laboratory (SRNL) was asked to evaluate the effect that adding both uncoated and nickel-coated boron carbide as a neutron poison to dilute and dispose material had on the neutron dose rate.[2] To perform this evaluation, SRNL created a batch of plutonium oxide diluted with a multi-component adulterant material similar to the diluted material produced by the dilute and dispose program in KAC.[3] Uncoated and nickel-coated boron carbide particles were added to separate batches of adulterated material for neutron measurements.

A secondary goal, especially for the future NNSA program, was to assess the ability to accurately measure the plutonium content of the blended material via neutron counting. The addition of boron carbide could negatively impact both the precision and accuracy of neutron-based non-destructive assays (NDA). Understanding the impact of the blending constituents on neutron generation and measurement is needed to guide future program decisions.

2.0 Theory

There are three concerns introduced by adding boron carbide to the dilute and dispose material. First, the (α,n) neutrons can significantly increase the dose rate of the material. Second, these neutrons can interfere with the assay of fission neutrons in a multiplicity counter by adding noise and reducing the precision of the assay. Third, as a neutron absorber, the boron may lower the efficiency of a neutron-based NDA. These last two effects combined could potentially make an item difficult to measure by NDA.

Most elements with low atomic numbers ($Z<18$) undergo the (α,n) reactions when mixed with plutonium. Even pure plutonium oxide exhibits a small generation rate of (α,n) neutrons due to the presence of oxygen. Because the plutonium is chemically bonded with the oxygen, the (α,n) reaction rate is well-known and easily calculated. However, when the low-Z elements are mechanically mixed, the interaction rate depends highly on the distribution of particle sizes and the quantity of material.

Both isotopes of boron (^{10}B and ^{11}B) have significant cross sections for (α,n) reactions, and neither has a threshold energy for the incident alpha particle. The thick target yield for plutonium alpha particles on boron is 17.5×10^{-6} neutrons per alpha, compared with 0.059×10^{-6} neutrons per alpha for plutonium alpha particles on oxygen. The neutron energy emitted from (α,n) reactions on boron (average 2.9 MeV) is somewhat higher than those emitted from fission (average 2.0 MeV) or from oxide (α,n) reactions (average 1.9 MeV).[4]

The total neutron rate of a plutonium sample is dependent on both the spontaneous fission rate (dominated by ^{240}Pu), and the (α,n) neutron rate due to low-Z elements in the sample. This rate can be expressed as follows:

$$R = (F \cdot \nu + A \cdot Y) \cdot M$$

where R is the neutron rate (neutrons / second / gram), F is the fission rate (fissions / second / gram), ν is the fission multiplicity (neutrons / fission), A is the alpha activity (alpha particles / second / gram), Y is the (α,n) reaction yield (neutrons / alpha), and M is the neutron multiplication in the sample. For this analysis, M is assumed to be 1, due to the low plutonium mass and dilute mixture. The parameters F, ν, and A are properties of the plutonium isotopics and cannot be changed, but Y can be changed by the addition or removal of interacting material.

In neutron assays, the parameter α (the "alpha ratio") is defined as the ratio between the (α,n) neutrons and the spontaneous fission neutrons:

$$\alpha \equiv \frac{A \cdot Y}{F \cdot \nu}$$

This parameter is easy to measure on a neutron multiplicity counter, and it is an excellent indicator of the presence of low-Z impurities that add to the neutron dose of the material.

To prevent the alpha particles from interacting with the boron, a coating of material can be added to the outside of the boron carbide particles. Coatings consisting of elements with $Z \geq 18$ will not increase the neutron rate of the material. Nickel ($Z=28$) was chosen as a convenient coating material for this application.

The highest major alpha energy in plutonium (originating from ^{241}Am daughter product) is 5.486 MeV; thus, the coating must be at least thick enough to stop alpha particles of this energy. Alpha stopping powers and ranges were taken from the ASTAR database at NIST.[5] The continuous-slowing-down-approximation (CSDA) range of ^{241}Am alpha particles is 0.01051 g/cm^2 in copper. ASTAR does not include data for nickel, but copper has an atomic number one higher than nickel, and a larger stopping range. Assuming a density of 8.9 g/cm^3 for nickel, this equates to a minimum required thickness of 11.8 μm.

3.0 Experimental Procedure

The experiment was conducted in three rounds of measurements.[2] In each round, material was prepared, divided into six roughly equal aliquots, and then assayed on a neutron multiplicity counter (NMC) to determine the neutron rates. The quantity of plutonium in each aliquot was chosen to be approximately one tenth of the amount that would be used in a full-sized blend can. Additionally, this quantity of plutonium contains almost exactly one gram of effective ^{240}Pu.

In the first round, a quantity of high-purity plutonium oxide was divided into aliquots and assayed. In the second round, the material was recombined, and the adulterant (without boron carbide) was added. The diluted mixture was again divided into six aliquots and assayed. In the third round, the material was again combined into a single batch and then split into two roughly equal batches. Uncoated boron carbide was added to one batch, and nickel-coated boron carbide was added to the other batch. Both batches were then divided into three aliquots each. All six aliquots were then assayed. Following the final round of neutron measurements, the material has been stored as the last six aliquots while waiting for final disposition.

3.1 Material Preparation

3.1.1 Adulterating recipe

The purpose of adding adulterant material to the plutonium oxide is to lower the composition of Special Nuclear Material (SNM) to 10 wt % or less, which reduces the safeguards attractiveness level and is a precursor for the future termination of safeguards.[3, 6] The plutonium was adulterated as described in G-ESR-K-00190, following the recipe in SRNS-N2000-2011-00161 (SRS Stardust), using a modified protocol to account for the smaller batch size. The recipe used in this work (NPT recipe) was modified to include the boron carbide as an additional adulterating component, and to introduce additional conservatism in the neutron generation rate. The differences between the two recipes are described below.

The original basis of the SRS Stardust recipe was 150 g (or less) of plutonium oxide. All other component amounts are specified as ranges, for example, 100 to 120 g of component X and 400 to 430 g of component Y. For the modified NPT recipe, the relative amounts of the components were held to the low ends of each range. For the remaining discussion, it is assumed that the SRS Stardust has three adulterating components which are held at a constant ratio of 4 parts of component A, 5 parts of component B and 6 parts of component C.

In the original SRS Stardust recipe, the target of less than 10 wt % SNM is conservatively achieved by basing the recipe on the weight of plutonium oxide, rather than the plutonium content of the oxide. The recipe specifies that no more than 150 g plutonium oxide could be used, which would contain at most 132 g of plutonium (if pure oxide). Using the minimum amount of adulterating materials, a mixture of less than 10 wt % Pu is produced even when using the maximum amount of plutonium oxide. For 150 g PuO_2, using the ratios set above would produce 360 g component A + 450 g component B + 540 g component C + 150 g PuO_2 = 1500 g total and resulting in 132 g Pu / 1500 g = 8.8 wt % Pu.

The NPT recipe was based on plutonium content, rather than plutonium oxide content. Thus, the oxygen content of the plutonium oxide is credited as part of the 90 wt % adulterating components. This introduced a slight conservatism to this work, since having a higher plutonium concentration increases the number of (α,n) reactions.

The final difference between the SRS Stardust recipe and the NPT recipe is that the SRS Stardust recipe did not include of boron carbide as an adulterant. In the NPT recipe, the boron carbide weight is used and credited as part of the 90 wt % diluent. However, in the NPT recipe, only the B_4C content of either boron carbide material was credited as part of the diluting material; neither the impurities nor the nickel, which do add weight, were credited as part of the adulterant in the NPT recipe. The boron carbide is added in the ratio of 50 grams of boron carbide to 380 grams of plutonium as recommended in the ORNL NCSA.[1] The differences between the two recipes are presented in Table 3-1, for a basis of 100 g of Pu. For comparison purposes, SRS Stardust with added boron carbide is also shown.

Table 3-1. Comparison of the NPT, SRS Stardust + B_4C, and SRS Stardust recipes (100 g Pu basis)

	NPT recipe	SRS Stardust	SRS Stardust + B_4C
Pu	100.00	100.00	100.00
PuO_2	113.39	113.39	113.39
Component A	232.39	272.13	272.13
Component B	291.15	340.17	340.17
Component C	349.38	408.20	408.20
B_4C	13.16	0	13.16
total	1000.00	1133.89	1147.05
Pu wt %	10.0	8.8%	8.7

3.1.2 Materials

3.1.2.1 Plutonium oxide

The plutonium oxide in these experiments (NPT1) was taken from a plutonium oxide shipment received from HB-Line (S042315) for use in this work. Prior to adding any adulterating material to NPT1, a disposition pathway for the final mixture had to be identified - after completion of these experiments, the adulterated NPT1 will be shipped to KAC.[7] For the adulterated material to comply with the KAC safety basis, the plutonium content must be documented as a Type S material as defined by the International Commission on Radiological Protection (ICRP) publication 66.[8] (Type S material has a slow rate of absorption into the bloodstream from inhaled particles.) ICRP publication 68 defines insoluble plutonium oxides as the only plutonium Type S material. Other (unspecified) plutonium compounds are Type M materials.[9] To confirm that the material received from HB-Line had been completely converted to plutonium oxide from the oxalate, an X-Ray Diffraction (XRD) measurement was performed to identify crystalline phases in the material. The XRD indicted there were no crystalline or amorphous materials present other than plutonium oxide. The process history of the material in HB-Line and characterization at SRNL are documented elsewhere.[10-11]

The plutonium content of S042315 was determined to be 85.8 wt % by dividing the plutonium elemental content of the item found in LANMAS by the measured weight. This ratio was used to determine the plutonium content of the subsequent sub samples. While this ratio is lower than the theoretical value of pure oxide, it is not unusual to see assays of 86 to 87 wt % for unprotected oxide. The oxide will absorb both moisture and carbon dioxide from the atmosphere, increasing the total weight, which, in turn, lowers the plutonium ratio. The NPT1 split used in this work was originally 116.28 g, with 100.17 g of plutonium as determined by employing the ratio.

3.1.2.2 Boron Carbide

The boron carbide and the nickel-coated boron carbide were purchased through Reade Advanced Materials. The boron carbide for both the plain and nickel-coated variants was the -30 +60 mesh refractory grade manufactured by U. K. Abrasives. The nickel coating was applied by Advanced Powder Solutions. A thickness of 12 µm was chosen for the nickel coating. The supplier reported that the nickel content was 30 wt % from X-ray fluorescence (XRF) analysis, which complies with the procurement specification. Due to a lack of precise analytical measurements, estimates were made for the actual boron carbide content of both materials. The plain boron carbide is assumed to be 89 wt % B_4C, based on labeling on the carton, and the nickel-coated boron carbide was assumed to be 61 wt % B_4C based on the mass of unidentified, low-Z compounds in the manufacturer's XRF analysis.

3.1.2.3 Adulterating material

The adulterating components had been previously purchased by SRNL for another study.

3.1.3 Round 1 – Plutonium Oxide Measurement

For the first round of measurements, NPT1 was divided into six aliquots. For this round and all subsequent rounds, each aliquot was placed into an individual slip lid can, and each can was removed from the glovebox as an individual piece. This allowed each piece to be measured separately, while preventing any contamination of the neutron multiplicity counter.

Between the first and second rounds, a small sample of material was removed for XRD analysis. After the sample was removed, the plutonium content of NPT1 was 99.94 g.

3.1.4 Round 2 – Adulterated Plutonium Oxide Measurement

Once the first round of neutron measurements was complete, the aliquots were returned to the glovebox and combined into a single container. Adulterant was prepared using the ingredients and recipe described above (omitting the boron carbide).

The preparation of the adulterating material and mixing of the adulterant with the plutonium oxide was performed per procedure L29, ITS-227.[12] After the plutonium was blended with all of the NPT recipe components except for the boron carbide, the diluted plutonium was again divided into six roughly equal aliquots by spooning the material into individual slip lid cans. This process did create some heterogeneity, which will be discussed in detail later. As in round one, each can was bagged out of the glovebox separately for measurement.

3.1.5 Round 3 – Adulterated Material with Boron Carbide

Once the second round of measurements was completed, the aliquots were again placed in the glovebox and combined into a single container. After mixing, the material was divided into two parts by the cone and split method. Each partition was weighed to determine the amount of boron carbide material to add. The nickel-coated boron carbide was added to the larger partition to enhance neutron detection. After the appropriate boron carbide material was added to each half, the halves were then divided into three aliquots each, using the cone and split methodology. After the division each aliquot was bagged out of the glove box individually and counted on the NMC.

The partitioning method was changed from the spooning method to the cone and split method (a variation of cone and quarter[13]) to try to reduce the heterogeneity in the round two measurements.

3.2 Non-Destructive Measurements

In each round, samples were measured using neutron multiplicity counting. This method counts time-correlated neutrons emitted by a sample, to determine not only the total neutron rate, but also the (α,n) rate, plutonium content, and neutron multiplication. The instrument used was an Active Well Coincidence Counter (AWCC) operated in the passive mode. Assays were performed for one hour each (60 cycles, 60 seconds each). The AWCC was in the "medium" configuration (25 cm tall cavity height, no nickel reflector).[14] Samples were placed in the center of the counting chamber. An Aquila Portable Shift Register (PSR-B) was used to collect multiplicity data, including the rates of the inner and outer rings of the AWCC.

The instrument was initially calibrated using a ^{252}Cf source placed at the center of the chamber. After the six pure oxide samples were measured, the triples gate fraction was adjusted such that the average bias between the measured values and values determined by mass was zero. These calibration parameters were then used for the remaining samples. The operating and calibration parameters used for this study are summarized in Table 3-2.

Table 3-2. AWCC operating and calibration parameters

Parameter	Value
High voltage	1080 V
Gate width	64 μs
Pre-delay	4.5 μs
Efficiency	0.296
Doubles fraction	0.604
Triples fraction	0.379
Fission neutron ring ratio	1.506

Isotopic data was obtained from an Isotope Dilution Mass Spectrometry (IDMS) analysis performed on the source material in HB-Line in December 2014. Values were decay corrected to June 2018 (the date that the pure oxide samples were measured) and are reported in Table 3-3. The pure samples were also measured on a coaxial, high-purity germanium (HPGe) detector for one hour each and analyzed with FRAM.[15] In this analysis, ^{242}Pu wt % from the IDMS data was used. No variance was found between the isotopics of the six samples. The six spectra were summed and analyzed again, and the results are reported in Table 3-3. The IDMS and FRAM results agreed extremely well. The IDMS value for effective ^{240}Pu was used in the neutron multiplicity analysis because of the lower uncertainty.

Table 3-3. Isotopic content of samples, measured by gamma and IDMS

	From FRAM	From IDMS
Pu-238 (wt %)	0.0275 ±5.0%	0.0277 ±1.48%
Pu-239 (wt %)	93.8846 ±0.2%	93.8609 ±0.03%
Pu-240 (wt %)	5.8766 ±1.0%	5.9365 ±0.42%
Pu-241 (wt %)	0.1413 ±0.3%	0.1413 ±3.96%
Pu-242 (wt %)	0.0336 (11.9%)	0.0336 ±11.9%
Am-241 (µg/g Pu)	269.9 ±1.0%	* 290.9 ±15.0%
Np-237 (µg/g Pu)	68.7 ±1.0%	
Pu-240-eff (%)	6.002 ±1.0%	6.063 ±0.43%
known-alpha	0.792 ±1.2%	0.786 ±0.61%

** Am-241 value for IDMS is primarily due to decay-correction of Pu-241; it also includes 15 µg/g measured by gamma in 2014.*

One sample from each of the four matrices was also measured on the HPGe for an hour with a range of 0-5 MeV to observe any prompt gamma production that would indicate alpha-induced reactions. None of the items exhibited any prompt gamma lines, which would have indicated gross amounts of impurities.

4.0 Results and Discussion

4.1 <u>Heterogeneity created during sample partitioning</u>

Partitioning of powders into aliquots and samples has long been recognized to be a non-trivial task. Many different techniques and devices have been developed to help take representative samples and produce uniform aliquots. Adequate care must be taken to prevent separation of the material during the splitting process. Self-separation of the material is greatly accentuated when the mixture has particles of different sizes and densities. The plutonium content of each aliquot is given in Table 4-1, and is shown graphically in Figure 4-1.

Table 4-1. Plutonium Content of Aliquots

Aliquot	**Round 1**		**Round 2**			**Round 3**		
	By mass	**Measured**	**By mass**	**Adjusted by mass**	**Measured**	**Plain B4C by mass**	**Ni-B4C by mass**	**Measured**
A	16.70	16.83	16.67	16.38	15.98	-	17.48	17.95
B	16.80	16.96	16.65	16.36	15.85	-	17.09	16.09
C	16.45	16.24	16.66	16.37	15.93	-	18.61	17.46
Average	**-**	**-**	**-**	**-**	**-**	**-**	**17.72**	**17.17**
D	16.81	16.96	16.65	16.37	16.00	15.57	-	14.84
E	16.47	16.43	16.66	16.38	16.22	15.41	-	16.37
F	16.82	16.63	16.57	18.00	17.63	15.59	-	14.87
Average	**-**	**-**	**-**		**-**	**15.52**	**-**	**15.36**
Overall Average	16.67	16.68	16.64		16.27	16.62		16.26

All values are in grams.

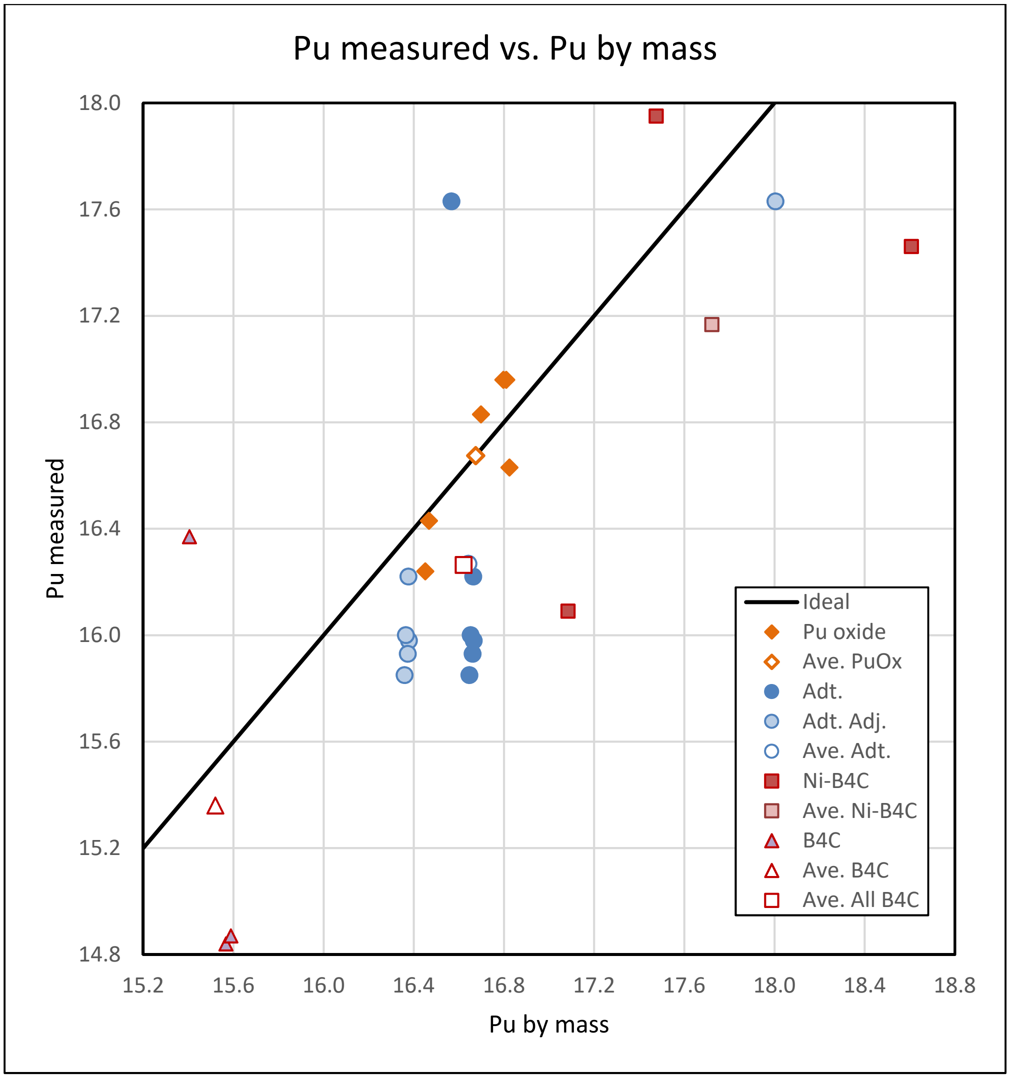

Figure 4-1. Plutonium by mass vs. by neutron measurement

While both the round 1 and round 2 aliquots were partitioned to the same dispensing precision, the small sizes of the aliquots in the round 1 measurements produced a larger spread in the plutonium content than found in round 2.

Since the round 2 samples were created by sequentially spooning aliquots from a jar, it is possible that heterogeneity was introduced during the partitioning process. The last sample had more fines and fewer large particles than the previous aliquots. Since the plutonium oxide is a fine powder, it could have been enriched in the final aliquot merely due to the absence of the larger particles, which do not contain plutonium. This interpretation would be a simple explanation as to why the neutron measurement of plutonium content of the last sample was higher than the others, since it actually contains more plutonium.

To explore this possibility, let us postulate that the ratio of total plutonium to total mass (r_T) remains unchanged, and that the ratio of plutonium to sample mass (r_i) is consistent for samples A-E but changed for sample F. Thus, we can write the plutonium balance as the equation

$$r_T m_T = r_{AE} m_{AE} + r_F m_F$$

where r_T is the total ratio, m_T is the total mass, r_{AE} is ratio for samples A-E, m_{AE} is the sum of the masses of samples A-E, r_F is the ratio for sample F, and m_F is the mass of sample F.

All variables except for r_{AE} and r_F, are known, so one additional relationship is required to determine them. Using least squares regression to minimize the difference between the calculated and measured plutonium content

$$w = \sum_{i=A}^{F} (r_i m_i - p_i)^2,$$

where p_i is the measured plutonium content for sample i, provides the necessary relationship.

The equation for r_{AE} is found to be

$$r_{AE} = \frac{\sum_{i=A}^{E} m_i p_i + m_{AE}(r_T m_T - p_F)}{\sum_{i=A}^{E} m_i^2 + (m_{AE})^2}.$$

The value of r_T was 0.101; solving for r_{AE} and r_F yields 0.0996 and 0.110, respectively. Using the values of r_{AE} and r_F to calculate new plutonium contents, we can adjust the plutonium content measured by mass. The adjusted values are also shown in Figure 4-1. Note the average does not change. The observed enrichment is striking; aliquot F has a 10% increase in plutonium content by mass over the base material.

To reduce self-separation and induced heterogeneity, a different partitioning technique, cone and split, was used to produce the round 3 aliquots. As can be seen in Figure 4-1, however, this technique did not succeed in reducing the heterogeneity compared to round 1 or round 2.

There was no significant change in the average of the measured plutonium values (indicative of the total plutonium amount) between rounds 2 and 3. This increases the confidence that the NMC measured plutonium results are consistent and represent the plutonium content of each aliquot. On this basis, the differences between the measured plutonium and plutonium by mass represent heterogeneity in the portioning rather than noise in the data.

Due to the observed heterogeneity in the partitioning, the masses determined by NDA are believed to be more accurate than the masses determined by weight for the individual diluted and poisoned samples. Biases on the neutron assay equipment are determined by comparing the sum of the weighted masses with the sum of the neutron-assayed masses.

4.2 Non-Destructive Measurements

Data analysis for the AWCC results was performed using the standard multiplicity equations.[16] The ring ratios are calculated by first subtracting the contribution from fission neutrons, as prescribed in the "dual energy passive multiplicity" algorithms in the *International Neutron Coincidence Counting (INCC) Software Users Manual*. Assay bias is calculated as the measured plutonium mass, divided by the expected plutonium mass, minus one.

Results for each of the four matrices are averaged and reported in Table 4-2. Total neutron rates are calculated based on the isotopic content and the plutonium mass as measured by the NMC. Uncertainties reported here are obtained by calculating the standard deviation of the mean within each matrix. Raw data are reported in Appendix A.

Table 4-2. Neutron multiplicity results for the pure, adulterated, and poisoned samples

	total neutron rate (n/s/g Pu)	alpha ratio	(α,n) ring ratio	multiplication	assay bias
pure oxide	113.3 ±0.49	0.811 ±0.007	1.409 ±0.003	1.012 ±0.001	0.0%
diluent added	133.5 ±0.47	1.153 ±0.007	1.415 ±0.002	1.003 ±0.001	-2.5%
diluent + coated B_4C	138.7 ±0.26	1.238 ±0.003	1.413 ±0.005	1.003 ±0.001	-3.2%
diluent + uncoated B_4C	177.3 ±1.15	1.856 ±0.017	1.397 ±0.006	1.004 ±0.000	-1.1%

First, note the difference between the pure and adulterated samples. As expected, the alpha ratio goes up due to other light elements in the adulterant. The increase in ring ratio is consistent with the lower-energy neutrons emitted by some low-Z elements. Multiplication drops dramatically due to the dilution of the plutonium in a much larger sample (with thirty times more volume). There is a detectable negative bias between the adulterated and pure samples. This bias is presumably due to the decrease in efficiency as the bulk material is moved away from the center of the AWCC chamber.

Next, the coated boron carbide results show an additional increase in alpha ratio. There are two potential explanations. The first is that the nickel coating is uneven, broken, or otherwise not sufficient to completely stop the alpha particles from reaching the boron. Second, vendor XRF analysis of the coated boron carbide indicates the presence of various low-Z impurities, such as magnesium and chlorine, which could contribute to the (α,n) rate if they are in the coating. However, boron has a significantly higher (α,n) cross section than any other element with the exception of beryllium.

Finally, the uncoated boron carbide results show an even larger increase in alpha ratio. The ring ratio is consistent with the higher-energy neutrons emitted in the boron reactions. The difference in the assay bias reported for the coated and uncoated boron carbide is likely due to heterogenous mixing, rather than a difference in performance by the AWCC. Averaged together, the assay bias is -2.2%, consistent with the diluent-only material. This suggests that the quantity added is not enough to significantly absorb the fission neutrons emitted by the material.

These results are predictive of an NMC behavior on a blend can (ten times larger) or a CCO (twenty times larger). The (α,n) production rate, whether from oxide, diluent, or boron carbide poison, will scale linearly with increased size. Multiplication due to induced fissions will not increase linearly, but this contribution to the total neutron rate is particularly small due to the dilution of the fissile material.

The consistency in assay bias between the poisoned and unpoisoned samples indicates that the boron will not reduce the efficiency of a larger counter. In addition, although the (α,n) rate is larger for the uncoated boron, this increase is not large enough to cause concern for multiplicity counting. Generally, alpha values greater than three indicate that the material will have significant loss of precision when measured by neutron multiplicity counting.

The neutron dose rate is expected to increase with the addition of boron, as well as with the adulterant alone. Dose rates were also measured by Radiological Protection (RP) personnel using a BF_3-based Thermo ASP-2e/NRD. Although the measured doses were consistent with the AWCC results, the rates were so low that the RP neutron rate was significantly noisy, and the results are not self-consistent. The observed data are shown in Table 4-3. An increase in the gamma dose rate was not observed, and HPGe measurements on the diluted and poisoned oxide did not show any additional prompt gamma-ray emissions.

Table 4-3. Neutron dose measured by Radiological Protection

	Uncoated B4C	Nickel coated B_4C
Background	0.2	
Before B4C addition	0.3	0.6
After B4C addition	0.3	1.2

All values in mRem/hr

4.2.1 Extrapolation to Other Material Types

The equations described previously can be used to predict the neutron production in materials with different isotopics or impurities. Recall that the (α,n) yields are given by

$$Y = \frac{\alpha \cdot F \cdot \nu}{A}.$$

The yields can now be calculated for the four matrices measured on the AWCC. These measured values for the pure, weapons-grade material are given in Table 4-4 below, along with the change in neutron rate as compared to pure oxide prior to dilution. Using these values, the change in rates can be estimated for other starting materials.

Table 4-4. Measured (α,n) yields and neutron rates for pure weapons-grade plutonium.

	alpha ratio	(α,n) yield ($n/10^6$ α)	total neutron rate (n/s/g)	compared to diluted oxide
pure oxide	0.811	0.0175	113.3	84.9%
diluent added	1.153	0.0249	133.5	100.0%
diluent + coated B_4C	1.238	0.0268	138.7	103.9%
diluent + uncoated B_4C	1.856	0.0401	177.3	132.8%

4.2.1.1 Pure, Fuel-Grade Plutonium

As a first case, assume the starting oxide were pure, fuel-grade plutonium, which has much higher alpha activity, spontaneous fission activity, and overall neutron dose rate. It is assumed that the measured yields are applicable for pure plutonium oxide of any isotopics. This assumption is valid because, to first order, the isotopes of plutonium and americium emit alpha particles at comparable energies, and thus interact in the oxygen and boron at approximately the same rate. However, the starting alpha ratio may be larger (perhaps around 1.2) for the fuel-grade material, due to increased ^{238}Pu and ^{241}Am.

In this case, the alpha ratio increases as more low-Z material is added to the matrix, at the same proportional rate as before. However, because of the increased alpha-particle emitters in the plutonium, the total neutron rate increases faster than in the case of the weapons grade material, as shown in Table 4-5.

Table 4-5. Predicted neutron rates for pure, fuel-grade plutonium.

	alpha ratio	(α,n) yield ($n/10^6\ \alpha$)	total neutron rate (n/s/g)	compared to diluted oxide
pure oxide	1.202	0.0175	521.6	81.3%
diluent added	1.709	0.0249	641.9	100.0%
diluent + coated B_4C	1.835	0.0268	671.7	104.6%
diluent + uncoated B_4C	2.752	0.0401	889.0	138.5%

4.2.1.2 Impure, Weapons-Grade Plutonium

As a second case, consider weapons-grade material with significant impurities such as magnesium or fluorine. For this material, the alpha ratio is already high (perhaps around α=10) due to the presence of low-Z impurities. Unlike pure, fuel-grade plutonium, this increased alpha ratio is not due to an increase in alpha-particle emitters, but rather an increase in the (α,n) yield. The addition of boron, as a potential neutron poison, does not increase the neutron rate as much as in the case of the pure material, because the additional yield is small compared to the impurities already present, as shown in Table 4-6.

Table 4-6. Predicted neutron rates for impure, weapons-grade plutonium.

	alpha ratio	(α,n) yield ($n/10^6\ \alpha$)	total neutron rate (n/s/g)	compared to diluted oxide
pure oxide	10.00	0.216	680.3	97.0%
diluent added	10.34	0.224	701.5	100.0%
diluent + coated B_4C	10.43	0.225	706.8	100.8%
diluent + uncoated B_4C	11.05	0.239	745.0	106.2%

5.0 Conclusions

Three rounds of measurements were performed to evaluate the effect of boron carbide as a neutron poison in the adulterant composition. Multiple batches of adulterated material were created with approximately one tenth of the total material of an actual dilute and dispose container. The neutron dose rates of these batches were measured as pure Pu oxide, adulterated using the NPT recipe without boron carbide, and adulterated using the complete NPT recipe.

Both uncoated and nickel-coated boron carbide particles were added to separate batches of adulterated material for neutron measurements. The thickness of the nickel coating was chosen to be greater than the range of alpha particles emitted by plutonium or americium.

Six aliquots were made for each round of measurements, the final batch containing three samples with uncoated boron carbide and three with nickel-coated boron carbide. The addition of the uncoated boron carbide increased the neutron rate by almost 33% over the material with diluent only. In comparison, the nickel-coated boron carbide increased the neutron rate by only 4%. Pure, weapons-grade material was used in this study; however, pure fuel-grade material with a higher quantity of alpha-emitters will experience an even greater increase in neutron count rate associated with (α,n) neutrons.

The addition of the uncoated boron carbide corresponds to an increase in the alpha ratio of 0.7 (from 1.153 to 1.856) for the weapons-grade material. For the fuel-grade material, the addition of boron carbide is predicted to increase the alpha by approximately 1.0 (from 1.709 to 2.757). Although these increases will affect the precision of neutron-based NDA measurements, values less than 3 are still able to be measured with relatively little difficulty. In addition, assay biases were consistent between the diluted and the poisoned samples. This indicates that this quantity of boron carbide did not negatively impact the accuracy of the NDA measurement.

6.0 Recommendations, Path Forward or Future Work

There are several recommendations for future work. Some will address immediate questions raised in this work, while others would support potential future strategies.

6.1 Measure neutron production with additional boron carbide

While the ORNL NCSA recommends 50 grams of boron carbide to 380 grams of plutonium, KAC may choose to use additional boron carbide as a conservative measure. This work suggests that neutron production should be linear with the addition of boron carbide. With the adulterated NPT1 still available, the effect of additional boron carbide could readily be verified.

6.2 Measure neutron production with higher purity boron carbide

The data supplied by the vendor of the nickel-coated boron carbide suggests there may be significant amounts of low Z impurities, which would increase the neutron generation from (α,n) reactions. Smaller scale (1 aliquot) measurements could be performed on samples where higher purity boron carbide is used to produce the nickel-coated boron carbide. The measurement could also be made with the higher-purity, plain boron carbide.

These measurements would require the formation of additional adulterated material, and additional procurements. While the labor and other resources required would be relatively minor, time for the procurement of materials may become significant.

6.3 Scanning Electron Microscope Analysis of the nickel-coated boron carbide

The material with the nickel-coated boron carbide has a neutron dose rate higher than the adulterated samples with no boron carbide. While this may be due to contaminants in the nickel coated boron carbide, it may also be caused by imperfections in the nickel coating, such as thin spots or gaps. Analysis of the nickel-coated boron carbide would reveal if flaws are present in the nickel coating. This knowledge may lead to simple changes in the production of the nickel coating which produce fewer imperfections, thereby lowering the neutron dose rate.

6.4 Consider alternatives for boron carbide

Other neutron poisons like cadmium sulfide are available at reasonable cost and may be considered if future changes to the adulterant formulation are warranted. Cadmium has a high cross section for absorbing thermal neutrons but does not have a significant (α,n) cross section. Using cadmium as a neutron absorber may produce a neutron dose similar to that of the material prior to the addition of any boron carbide and may even be lower due to the displacement of low-Z elements in the diluent.

One concern associated with using cadmium is potentially having the final material qualify as hazardous waste under the Resource Conservation and Recovery Act (RCRA). Determination of RCRA hazardous waste material is based on the results of a Toxicity Characteristic Leaching Procedure (TCLP) test. Cadmium sulfide is a commonly used pigment, and readily available in forms that will pass a TCLP test. Cadmium sulfide is generally considered highly insoluble and shows excellent stability.

If it is desired to develop cadmium sulfide as an eventual replacement for boron carbide, SRNL can substitute cadmium sulfide for plain boron carbide in a single aliquot test. A TCLP test on the diluting material with the cadmium sulfide can also be performed to verify the resulting material will not be hazardous under RCRA.

7.0 References

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Appendix A. Neutron Multiplicity Data

Table A-1. Shift Register Results as Reported by INCC

Sample	Singles (s^{-1})	Doubles (s^{-1})	Triples (s^{-1})	Scalar 1 (s^{-1})	Scalar 2 (s^{-1})
A-pure	558.517 ±0.540	51.333 ±0.185	4.865 ±0.069	331.834 ±0.320	226.660 ±0.308
B-pure	563.285 ±0.507	51.994 ±0.211	4.973 ±0.082	334.902 ±0.327	228.365 ±0.283
C-pure	547.943 ±0.558	50.658 ±0.195	4.990 ±0.085	325.616 ±0.360	222.306 ±0.296
D-pure	567.427 ±0.606	53.213 ±0.190	5.303 ±0.084	337.274 ±0.424	230.127 ±0.315
E-pure	550.767 ±0.505	49.185 ±0.163	4.507 ±0.078	326.591 ±0.281	224.143 ±0.307
F-pure	567.661 ±0.484	52.670 ±0.171	5.323 ±0.067	337.219 ±0.280	230.416 ±0.276
A-diluent	625.186 ±0.562	46.084 ±0.159	3.935 ±0.078	370.995 ±0.326	254.144 ±0.348
B-diluent	630.324 ±0.628	46.750 ±0.131	4.150 ±0.071	373.464 ±0.363	256.824 ±0.381
C-diluent	626.433 ±0.628	46.320 ±0.198	4.013 ±0.072	371.090 ±0.419	255.305 ±0.339
D-diluent	640.317 ±0.550	47.353 ±0.185	4.231 ±0.078	380.040 ±0.340	260.226 ±0.327
E-diluent	642.488 ±0.603	47.885 ±0.157	4.261 ±0.073	381.036 ±0.306	261.403 ±0.385
F1-diluent*	685.400 ±0.529	51.797 ±0.183	4.572 ±0.085	406.373 ±0.336	278.968 ±0.323
F2-diluent*	684.785 ±0.440	51.584 ±0.172	4.695 ±0.080	406.659 ±0.294	278.067 ±0.259
A-coated	738.233 ±0.627	52.552 ±0.206	4.604 ±0.317	437.984 ±0.317	300.158 ±0.340
B-coated	658.073 ±0.749	46.759 ±0.175	4.048 ±0.056	389.433 ±0.391	268.589 ±0.375
C-coated	718.855 ±0.608	51.574 ±0.182	4.586 ±0.074	426.016 ±0.347	292.767 ±0.284
D-uncoated	783.834 ±0.851	44.049 ±0.155	3.926 ±0.077	461.534 ±0.392	322.212 ±0.502
E-uncoated	847.642 ±0.900	48.170 ±0.177	4.241 ±0.078	500.609 ±0.516	346.923 ±0.416
F-uncoated	785.126 ±0.662	44.242 ±0.155	3.958 ±0.074	462.036 ±0.376	322.992 ±0.337

* F-diluent was measured twice, to ensure that the observed plutonium segregation between samples was not simply due to measurement error.

Table A-2. Multiplicity Analysis Results

Sample	Declared Pu mass (g)	Assay Pu mass (g)	Assay bias	Multipli-cation	alpha ratio	total n/s/g	Ring Ratio
A-pure	16.70	16.83	0.80%	1.010	0.795	112.1	1.409
B-pure	16.80	16.96	0.98%	1.011	0.795	112.2	1.415
C-pure	16.45	16.24	-1.26%	1.014	0.818	114.0	1.412
D-pure	16.81	16.96	0.86%	1.015	0.801	113.1	1.413
E-pure	16.47	16.43	-0.25%	1.007	0.820	113.3	1.396
F-pure	16.82	16.63	-1.14%	1.016	0.835	115.3	1.410
A-diluent	16.67	15.98	-4.13%	1.000	1.137	132.2	1.418
B-diluent	16.65	15.85	-4.79%	1.004	1.164	134.3	1.409
C-diluent	16.66	15.93	-4.37%	1.002	1.145	132.8	1.407
D-diluent	16.65	16.00	-3.93%	1.004	1.178	135.2	1.421
E-diluent	16.66	16.22	-2.66%	1.004	1.156	133.9	1.415
F1-diluent	16.57	17.63	6.38%	1.003	1.118	131.4	1.412
F2-diluent	16.57	17.24	4.03%	1.006	1.158	134.2	1.424
A-coated	17.48	17.95	2.70%	1.003	1.241	138.9	1.421
B-coated	17.09	16.09	-5.88%	1.002	1.232	138.2	1.404
C-coated	18.61	17.46	-6.16%	1.004	1.240	139.1	1.413
D-uncoated	15.57	14.84	-4.68%	1.004	1.875	178.4	1.393
E-uncoated	15.41	16.37	6.21%	1.003	1.822	175.0	1.408
F-uncoated	15.59	14.87	-4.62%	1.004	1.873	178.4	1.390

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