*Production of Working Reference Materials
for the Capability Evaluation Project*

*Phillip D. Noll, Jr.
Robert S. Marshall*

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Contents

ABSTRACT	1
I. INTRODUCTION	2
II. STARTING MATERIALS	3
A. Plutonium Dioxide	3
1. Preparation	3
a. Blending and Calcining	3
b. Pulverization	3
c. Screening	3
d. Documentation and Records	3
2. Sampling	3
a. Sampling Method	4
b. Number of Samples	4
c. Packaging and Labeling	4
d. Documentation and Records	4
3. Characterization	4
a. Particle Size Distribution	4
b. Plutonium Assay	5
c. Plutonium Isotopic Distribution and ²⁴¹ Am Analysis	6
d. DC Arc Emission Spectroscopy Determination of Impurities	8
e. Inductively-Coupled Plasma Mass Spectrometry Determination of Impurities	8
f. Radiometric Analyses	10
g. Anionic Impurity Analyses	10
B. Americium Dioxide	11
1. Preparation	11
a. Initial Preparation	11
b. Blending	11
c. Sampling	12
2. Characterization	12
a. Sampling for Analytical Chemistry	12
b. Particle Size Distribution	12
c. Americium Assay, Pu and U Content, and Isotopic Distribution	13
d. Inductively-Coupled Plasma Mass Spectrometry Determination of Impurities	15
e. Radiometric Analysis	17
f. Loss on Ignition	18
g. Anionic Impurity Analyses	18
C. Depleted Uranium Oxide	19
1. Preparation	19
a. Initial Preparation	19
b. Packaging	19
c. Blending	19

2. Characterization	19
a. Sampling for Analytical Chemistry	19
b. Particle Size Distribution	20
c. Uranium Assay and Isotopic Distribution	20
d. ICP-AES Determination of Impurities	21
e. ICP-MS Determination of Impurities	22
f. Radiometric Analysis	23
g. Anionic Impurity Analyses	24
D. Diatomaceous Earth	24
1. Preparation	24
a. Sampling	25
2. Characterization	25
a. Glow Discharge Mass Spectrometry	25
b. Loss on Ignition	26
III. BLENDING	26
A. Blending Parameters and Blend Evaluations	27
1. Summary of Initial Tests for PDP	27
2. Alpha-Neutron Yield of AmO ₂ -DE Blends	27
3. Gas-Generation Evaluation of the IAP WRMs	27
B. IAP WRM Blend Preparation	27
1. Nuclear Material and DE Addition	28
2. Blending	28
3. Documentation and Reporting	28
C. DU WRM Blend Preparation	28
1. Nuclear Material and DE Addition	28
2. Blending	28
3. Documentation and Reporting	29
IV. STAINLESS STEEL ASSEMBLIES	29
A. Fabrication	29
B. Receiving Inspection	29
V. STAINLESS STEEL CYLINDER FILLING	30
A. Cylinder Preparation	30
B. Cylinder Filling and Packing	30
C. Frit Insertion	32
D. Waste Materials Measurement	32
1. IAP WRMs	32
2. DU WRMs	32
E. Documentation and Records	33
VI. INERTING	33
A. IAP WRMs	33
B. DU WRMs	33

VII. WELDING	34
A. Welding Setup	34
1. IAP Inner Stainless Steel Tube Welds	34
2. IAP Outer Stainless Steel Tube Welds	34
3. DU Stainless Steel Tube Welds	34
B. Establishing Weld Parameters	34
C. Welding Qualification	35
D. Documentation and Records	35
VIII. DECONTAMINATION OF THE IAP INNER CYLINDERS	36
IX. LEAK CHECKING	36
A. Helium Leak Check	36
B. Documentation and Records	36
X. FINAL INSPECTION	36
A. Alpha Contamination Checks	36
B. Dimensional and Identification Checks	37
C. Nuclear Material Uniformity Checks and Verification	37
1. IAP WRMs	37
a. Uniformity Check	37
b. Verification	38
2. DU WRMs	39
a. Uniformity Check	39
b. Verification	39
XI. PACKAGING AND SHIPPING	40
XII. WRM CERTIFICATION	41
A. Nuclide Mass and Alpha Activity Calculations	41
1. Pu Mass	42
2. Am Mass	42
3. DU Mass	42
4. Alpha Activity	42
B. Uncertainty Data and Calculations	43
1. Weighing Uncertainty	43
2. Assay Uncertainty	45
a. Pu Oxide	45
b. Am Oxide	45
c. U Oxide	46
3. Isotopic Uncertainty	46
a. Pu Oxide	46
b. U Oxide	46
4. Transfer and Hold-Up Loss	47
5. Alpha Activity Uncertainty	47
6. Combined Uncertainty for Mass and Alpha Activity	48
C. WRM Certification	48
XIII. REFERENCES	49

Appendices

- A. CEP SOW
- B. Pu Preparation at TA-55
- C. Final Chemical Analysis Report
- D. Am Preparation at TA-55
- E. AmO₂ Particle Size Distribution
- F. UO₂ Particle Size Distribution
- G. Am + DE Neutron Shuffler Measurements
- H. Gas-Generation Report
- I. Final Weights (IAP)
- J. Final Weights (DU)
- K. Hold-up Measurements
- L. IAP Uniformity/Verification Measurements
- M. DU Uniformity/Verification Measurements
- N. Shipping Information
- O. Uncertainty Calculations on Mass and Alpha Activity
- P. Alpha Activity for Each WRM through 2001
- Q. Weighing Data
- R. Certificates for All Standards Used
- S. NBS 948 and NBS U005 Standard Data
- T. Description of Hold-up Measurements and Uncertainty
- U. Description of Uncertainty Propagation
- V. WRM Certificates

List of Tables

Table 1.	Pu Assay Results, Wt. % Pu	5
Table 2.	Summary of Pu Isotopic Distribution As Weight Percent	7
Table 3.	²⁴¹ Am/Pu Analysis Results	7
Table 4.	Emission Spectroscopic Analysis Results in Micrograms Impurity per Gram PuO ₂ ...	8
Table 5.	ICP-MS Analysis Results in Micrograms Impurity per Gram PuO ₂	9
Table 6.	Gamma Radiometric Analysis of PuO ₂ for ²⁴¹ Am	10
Table 7.	Ion Chromatographic Analyses of PuO ₂ for Cl and F	11
Table 8.	Results of the ²⁴¹ Am Assay of the AmO ₂	13
Table 9.	Results of the Pu Assay of the AmO ₂	14
Table 10.	Summary of Pu Isotopic Distribution of the AmO ₂ As Weight Percent	14
Table 11.	Results of the U Assay of the AmO ₂	14
Table 12.	Summary of U Isotopic Distribution of the AmO ₂ As Weight Percent	14
Table 13.	ICP-MS Analysis Results in Micrograms Impurity per Gram AmO ₂	16
Table 14.	Liquid Scintillation and Gamma Radiometric Analysis of AmO ₂ for ²⁴¹ Am	17
Table 15.	Ion Chromatographic Analyses of AmO ₂ for Cl and F	18
Table 16.	Summary of U Isotopic Distribution As Weight Percent	20
Table 17.	U Assay Results	21
Table 18.	ICP-AES Analysis Results in Micrograms Impurity per Gram UO ₂	22
Table 19.	ICP-MS Analysis Results in Micrograms Impurity per Gram UO ₂	23
Table 20.	Ion Selective Electrode Analyses of UO ₂ for Cl and F	24
Table 21.	GDMS Analysis Results on Kieselguhr Diatomaceous Earth	25
Table 22.	Uniformity Measurement Data Summary	37
Table 23.	Verification Measurement Data Summary	38
Table 24.	Uniformity Measurement Data Summary for the DU WRMs	40
Table 25.	Verification Measurement Data Summary for DU WRMs	40
Table 26.	Atomic Masses, Half-Lives, and Alpha Activities of the Isotopes Present in the CEP WRMs	43
Table 27.	Precision on Standard-Control Weight Weighings	44
Table 28.	Upper Bound Error Estimates for Mass Spectrometric Pu Isotopic Distribution	46
Table 29.	Upper Bound Error Estimates for Mass Spectrometric U Isotopic Distribution	47
Table 30.	Components Included in Uncertainty Calculations	48

List of Figures

Figure 1.	Turbula Blender with Blend Bottle	26
Figure 2.	Packaged Assemblies with Inspection Forms	29
Figure 3.	Labeled Assembly with Endcaps	30
Figure 4.	Cylinder Ready for Filling	31
Figure 5.	Fully Loaded and Tap-Packed	31
Figure 6.	Packaged Wasted and Filled Cylinder Ready for Bagout	32
Figure 7.	The Six DU WRMs in the Desiccator for Inerting	33
Figure 8.	In-Glovebox Welding Electrode	34
Figure 9.	Completed IAP WRM after Welding and Inspection	35
Figure 10.	Inner Cylinder-Endcap Weld Cross-section	35
Figure 11.	IAP WRM Undergoing Uniformity/Verification Measurement	38
Figure 12.	DU WRM (In Gray Can) Undergoing Uniformity/Verification Measurement on a Segmented Gamma Scanner	39
Figure 13.	Bubble Wrapping Two WRMs	41
Figure 14.	Inserting WRMs into a 2R Cylinder for Shipment	41

PRODUCTION OF WORKING REFERENCE MATERIALS FOR THE CAPABILITY EVALUATION PROJECT

Phillip D. Noll, Jr. and Robert S. Marshall

ABSTRACT

Nondestructive waste assay (NDA) methods are employed to determine the mass and activity of waste-entrained radionuclides as part of the National TRU (Trans-Uranic) Waste Characterization Program. In support of this program the Idaho National Engineering and Environmental Laboratory Mixed Waste Focus Area developed a plan to acquire capability/performance data on systems proposed for NDA purposes. The Capability Evaluation Project (CEP) was designed to evaluate the NDA systems of commercial contractors by subjecting all participants to identical tests involving 55 gallon drum surrogates containing known quantities and distributions of radioactive materials in the form of sealed-source standards, referred to as working reference materials (WRMs). Although numerous Pu WRMs already exist, the CEP WRM set allows for the evaluation of the capability and performance of systems with respect to waste types/configurations which contain increased amounts of ^{241}Am relative to weapons grade Pu, waste that is dominantly ^{241}Am , as well as wastes containing various proportions of depleted uranium. The CEP WRMs consist of a special mixture of $\text{PuO}_2/\text{AmO}_2$ (IAP) and diatomaceous earth (DE) or depleted uranium (DU) oxide and DE and were fabricated at Los Alamos National Laboratory. The IAP WRMs are contained inside a pair of welded inner and outer stainless steel containers. The DU WRMs are singly contained within a stainless steel container equivalent to the outer container of the IAP standards. This report gives a general overview and discussion relating to the production and certification of the CEP WRMs.

I. INTRODUCTION

This Nondestructive Waste Assay (NDA) Capability Evaluation Project (CEP) Production Plan addresses the key elements in the production of 16 Working Reference Materials (WRMs) as defined in the LITCO Statement of Work (SOW, see Appendix A). Guidelines in the ASTM Standard Guide C1128-89 were used to ensure that this plan addresses all the subjects appropriate to a production plan. This document serves two purposes: first, it defines all the operations required to meet the SOW objectives, task requirements, and technical specifications as envisioned prior to the start of WRM production; and second, it provides a record of the actual production and certification of the WRMs after they were produced. The SOW specified the WRM nuclear material masses, traceability to Certified Reference Materials, physical, chemical, and isotopic ranges, and total uncertainty. Additionally, specifications addressed the matrix material, matrix uniformity, containment, verification measurements, WRM shipping, and records disposition. The sixteen WRMs fabricated for the CEP project are analogous to those fabricated for the Performance Demonstration Program (PDP) Phase I (See LA-UR-96-2277, "NDA PDP Working Reference Materials Production Plan-Phase I") and PDP Phase II WRMs with the following exceptions: 1) eight of the WRMs contain both AmO_2 and PuO_2 in an Increased Am/Pu (IAP) ratio; 2) two of the WRMs contain Am only (these two are also considered "IAP" WRMs); 3) six of the WRMs contain Depleted Uranium oxide (UO_2); and 4) the six DU WRMs are encapsulated within a single layer of stainless steel (the PDP and IAP WRMs are encased in two layers of stainless steel). Since the fabrication of the CEP WRMs is nearly identical to that of previous PDP WRMs, some of the documentation associated with the production of the CEP WRMs was originally created during the production of the PDP WRMs. This being the case, there are numerous references within this document to data and/or documentation that is located within the PDP Phase I files and/or the PDP Phase I Production Plan (LA-UR-96-2277) rather than contained within the CEP files. Even though WRMs meeting the specifications of the SOW had been produced before (PDP Phase I, II), the SOW was continually updated during the course of WRM production and certification. Throughout the WRM production, the production plan was also updated to provide a complete and accurate description of the production and certification processes used to meet the SOW objectives.

This production plan was written to provide a general description of processes, steps, files, quality control, and certification measures. This plan identifies the files where detailed procedures, data, quality control and certification documentation, and forms are retained. The first several sections of this production plan describe the production and distribution of the WRMs, including starting materials, blending, loading, packaging, and shipping. The final sections address WRM certification, including WRM plutonium, americium, and depleted uranium content calculations, uncertainties and propagation of bias and precision, components, and the WRM certification certificates. Appendices for this production plan include extensive, detailed data that is identified and often summarized in the text. Four other documents complement the production plan: a quality assurance plan defining those quality assurance measures implemented to ensure the WRMs meet all the quality objectives stated in the SOW; a management plan which defines the management methods and tools used to ensure the WRMs are produced in a cost effective manner while meeting all milestones; a document control/records management plan that defines those measures taken to ensure that all procedures, records, and data required to document the production and demonstrate the pedigree of the WRMs are properly generated, documented, retained, and protected until delivered to LITCO; and finally, PERT Charts showing all tasks (with start and finish dates) that facilitate the scheduling and management of the project.

II. STARTING MATERIALS

A. Plutonium Dioxide

Specifications for the plutonium dioxide (PuO_2) to be used for the CEP WRM production are listed in sections 5.6-5.8 of the SOW. Characteristics of the PuO_2 used for the WRM production are typical of material used in the fabrication of nuclear weapons. Since waste materials currently being measured were typically generated on the order of ten to twenty years ago, the 0.085 - 0.25 weight percent ^{241}Am specified in the CEP WRM Production SOW (section 5.6) corresponds to a high purity PuO_2 starting material purified in 1986 that now contains about ten years of daughter product in-growth. The PuO_2 batch initially selected and used for the production of NDA PDP Phase I WRMs (LANL PEOR3258) was also used for the NDA CEP WRM production. This material (see LA-UR-96-2277 "NDA PDP Working Reference Materials Production Plan-Phase One") was initially prepared by the anion exchange purification of a plutonium nitrate solution, followed by precipitation as plutonium oxalate. The resultant Pu oxalate was calcined to PuO_2 and packaged in hermetically sealed food-pack cans. The 1988 Pu assay, plutonium isotopic, and impurity analyses records on this material indicates a suitable starting material. Approximately 120 grams was prepared, sampled, and characterized as described below.

1. Preparation. Appendix B contains a detailed report describing all preparation steps taken at the Los Alamos National Laboratory (LANL) TA-55 Plutonium Facility on the PuO_2 used for the WRM production. These steps are summarized as follows:

a. Blending and Calcining. The PuO_2 batch was calcined during initial Pu oxalate-to- PuO_2 production; however, the calcination was not sufficiently rigorous to generate a stable (low surface area) material. Therefore, the batch was further calcined between 900°C and 960°C for nine hours. Prior to calcining, the PuO_2 was blended in a V blender for approximately 30 minutes and then screened through a #40 mesh sieve to eliminate any coarse PuO_2 particles. The blended PuO_2 was placed in a fused silica furnace boat and calcined. Furnace temperature history and calcining time was recorded, and is detailed in Appendix B.

b. Pulverization. The PuO_2 was a uniform, finely divided powder when initially produced. The PuO_2 was examined after calcining to determine whether the material homogeneity might be enhanced by grinding. The material appeared highly uniform; hence, no grinding was required.

c. Screening. The calcined PuO_2 was screened through a #80 mesh sieve. All the material passed through the sieve.

d. Documentation and Records. Records of the calcining time and temperature history, Loss-On-Ignition (LOI) analysis, and particle size analysis were generated and initialed upon completion of the calcining operation. The original report on the calcining, final screening, LOI, and particle size analyses are in the NDA PDP Phase I WRM files.

2. Sampling. Quality assurance and quality control are incorporated into the procedures used to characterize the PuO_2 . The accuracy and precision of these methods have been established via ongoing quality control programs, and maintained through the routine practice of

analyzing control standards along with samples. Hence, multiple sampling of the selected PuO₂ batch was not required to establish analytical method precision. However, multiple sampling was performed to verify batch homogeneity.

a. Sampling Method. The entire 120 gram batch of PuO₂ was placed in a container of sufficient diameter to allow removal of five samples from different areas throughout the volume of PuO₂. A spatula of suitable size was used to withdraw samples of the desired mass. The spatula was inserted into the PuO₂ to obtain samples from different vertical and horizontal regions of the PuO₂ batch.

b. Number of Samples. The number of samples selected for the measurement of each attribute was based on SOW quality objectives, and on the demonstrated performance of the instrument and method used to measure the attribute. Statistical considerations were used to assure that the samples taken for particle size determination, Pu assay, Pu isotopic distribution, Am Assay, and impurity analyses provided the desired level of confidence in the data and its subsequent application and propagation.

Three samples were taken for each of the following analysis methods: particle size distribution, DC Arc Emission Spectroscopy of metallic impurities, Inductively-Coupled Plasma (ICP)-Mass Spectrometry (MS) of metallic impurities, and Ion Chromatography of anionic impurities. Five samples were taken for each of the following determinations: Pu Assay, Pu isotopic distribution, and ²⁴¹Am assay (sample dissolution for Pu assay provides aliquots for Pu isotopic distribution and ²⁴¹Am assay). Samples taken for Pu Assay and for blend preparation were taken from the batch only after the batch equilibrated in the glovebox inert-gas atmosphere for at least 24 hours. This precaution assured that the batch PuO₂ samples taken for critical analyses and for blend preparation had exactly the same moisture content.

c. Packaging and Labeling. All samples were promptly sealed in glass or plastic screw-cap vials after removal from the PuO₂ batch and before removal from the inert-gas atmosphere glovebox. The vials removed from the sample distribution room were clearly labeled with bar-coded labels containing sequential numbers that uniquely defined each individual sample. Sample tracking was provided by the NMT-1 Analytical Laboratory Information Management System (LIMS).

d. Documentation and Records. Records were made showing sample identification, sample mass, date, and requested analyses. These records are retained in the NDA PDP Phase I WRM file.

3. Characterization.

a. Particle Size Distribution.

Method. A LAB-TEC 1000 Particle Size Analyzer was used to determine the sample particle size distribution. This instrument is a scanning laser microscope that measures particles suspended in a liquid medium. The procedure used for particle size determination is NMT-3-281-MRD-R00. The particle count fraction and mass fraction were determined for particle size distributions with resolution of < 1 micron in 28 increments over the 0.5 to 250 micron range.

Calibration. Quartz certified reference materials BCR Nos. 67 and 69 were used for the particle size analyzer calibration and performance checks. The instrument laser, detectors, and focal point are fixed in position and cannot be manipulated or adjusted. This very stable instrument is checked using the BCR quartz certified reference materials on a biweekly basis. The performance check was performed on the same day as, and prior to, the PuO₂ particle size determination.

Documentation and Reporting. The particle size distribution was a technical **HOLD POINT**. The particle size distribution data was reviewed by the NDA CEP Technical Team prior to using the PuO₂ for preparing the WRMs. The Spherical Equivalent Mean Diameter of the three samples ranged from 32.5 to 34.4 microns. On a mass basis, the mean particle size was 125 microns for all three samples. Tables showing the detailed particle size data on both a particle count and mass basis can be found in the PDP Phase I file and in LA-UR-96-2277, "NDA PDP Working Reference Materials Production Plan-Phase I." A copy of procedure NMT-3 281-MRD-R00 and a brief description of the instrument operation and calibration are retained in the NDA PDP Phase I WRM file. The particle size distribution determined for each sample and the instrument calibration control data are retained in the NDA PDP Phase I WRM file.

b. Plutonium Assay.

Dissolution. The five sample aliquots taken for Pu assay were independently dissolved. Each sample aliquot was weighed to the nearest 0.01 milligram and the weight recorded. The dissolution method was ASTM C1168-90 "Standard Practice for Preparation and Dissolution of Plutonium Materials for Analyses," which required a sealed reflux tube dissolution incorporating a mixture of HCl, HNO₃, and HF under pressure to ensure complete dissolution.

Solution Preparation. After dissolution, each aliquot was quantitatively transferred to a tarred dispensing bottle. Multiple rinses of the dissolution vessel were performed to ensure quantitative transfer. After transfer, the dispensing bottle was weighed to the nearest 0.1 milligram, and the net weight of the solution was recorded. The solution was then thoroughly mixed by swirling and shaking the dispensing bottle for at least 30 seconds.

Coulometric Titration. Two aliquots of each of the five samples were weight dispensed from the dispensing bottle for coulometric assay as described in procedure ANC125, "Controlled-Potential Coulometric Determination of Plutonium in Sulfuric Acid Electrolyte." The average analysis results for the five samples are summarized in Table 1.

Standards and Calibration. As described in procedure ANC125, "Controlled-Potential Coulometric Determination of Plutonium in Sulfuric Acid Electrolyte" the coulometric titrator was calibrated with NBS Standard Reference Material (SRM) #949f or NBL Certified Reference Material (CRM) 126 (see Appendix R), which provides SRM traceability for sample analysis. Further, titration of the sample aliquots were preceded and followed by titrations of aliquots of a well characterized, high purity PuO₂ control material. The analyses of the control material

Table 1. Pu Assay Results, Wt. % Pu.					
Sample	1	2	3	4	5
Wt. % Pu	87.8317	87.8466	87.7717	87.7817	87.7867
Ave. % Pu: 87.8036				Effective: 6/1/97	

aliquots were recorded and used as quality control samples to determine the analysis precision on the five samples. The control material assays fell within the 0.5%, three relative standard deviation, coulometric assay control limit. The historical precision of the method is 0.07% at one relative standard deviation. The overall method relative random uncertainty is 0.08%. Inter-laboratory studies have shown that the method has no significant bias. The mean replicate assay result (87.804 % Pu) for the five samples had a one relative standard deviation precision (RSD) of 0.038% on 6/1/97. The mean assay values for all five samples fell within the one standard deviation range of all five samples; hence, the batch homogeneity can be bounded by a RSD of 0.038%.

Documentation and Reporting. The sample aliquots' weights, dissolution date, net solution weights, coulometric assay results and dates, certified reference material aliquot assay results, and operator and supervisor initials are recorded in formal LANL notebooks with appropriate page copies introduced into NDA PDP Phase I WRM files. The results of the replicate assays for the five samples are summarized in Table 1 and presented in Appendix C. These data were a technical **HOLD POINT** and were reviewed by the NDA CEP Technical Team prior to preparing the WRM blends.

c. Plutonium Isotopic Distribution and ^{241}Am Analysis. The same sample preparation and mass spectrometer operations were used for both Pu isotopic distribution and ^{241}Am analysis. The sample preparation and mass spectrometer operations are described in LANL procedures ANC 131, "U & Pu Isotopic Distribution and Assay by Isotope Dilution Mass Spectrometry," ANC 1312, "Operation of the VG-2 Mass Spectrometer," and ANC 130, "Purification of Plutonium for Mass Spectrometric Analysis by Ion Exchange Separation."

Samples. Aliquots of the five solutions prepared for Pu assay (see section II.A.3.B., "Plutonium Assay") were used for the Pu isotopic distribution and for the ^{241}Am assay.

Sample Preparation. For the Pu isotopic distribution determinations, aliquots of the dissolved samples were purified by ion exchange chromatography to remove interfering uranium isotopes. The ^{241}Am analysis was performed by Isotope Dilution Mass Spectrometry (IDMS), and a weight aliquot of each sample was spiked with a known quantity of ^{243}Am (spike). Using ion exchange chromatography, the Pu fraction and Am fraction were separated, and an aliquot of the Am fraction was mounted on filaments for the $^{241}\text{Am}/^{243}\text{Am}$ ratio determination.

Pu Isotopic Distribution Analysis. The Pu fraction filaments containing approximately 20 ng of Pu were mounted in the VG-354 instrument (a.k.a. VG-2). This instrument is equipped with a multicollector (5) Faraday system which permits the simultaneous collection of up to five isotopes. The entire Pu aliquot is evaporated (Total Evaporation Mass Spectrometry), and the subsequent ion signal is collected, thereby yielding a very high precision, high accuracy analysis (see Ref. 2). The relative gain of the five measuring channels was determined at the start of each analysis with a built-in constant current source. The integrated intensity from each isotope was proportional to the abundance of that isotope. The results of these analyses are summarized in Table 2.

^{241}Am Assay. The Am fraction filaments were mounted in the VG-354 Mass Spectrometer and processed as described in II.A.3.C., above. The $^{241}\text{Am}/^{243}\text{Am}$ mass ratio was measured and the ^{241}Am concentration in the dissolved sample was calculated using the $^{241}\text{Am}/^{243}\text{Am}$ ratio and the ^{243}Am concentration of the spike. Since the Pu concentration in the dissolved sample was known from the Pu assay of the same solution, the $^{241}\text{Am}/\text{Pu}$ mass ratio could be calculated. The $^{241}\text{Am}/\text{Pu}$ assay and g $^{241}\text{Am}/\text{g PuO}_2$ results are summarized in Table 3.

Table 2. Summary of Pu Isotopic Distribution As Weight Percent.*						
Sample	²³⁸ Pu	²³⁹ Pu	²⁴⁰ Pu	²⁴¹ Pu	²⁴² Pu	²⁴⁴ Pu
1	0.0148	93.7806	5.9448	0.2037	0.0561	0.0000
2	0.0138	93.7821	5.9443	0.2040	0.0558	0.0000
3	0.0147	93.7812	5.9446	0.2037	0.0558	0.0000
4	0.0141	93.7812	5.9451	0.2036	0.0560	0.0000
5	0.0141	93.7804	5.9455	0.2038	0.0562	0.0000
Average	0.0143	93.7811	5.9448	0.2038	0.0560	0.0000
* Effective: 6/1/97						

Table 3. ²⁴¹ Am/Pu Analysis Results.*					
Sample	1	2	3	4	5
μg ²⁴¹ Am/g PuO ₂	938.9	939.5	938.8	939.1	939.0
μg ²⁴¹ Am/g Pu	1069.0	1069.5	1069.6	1069.8	1069.7
Mean μg ²⁴¹ Am/ g PuO ₂ : 939.1* One standard deviation: 0.27					
Effective: 6/1/97 Mean μg ²⁴¹ Am/ g Pu: 1069.5					

Standards and Calibration. The Mass Spectrometer is calibrated using NBS SRM-948 (see Appendix R) aliquots run with every batch of samples receiving Pu isotopic distribution and ²⁴¹Am assay analysis. These data have formed the basis for control charts and were used to assure the method is meeting performance criteria, and to determine the method's historical performance. Historical quality check (QC) data demonstrates that this method (sample preparation and instrument performance) is superior to the manufacture's stated precision of 0.05% at one standard deviation.

Documentation and Reporting. The SRM and sample replicate Pu isotopic distribution (both as atomic percent and weight percent) and ²⁴¹Am assay results, analysis dates, SRM aliquot, and operator and supervisor initials are recorded in formal LANL notebooks with copies introduced into NDA PDP Phase I WRM files. The Pu isotopic distribution results and ²⁴¹Am/Pu ratio results for the five samples are summarized in Tables 2 and 3 above and are contained in the "Final Chemical Analysis Report," included as Appendix C. Copies of procedures ANC 130, ANC 131, ANC 1312 are in the NDA CEP WRM file. The ²⁴¹Am/Pu ratio and Pu isotopic distribution results were a technical **HOLD POINT** and were reviewed by the NDA CEP Technical Team prior to using the PuO₂ to prepare AmO₂-PuO₂-DE WRM blends.

d. DC Arc Emission Spectroscopy Determination of Impurities. The DC Arc Emission Spectroscopy (ES) method is well suited for, and was used to determine, the concentration of aluminum, calcium, iron, nickel, and silicon impurities in the PuO₂. The LANL procedure ANC-DE-1-SC-2S;R3 was used for sample preparation and emission spectroscopic analyses.

Sample Preparation. Single aliquots of PuO₂ from each of three samples were mixed with gallium and cobalt (internal standards) and transferred into cupped graphite electrodes.

Emission Spectroscopic Analysis. The electrodes were placed in a Jarrell-Ash emission spectrograph and ionized. The resulting spectra were analyzed to determine the PuO₂ impurity concentrations.

DC Arc ES Impurity Analysis Results. The analysis results for Al, Ca, Fe, Ni, and Si for the three samples are listed in Table 4 below.

Standards and Calibration. The emission spectrograph was calibrated with a standard prepared from a high purity PuO₂ matrix spiked with known quantities of elemental impurities from NIST-traceable emission spectroscopy standards. The precision and accuracy of the LANL DC Arc Emission Spectroscopic method has been established through a large number of control checks using the PuO₂ based in house standard Pu-U-H-RMP2323 and verified through LANL participation in interlaboratory studies.

Documentation and Reporting. The emission spectroscopic data for the three samples, the instrument calibration data, calibration and sample analysis dates, and operator and supervisor initials are recorded in formal LANL notebook 26648, p. 96, and the analytical results are recorded in the LANL NMT LIMS under sample #200016529. A copy of the "Final Chemical Analysis Report" is included in Appendix C. A copy of LANL procedure ANC-DE-1-SC-2S;R3 is contained in the NDA PDP Phase I WRM file. The results for all three samples were reviewed by the NDA CEP Technical Team to ensure the impurity levels and batch homogeneity were satisfactory. This review was a technical **HOLD POINT** prior to using the PuO₂ to prepare WRM AmO₂-PuO₂-DE blends.

e. Inductively-Coupled Plasma Mass Spectrometry Determination of Impurities. The ICP-MS method is well suited for, and was used to determine, the concentration of the following impurities in the PuO₂: B, Be, Cd, Ga, Gd, Mg, Li, ²³⁵U, ²³⁷Np, ²⁴⁴Cm, and ²⁵²Cf. The LANL procedure ANC102, "Inductively Coupled Plasma-Mass Spectrometry Using the VG Plasma Quad" was used for sample preparation and ICP-MS analyses.

Table 4. Emission Spectroscopic Analysis Results in Micrograms Impurity per Gram PuO₂.

Impurity	Sample 1	Sample 2	Sample 3	Average
Al	35	19	19	24
Ca	1000	770	750	840
Fe	99	89	100	96
Ni	245	280	285	270
Si	205	130	155	163

Sample Preparation. Three samples of the PuO₂ were dissolved in concentrated nitric acid using procedure ANC142, "Trace-Element Analysis Microwave Assisted Sample Preparation." The dissolved samples were aspirated into the ICP-MS for elemental and isotopic analyses.

ICP-MS Analysis. The samples were atomized and ionized in the plasma, which is sampled by a mass spectrometer. Mass detection and concentration measurements were made, relative to external calibration for each element. The analysis results for the three samples are summarized in Table 5.

Standards and Calibration. Standard Reference Materials traceable to the National Institute of Standards and Technology (NIST) standards for each element were used to calibrate the ICP-MS instrument and to assess instrument performance. The precision and accuracy of this method, based on ongoing calibration and verification standards, is $\pm 10\%$. Neptunium, Cm, and Cf are reported as semiquantitative values, due to lack of NIST standards for these elements. Analyses for ²⁴²Cm and ²³⁸U were performed, but were of limited value because ²⁴²Pu and ²³⁸Pu interfere directly with these mass values.

Documentation and Reporting. The sample handling, weights, and dissolution volumes are recorded in formal LANL notebook 10733, pp. 11 and 120, and in the LANL NMT LIMS system. Appendix C contains copies of the "Final Chemical Analysis Report." A memorandum discussing the analysis results for elements measured by ICP-MS is contained within the NDA

Table 5. ICP-MS Analysis Results in Micrograms Impurity per Gram PuO₂.				
Impurity	Sample 1	Sample 2	Sample 3	Average
B	<8	<8	<8	<8
Be	<2	<2	<2	<2
Cd	8.5	0.4	7.7	5.5
²⁵² Cf	<4	<4	<4	<4
²⁴⁴ Cm	0.10	0.095	2.3	0.8
Ga	18	17	17	17
Gd	0.6	<0.4	<0.4	<0.6
Li	8.5	<6	<7	<8.5
Mg	34	33	32	33
²³⁷ Np	94	96	96	95
Th	1.1	2.8	0.95	1.6
²³⁵ U	310	310	320	313
Note: values preceded by the symbol < represent ICP-MS limits of detection				

PDP Phase I WRM files. Copies of procedures ANC102 and ANC142 are contained in the NDA CEP WRM files. The sample impurity analysis results were reviewed by the NDA CEP Technical Team. The impurity levels, analysis data, and batch homogeneity were found to be satisfactory. This review was a technical **HOLD POINT** prior to using the PuO₂ to prepare AmO₂-PuO₂-DE WRM blends.

f. Radiometric Analyses. Radiometric analyses were performed to verify that the ²⁴⁴Cm and ²⁵²Cf levels were not high enough to adversely effect neutron or gamma based measurements of the WRMs, and to confirm the Mass Spectrometric/IDMS assay of ²⁴¹Am.

Sample Preparation. An aliquot of each of the five PuO₂ samples dissolved for Pu Assay (see section II.A.3.B, "Plutonium Assay") were pipetted onto glass slides and evaporated to dryness for alpha spectra analysis. To determine the ²⁴¹Am concentration, an aliquot from each of the dissolved samples was diluted to 10.0 ml with nitric acid. Gamma spectroscopy analysis was performed on the resultant solutions.

Radiometric Analyses. The alpha spectra from five sample aliquots showed no alpha peaks in the energy region 5.7 to 6.5 meV. Based on the alpha background in this energy region and the alpha peak from ²³⁹Pu and ²⁴⁰Pu at 5.155 meV, the measurement established that the ²⁴⁴Cm was <0.5 µg/g Pu and the ²⁵²Cf was <0.1 µg/g Pu. The gamma analysis of the five sample aliquots showed the ²⁴¹Am/PuO₂ ratios listed in Table 6. The mean value of the five analyses is 934.4 µg ²⁴¹Am/g PuO₂. This value confirms the more precise and accurate mass spectrometric value of 939.1 µg ²⁴¹Am/g PuO₂.

Standards and Calibration. The alpha spectrometer was energy calibrated with a source prepared from weapons grade Pu and ²⁴⁴Cm. This energy calibration was validated with NIST Standard Reference Material 4906-C-9, a ²³⁸Pu source. The gamma spectrometer was energy calibrated using an Amersham QCY.44 mixed radionuclide gamma-ray reference solution (see Appendix R).

Documentation and Reporting. The results of the ²⁴⁴Cm and ²⁵²Cf alpha spectrometric analysis and the ²⁴¹Am assay are contained in the Appendix C "Final Chemical Analysis Report" and are recorded in the LANL NMT LIMS system. The alpha spectrometric results for ²⁴⁴Cm and ²⁵²Cf were reported to the NDA CEP Technical team and was a technical **HOLD POINT** prior to using the PuO₂ to preparing AmO₂-PuO₂-DE WRM blends.

g. Anionic Impurity Analyses. Chlorine and fluorine potentially add random uncertainty to neutron based waste assays; hence, analyses were performed to determine their concentration in the PuO₂.

Sample Preparation. The chlorine and fluorine constituents of three representative samples of PuO₂ were separated from each PuO₂ sample by pyrohydrolysis and collected in a condensate. The pyrohydrolysis procedure is ANC115, "Determination of Chloride and Fluoride in Uranium-Plutonium Fuel Materials and Boron Carbide."

Table 6. Gamma Radiometric Analysis of PuO₂ for ²⁴¹Am.

Sample	µg ²⁴¹ Am/g PuO ₂
1	936
2	936
3	934
4	930
5	936
Average	934
Effective: 6/1/97	

Ion Chromatography Analysis. The chloride and fluoride concentrations were determined in the condensates using a Dionex DX-100 ion-chromatography instrument with a sensitivity of better than 1 microgram analyte per milliliter of solution. The three PuO₂ sample analyses for chloride and fluoride are listed in Table 7.

Table 7. Ion Chromatographic Analyses of PuO₂ for Cl and F.		
Sample	Cl, µg/g PuO₂	F, µg/g PuO₂
1	31.9	36.3
2	23.6	28.3
3	51.6	54.6
Average	35.7	39.7

Standards and Calibration. The Dionex-100 instrument was calibrated using a three point calibration curve with VHG standards that are traceable to NIST. The instrument was calibrated the day it was used.

Documentation and Reporting. The sample aliquots' weights and volumes, solution volume adjustments, sample preparation and analysis dates, VHG standards assay results, calculations, and operator and supervisor initials were recorded in formal LANL notebook #10733, pp. 117 and 120, and copies of these pages are in NDA PDP Phase I WRM files. Final data results are recorded in the LANL NMT LIMS system and found in the Appendix C "Final Chemical Analysis Report." A copy of procedure ANC115 is in the NDA CEP WRM file. The data was reviewed by the NDA CEP Technical Team and was a technical **HOLD POINT** prior to using the PuO₂ to prepare WRM AmO₂-PuO₂-DE blends.

B. Americium Dioxide

Specifications for the americium oxide (AmO₂) to be used for the CEP WRM production are listed in sections 5.9-5.14 of the SOW. The material (LANL FZG45-AMP138-2) was initially prepared at the LANL TA-55 Plutonium Facility as described below. A copy of the TA-55 report is included as Appendix D.

1. Preparation

a. Initial Preparation. Am (and any accompanying Pu) was separated from solution using a hydrogen peroxide precipitation. The hydroxide was redissolved in 7M HNO₃. Am was then separated from Pu via ion exchange. Am in the eluate was precipitated using oxalic acid. The Am oxalate was calcined to AmO₂ and screened through a 100 mesh screen and then packaged. Approximately 32 grams AmO₂ was prepared.

b. Blending. The AmO₂ was placed within a 1 pint container and blended using a rod mill without the rods. The material was blended for 1 hour.

c. Sampling. A 2 gram sample (LANL FZG45-AMP138-2S2) was used for production of the CEP WRMs and for characterization (described below). In addition, a 3 gram sample (LANL FZG45-AMP138-2S1) was sent out for particle size distribution analysis (also described below). Two additional 2 gram samples (LANL FZG45-AMP138-2S3 and LANL FZG45-AMP138-2S4) were placed in storage.

2. Characterization

a. Sampling for Analytical Chemistry. Quality assurance and quality control are incorporated into the procedures used to characterize the AmO_2 . The accuracy and precision of these methods have been established via ongoing quality control programs, and maintained through the routine practice of analyzing control standards along with samples. Hence, multiple sampling of the selected AmO_2 batch was not required to establish analytical method precision. However, multiple sampling was performed to verify batch homogeneity.

Four 50 mg samples were taken from the vial containing the 2 g of AmO_2 . These were dissolved and diluted (first dilution). From this, one sample from each bottle was taken for trace element analysis by ICP-MS (roughly 5 mg of AmO_2 in each sample) and two samples from each bottle were taken for mass spectral analysis (roughly 2 mg of AmO_2 in each sample) of the Pu fraction and Pu isotopic distribution of the AmO_2 . In addition, one sample from each bottle was placed into 250 mL plastic bottles for a second dilution. From each of these four bottles (second dilution), one sample each was taken for radiochemistry. In addition, two samples were taken from each of the four bottles for mass spectrometric determination of total Am as well as two samples from one of the bottles for U assay and U isotopic distribution of the AmO_2 .

b. Particle Size Distribution. A particle size distribution was performed on a separate 3 gram sample (LANL FZG45-AMP138-2S1) of AmO_2 . Two portions of this material were analyzed and the reports are included in Appendix E.

Method. A LAB-TEC 1000 Particle Size Analyzer was used to determine the sample particle size distribution. This instrument is a scanning laser microscope that measures particles suspended in a liquid medium. The procedure used for particle size determination is NMT-3-281-MRD-R00. The particle count fraction and mass fraction were determined for particle size distributions with resolution of < 1 micron in 28 increments over the 0.5 to 250 micron range.

Calibration. Quartz certified reference materials BCR Nos. 67 and 69 were used for the particle size analyzer calibration and performance checks. The instrument laser, detectors, and focal point are fixed in position and cannot be manipulated or adjusted. This very stable instrument is checked using the BCR quartz certified reference materials on a biweekly basis.

Documentation and Reporting. The particle size distribution was a technical **HOLD POINT**. The particle size distribution data was reviewed by the NDA CEP Technical Team prior to using the AmO_2 for preparing WRMs. The Spherical Equivalent Mean Diameter of the two samples ranged from 5.7 to 6.1 microns. Appendix E contains tables showing the detailed particle size data on both a particle count and mass basis.

A copy of procedure NMT-3 281-MRD-R00 and a brief description of the instrument operation and calibration are retained in the NDA PDP Phase I WRM file. The particle size distribution determined for each sample and the instrument calibration control data are retained in the NDA CEP WRM file.

c. Americium Assay, Pu and U Content, and Isotopic Distribution. The mass spectrometer operations are described in LANL procedure ANC 1312, "Operation of the VG-2 Mass Spectrometer." Procedures ANC 130, "Purification of Plutonium for Mass Spectrometric Analysis by Ion Exchange Separation" and ANC 141, "Separation of Trace Quantities of Uranium From Plutonium" describe the separation of Am from Pu and the separation of U from Pu by ion exchange. The principles discussed in procedure ANC 131, "U & Pu Isotopic Distribution and Assay by Isotope Dilution Mass Spectrometry" are directly applicable to the determination of Am in AmO₂ by IDMS except that the isotopic spike in this case would be ²⁴³Am.

Samples. Aliquots of the four AmO₂ solutions (second dilution) prepared for characterization were used for Am assay. Aliquots from one of the four solutions (second dilution) was used to determine the U content and U isotopic distribution of the AmO₂. Aliquots from the four AmO₂ solutions (first dilution) were used to determine the total Pu content and Pu isotopic distribution of the AmO₂.

Sample Preparation. The ²⁴¹Am analysis was performed by IDMS, and a weight aliquot of each sample was spiked with a known quantity of ²⁴³Am. Using ion exchange chromatography, the Pu fraction and Am fraction were separated, and an aliquot of the Am fraction was mounted on Re filaments for the ²⁴¹Am/²⁴³Am ratio determination. For the Pu isotopic distribution determinations, aliquots of the dissolved samples were purified by ion exchange chromatography to remove interfering uranium and americium isotopes. Samples used for Pu assay by IDMS were spiked with a known quantity of ²⁴⁴Pu and then interfering U and Am isotopes were removed by ion exchange. The resulting Pu fractions were mounted on Re filaments for analysis. For U isotopic distribution determinations, an aliquot of the dissolved sample was purified by removing interfering Pu isotopes by ion exchange. For U assay by IDMS, the sample was spiked with a known quantity of ²³³U followed by ion exchange as above. The U fractions were mounted on Re filaments for the analysis.

²⁴¹Am Assay. The Am fraction filaments were mounted in the VG-354 Mass Spectrometer. This instrument is equipped with a multicollector (5) Faraday system which permits the simultaneous collection of up to five isotopes. The entire Am aliquot is evaporated (Total Evaporation Mass Spectrometry), and the subsequent ion signal is collected, thereby yielding a very high precision, high accuracy analysis. The relative gain of the five measuring channels was determined at the start of each analysis with a built-in constant current source. The integrated intensity from each isotope was proportional to the abundance of that isotope. The ²⁴¹Am/²⁴³Am mass ratio was measured and the ²⁴¹Am concentration in the dissolved sample was calculated using the 241/243 ratio and the known ²⁴³Am concentration of the spiked sample. ²⁴¹Am assay results are summarized in Table 8.

Pu Assay and Isotopic Distribution Analysis. The Pu fraction filaments containing approximately 20 ng of Pu (mostly ²³⁹Pu) were mounted in the VG-354 instrument and analyzed by Total Evaporation Mass Spectrometry as described above for Am. The integrated intensity from

Table 8. Results of the ²⁴¹Am Assay of the AmO₂.*					
Sample	1	2	3	4	Mean
Wt. % Am	78.605	79.045	78.543	78.718	78.728
* Effective: 6/1/97					

each isotope was proportional to the abundance of that isotope. The total Pu concentration in the dissolved sample was calculated using the Pu isotopic ratios and the known ^{244}Pu concentration of the spiked sample. The results of these analyses are summarized in Tables 9 and 10.

U Assay and Isotopic Distribution Analysis. The U fraction filaments containing approximately 200 ng of U were mounted in the VG-354 instrument and analyzed by Total Evaporation Mass Spectrometry as described above for Am and Pu. The total U concentration in the dissolved sample was calculated using the U isotopic ratios and the known ^{233}U concentration of the spiked sample. The results of these analyses are summarized in Tables 11 and 12.

Table 9. Results of the Pu Assay of the AmO₂.*

Sample	1	2	3	4	Mean
Wt. % Pu	0.403	0.414	0.421	0.421	0.415
* Effective: 6/1/97					

Table 10. Summary of Pu Isotopic Distribution of the AmO₂ As Weight Percent.*

Sample	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu	^{244}Pu
1	0.0438	89.3626	9.9459	0.4623	0.1854	0
2	0.0457	89.1017	10.1855	0.4784	0.1887	0
3	0.0459	89.1031	10.1839	0.4778	0.1894	0
4	0.0453	89.0381	10.2437	0.4821	0.1908	0
Mean	0.0451	89.1514	10.1398	0.4751	0.1886	0
* Effective: 6/1/97						

Table 11. Results of the U Assay of the AmO₂.

Sample	1
Wt. % U	0.0035

Table 12. Summary of U Isotopic Distribution of the AmO₂ As Weight Percent.

Sample	^{233}U	^{234}U	^{235}U	^{236}U	^{238}U
1	0.1904	5.3044	42.8841	17.1893	34.4319

Standards and Calibration. For Pu and U, the mass spectrometer is calibrated using NBS Standard Reference Materials (SRMs) which are run with every batch of samples. These data have formed the basis for control charts and were used to assure the method is meeting performance criteria and to determine the method's historical performance. Historical QC data demonstrates that this method (sample preparation and instrument performance) is superior to the manufacture's stated precision of 0.05% at one standard deviation. For Pu and U, the standards used include NBS 948 (Pu standard) and NBS U005 (U standard), respectively (Appendix R). However, since there are no SRMs for Am, NBS 948 is used. This is acceptable because the isotopic range measured for Am (241-243 a.m.u.) overlaps that of the Pu measurements (238-244 a.m.u.). In addition, because of the use of the total evaporation method, the measurements are near absolute, i.e., no correction factors for mass discrimination are needed (see Ref. 2).

Documentation and Reporting. The SRM and sample replicate Pu and U isotopic distribution (both as atomic percent and weight percent), Pu and U assay, and ^{241}Am assay results, analysis dates, SRM aliquots, and operator and supervisor initials are recorded in formal LANL notebooks with copies introduced into NDA CEP WRM files. The Pu and U isotopic distribution and assay results and ^{241}Am assay ratio results for the five samples are summarized in Tables 8-12 above, and are contained in the "Final Chemical Analysis Report," included as Appendix C. Copies of procedures ANC 1312, ANC 130, ANC 141, and ANC 131 are included in the NDA CEP WRM file. The ^{241}Am assay, Pu isotopic distribution and assay, and the U isotopic distribution and assay results were technical **HOLD POINTS** and were reviewed by the NDA CEP Technical Team prior to using the AmO_2 to prepare AmO_2 - PuO_2 -DE WRM blends.

d. Inductively-Coupled Plasma Mass Spectrometry Determination of Impurities. The ICP-Mass Spectrometry (MS) method is well suited for, and was used to determine the concentration of the following impurities in the AmO_2 : Fe, Ni, Ca, Th, Al, Be, Ga, Mg, ^{235}U (semi-quantitative), ^{238}U , and ^{237}Np . The LANL procedures ANC102, "Inductively Coupled Plasma-Mass Spectrometry Using the VG Plasma Quad" and ANC142, "Trace Element Analysis-Microwave-Assisted Sample Preparation" were used for sample preparation and ICP-MS analyses. In addition, Bi, K, La, Na, Pb, Np, and Y were determined, for information only, using full quality assurances.

Sample Preparation. Three samples of the AmO_2 were dissolved in concentrated nitric acid. The dissolved samples were diluted and aspirated into the ICP-MS for elemental and isotopic analyses.

ICP-MS Analysis. The samples were atomized and ionized in the plasma, which is sampled by a mass spectrometer. Mass detection and concentration measurements were made, relative to external calibration for each element. The analysis results for the three samples are summarized in Table 13 below.

Standards and Calibration. Standard Reference Materials, traceable to NIST standards, for each element were used to calibrate the ICP-MS instrument and to assess instrument performance. The precision and accuracy of this method, based on ongoing calibration and verification standards, is +/- 10%.

Table 13. ICP-MS Analysis Results in Micrograms Impurity per Gram AmO₂.				
Impurity	Sample 1	Sample 2	Sample 3	Average
Fe	287	264	259	270
Ni	97	95	87	83
Be	<0.5	<0.5	<0.5	-----
Mg	58	52	45	52
Ca	1298	1257	1277	1277
Np	19976	20201	19150	19776
Th	35	36	35	35
Ga	3.2	3.3	3.0	3.0
Al	144	121	102	122
²³⁵ U	18*	20*	18*	19*
²³⁸ U	18	20	19	19
Bi	576	584	552	571
K	4821	5640	4124	4862
La	190	198	180	189
Na	2981	2223	1438	2214
Pb	4385***	1761	1619	1690
Np**	19590	19631	18718	19313
Y	43301	45056	42890	43749
Note: values preceded by the symbol < represent ICP-MS limits of detection; * = Semi-quant. **Repeat Np analysis to verify original results. ***Suspect result - not included in average.				

Documentation and Reporting. The sample handling, weights, and dissolution volumes are recorded in formal LANL notebook ANC52, p.115, and in the LANL NMT LIMS system. Appendix C contains copies of the "Final Chemical Analysis Report." Copies of procedures ANC102 and ANC142 are contained in the NDA CEP WRM file. The sample impurity analysis results were reviewed by the NDA CEP Technical Team. The impurity levels, analyses data, and batch homogeneity were found to be satisfactory. This review was a technical **HOLD POINT** prior to using the AmO₂ to prepare AmO₂-PuO₂-DE WRM blends.

e. Radiometric Analysis. Radiometric analyses were performed to verify that the ^{244}Cm and ^{252}Cf levels were not high enough to adversely effect neutron or gamma based measurements of the WRMs, and to confirm the Mass Spectrometric/IDMS assay of the Am.

Sample Preparation. 100 μL of the ^{241}Am sample solution (2nd dilution) was diluted with 1 N HNO_3 to 10 mL. Two cuts of 25 μL of diluted solutions were analyzed for total alpha (assume all alpha activity is due to ^{241}Am) by liquid scintillation counting. Two cuts of 25 μL of the ^{241}Am sample solution were diluted with 2 mL 1 N HNO_3 for ^{241}Am determination by gamma ray spectrometry.

Radiometric Analyses. The amount of ^{241}Am isotope within the sample was determined by radiochemical methods. Duplicate analyses were performed on each cut of sample. The total alpha activity was determined by liquid scintillation counting and ^{241}Am -gamma activity was determined by gamma spectrometry.

The wt. % Am determined by radiochemistry, 79.27%, is slightly larger than the more precise IDMS value 78.73% (see Table 14). This is likely due to the assumption that all alpha activity in the sample is due to Am decay. The presence of small amounts of Pu, Np, and U in the AmO_2 (Tables 9, 11, 13) add slightly to the total alpha activity thus increasing the radiochemistry assay value. The amounts of ^{244}Cm and ^{252}Cf in the sample were estimated based on their half-lives, and activity ratio to the ^{241}Am isotope using alpha spectrometry. This method provides a rough upper limit estimate of ^{244}Cm and ^{252}Cf concentrations. No actual peaks were observed in the 5.7 to 7.5 MeV region so an integrated background area (5 counts) in this energy region was used to bound the ^{244}Cm and ^{252}Cf concentrations. The estimated ^{244}Cm concentration in the sample was less than 2 ppm based on the half-lives and activity ratio of ^{244}Cm to ^{241}Am . The estimated ^{252}Cf concentration in the sample was less than 3 ppm based on the half-lives and activity ratio of ^{252}Cf to ^{241}Am .

Table 14. Liquid Scintillation and Gamma Radiometric Analysis of AmO_2 for ^{241}Am .

Cut	Sample wt., mg	LSC, Am mg*	Wt. % Am	Gamma, Am mg	Wt. % Am
14a	21.09	16.69	79.14	16.63	78.85
14b	21.09	16.54	78.43	16.60	78.71
15a	17.37	13.83	79.62	13.77	79.27
15b	17.37	13.83	79.62	13.91	80.08
16a	24.33	19.46	79.98	19.19	78.87
16b	24.33	19.19	78.87	19.28	79.24
17a	17.82	14.09	79.07	14.26	80.02
17b	17.82	14.15	79.41	14.18	79.57
		AVG	79.27 ± 0.49	AVG	79.33 ± 0.53
		% RSD	0.62	% RSD	0.66
* Assumes all alpha activity due to ^{241}Am .				Effective: 6/1/97	

The half-life ratios of ^{244}Cm and ^{252}Cf to ^{241}Am were very small due to small magnitude differences in the isotope half-lives. Therefore, the estimated detection limits for ^{244}Cm and ^{252}Cf were higher than the expected or real concentrations. If the detection limits were estimated based on $^{239,240}\text{Pu}$ alpha peak, then the estimated ^{244}Cm was less than 0.02 ppm and ^{252}Cf was less than 0.003 ppm. However, the counting statistics of the Pu peak was poor due to the tailing effect of high ^{241}Am alphas.

Standards and Calibration. Alpha spectrometry was performed using the surface barrier silicon detectors in an Oxford alpha spectrometry system (OASIS). The alpha spectrometers were energy calibrated with a source prepared from ^{239}Pu , ^{241}Am , and ^{244}Cm . The energy calibrations were validated with a NIST ^{238}Pu source (SRM 4906-C-9; see Appendix R). The liquid scintillation counter (Packard Tri-Carb 2200C) was calibrated using a NIST traceable in-house Radiochemistry Primary plutonium oxide standard (PEOR3258-STD1). The NaI based gamma-ray spectrometer was calibrated using an NIST traceable ^{241}Am standard solution (Amersham, AMP10040, batch 95/241/49; see Appendix R).

Documentation and Reporting. The results of the ^{244}Cm and ^{252}Cf alpha spectrometric analysis and the ^{241}Am assay are contained in Appendix C "Final Chemical Analysis Report," recorded in notebook AW-RC-5, pp. 91-102, and are recorded in the LANL NMT LIMS system. The alpha spectrometric results for ^{244}Cm and ^{252}Cf were reported to the NDA CEP Technical team and was a technical **HOLD POINT** prior to using the AmO_2 to prepare $\text{AmO}_2\text{-PuO}_2\text{-DE WRM}$ blends.

f. Loss on Ignition. LOI was determined for mass balance considerations. A Pt dish was prepared by washing in water, dried, and heated to approximately 950°C , cooled 0.5 hr. in oven, and then placed into a desiccator for approximately 2 hr. A nominally 1.7 g aliquot of AmO_2 was weighed into this Pt dish. The AmO_2 and Pt dish were then heated to 950°C for 2 hrs. After cooling in a desiccator, the AmO_2 and Pt dish were reweighed and the weight difference recorded. The percent LOI was calculated as 2.19%.

g. Anionic Impurity Analyses. Chlorine and fluorine potentially add random uncertainty to neutron based waste assays; hence, analyses were performed to determine their concentration in the AmO_2 .

Sample Preparation. The chlorine and fluorine constituents of a 99 mg sample of AmO_2 were separated from the AmO_2 sample by pyrohydrolysis and collected in a condensate. The pyrohydrolysis procedure is ANC115, "Determination of Chloride and Fluoride in Uranium-Plutonium Fuel Materials and Boron Carbide."

Ion Chromatography Analysis. The chloride and fluoride concentrations were determined in the condensates using a Dionex DX-100 ion-chromatography instrument with a sensitivity of better than 1 microgram analyte per milliliter of solution. The results of the AmO_2 sample analysis for chloride and fluoride are listed in Table 15.

Table 15. Ion Chromatographic Analyses of AmO_2 for Cl and F.		
Sample	Cl, $\mu\text{g/g AmO}_2$	F, $\mu\text{g/g AmO}_2$
1	40	654

Standards and Calibration. The Dionex-100 instrument was calibrated using a three point calibration curve with VHG or SPEX standards that are traceable to NIST. The instrument was calibrated the day it was used.

Documentation and Reporting. The sample aliquot weight and volume, solution volume adjustments, sample preparation and analysis dates, VHG or SPEX standards assay results, calculations, and operator and supervisor initials were recorded in formal LANL notebook 4070, pp. 142-143. Final data results are recorded in the LANL NMT LIMS system and found in Appendix C "Final Chemical Analysis Report." A copy of procedure ANC115 is in the NDA CEP WRM file. The data was reviewed by the NDA CEP Technical Team and was a technical **HOLD POINT** prior to using the AmO₂ to prepare WRM AmO₂-PuO₂-DE blends.

C. Depleted Uranium Oxide

Specifications for the Depleted Uranium (DU) oxide (UO₂) to be used for the CEP WRM production are listed in sections 5.15-5.19 of the SOW. The material (LANL LOT 0907-43 and 0907-44) was initially prepared at LANL. Two, nominally 3 kg each, bottles of the DU were delivered for CEP WRM production.

1. Preparation.

a. Initial Preparation. This material was initially prepared at Los Alamos in 1986 as fuel feedstock. U₃O₈ was dissolved in demineralized water. UO₄ was then precipitated using H₂O₂-NH₄OH. The precipitate was then filtered and dried to "yellow cake." The yellow cake was then calcined at 900°C for 16 hours in air to oxidize the material to U₃O₈. The U₃O₈ was then reduced to UO₂ by heating to 700°C for 1 hour in an H₂ atmosphere. The temperature was then decreased to 400°C and the material oxidized in air for 6 hours. Further reduction of the U oxide was accomplished by heating the material to 700°C for 1 hour in H₂ then sintering for 1 hour in CO₂ and allowed to cool in CO₂.

b. Packaging. A total of 6 kg were requested for production of the CEP WRMs. The material was packaged in two plastic bottles each containing nominally 3 kg.

c. Blending. To ensure homogeneity of the total 6 kg of DU, the entire batch was blended in a Turbula Model T2C blender. Approximately half of the contents of the two bottles were placed within a blending bottle and blended for 30 minutes. The same was done with the remainder of the material. Next, one half of the material blended first is combined with one half of the material blended second and this was blended for 30 min. The same was done with the remainder of the material. The DU was then left in the blending bottles.

2. Characterization

a. Sampling for Analytical Chemistry. Aliquots of the DU oxide were weighed for analytical chemistry as follows: four 250 mg samples for trace element analysis, one 30 mg sample for particle size distribution, three 100 mg samples for pyrohydrolysis/ion selective electrode analysis, and eight 150 mg samples (total) for mass spectrometry and radiochemistry.

b. Particle Size Distribution. A particle size distribution was performed on a 30 mg sample of DU oxide. The report is included in Appendix F.

Method. An Olympus Vanox microscope (reflected light) was used to determine the sample particle size distribution. The optical images were collected and digitized by a Scion video capture board. The digital images were processed and sized using NIH Image software.

Calibration. The calibration was done using a B&L optical standard.

Documentation and Reporting. The particle size distribution was a technical **HOLD POINT**. The particle size distribution data was reviewed by the NDA CEP Technical Team prior to using the DU for preparing WRMs. The Spherical Equivalent Mean Diameter is 11 microns with a maximum diameter of 43 microns. See Appendix F for details.

c. Uranium Assay and Isotopic Distribution. The same sample preparation and mass spectrometer operations described previously for the PuO₂ and AmO₂ analyses were used for U assay and U isotopic distribution of the UO₂. The sample preparation and mass spectrometer operations are described in LANL procedures ANC 131, "U & Pu Isotopic Distribution and Assay by Isotope Dilution Mass Spectrometry," ANC 1312, "Operation of the VG-2 Mass Spectrometer," and ANC 130, "Purification of Plutonium for Mass Spectrometric Analysis by Ion Exchange Separation."

Samples. Five 150 mg aliquots of the DU oxide powder were dissolved in concentrated HNO₃ with a few drops of dilute HF and diluted with 1N HNO₃ for mass spectrometric determination of total U and U isotopic distribution.

Sample Preparation. For the U isotopic distribution determinations, aliquots of the dissolved samples were purified by ion exchange chromatography to remove any interfering plutonium isotopes. The U assay analysis was performed by IDMS, and a weight aliquot of each sample was spiked with a known quantity of ²³³U. Using ion exchange chromatography, the U sample plus spike was purified, and an aliquot of the U sample plus spike fraction was mounted on Re filaments for the isotopic ratio determinations.

U Isotopic Distribution Analysis. The U fraction filaments containing approximately 200 ng of U were mounted in the VG-354 instrument. This instrument is equipped with a multicollector (5) Faraday system which permits the simultaneous collection of up to five isotopes. The entire U aliquot is evaporated (Total Evaporation Mass Spectrometry), and the subsequent ion signal is collected, thereby yielding a very high precision, high accuracy analysis (see Ref. 2). The relative gain of the five measuring channels was determined at the start of each analysis with a built-in current source. The integrated intensity from each isotope was proportional to the abundance of that isotope. Results of these analyses are summarized in Table 16.

Table 16. Summary of U Isotopic Distribution As Weight Percent.					
Sample	²³³U	²³⁴U	²³⁵U	²³⁶U	²³⁸U
1	0.0000	0.0008	0.1901	0.0035	99.8057
2	0.0000	0.0008	0.1906	0.0034	99.8053
3	0.0000	0.0002	0.1899	0.0036	99.8064
4	0.0000	0.0014	0.1898	0.0029	99.8060
5	0.0000	0.0008	0.1905	0.0037	99.8051
Average	0.0000	0.0008	0.1902	0.0034	99.8057

U Assay. The U fraction (plus spike) filaments were mounted in the VG-354 Mass Spectrometer and processed as described above. The U isotope ratios were measured and the total U concentration in the dissolved sample was calculated using the 238/233 ratio and the ²³³U concentration of the spike. The U assay results are summarized in Table 17.

Table 17. U Assay Results.					
Sample	1	2	3	4	5
Wt. % U	87.464	87.497	87.496	87.525	87.485
Average: 87.493					

Standards and Calibration. The Mass Spectrometer is calibrated using NBS SRM-U005 (Appendix R) aliquots run with every batch of depleted uranium samples receiving U isotopic distribution analysis. These data have formed the basis for control charts and were used to assure the method is meeting performance criteria, and to determine the method's historical performance. Historical QC data demonstrates that this method (sample preparation and instrument performance) is superior to the manufacture's stated precision of 0.05% at one standard deviation.

Documentation and Reporting. The SRM and sample replicate U isotopic distribution (both as atomic percent and weight percent) and U assay results, analysis dates, SRM aliquot, and operator and supervisor initials are recorded in formal LANL notebooks with copies introduced into NDA CEP WRM files. The U isotopic distribution results and U isotopic ratio results for the five samples are summarized in Tables 16 and 17 above and are contained in the "Final Chemical Analysis Report," included as Appendix C. Copies of procedures ANC 130, ANC 131, ANC 1312 are in the NDA CEP WRM file. The total U and U isotopic distribution results were a technical **HOLD POINT** and were reviewed by the NDA CEP Technical Team prior to using the UO₂ to prepare UO₂-DE WRM blends.

d. ICP-AES Determination of Impurities. The ICP Atomic Emission Spectroscopy (AES) method is well suited for, and was used to determine, the concentration of the following impurities in the UO₂: Al, Ca, Fe, Mg, and Na. The LANL procedure ANC189, "Inductively Coupled Plasma—Atomic Emission Spectroscopy Using the Thermo Jarrell-Ash IRIS Spectrometer" was used for sample preparation and ICP-AES analyses.

Sample Preparation. Three samples of the UO₂ were dissolved in concentrated nitric acid with a few drops of dilute HF following LANL procedure ANC142, "Trace-Element Analysis—Microwave-Assisted Sample Preparation." The dissolved samples were diluted and aspirated into the ICP-AES for elemental analyses.

ICP-AES Analysis. The samples were atomized and ionized in the plasma; atomic emission lines were detected optically and intensities measured by a charge injection device. Elemental concentration measurements were made, relative to external calibration for each element. The analysis results for the three samples are summarized in Table 18 below.

Standards and Calibration. Standard Reference Materials (e.g., SPEX) traceable to NIST standards for each element were used to calibrate the ICP-AES instrument and to assess instrument performance. The precision and accuracy of this method, based on ongoing calibration and verification standards, is 5-10%.

Table 18. ICP-AES Analysis Results in Micrograms Impurity per Gram UO₂.				
Impurity	Sample 1	Sample 2	Sample 4	Average
Al	99	108	102	103
Ca	21	23	25	23
Fe	46	50	49	48
Mg	4.1	5.4	4.5	4.7
Na	171	175	157	168

Documentation and Reporting. The sample handling, weights, and dissolution volumes are recorded in formal LANL notebooks ANC52, p. 116, ANC6, p. 211, and F1994, p. 129, and in the LANL NMT LIMS system. Appendix C contains copies of the “Final Chemical Analysis Report.” Copies of the procedures ANC189 and ANC142 are contained in the NDA CEP WRM file. The sample impurity analysis results were reviewed by the NDA CEP Technical Team. The impurity levels, analyses data, and batch homogeneity were found to be satisfactory. This review was a technical **HOLD POINT** prior to using the UO₂ to prepare UO₂-DE WRM blends.

e. ICP-MS Determination of Impurities. The ICP-MS method is well suited for, and was used to determine, the concentration of the following impurities in the UO₂: B, Be, Cd, Ni, Cr, Li, Pb, W, V. The LANL procedure ANC102, “Inductively Coupled Plasma-Mass Spectrometry Using the VG Plasma Quad” was used for sample preparation and ICP-MS analyses.

Sample Preparation. Three samples of the UO₂ were dissolved in concentrated nitric acid with a few drops of dilute HF following LANL procedure ANC142, “Trace-Element Analysis—Microwave-Assisted Sample Preparation.” The dissolved samples were diluted and aspirated into the ICP-MS for elemental and isotopic analyses.

ICP-MS Analysis. The samples were atomized and ionized in the plasma, which is sampled by a mass spectrometer. Mass detection and concentration measurements were made, relative to external calibration for each element. The analysis results for the three samples are summarized in Table 19.

Standards and Calibration. Certified Reference Materials (e.g., SPEX) traceable to NIST standards for each element were used to calibrate the ICP-MS instrument and to assess instrument performance. The precision and accuracy of this method, based on ongoing calibration and verification standards, is +/- 10%.

Documentation and Reporting. The sample handling, weights, and dissolution volumes are recorded in formal LANL notebooks ANC52, p. 116, ANC6, p. 211, and F1994, p. 129, and in the LANL NMT LIMS system. Appendix C contains copies of the “Final Chemical Analysis Report.” Copies of procedures ANC102 and ANC142 are contained in the NDA CEP WRM file. The sample impurity analysis results were reviewed by the NDA CEP Technical Team. The impurity levels, analyses data, and batch homogeneity were found to be satisfactory. This review was a technical **HOLD POINT** prior to using the UO₂ to prepare UO₂-DE WRM blends.

Table 19. ICP-MS Analysis Results in Micrograms Impurity per Gram UO ₂ .				
Impurity	Sample 1	Sample 2	Sample 4	Average
B	297	307	307	304
Be	<0.5	<0.5	<0.5	
Cd	0.22	0.13	0.31	0.22
Ni	7.7	5.7	4.9	6.1
Cr	1.6	1.7	2.0	1.8
Li	<0.5	<0.5	<0.5	
Pb	7.2	5.5	5.4	6.0
W	3.0	2.8	3.1	3.0
V	1.6	1.4	1.6	1.5
Note: Values preceded by the symbol < represent ICP-MS limits of detection.				

f. Radiometric Analysis. Radiometric analyses were performed to verify that the ²⁴⁴Cm and ²⁵²Cf levels were not high enough to adversely effect neutron or gamma based measurements of the WRMs.

Sample Preparation. Two cuts (200 µL each) of the DU sample solution were analyzed for ²⁴⁴Cm and ²⁵²Cf by alpha spectrometry.

Radiometric Analyses. The amounts of ²⁴⁴Cm and ²⁵²Cf in the sample were estimated based on their half-lives and activity ratio to the ²³⁸U isotope using alpha spectrometry. This method provides a rough upper limit estimate of ²⁴⁴Cm and ²⁵²Cf concentrations. No actual peaks were observed in the 5.7 to 7.5 MeV region so an integrated background area (5 counts) in this energy region was used to bound the ²⁴⁴Cm and ²⁵²Cf concentrations. The estimated ²⁴⁴Cm concentration in the sample was less than 1×10^{-5} ppm based on the half-lives and activity ratio of ²⁴⁴Cm to ²³⁸U. The estimated ²⁵²Cf concentration in the sample was less than 1×10^{-6} ppm based on the half-lives and activity ratio of ²⁵²Cf to ²³⁸U.

The half-life ratios of ²⁴⁴Cm and ²⁵²Cf to ²³⁸U were very large due to a very long half-life of ²³⁸U. Therefore, conservative estimates of detection limits for ²⁴⁴Cm and ²⁵²Cf were reported. If the detection limits were estimated based on ²³⁴U alpha peak, then the estimated ²⁴⁴Cm was less than 0.001 ppm and ²⁵²Cf was less than 0.001 ppm.

Standards and Calibration. Alpha spectrometry was performed using the surface barrier silicon detectors in an OASIS. The alpha spectrometers were energy calibrated with a source prepared from ²³⁸Pu, ²³⁹Pu and ²⁴⁴Cm. The energy calibrations were validated with an NIST ²³⁸Pu source (SRM 4906-C-9; see Appendix R).

Documentation and Reporting. The results of the ²⁴⁴Cm and ²⁵²Cf alpha spectrometric analysis are contained in the Appendix C "Final Chemical Analysis Report" and are recorded in the LANL NMT LIMS system. The alpha spectrometric results for ²⁴⁴Cm and ²⁵²Cf were reviewed by the NDA CEP Technical team and was a technical **HOLD POINT** prior to using the DU to prepare DU WRM blends.

g. Anionic Impurity Analyses. Chlorine and fluorine potentially add random uncertainty to neutron based waste assays; hence, analyses were performed to determine their concentration in the UO_2 .

Sample Preparation. The chlorine and fluorine constituents of three representative samples of UO_2 were separated from each UO_2 sample by pyrohydrolysis and collected in a condensate. The pyrohydrolysis procedure is ANC115, "Determination of Chloride and Fluoride in Uranium-Plutonium Fuel Materials and Boron Carbide."

Ion-Selective Electrode Analysis. The chloride and fluoride concentrations were determined in the condensates using ion-selective electrodes. Either a fluoride or chloride ISE is plugged into a pH/ISE meter and calibration curve standards are directly read and recorded into computer memory followed by sample analysis. The three UO_2 sample analyses for chloride and fluoride are listed in Table 20.

Standards and Calibration. The ISEs are calibrated using a three point calibration curve with VHG or SPEX standards that are traceable to NIST. The instruments were calibrated the day they were used.

Documentation and Reporting. The sample aliquots' weights and volumes, solution volume adjustments, sample preparation and analysis dates, VHG or SPEX standards assay results, calculations, and operator and supervisor initials were recorded in formal LANL notebook #4070, pp. 142-143. Final data results are recorded in the LANL NMT LIMS system and found in Appendix C "Final Chemical Analysis Report." A copy of ANC115 is in the NDA CEP WRM file. The data was reviewed by the NDA CEP Technical Team and was a technical **HOLD POINT** prior to using the UO_2 to prepare WRM UO_2 -DE blends.

Table 20. Ion Selective Electrode Analyses of UO_2 for Cl and F.		
Sample	Cl, $\mu\text{g/g UO}_2$	F, $\mu\text{g/g UO}_2$
1	6	4
2	3	4
3	3	<1
Mean	4	4

D. Diatomaceous Earth

J.T. Baker Co. #2244-05 diatomaceous earth (DE) (Kieselguhr) was obtained in eleven, one kg batches. This type of DE was selected because previous analyses demonstrated that Kieselguhr contained the lowest chloride and fluoride content of the four types of DE evaluated.

1. Preparation

The diatomaceous earth (DE) batches were sealed inside a clean, 55 gallon steel drum. The DE was blended for ten hours with a rotating drum mixer.

a. Sampling. After blending, the DE was poured into three plastic containers. While the DE was being poured, two samples were taken from each receiving container. The first sample was taken when the receiving container was 1/5 full, and the second sample was taken when the receiving container was full. The blending and sampling are described in further detail in the August 17, 1995 memo, "Don Vance to File," which is contained in Appendix D of LA-UR-96-2277, "NDA PDP Working Reference Materials Production Plan-Phase I."

2. Characterization

Characterization of the DE was important for two reasons: to ensure that the material was adequately blended, and to ensure that the concentration of elements interfering with neutron and gamma based analyses were at or below levels which might introduce significant interference via alpha-n reactions.

a. Glow Discharge Mass Spectrometry. The constituents of the DE were determined by Glow Discharge Mass Spectrometry (GDMS), with the procedure described (in Ref. 3). The GDMS analysis involved quantification of 29 constituents, including the alkaline metals Be, B, Si, S, Cl, F, Fe, Al, Th, and U.

Sample Preparation. Each DE sample (and the SRM materials) was blended with high purity tantalum metal and mounted in the Glow Discharge Mass Spectrometer. Two of the DE samples were prepared in duplicate to provide analysis precision data.

Glow-Discharge Mass Spectrometry Analysis. Glow discharge mass spectrometry is a sputtering technique whereby atoms from an electrically conducting sample are introduced into a high-energy argon plasma, ionized and mass analyzed. The GDMS instrument at LANL is a double focusing, magnetic sector, high resolution instrument capable of quantification to the ppb level. The complete report of GDMS data is contained in Appendix E of LA-UR-96-2277 (Ref. 1). Table 21 contains a summary of GDMS analysis results on constituents with potential neutronic interference. Most elements are reported as weight percent oxides which is a geological convention. This does not mean that the oxides listed in Table 21 are actually present in the DE.

Table 21 lists a F content of 0.2 weight percent. This content is probably biased high; analysis of F on the NIST SRM 278 was biased almost a factor of 10 high. However, the F content for the SRM is listed as a semiquantitative value. Hence, we cannot unequivocally bias correct the GDMS F value.

Table 21. GDMS Analysis Results on Kieselguhr Diatomaceous Earth.			
Constituent	Wt %	Constituent	µg/g (ppm)
SiO	88	BeO	<0.1
Fe ₂ O ₃	1.7	B ₂ O ₃	18
CaO	4.2	ThO ₂	<0.2
Na ₂ O	1.2	UO ₂	<0.3
Cl	<0.006		
F	0.2		

Standards and Calibration. The high purity tantalum binder is routinely analyzed for impurities to provide background correction data. NIST SRMs 89, 91, 92, and 98b (Appendix R) were combined with the tantalum binder and analyzed to provide calibration sensitivity factors. Further, NIST SRM 278 (Appendix R) was analyzed along with the DE samples as a QC check. To ensure that the instrument precision was adequate, two of the six DE samples were analyzed in duplicate.

Documentation and Reporting. All pertinent data, including calibration, sample weights, sample identification, sample preparation and analysis dates, and QC sample identification and sample analyses results are recorded on both the GDMS hard disk and the backup floppy disks of the LANL Analytical Chemistry Group (NMT-1). A copy of the procedure described in Ref. 3 is contained in the NDA PDP Phase I WRM file. A copy of the complete analytical results for all six samples and the control samples is found in Appendix E of LA-UR-96-2277 (Ref. 1). The DE impurity analysis results were reviewed by the NDA CEP Technical Team to ensure the DE homogeneity and impurity content were satisfactory. This review was a technical **HOLD POINT** prior to usage of the DE for preparing WRM blends.

b. Loss on Ignition. To ensure that the DE did not contain appreciable adsorbed moisture or other volatile species, two samples of the DE were heated to 102° C for 16 hours. The resulting LOI was 0.29% and 0.17%, indicating that the DE was relatively free of moisture or volatile materials. This measurement data and calculation is found in both Appendix F of LA-UR-96-2277 (Ref. 1) and the NDA PDP Phase I WRM file.

III. BLENDING

A Turbula Model T2C blender, shown in Figure 1, was used for blending. Separate blends were prepared for each individual WRM to ensure that the amount of nuclear material in each WRM was known with maximum certainty.

Figure 1. Turbula Blender with Blend Bottle.

A. Blending Parameters and Blend Evaluations

Blending parameters were determined and blend evaluations carried out during the initial stages of the PDP WRM production (PDP Phase I) and these results are directly applicable to the CEP WRM production. The following summary is based on detailed descriptions that are found in LA-UR-96-2277, "NDA PDP Working Reference Materials Production Plan-Phase I" (see Ref. 1).

1. Summary of Initial Tests for PDP

To establish the blender speed and length of time required to produce homogeneous blends of nuclear material with DE, cold tests were initially performed using iron powder as a surrogate for the nuclear material. Following this, actual test blends using PuO_2 and DE were prepared following procedure ANC1412, "Blending the Nuclear Material and Matrix for the Nondestructive Assay Standards." The homogeneity of these blends was determined via gamma-ray spectrometry following procedure ANC1413, "Nuclear Material-Diatomaceous Earth Homogeneity Determination." The homogeneity of the blends was better than 2% RSD. Concerns of nuclear material particle agglomeration during blending were evaluated by performing a particle size distribution analysis of the PuO_2 and of the PuO_2 -DE blend. No indication of agglomeration of the nuclear material particles was found. The alpha-neutron yield of the blend was also evaluated employing a Shuffler neutron assay instrument (passive mode). The total neutron flux ratio of the PuO_2 -DE blend divided by the flux for PuO_2 alone was found to be 1.37 indicating some increased neutron flux from the blend. However, this level of added random neutron flux would not appreciably degrade the neutron-based assay system's precision.

2. Alpha-Neutron Yield of AmO_2 -DE Blends

A prototype IAP WRM (58 mg Am) was analyzed on the Shuffler neutron assay instrument. The counts per second of the WRM were indistinguishable from background implying that the alpha-neutron yield from AmO_2 blends is insignificant. The data (see Appendix G) were reviewed by the NDA CEP Technical Team and was a technical **HOLD POINT** prior to the preparation of IAP WRMs.

3. Gas-Generation Evaluation of the IAP WRMs

Gas generation within the WRMs due to alpha radiolysis of hydrogenous material was evaluated through computational studies. These studies reveal that the pressure within the WRMs will rise to 22.6 psig within 27.9 years due primarily to the radiolysis of residual water. After 27.9 years the water will be consumed and the pressure will continue to rise very slowly due to formation of helium gas formed by the decay of alpha-emitting radionuclides. The pressure within the stainless steel WRMs will reach the maximum design and failure (yield) pressures within 83 and 176 years, respectively. These values are affected by assumptions made for the evaluations. A full report on the gas generation evaluation is included in Appendix H. These data were reviewed by the NDA CEP Technical Team and was a technical **HOLD POINT** prior to the preparation of IAP WRMs.

B. IAP WRM Blend Preparation

The blending procedure ANC1412, "Blending the Nuclear Material and Matrix for the Nondestructive Assay Standards" was followed to produce homogeneous blends with Am and Pu masses within the ranges listed in the SOW section 4.1.a-4.1.e (see Appendix A).

1. Nuclear Material and DE Addition

Prior to producing the first blend, the quantity of DE required to fill a tap-packed stainless steel container to within 0.5 +/- 0.05 inches of the top was determined in cold testing to be 134.0 grams. The DE was weighed out to the nearest 0.1 gram and recorded. The DE was placed in a uniquely identified blend container. The containers were introduced into the glovebox train and transferred into the weighing glovebox. Weighed quantities of PuO₂ and AmO₂ were transferred to the blend bottle containing the DE.

2. Blending

After DE, AmO₂, and PuO₂ addition, the blend container lid was secured, and the container was removed from the weighing glovebox, transferred into the blending glovebox, and introduced into the blender. The blending was performed for 60 minutes at the medium rotational speed of the Turbula Blender, under the conditions established in LANL procedure ANC1412, "Blending the Nuclear Material and Matrix for the Nondestructive Assay Standards."

3. Documentation and Reporting

Diatomaceous earth weight, AmO₂ weight, PuO₂ weight, blend identification, blend time, date, and operator signature were recorded on the work form from procedure ANC1412. These forms are retained in the NDA CEP WRM file. A summary of the DE, AmO₂, and PuO₂ weights for each of the 16 blends is found in Appendix I.

C. DU WRM Blend Preparation

The blending procedure ANC1412, "Blending the Nuclear Material and Matrix for the Non-destructive Assay Standards" was followed to produce homogeneous blends with DU masses within the ranges listed in the SOW section 4.1.f-4.1.h (see Appendix A).

1. Nuclear Material and DE Addition

Prior to producing the first blend, the quantity of DE to be mixed with the nuclear material required to fill a tap-packed stainless steel container to within 0.5 +/- 0.05 inches of the top was estimated based on experience with PuO₂ PDP WRMs and was adjusted for each mass range. Since the DU WRMs have such large mass ranges for U, the amount of DE in these WRMs is far less than the amount of nuclear material (opposite from the PDP and IAP WRMs). Thus, errors were encountered in terms of the proper amount of DE to use. These errors will be discussed below in the section pertaining to filling the cylinders and WRM certification. The DE was weighed out to the nearest 0.1 gram and recorded. The DE was placed in a uniquely identified blend container. The containers were then introduced into the glovebox train and transferred into the weighing glovebox. Weighed quantities of UO₂ were transferred to the blend bottles containing the DE. The total weight of the blend bottle plus the blend was then recorded on the blend preparation form.

2. Blending

After DE and UO₂ addition, the blend container lid was secured, and the container was removed from the weighing glovebox, transferred into the blending glovebox, and introduced into the blender. The blending was performed for 60 minutes at the medium rotational speed of the Turbula Blender, under the conditions established in LANL procedure ANC1412, "Blending the Nuclear Material and Matrix for the Nondestructive Assay Standards."

3. Documentation and Reporting

Diatomaceous earth weight, DU weight, blend bottle tare, blend identification, blend time, date, and operator signature were recorded on the work form from procedure ANC1412. These forms are retained in the NDA CEP WRM file. A summary of the DE and DU weights for each of the 16 blends is found in Appendix J.

IV. STAINLESS STEEL ASSEMBLIES

A. Fabrication

Sixteen stainless steel assemblies were fabricated from #304 stainless steel tubing (seamless) and sheet and bar stock in the same manner as those prepared for PDP Phases I and II. The fabrication was performed at the Los Alamos National Laboratory main machine shop as described in the Statement of Work PLA-STD-103/1 (Appendix K in LA-UR-96-2277; Ref. 1). The Statement of Work and all QA documentation (manufacture certificates, initial assembly inspection, test component inspection, welder certification, and leak check records) are contained in the NDA PDP Phase I WRM file.

B. Receiving Inspection

The 16, #304 stainless steel tubing assemblies and upper endcaps were inspected for cleanliness, identification marks, dimensions (wall thickness, length, diameter), endcap fit, damage, and weld quality. Inspection was performed using LANL procedure ANC1408, "Receiving Inspection of NDA Standard Containers" which includes an inspection form. A copy of this procedure and the initialed and dated inspection forms for all assembly inspections are retained in the NDA CEP WRM files. Figure 2 shows several of the as-received assembly packages for the PDP Phase I WRMs with inspection documentation. Those received for the CEP WRMs were similar.

Figure 2. Packaged Assemblies with Inspection Forms.

The 16 assemblies were identified with the following sequential alpha-numeric system: CEP-001, ..., CEP-010, ...CEP-016. The identification was laser-etched into both the assembly inner and outer cylinder endcaps for the IAP WRMs and the outer assembly and endcap for the DU WRMs (Note: the DU WRMs are singly contained). Figure 3 shows a labeled DU WRM assembly with endcaps.

V. STAINLESS STEEL CYLINDER FILLING

The WRM cylinder preparation (inner cylinder for IAP WRMs), filling, packing, frit insertion, bagout for transfer, and cleanup operations are fully described in LANL procedures ANC1410, "Filling the Containers with the Reference Materials" and ANC1411, "Decontaminating Containers for NDA Standards."

Figure 3. Labeled Assembly with Endcaps.

A. Cylinder Preparation

To minimize the spread of contamination, the cylinders were wrapped in aluminum foil and taped in a manner to cover all the cylinder outer surfaces while leaving the open end of the tube exposed for filling. The tube was then placed in the filling glovebox. The cylinder was fitted with a plastic sleeve which covered the lip of the open end and extended 12 mm down into the cylinder. This sleeve prevented blend powder from contacting the upper 12 mm of the cylinder, where the graphite frit and endcap would be placed.

B. Cylinder Filling and Packing

For stability, the cylinders were placed inside an approximately six cm (ID) graduated cylinder of about 15 cm in height. An approximately 30 X 30 cm sheet of brown paper was positioned under the graduated cylinder so that any spillage could be readily detected and recovered. Tests with DE demonstrated that one mg of DE (<0.001% of the blend mass) was highly visible when spilled on this paper. All implements used in the loading process were kept on the paper. A paper funnel was then inserted into the open cylinder and the designated blend powder was added incrementally via a spatula into the cylinder. Figure 4 shows a cylinder ready for filling, an opened blend bottle, filling implements and a graphite frit on spillage-catch paper.

After each incremental addition, the cylinder was repetitively tapped against the glovebox floor to firmly pack the powder. Figure 5 shows a fully loaded cylinder.

Figure 4. Cylinder Ready for Filling.

Figure 5. Fully Loaded and Tap-Packed.

To prevent loss of blend powder during tap-packing, a small watch glass was held over the open end of the cylinder. Any detected spillage was immediately returned to the blend bottle or the cylinder. The incremental addition and tap packing steps were continued until all the blend powder was introduced into the cylinder (Note: For some of the DU WRMs, not all of the blend was transferred into the cylinder due to errors in determining the proper amount of DE to use for blending. Since not all of the blend is incorporated into the WRM, the total mass of DU in the WRM was adjusted and the homogeneity of the blend was determined and incorporated into the total uncertainty calculations discussed in the section on WRM Certification.). The cylinder was finally tap packed until the surface of the powder was just below the bottom lip of the plastic sleeve insert (see Figure 5).

C. Frit Insertion

After the blend powder was transferred into the cylinder, the plastic sleeve was removed and a precut graphite felt frit was carefully inserted and gently pressed down onto the surface of the powder. The loaded WRM cylinder was now ready for bagout.

D. Waste Materials Measurement

1. IAP WRMs

After each cylinder was fully loaded, the paper funnel, paper tissues used to clean the plastic sleeve, watch glass, and spatula, and the paper sheet under the loading area were inserted into the now-empty blending bottle (see Figure 6). These materials were bagged out of the filling glovebox and submitted to radiochemistry to determine the amount of nuclear material adhering to these materials. This measurement was performed using the far-field gamma counting method. Appendix K describes the measurement of the waste materials.

2. DU WRMs

After each cylinder was fully loaded, the paper funnel, paper tissues used to clean the plastic sleeve, watch glass, and spatula, and the paper sheet under the loading area were inserted into a CLEAN blending bottle (except for CEP011 which was handled like the IAP WRMs discussed above). These materials were bagged out of the filling glovebox and submitted to radiochemistry to

Figure 6. Packaged Wasted and Filled Cylinder Ready for Bagout.

determine the amount of nuclear material adhering to these materials. This measurement was performed using the far-field gamma counting method. Appendix K describes the measurement of the waste materials.

Since, in most cases, not all of the DU-DE blend was delivered into the steel cylinder, the remaining blend in the blend bottle was reweighed and the amount of DU remaining was subtracted from the total DU in the blend. This correction was then applied to the total mass of DU in the WRM.

E. Documentation and Records

Records were prepared showing that all the steps pertaining to the cylinder preparation, filling, frit insertion, and cleanup were performed. These records list blend and cylinder identification numbers, and they were initialed and dated by the operator performing the operations. The final calculated quantity of nuclear material in the WRMs, including the weight of the nuclear materials and corrections for the amount of nuclear material in the waste materials, are contained in the NDA CEP WRM files and summarized in Appendices I and J. The cylinder filling records are retained in the NDA CEP WRM files.

VI. INERTING

A. IAP WRMs

The prepared IAP WRM cylinders (10) were transferred into the inert gas airlock of the welding glovebox. The airlock was gently evacuated and backfilled with helium gas. The evacuation and backfilling operation was repeated twice. This operation replaced the air in the cylinders with helium, which prevented oxidation of the stainless steel cylinders during TIG welding and acted as a fill gas for leak inspection after welding.

B. DU WRMs

The prepared DU WRM cylinders (6) were transferred into a large desiccator which was then evacuated using a rotary pump. The desiccator was then backfilled with helium gas. The evacuation and back filling was repeated twice. This operation replaced the air in the cylinders with helium which acted as a fill gas for leak inspection. Figure 7 shows the desiccator with the DU WRMs.

Figure 7. The Six DU WRMs in the Desiccator for Inerting.

VII. WELDING

A. Welding Setup

A Programmable TIG welder located within an inert atmosphere glovebox was used to perform the TIG welds on both the inner and outer stainless steel tubes for the IAP WRMs. A similar TIG welder mounted on a bench top was used to seal the DU WRMs.

1. IAP Inner Stainless Steel Tube Welds

The power supply was located outside of the welding glovebox, and the welding cables fed into the welding glovebox. Located inside the glovebox was a speed-controlled rotational chuck and a stand holding the welding torch. The stand allowed precise, three directional positioning of the welding electrode. Since the welding was performed in an inert-gas glovebox, no additional inert cover gas was required. Figure 8 shows the in-glovebox welding electrode being positioned for an IAP inner cylinder endcap weld.

Figure 8. In-Glovebox Welding Electrode.

2. IAP Outer Stainless Steel Tube Welds

The IAP outer tube welds were performed outside the glovebox. A speed-controlled rotational chuck and a stand holding the welding torch was used to ensure reproducible welds. An inert cover gas was used to minimize oxidation of the stainless steel during welding. Figure 9 shows a completed WRM after outer cylinder-endcap welding.

3. DU Stainless Steel Tube Welds

The DU tube welds were performed outside the glovebox. A speed-controlled rotational chuck and a stand holding the welding torch was used to ensure reproducible welds. An inert cover gas was used to minimize oxidation of the stainless steel during welding.

B. Establishing Weld Parameters

Using the #304 stainless steel cylinders and end caps that comprised the test components (see Appendix K of LA-UR-96-2277; Ref. 1), practice welds were performed to establish welding power supply amperage, program cylinder rotational speed, and welding electrode-to-cylinder distance and position following procedure ANC1404, "Weld Development Procedure-NDA Standards." To ensure acceptable welds, end cap welds were performed at different current levels, and a current operating range was established. These parameters were established for both

the inner and outer cylinders and end caps.

C. Welding Qualification

The welding equipment, welding parameters, welding procedure and weld operators were qualified as detailed in LANL procedure ANC1402, "Weld Qualifications Procedure-NDA Standards." Once they had demonstrated operational welding parameters by performing test welds, the two welding operators performed qualification welds on both the inner and outer cylinders with appropriate end caps. The welds extended 360° around the cylinders, with adequate over-run to assure a complete seal and controlled arc break. The weld qualification acceptance criteria includes visual examination at 10X magnification, helium leak testing, and metallographic examination as defined in LANL procedure CST-STD-005/0. Figure 10 shows a photomicrograph of an inner-cylinder endcap weld cross-section with 0.040" penetration.

D. Documentation and Records

Dated and signed records were prepared on the weld qualification visual inspection and leak testing. Metallographic examination records include dated and signed photomicrographs of various sections of the qualification weld specimens. These evaluations were performed as part of PDP Phase I and the records are retained in the NDA PDP Phase I WRM files. The visual inspection and leak testing results, and the photomicrographs were reviewed to assess weld quality. A weld qualification report was prepared and is included in the NDA PDP Phase I WRM files. These reviews were a technical **HOLD POINT** prior to welding of the blend-filled CEP WRM cylinders.

Figure 9. Completed IAP WRM after Welding and Inspection.

Figure 10. Inner Cylinder-Endcap Weld Cross-section.

VIII. DECONTAMINATION OF THE IAP INNER CYLINDERS

After the inner cylinder end caps were welded in the inert atmosphere glovebox, the cylinders were removed from the glovebox through a series of cleaning operations. The cleaning was assessed by smearing the cylinders with a Health Physics swipe cloth, and then counting the swipe with a portable alpha survey instrument. Once the cylinders were cleaned to <20 dpm smearable alpha contamination, they were removed from the open-front glovebox and placed in plastic bags. The cleaning and contamination monitoring procedure was ANC1411, "Decontaminating Containers for NDA Standards." This procedure and records of the decontamination assessment are retained in the NDA CEP WRM files.

IX. LEAK CHECKING

A. Helium Leak Check

Helium leak checks were performed on the welder qualification welds for the inner and outer test cylinders, and for the inner cylinder welds for every IAP WRM and the cylinder welds for the DU WRMs. The helium leak checks were performed following LANL procedure ANC1401, "Leak-Checking the NDA Standard Containers," with a leak rate pass criteria of $< 1 \times 10^{-6}$ atm-cc/second. All cylinders demonstrated a helium leak rate of $< 1 \times 10^{-8}$ atm-cc/second, meeting Department of Transportation requirements for shipping in Class B containment.

B. Documentation and Records

Dated and initialed leak check records including leak detector calibration data and leak rate data for each WRM are retained in the NDA CEP WRM files. These records were a technical **HOLD POINT** and were reviewed prior to WRM shipment.

X. FINAL INSPECTION

Final WRM inspection included alpha contamination checks, dimensional checks, and gamma-ray measurements to both evaluate WRM nuclear material uniformity and verify WRM nuclear material loadings. Each WRM was placed in a plastic bag and sealed after final inspection. The bags were identified using a flow-pen with the same alphanumeric identifier as the WRM.

A. Alpha Contamination Checks

Thorough alpha contamination measurements were made on every WRM using both swipe methods sensitive to 6 dpm/100 cm² removable alpha activity and direct alpha surveys sensitive to 300 dpm/100 cm². No detectable alpha contamination was found on any WRM. These survey results were recorded, signed, and dated for every WRM and are contained in the NDA CEP WRM files.

B. Dimensional and Identification Checks

Every WRM was checked for length and diameter to ensure a length of 8.99 to 9.01 inches and a diameter of <1.947 inches. The final weld on the endcap of the outer cylinder added up to 0.04 inches to the length of some of the WRMs. With permission from the Idaho National Engineering and Environmental Laboratory (INEEL) technical manager, this increased length was accepted as being preferable to machining the weld down to the 9.01 inch specification length. Further, every WRM was visually inspected for any obvious defects such as dents or scratches, and to ensure the identification markings were clear and robust. These data are recorded, dated, signed, and retained in the NDA CEP WRM files.

C. Nuclear Material Uniformity Checks and Verification

Each WRM was subjected to collimated gamma-ray intensity measurements focused on either a 4.5 cm zone of the left and right (upper and lower if the cylinder is vertically oriented) segments of the cylinder (IAP WRMs) or three 5 cm segments near the center of the WRM (DU WRMs). These measurements were used to determine the uniformity of the nuclear material within the WRM and to verify the contents of the WRM.

1. IAP WRMs

a. Uniformity Check. Peak areas for the 59.5 keV gamma peak of ^{241}Am and the 129 keV peak of ^{239}Pu were integrated. Table 22 contains representative uniformity data for the WRMs at 59.5 keV and 129 keV. All counting data (including multiple runs for certain WRMs) are included in Appendix L.

Table 22. Uniformity Measurement Data Summary.				
WRM	mg Am	L/R Ratio @59.5keV	g Pu	L/R Ratio @129keV
CEP001	10.4778	0.98	0.0854	1.09
CEP002	9.9121	1.00	0.0788	1.09
CEP003	54.1621	0.99	1.9880	1.01
CEP004	54.7379	1.01	2.0089	1.03
CEP005	92.2422	1.00	1.9934	0.98
CEP006	93.2348	1.06	1.9915	1.02
CEP007	54.7680	1.01	0.3957	0.99
CEP008	52.1327	1.01	0.4136	1.02
CEP009	103.3964	1.02	-----	-----
CEP010	99.7750	1.01	-----	-----

Figure 11 shows a verification/uniformity measurement being performed with the IAP WRM positioned behind lead bricks with a 4.5 cm wide aperture. Appendix L summarizes the measurement details and contains a complete compilation of the left-right zone counting data for each WRM. This record showing WRM identification and two-zone gamma count data is also contained in the NDA CEP WRM files.

b. Verification. The same measurements used above for uniformity were also applied to the verification of the nuclear material content of the WRMs. The left and right side counts are added together which is then divided by the total mass of Am or Pu as the case may be. By dividing the total peak areas by the mass of nuclear material, one would expect a constant count rate/mass value for all the WRMs. In no case was the Count Rate/Mass significantly different from the mean (Appendix L). Table 23 displays this data for both Am and Pu.

Figure 11. IAP WRM Undergoing Uniformity/Verification Measurement.

Table 23. Verification Measurement Data Summary.				
WRM	Am L + R Counts 59.9 keV	Count Rate/Mass	Pu L + R Counts 129 keV	Count Rate/Mass
CEP001	23787	2270	750	8786
CEP002	21597	2179	545	6921
CEP003	119288	2202	14316	7201
CEP004	107893	1971	14001	6970
CEP005	205095	2223	14445	7247
CEP006	190381	2042	13966	7013
CEP007	137330	2507	2906	7343
CEP008	117226	2249	3295	7966
CEP009	233310	2256		
CEP010	252694	2533		

2. DU WRMs

The DU WRMs were counted on a segmented gamma scanner (Figure 12). Instead of counting the left and right halves of each WRM as was done for the IAP WRMs, three 5 cm segments were counted (2 hours/segment) and compared for each DU WRM. Acquiring accurate data for the DU WRMs was problematic due to the weak gamma signal from the ^{235}U coupled with an excessive attenuation correction applied to the count data. The attenuation correction in most cases was $> 90\%$ thus rendering the quality of the data suspect. With this in mind, the uniformity and verification data are presented below.

Figure 12. DU WRM (In Gray Can) Undergoing Uniformity/Verification Measurement on a Segmented Gamma Scanner.

a. Uniformity Check. Peak areas for the 186 keV gamma peak of ^{235}U were integrated. Table 24 contains representative uniformity data for the WRMs at 186 keV. All counting data (including multiple runs for certain WRMs) are included in Appendix M.

Even with the poor quality count data it is evident that the WRMs are uniform to better than 18% (worst case). Higher quality gamma count data would likely show uniformity to better than 10%.

Appendix M summarizes the measurement details and contains a complete compilation of the segmented gamma scanner data for each WRM. This record showing WRM identification and three-zone gamma count data is also contained in the NDA CEP WRM files.

b. Verification. The same measurements used above for uniformity were also applied to the verification of the uranium content of the WRMs. The three segment counts are added together and then divided by the total mass of U. Table 25 displays this data for the U WRMs.

In only one case (CEP011) was the Count Rate/Mass apparently (not statistically significant) different from the mean (Appendix M). This was to be expected as this WRM has the highest DE/DU ratio and thus less attenuation of the 186 keV gamma due to self-shielding. The other

Table 24. Uniformity Measurement Data Summary for the DU WRMs.				
WRM	g U	Seg. 1/Seg. 2 Ratio	Seg. 1/Seg. 3 Ratio	Seg. 2/Seg. 3 Ratio
CEP011	499.18	0.98	1.04	1.06
CEP012	883.94	0.85	0.93	1.09
CEP013	957.60	1.00	1.13	1.14
CEP014	959.51	0.85	1.01	1.19
CEP015	972.64	0.88	0.98	1.11
CEP016	948.66	0.99	0.97	0.98
				Mean ratio = 1.01

Table 25. Verification Measurement Data Summary for DU WRMs.		
WRM	U 1 + 2 +3 Counts 186 keV	Count Rate/Mass
CEP011	40104	80
CEP012	41472	47
CEP013	42192	44
CEP014	45000	47
CEP015	40824	42
CEP016	45360	48

five DU WRMs, taken as a group, show no significant deviation from the mean which verifies the U content of the WRMs.

XI. PACKAGING AND SHIPPING

Each WRM was sealed in a plastic bag, and each bag was marked with the WRM identification number, plus an “OK” sticker signifying the WRM had passed all inspection and verification steps. The WRMs were wrapped in bubble wrap, inserted into the 2R inner container of a certified DOT 6M shipping container, and shipped via authorized commercial carriers Appendix N is a record of the WRMs sent to INEEL. Also included in Appendix N is a listing of the NDA CEP Program site personnel, including the individual designated for receiving the WRMs. Figures 13 and 14 show WRMs being protected by bubble wrap and inserted into a 2R cylinder.

Figure 13. Bubble Wrapping Two WRMs.

XII. WRM CERTIFICATION

The WRM production process was designed to meet all SOW specified design attributes. To certify the WRMs, critical information and data generated during the WRM production process was formally documented, filed and reduced through appropriate calculations to determine the Pu, Am, and U mass values and the total alpha activity for each WRM. Further, uncertainty data for the critical information and data was propagated to determine the statistical uncertainties of the calculated masses and alpha activities of each completed WRM. Finally, for certification purposes, all critical measurements are traceable to nationally recognized reference materials.

A. Nuclide Mass and Alpha Activity Calculations

The calculation algorithms for Pu, Am, and U mass and alpha activity for the WRMs are presented below. These calculations were executed in the spreadsheet format contained in Appendix O. Also listed in Appendix O are the calculated Pu, Am, U mass, and alpha activities for all 16 WRMs. It should be noted that the AmO₂ used for CEP WRM production does contain minor amounts of Pu, U, and Np (see Appendix C). The small concentration of these elements in the AmO₂ result in negligible contributions to the total alpha activity and are hence ignored in the mass and alpha activity calculations.

Figure 14. Inserting WRMs into a 2R Cylinder for Shipment.

1. Pu Mass

The Pu mass for each WRM is calculated as follows:

$$\text{Pu Mass (g)} = (\text{Wt. PuO}_2 \text{ (g)} \times \text{Pu assay}) - \text{T\&H Loss (g Pu)}$$

where: **Wt. PuO₂** is the weight of PuO₂ (g) weighed out for the specific WRM.

Pu assay is the mean value of the Pu content of the PuO₂ in g Pu/g PuO₂ (decay corrected to 6/1/97).

T&H Loss is the transfer and holdup loss as determined by the gamma spectroscopy assay of the transfer container, the blending container, funnel, cleaning tissues, and the transfer spillage catch paper.

2. Am Mass

The ²⁴¹Am mass for each WRM is calculated as follows:

$$^{241}\text{Am Mass (g)} = [(\text{Wt. AmO}_2 \text{ (g)} \times \text{Am assay1}) + (\text{Wt. Pu (g)} \times \text{Am assay2})] - \text{T\&H Loss (g Am)}$$

where: **Wt. AmO₂** is the weight of AmO₂ weighed out for the specific WRM.

Am assay1 is the mean value of the Am content of the AmO₂ in g Am/g AmO₂ (decay corrected to 6/1/97).

Wt. Pu is the weight of elemental Pu in the WRM (i.e., Wt. PuO₂ (g) x Pu assay).

Am assay2 is the mean value of the Am content of the PuO₂ in g Am/g Pu (decay corrected to 6/1/97).

T&H Loss is the transfer and holdup loss as determined by the gamma spectroscopy assay of the transfer container, the blending container, funnel, cleaning tissues, and the transfer spillage catch paper.

3. DU Mass

The DU mass for each WRM is calculated as follows:

$$\text{DU Mass} = (\text{Wt. UO}_2 \times \text{U assay}) - \text{T\&H Loss (g U)}$$

where: **Wt. UO₂** is the weight of UO₂ weighed out for the specific WRM.

U Assay is the mean value of the U content of the UO₂ in g U/g UO₂.

T&H Loss is the transfer and holdup loss as determined by the gamma spectroscopy assay of the transfer container, the blending container, funnel, cleaning tissues, and the transfer spillage catch paper.

4. Alpha Activity

The WRM alpha activity is the summation of the alpha activity contribution from each of the Pu, Am, and U isotopes in the WRM. To determine the alpha activity for each WRM, the alpha *specific* activity (alpha curies per gram Pu, Am, or U multiplied by the g Pu, Am, or U, respectively) for the nuclear material feed stock was calculated, effective 6/1/97 (see Table 26). Each WRM alpha activity was calculated by multiplying the WRM Pu, Am, or U isotopic mass times the appropriate factor (see Table 26). Appendix P lists the WRM's alpha specific activity for the initial date of 6/1/97, plus annual calculations from 6/1/97 through 6/1/2001.

Table 26. Atomic Masses, Half-Lives, and Alpha Activities of the Isotopes Present in the CEP WRMs.

Isotope	At. Mass (a.m.u.)	At. Mass Uncert. (a.m.u.)	$T_{1/2}$ (yrs)	$T_{1/2}$ Uncert. (yrs)	$T_{1/2}$ (s)	Total Specific Activity (Ci/g)	Branching Ratio % Alpha	Alpha Activity (Ci/g)
²³³ U	233.03963	3.006E-06	1.592E+05	2.0E+02	5.021E+12	9.644E-03	100	9.644E-03
²³⁴ U	234.04095	2.147E-06	2.455E+05	6.0E+02	7.742E+12	6.227E-03	100	6.227E-03
²³⁵ U	235.04392	2.147E-06	7.038E+08	5.0E+05	2.220E+16	2.163E-06	100	2.163E-06
²³⁶ U	236.04556	2.040E-06	2.342E+07	3.0E+04	7.386E+14	6.472E-05	100	6.472E-05
²³⁸ U	238.05078	2.147E-06	4.468E+09	3.0E+06	1.409E+17	3.364E-07	100	3.364E-07
²³⁸ Pu	238.04955	2.147E-06	8.770E+01	3.0E-01	2.766E+09	1.714E+01	100	1.714E+01
²³⁹ Pu	239.05216	2.147E-06	2.411E+04	3.0E+01	7.603E+11	6.208E-02	100	6.208E-02
²⁴⁰ Pu	240.05381	2.040E-06	6.563E+03	7.0E+00	2.070E+11	2.271E-01	100	2.271E-01
²⁴¹ Pu	241.05685	2.040E-06	1.435E+01	1.0E-01	4.525E+08	1.034E+02	0.0025	2.534E-03
²⁴² Pu	242.05874	2.147E-06	3.733E+05	1.2E+03	1.177E+13	3.960E-03	100	3.960E-03
²⁴¹ Am	241.05682	2.147E-06	4.322E+02	7.0E-01	1.363E+10	3.434E+00	100	3.434E+00
Ref:	4	4	5	5	5		5	

B. Uncertainty Data and Calculations

To meet the SOW (Appendix A) section 4.1 criteria for alpha activity and uncertainty, the following information was collected and data reduction performed.

1. Weighing Uncertainty

Random uncertainties for weighing were estimated from repeated weighings using a standard weight that most closely matched the weight of the target quantity of nuclear material. These weighings were performed before each set of same-target-mass nuclear material were weighed. The balance performance is summarized in Table 27 below and full control weighing data is listed in Appendix Q.

All but one of the standard weight sets used for control weighings were certified against NIST traceable weights. Copies of the certifications for the weight sets and analytical balances are also contained in Appendix Q. The observed control weight checks (Table 27) show no statistically significant bias from the certified mass. Hence, the WRM nuclear material weights were considered free of significant bias.

Table 27. Precision on Standard-Control Weight Weighings.				
Diatomaceous Earth Weighings				
<i>Balance: 13045 (Certified)</i>				
<i>Check Wts: Cenco Class S (Certified)</i>				
Standard Wt.	Number of Measurements	Mean	Std. Dev.	% Uncert.
30 g	15	29.99853	5.16E-04	0.009
50 g	3	50.001	0.002	0.014
100 g	20	99.993	0.004	0.018
Diatomaceous Earth Adjustment Weighings (DU WRMs)				
<i>Balance: 015233(Certified)</i>				
<i>Check Wts: Not Certified</i>				
Standard Wt.	Number of Measurements	Mean	Std. Dev.	% Uncert.
10 g	15	10.00094	1.46E-05	0.010
200 g	12	200.0143	4.93E-05	0.007
Pu Oxide and Am Oxide Weighings				
<i>Balance: 013599 (Certified)</i>				
<i>Check Wts: 15082 (Certified)</i>				
Standard Wt.	Number of Measurements	Mean	Std. Dev.	% Uncert.
10 mg	5	0.01001	4.47E-06	0.204
50 mg	5	0.05002	5.48E-06	0.062
100 mg	10	0.10002	6.75E-06	0.032
1 g	6	1.00001	5.16E-06	0.003
2 g	10	2.00003	6.75E-06	0.002
U Oxide Weighings				
<i>Balance: 016834 (Certified)</i>				
<i>Check Wts: 4482,3648 (Certified)</i>				
Standard Wt.	Number of Measurements	Mean	Std. Dev.	% Uncert.
500 g	41	499.954	5.91E-03	0.012
1 kg	38	999.899	6.49E-03	0.012

2. Assay Uncertainty

a. Pu Oxide

Pu Assay. Pu assay values on five samples of the PuO₂ starting material are listed in Table 1. The assays were performed by coulometry. The individual assay values on the five samples can be found in Appendix C and a copy of the certificate of the SRM used is in Appendix R.

The mean assay of the five samples was 0.87804 grams Pu/gram PuO₂ (6/1/97). The observed standard deviation of the five assays was 0.00033 (6/1/97). This value was deemed too small for the uncertainty to be associated with the plutonium assay, for two reasons. First, it does not reflect systematic errors that can effect an assay; and second, it probably does not capture anything other than short-term variation (repeatability). Because this PuO₂ was made to be a standard material, extra care was taken during analyses to ensure homogeneity and representative sampling. Based on the analysis of SRMs, it was believed that a standard deviation value in the range 0.0006 to 0.0008 (0.07-0.09%RSD) would be more reasonable. To be conservative, values at the high end of this interval were chosen. (Experience with propagation of variation (POV) has shown that in spite of efforts to include all sources of uncertainty, POV estimates are often understatements.) Since assays from five independent samples were averaged to get the 0.87804 value, the standard deviation of the average was taken as the standard deviation of SRM control data from a 2 year period divided by the square root of five. This was then multiplied by the student's t corresponding to n = 5 to get the 95% confidence bounds. This error was then propagated (n = 5) to result in an overall uncertainty on the Pu assay of 0.036%.

Am Assay. The ²⁴¹Am assay values for the five samples of PuO₂ analyzed are listed in Table 3. The mean assay value of 939.1 µg ²⁴¹Am/g PuO₂ (6/1/97) has a one random standard deviation uncertainty of 0.028 %. The isotope dilution mass spectrometric (IDMS) method uses a ²⁴³Am spike as an internal standard. Since no nationally traceable mass spectrometry (isotopic) standards exist for Am, the relative uncertainty of the ²⁴³Am spike solution concentration has been conservatively estimated as 0.25% at 95% confidence. This estimate was made based on (1) the original calibration of the ²⁴³Am spike using a high purity ²⁴¹Am material which itself was assayed by EDTA titration and (2) comparative studies on EURATOM samples (PIDIE) (Ref. 6). The 0.25% uncertainty serves as a systematic error, as applied to the ²⁴¹Am assay. The random error associated with the Am assay is 0.028% as obtained from multiple determinations (five by IDMS) of the Am content of the PuO₂ (see Appendix C). The random error (0.028%) is added after using the systematic error (0.25%) to bound the expected value resulting in an overall error of 2.8 mg/g Pu. For details see Appendices O and U.

b. Am Oxide

Am Assay. Four samples of Am oxide were assayed by IDMS. The mean assay value of 78.728 % Am (6/1/97) has a one random standard deviation uncertainty of 0.284 %. Again, since no nationally traceable mass spectrometry (isotopic) standards exist for Am, the relative uncertainty of the ²⁴³Am spike solution concentration has been conservatively estimated as 0.25% at 95% confidence as described above. The random uncertainty associated with this assay is 0.28% as obtained from multiple determinations (four by IDMS) of the Am content of the AmO₂ (see Appendix C). The random error (0.28%) is added after using the systematic error (0.25%) to bound the expected value resulting in an overall error of 0.6%. More details can be found in Appendices O and U.

c. U Oxide

U Assay. The uncertainty on the U assay (87.493% U) at the 95% confidence level is taken the standard deviation of the five assay values (0.022) divided by the square root of five. This is then multiplied by the corresponding student's t value for $n = 5$ to obtain the 95% confidence bounds. The overall error on the U assay is calculated as 0.03%.

3. Isotopic Uncertainty

a. Pu Oxide. The Total Evaporation Mass Spectrometric method used for the Pu isotopic distribution and ^{241}Am assay analyses is an absolute method with no known bias. The method was calibrated with the Plutonium Isotopic Standard NBS SRM 948 (Appendix R), which has a Pu distribution similar to the PuO_2 starting material. The ^{239}Pu weight percents of the NBS SRM 948 and the PuO_2 are 91.8284 % and 93.7811 %, respectively (on 6/1/97).

The five samples analyzed for total plutonium were also analyzed for Pu isotopic fraction using Thermal Ionization Mass Spectrometry. The summary statistics provide estimates of isotopic standard deviations that were 25 to 67% less than the standard deviations obtained on the measurement control standard (NBS 948), measured over a four month period immediately prior to the date of the PuO_2 isotopic measurements (Appendix S). To be conservative, it was decided to use the isotopic averages of the five samples and the standard deviations calculated from the measurement control data. To obtain the standard deviations of the averages, the measurement control standard deviations were divided by the square root of five, the sample size. These estimates of uncertainties for the stated PuO_2 isotope weight percents are then multiplied by the corresponding student's t value for $n = 5$ to provide 95% confidence bounds, and are listed in Table 28.

Table 28. Upper Bound Error Estimates for Mass Spectrometric Pu Isotopic Distribution.					
Isotope	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
Mean	0.0143	93.7811	5.9448	0.2038	0.0560
Std. Dev	0.0014	0.0022	0.0008	0.0015	0.0003
95% C.B.	+/-0.0018	+/-0.0028	+/-0.0010	+/-0.0019	+/-0.0003

b. U Oxide. The Total Evaporation Mass Spectrometric method used for the U isotopic distribution is an absolute method with no known bias. The method was calibrated with the Uranium Isotopic Standard NBS SRM U005 (Appendix R), which has a U distribution similar to the UO_2 starting material. The ^{238}U weight percents of the NBS SRM U005 and the UO_2 are 99.504 % and 99.806 %, respectively (on 6/1/97).

The five samples analyzed for total uranium were also analyzed for U isotopic fraction using Thermal Ionization Mass Spectrometry. The summary statistics provide estimates of isotopic standard deviations that were generally similar to the standard deviations obtained on the measurement control standard (NBS U005), measured over a 1.5 year period immediately prior to the date of the UO_2 isotopic measurements (Appendix S). It was decided to use the isotopic averages of the five samples along with their standard deviations. These estimates of uncertainties for the stated UO_2 isotope weight are divided by the square root of five and then multiplied by the

corresponding student's t value for $n = 5$ to provide the 95% confidence bound and are listed in Table 29.

Table 29. Upper Bound Error Estimates for Mass Spectrometric U Isotopic Distribution.					
Isotope	^{233}U	^{234}U	^{235}U	^{236}U	^{238}U
Mean	0.0000	0.0008	0.1902	0.0034	99.8057
Std. Dev	0.0000	0.0004	0.0004	0.0003	0.0005
95% C.B.	+/-0.0000	+/-0.0005	+/-0.0004	+/-0.0004	+/-0.007

4. Transfer and Hold-Up Loss

Transfer and holdup loss quantities were measured by counting the 59.5 keV peak of the ^{241}Am (IAP WRMs) and the 63.32 keV peak of ^{234}Th (DU WRMs) contained in the funnels, cleaning tissues, and spillage catch paper used to transfer the blend material into the stainless steel cylinder. For the IAP WRMs, since all of the blend produced was incorporated into the steel cylinders, the blending bottle was also counted to determine the amount of Am and Pu that was adhered to the blending bottle. For the DU WRMs, The blending bottle was not counted (except for CEP011). This is because there was always a portion of the blend that was not incorporated into the WRMs (except for CEP011 in which all of the blend was incorporated into the WRM), and this excess amount of blend was determined gravimetrically and the amount of U in the WRM was adjusted accordingly. These loss corrections were relatively small; the largest loss was 0.063 % for WRM CEP003.

Uncertainty terms for these waste item measurements include the preparation of the Am and Th standards, the gamma spectroscopy instrumentation calibration, and the uncertainty associated with the counting of each waste item. From a positional/rotational study, the uncertainty in the counts appeared to be the dominating term, especially for those items with the smallest amount of Pu.

The uncertainty associated with the gamma spectroscopy counter calibration includes uncertainty in the standard value used for calibration, uncertainty in the calibration curve shape, and uncertainty in the calibration short-term variability. From prior studies on pipette calibration, weighing, and the calibration curve fit, the calibration *short-term* random variability was estimated to be less than 1.6%. The relative effect of the uncertainty on the calibration curve shape and the standard value depends on the count rate of the waste item, and was assumed to be a systematic error. Both the random variation and the systematic error are thought to act as a bias effect on the final calculation of the grams of ^{241}Am , Pu, or U in the WRM.

The WRM waste item uncertainty terms used for calculating the final Pu, Am, and U content, and alpha activity for each WRM are listed in Appendix K. Appendix T includes a detailed description and discussion of the waste item uncertainty terms.

5. Alpha Activity Uncertainty

Alpha half-life values for converting Pu, Am, and U isotope quantities in the WRMs to alpha activity are those presented in Table 26. The total alpha activity for the various WRMs has two uncertainty components, the alpha half-life and the isotopic abundance. These uncertainty terms

are incorporated into the total uncertainty associated with the total alpha activity of each WRM (see Appendix O).

6. Combined Uncertainty for Mass and Alpha Activity

Standard uncertainty estimation methods were used to calculate individual random and systematic uncertainties for Pu, PuO₂, Am, AmO₂, U, UO₂ mass, total alpha activity, and Pu and U isotope ratios. POV techniques were used to appropriately combine the uncertainties for all the components used to calculate nuclear quantities. Where well-supported estimates on uncertainties for components were not available, a conservative approach was taken to use values that are believed to exceed the actual values. Therefore the 95% confidence intervals (bounds) presented in this plan and listed on the WRM Certificates are believed to be larger than they would be if exhaustive data was available for every uncertainty component.

Identified in Table 30 are uncertainty components included in estimates of the 95% confidence interval for the nuclear quantities listed on the WRM Certificates.

Appendix U presents all the equations used to estimate the 95% confidence limits for the quantities listed in Table 30. Appendix U provides values for all the parameters used in the propagated uncertainty calculations listed in Appendix O.

C. WRM Certification

The WRMs are formally certified by a combination of the multiple analyses characterizing the PuO₂ and DE starting materials, the calibration and check of measurement methods with Certified or Standard Reference Materials, the Project Quality Assurance Plans, the numerous signed and dated QA forms used to document inspections and completion of fabrication steps, the review of calculations and critical documents, the statistical analysis of data using standard

Table 30. Components Included in Uncertainty Calculations.						
Source of Uncertainty						
	Weighing	Pu, Am, or U Assay	Pu or U Isotopic Ratio	Transfer	Homogeneity	Counting
PuO ₂ Mass	X					
Pu Mass	X	X		X		X
Pu Isotopic			X			
AmO ₂ Mass	X					
Am Mass	X	X		X		X
UO ₂ Mass	X				X	
U Mass	X	X		X	X	X
U Isotopic			X		X	
Alpha Activity	X	X	X	X	X	X

data reduction methods, the qualification and training of persons responsible for analysis and fabrication operations, and the formal control and filing of documents. To provide a single document attesting to the formal certification of the 16 WRMs, Certificates of Content and Traceability for each WRM were prepared, signed, and dated by the LANL Analytical Chemistry (NMT-1) Group Leader, Bill J. McKerley and the LANL Project Leader for NDA CEP WRM production, Robert S. Marshall. Copies of the WRM Certificates are included as Appendix V.

The reduction of PuO_2 , AmO_2 , and UO_2 characterization, mass transfer data, and control data generated during the production of the WRMs produces Pu, Am, and U isotope mass and alpha activity, as well as uncertainty data required for the WRM certification. These data and calculations are formally recorded, signed and stored in the NDA CEP WRM file. Certification also requires that the critical characterization measurements (Pu assay, Pu isotopic distribution, Am assay, PuO_2 , AmO_2 , and UO_2 weighings, U assay, and U isotopic distribution) be traceable to nationally recognized reference materials. This traceability has been demonstrated by identifying these reference materials in this production plan (Appendix R) and in the analytical procedures used. In addition, the DOE site that received WRMs also received Certificates of Control and Traceability for those WRMs. Copies of these reference materials' certificates are retained in the NDA CEP WRM reference file. To the highest degree possible, traceability to nationally recognized reference materials was established for less critical parameters, including particle size analysis, impurity analyses, isotope half-life and alpha specific activity, and helium leak check. Certificates or statements addressing traceability are retained in the NDA CEP WRM file for the less critical parameters.

XIII. REFERENCES

1. Marshall, "NDA PDP Working Reference Materials Production Plan-Phase I," Los Alamos National Laboratory document LA-UR-96-2277, (1996).
2. Callis & Abernathy, High-Precision Isotopic Analyses of Uranium and Plutonium by Total Sample Volatilization and Signal Integration., International Journal of Mass Spectrometry and Ion Processes, 103, pp. 93-105, (1991).
3. Duckworth, Borshick & Smith, Analysis of Soils by Glow-Discharge Mass-Spectrometry, Journal of Analytical Atomic Spectrometry, Vol. 8, pp. 875-879, (1993).
4. Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p. 409, (1995).
5. Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996).
6. Chauvenet & Morel, "Intercomparaison des mesures de Composition Isotopique du Plutonium par Spectrometrie X et Gamma Resultats de L'action 'Pidie,' Raport Final," report CEA-R-5582.

A

Statement of Work

Idaho National Engineering and Environmental Laboratory
Nondestructive Waste Assay
Capability Evaluation Project

Working Reference Material
Production

Increased Am/Pu and DU WRMs

Revision 1.15

July 24, 1997

1. Introduction

Non-destructive waste assay (NDA) methods are used in the National TRU Waste Characterization Program to determine the mass and associated alpha activity of waste entrained radionuclides. The capability and performance of waste NDA systems employed in the Program must comply with requirements as set forth in the TRU Waste Characterization Program Quality Assurance Program Plan (QAPP)¹. As part of the overall effort to assess waste NDA system utility and capability, the Idaho National Engineering and Environmental Laboratory (INEEL) Mixed Waste Focus Area (MWFA) has developed a plan to acquire capability/performance data on systems proposed for nondestructive assay characterization purposes. Such systems include those purveyed by commercial entities as well as Department of Energy (DOE) developed systems. The INEEL waste NDA system Capability Evaluation Project (CEP), scheduled to commence in August of 1997, is not designed to certify waste NDA systems per QAPP waste NDA requirements. Rather the goal of the CEP is to acquire objective data/information useful for determining the potential utility of a given system benchmarked to QAPP quality assurance objectives for specified waste type subpopulations.

The CEP test sequence subjects all participants to identical test sample configurations thereby allowing an assessment of NDA system performance relative to a common basis. The common basis is derived from test sample configurations comprised of surrogate and/or actual

waste containers selected from the inventory at the INEEL Radioactive Waste Management Complex (RWMC). The surrogates are specified, and the actual RWMC waste from drums are selected, to test fundamental waste NDA system properties and performance relative to actual waste form attributes.

A fraction of the test samples are derived from surrogate waste matrices combined with known quantities and distributions of radioactive material. Due to limited radioactive standard set present at the INEEL RWMC, additional standards are required to meet the objective of the INEEL Capability Evaluation Project. These standards, hereafter referred to as Working Reference Materials (WRMs), are specified to complement both existing and scheduled fabrication of standards made available through the Nondestructive Assay Performance Demonstration Program².

2. Objectives

The objective of this work statement is to support implementation of the INEEL Nondestructive Waste Assay CEP project through the production of an Increased Am/Pu mass ratio (IAP) and depleted uranium (DU) WRM set. The CEP IAP/DU WRM set provides radioactive material standards for use in combination with 55 gallon drum waste matrix surrogates. The CEP IAP/DU WRM set allows an assessment of system capability and performance specific to waste types/configurations containing increased amounts of ²⁴¹Am relative to weapons grade Pu, (i.e. ²⁴¹Am/WG Pu mass ratios greater than 0.2%), wastes containing principally ²⁴¹Am, and wastes containing DU of varying weight proportions relative to other radionuclides. The specifications for the enhanced ratios and americium masses used to fabricate the CEP IAP/DU WRM set are derived from knowledge of radionuclidic compositions of actual waste forms. The CEP IAP/DU WRM set can be utilized in combination with other NDA PDP WRM types, e.g. NDA PDP Phase I/II.A weapons grade specification WRMs to achieve a variety of source configurations thereby enhancing the assessment variable range of the INEEL CEP program.

3. Scope

A total of sixteen CEP IAP/DU WRMs are to be produced to achieve the objectives of and support implementation of the INEEL Capability Evaluation Project. Ten of the CEP WRMs are of an IAP composition

designed to complement the WRM attributes specified for the NDA PDP Phase III.A production campaign. The remaining six CEP WRMs are of a DU composition provided to assess system capability and performance for waste forms which contain radionuclidic compositions comprised of DU in various combinations with other radionuclides and waste forms of a predominantly DU composition.

The CEP IAP/DU WRM production techniques and procedures are analogous to those prepared and employed in the NDA PDP Phase I WRM set in that the principle attribute varied for the CEP IAP Project WRM set is the addition of ^{241}Am to WG Pu. The DU WRMs are also similar to the Phase I WRMs in that the principle attribute varied is the type of the radioactive material. To this extent the CEP IAP/DU WRM production specifications and task outline is similar in content and structure to the PDP WRM Phase I statement of work³.

A fundamental requirement of all phases of the CEP IAP/DU WRM production project driving scope definition and the associated project task specification, is the need to ensure the completed WRMs have pedigrees specifying a primary attribute traceable to a recognized reference base. The precision and accuracy of the attribute value is to be commensurate with the intended application, i.e. qualification of measurement methods/systems. To this end all pertinent aspects of the production of the CEP IAP/DU WRM set requires carefully specified quality assurance and control measures to ensure data supporting attribute quantification, uncertainty and traceability are produced, analyzed and documented in an auditable form.

The overall project scope is delineated as a set of general tasks as follows:

- (1) review CEP IAP/DU WRM data quality objectives,
- (2) production of the Phase CEP IAP/DU WRMs,
- (3) assignment of a CEP IAP/DU WRM mass/alpha activity values and uncertainties,
- (4) documentation of the CEP IAP/DU WRM production plan,
- (5) preparation of CEP IAP/DU WRM content and traceability certificates,
- (6) waste NDA physics support/review of fabrication process and resulting CEP IAP/DU WRMs,
- (7) packaging/transportation of the CEP IAP/DU WRMs to the INEL RWMC facility.

The capability to produce the CEP IAP/DU WRM set requires expertise in the area of waste NDA expert and the objectives of the capability evaluation project. Successful completion of the project necessitates that an individuals of this capacity interface with the LANL CST-8 technical production team as issues regarding waste NDA measurements arise. The scope of work for this preselected complement of personnel and resources is outlined in the general task tabulation above. LMITCO will specify WRM data quality objectives and is responsible for coordination, technical/managerial review of subcontractor performed activities, and completion of the indicated tasks.

4. Technical Tasks

The technical tasks address the completion of the sixteen CEP IAP/DU WRM set. The technical tasks are dependent on the data quality objectives (DQOs) specified for the project. For this reason the DQOs are presented first, followed by the task breakdown.

Data Quality Objectives

- 4.1 Produce sixteen CEP IAP/DU WRMs of the following quantity/mass denominations:

IAP WRMs

- 4.1.a) two WRMs with an ^{241}Am mass in the range of (8 - 11) milligrams combined with WG Pu in the mass range of (60 - 110) milligrams,
- 4.1.b) two WRMs with an ^{241}Am mass in the range of (45 - 55) milligrams combined with WG Pu in the mass range of (1.50 - 2.50) grams,
- 4.1.c) two WRMs with an ^{241}Am mass in the range of (85 - 95) milligrams combined with WG Pu in the mass range of (1.50 - 2.50) grams,
- 4.1.d) two WRMs with an ^{241}Am mass in the range of (45 - 55) milligrams combined with WG Pu in the mass range of (0.3 - 0.5) grams,
- 4.1.e) two WRMs with an ^{241}Am mass in the range of (95 - 105) milligrams.

Note: maximum allowable total uncertainty at the 95% confidence level per CEP IAP WRM as specified in items 4.1.b through 4.1.e is $\pm 0.5\%$ in terms of WRM alpha activity. Due to limitations in mass determinations for 4.1.a ^{241}Am and Pu components of the WRM the allowable alpha activity uncertainty is relaxed to $\pm 0.75\%$ at the 95% confidence level.

DU WRMs

- 4.1.f) one WRM with a DU mass in the range of (490 - 510) grams,
- 4.1.g) one WRM with a DU mass in the range of (850 - 900) grams,
- 4.1.h) four WRM with a DU mass in the range of (925 - 1000) grams.

Note: maximum allowable total uncertainty at the 95% confidence level per DU WRM as specified in items 4.1.f through 4.1.h is $\pm 0.5\%$ in terms of WRM DU mass.

- 4.2 Item (4.1) criteria is provided to specify an acceptable range within which a suitable ^{241}Am , WG Pu and DU mass value can be realized in consideration of production procedures. The ^{241}Am , WG Pu and DU mass ranges are not to be construed as the allowable uncertainty per IAP or DU WRM.
- 4.3 For Project purposes, the CEP IAP/DU WRMs must be traceable to a certificate or other documentation that is issued by a technically competent certifying body such as the New Brunswick Laboratory. The waste NDA CEP project need is for traceable standards in accordance with the reference material hierarchy level classified as WRMs. The WRM level specification is based on the intended application, i.e., qualification of a measurement method.⁴ The IAP/DU WRMs are not intended to be used for calibration purposes. Hence all aspects of the planning, preparation, fabrication, packaging, characterization, statistical analyses, and documentation shall be consistent with the rigor and completeness typical of establishing and demonstrating traceability for WRMs. It is the responsibility of LMITCO to ensure that project production procedures, quality assurance measures, and documentation are adequate to demonstrate that degree of WRM credibility needed to support

the intended application, measurement system qualification.

5. Project Tasks/Requirements

Project tasks/requirements/documentation to be performed by the LANL CST-8 group are largely based on techniques developed and approved for the Phase I PDP WRM production project. The CEP IAP/DU WRM Production Plan will detail the indicated tasks and fully document the embedded activities and derived data. Where indicated, documented procedures or descriptions necessary to sufficiently detail the indicated tasks must be prepared and submitted by LANL to LMITCO for review and concurrence prior to commencement of the activity.

- 5.1 General - Establish in advance required resources and personnel to complete the production and delivery of the CEP IAP/DU WRMs in accordance with the specified completion date; July 21, 1997. Ensure contingency plans have been considered in the event of unanticipated delays. Notification of LMITCO of the occurrence of delays impacting the completion date is required. Production unknowns and open-ended technical issues to be identified and resolved by production team prior to commencement of fabrication activities.
- 5.2 Prepare and submit a short form project quality assurance plan in accordance with requirements as provided by LMITCO.
- 5.3 Prepare and submit a short form project management plan, requires LMITCO concurrence.
- 5.4 Prepare and submit documentation control and records management plan. Records repository at CEP IAP/DU WRM project completion is the MWFA at LMITCO.
- 5.5 Develop CEP IAP/DU WRM Production Plan complete with all ²⁴¹Am, Pu and DU feedstock characterization, chemical and physical manipulations to support the process of feedstock fixation/encapsulation, completed assembly characterization and documentation/quality control measures. The production plan shall accommodate the following IAP/DU WRM material attribute requirements:

WRM Attribute Requirements

IAP WRMs

- 5.6 Isotopic/radionuclidic composition of the Pu feedstock component of the IAP WRM at the time of incorporation into the WRM must be within the following nominal ranges:

Table 1. Allowable Pu Isotopic/nuclidic Composition Range

Radionuclide	Mass fraction range (%)
^{238}Pu	0.01 - 0.02
^{239}Pu	93.0 - 94.0
^{240}Pu	5.0 - 6.0
^{241}Pu	0.1 - 0.25
^{242}Pu	0.02 - 0.06
^{241}Am	0.085 - 0.25

- 5.7 Chemical composition of Pu feedstock to be PuO_2 . Certification of elemental impurity concentration must be provided and consistent with Table 2 limits below. Other impurity elements will be considered on a case by case basis.

Table 2. Allowable PuO_2 Feedstock Impurity Limits

Impurity Element	Concentration Limit (ppm)
Fe + Ni	<400
Be	<2
Mg	<500
Ca	<1000
Np	<100
Th	<50

Ga	<25
F	<85
Cl	<70
Si	<300
Al	<50
U	<500
²⁴⁴ Cm	<1
²⁵² Cf	<0.5
any significant individual remaining impurity	<100

- 5.8 WG PuO₂ physical form to be particulate with a particle size distribution mean less than 100 microns and no more than 2% of the PuO₂ particle population exceeding 250 microns in diameter. An evaluation of the PuO₂ particle size data is required prior to continuance of the fabrication process. (QA documentation required).
- 5.9 Isotopic/radionuclidic composition of the Am feedstock component of the IAP WRM at the time of incorporation into the WRM must be within the following nominal ranges:

Table 3. Allowable Am Isotopic/nuclidic Composition Range

Radionuclide	Mass fraction range (%)
²⁴¹ Am	99.9 - 100
²⁴² Am	< 0.001
²⁴³ Am	< 0.001

- 5.10 Pu component of Am feedstock to be accounted for in complying with WRM data quality objective specifications

item 4.1.

- 5.11 Chemical composition of Am feedstock to be Am oxide. Certification of elemental impurity concentration must be provided and consistent with Table 4 limits below. Other impurity elements will be considered on a case by case basis.

Table 4. Allowable Am Oxide Feedstock Impurity Limits

Impurity Element	Concentration Limit (ppm)
Fe + Ni	<400
Be	<2
Mg	<500
Ca	<1500
Np	N/A ¹
Th	<50
Ga	<25
F	<85
Cl	<70
Si	<300
Al	<150
U	<500
²⁴⁴ Cm	<1
²⁵² Cf	<0.5
any significant individual remaining impurity	<100

1 - the fraction of the total alpha activity in any given WRM attributable to Np²³⁷ is on the order of 10⁻⁶ and hence does not affect performance assessment considerations; additionally its concentration varies with time as it is a daughter of Am²⁴¹ making it difficult to specify an impurity limit on concentration.

- 5.12 Am oxide physical form to be particulate with no more than 2% of the Am oxide particle population exceeding 250 microns in diameter. This specification may be met by sieving the Am oxide through an appropriate sized sieve.
- 5.13 The Am/Pu diluent is to be comprised of diatomaceous earth of the same quality and composition as utilized in the fabrication of the NDA PDP Phase II.A WRM set.
- 5.14 Uniformity of the IAP WRM Am/Pu diluent mixture throughout inner cylinder stainless steel encapsulation volume to be within $\pm 2.5\%$ of mean concentration for any given 30 cm³ volume for each mass range delineated in item 4.1. (QA checkpoint)

DU WRMs

- 5.15 Isotopic/radionuclidic composition of the DU feedstock at the time of incorporation into the WRM must be within the following nominal ranges:

Table 5. Allowable DU Isotopic/nuclidic Composition Range

Radionuclide	Mass fraction range (%)
²³⁴ U	0.000 - 0.001
²³⁵ U	0.10 - 0.30
²³⁶ U	0.001 - 0.006
²³⁸ U	99.6 - 99.9

- 5.16 Chemical composition of DU feedstock to be U oxide. Certification of elemental impurity concentration must be provided and consistent with Table 6 limits below. Other impurity elements will be considered on a case by case basis.

Table 6. Allowable DU Oxide Feedstock Impurity Limits

Impurity Element	Concentration Limit (ppm)
Al	<200

B	<350
Be	<1
Ca	<200
Cl	<70
F	<85
Cd	<2
Fe + Ni + Cr	<250
Li	<1
Mg	<50
Na	<200
Si	<300
Pb	<20
W	<350
V	<100
²⁴⁴ Cm	<1
²⁵² Cf	<0.5
any significant individual remaining impurity	<100

- 5.17 DU oxide physical form to be particulate with a particle size distribution mean less than 100 microns and no more than 2% of the DU oxide particle population exceeding 250 microns in diameter. An evaluation of the DU oxide particle size data is required prior to continuance of the fabrication process. (QA documentation required).
- 5.18 The DU oxide diluent is to be comprised of diatomaceous earth of the same quality and composition as utilized in the fabrication of the NDA PDP Phase II.A WRM set.

- 5.19 Uniformity of the DU oxide/diluent mixture throughout inner cylinder stainless steel encapsulation volume to be within $\pm 5.0\%$ of mean concentration for any given 30 cm³ volume for each mass range delineated in item 4.1. (QA checkpoint)

WRM Production Requirements

Prior to commencing the production of the as specified IAP WRMs, scoping studies regarding WRM configuration gas generation and (α, n) reaction yields are required. Gas generation data can be acquired through computational studies and from existing studies where an appropriate margin of safety is represented in the data relative to the IAP WRM configuration. The gas generation data shall be reviewed by LMITCO and concurrence acquired before commencing production. The data may require supplemental experimental data to conclusively ensure gas generation does not compromise the value and safety of the IAP WRMs. Such being the case, LANL CST-8 will collaborate with the LMITCO technical representative to devise an appropriate test.

In addition to gas generation considerations, the IAP WRM design may complicate the the CEP NDA capability evaluation process due to the potential for high (α, n) yields resulting from the blend process. In order to assess the magnitude of this effect in the specified WRM configuration, measurements of a test blend is required. A technical review of the resulting measurement data is required. Based on the results of such measurements, modification to the radioactive Am/Pu substrate may be required in some of the IAP WRM mass ranges.

The following is the general production sequence with specific quality assurance or test case holdpoints for technical review.

- 5.20 Process PuO₂, Am oxide and DU oxide feedstock as appropriate and characterize isotopic, chemical, and elemental impurity homogeneity throughout the batch. Accepted methods of establishing these parameters for PuO₂ and Am oxide are as determined during the Phase I WRM production project as documented in the Phase I NDA PDP WRM Production Plan⁵. Similar methods are required for DU characterization per LMITCO concurrence. Documentation of the quantitation procedure and results is to be provided in the CEP IAP/DU WRM Production Plan. Requirements itemized below:

- 5.20.1 Pu, Am and DU feedstock isotopic/radionuclidic composition analysis, traceable documentation required, technical and QA checkpoint.
- 5.20.2 Pu, Am and DU feedstock chemical composition analysis, traceable documentation required, technical and QA checkpoint.
- 5.20.3 Pu, Am and DU feedstock elemental impurities analysis, traceable documentation required, technical and QA checkpoint.
- 5.20.4 Pu, Am oxide and DU oxide feedstock particle size characterization, documentation required, technical and QA checkpoint.
- 5.21 Determine blend parameters required to achieve the acceptable Am/Pu/diatomaceous earth uniformity as specified in 5.14. Determine blend parameters for the DU/diatomaceous earth mixture per item 5.19. Acquire samples to validate compliance, documentation required - (QA checkpoint).
- 5.22 Procure stainless steel WRM encapsulation assemblies per Phase II NDA PDP WRM Statement Of Work for Stainless Steel Assemblies⁶.
- 5.23 Determine weld configuration and associated parameters and perform test weld analyses to assure weld integrity. Documentation of weld integrity analysis results required.
- 5.24 A unique indelible identification number is to be marked on the outer stainless steel encapsulation assembly endcaps. The identification number is to be a physical attribute of the material, e.g. chemically etched or embedded, into the external surface of the selected endcaps. The height of the identification numbers/characters are to occupy a maximum area on the outer cylinder endcaps without compromising endcap weld regions. The identification serial numbers of the thirteen IAP/DU WRM encapsulation assemblies are as follows: CEP001, CEP002, ... CEP016. WRM specific identifications are as tabulated below:

Table 7. CEP WRM Identification

CEP SOW WRM Specification Item	Identification
4.1.a -- WRM 1	CEP001
4.1.a -- WRM 2	CEP002
4.1.b - WRM 1	CEP003
4.1.b - WRM 2	CEP004
4.1.c - WRM 1	CEP005
4.1.c - WRM 2	CEP006
4.1.d - WRM 1	CEP007
4.1.d - WRM 2	CEP008
4.1.e - WRM 1	CEP009
4.1.e - WRM 2	CEP010
4.1.f - WRM 1	CEP011
4.1.g - WRM 1	CEP012
4.1.h - WRM 1	CEP013
4.1.h - WRM 2	CEP014
4.1.h - WRM 3	CEP015
4.1.h - WRM 4	CEP016

- 5.25 Quantitatively dispense IAP/diatomaceous earth prepared blend into inner stainless steel cylinder/endcap assembly (QA documentation required), install graphite felt pad, and weld inner top endcap in place. Deburr and smooth external surface of welded inner cylinder to specified dimensions.
- 5.26 Quantitatively dispense DU/diatomaceous earth prepared blend into stainless steel cylinder/endcap assembly (QA documentation required), install graphite felt pad, and weld top endcap in place. The exterior surface of the DU WRM encapsulation cylinder is then finished with particular

attention being given to the weld point to ensure the O.D. of the tube does not exceed 1.955 inches.

- 5.27 Perform helium leak test on IAP inner cylinder at 10^{-8} atm-cc/sec, QA documentation required. Data generated during weld parameter determinations, associated evaluations and any nondestructive testing is to be maintained to support proof of seal integrity.
- 5.28 Perform helium leak test on DU single encapsulation cylinder at 10^{-8} atm-cc/sec, QA documentation required. Data generated during weld parameter determinations, associated evaluations and any nondestructive testing is to be maintained to support proof of seal integrity.
- 5.29 Insert IAP assembled inner stainless steel cylinder into outer cylinder and weld top outer endcap in place. The exterior surface of the outer tube is then finished at the weld point to ensure the O.D. of the tube at the weld location does not exceed 1.955 inches.
- 5.30 Determine Am and Pu mass and alpha activity values and associated uncertainties for each radionuclidic/isotopic feedstock component, i.e. Am and Pu, individually and combined for each IAP WRM. Assemble all pertinent QA records and statistical analyses used in the mass/activity value/uncertainty determination, and establish traceability of assigned WRM values to recognized reference base.
- 5.31 Determine DU mass and associated uncertainty for each radionuclidic/isotopic feedstock component individually and combined. Assemble all pertinent QA records and statistical analyses used in the mass value/uncertainty determination, and establish traceability of assigned WRM values to recognized reference base.
- 5.32 Perform WRM attribute verification measurements on the completed units as specified by LANL and concurred with by LMITCO. Attributes to be verified will be specified by LANL and LMITCO, i.e., vertical IAP and DU WRM uniformity. Include results of verification in CEP IAP/DU WRM Production document.

- 5.33 Prepare WRM Certificates of Content and Traceability and SNM accountability documentation.
- 5.34 Perform internal independent physics review of completed WRMs.
- 5.35 Acquire transport containers, package and ship CEP WRMs to the INEEL RWMC site contact.
- 5.36 Formally transmit all records and documentation to the INEEL MWFA LMITCO project management office.
- 5.37 Prepare a CEP IAP/DU WRM Production document providing details of the production/fabrication process, mass value determination method and data and traceability process. Method and document is to be reviewed and concurred with by LMITCO and to serve as the source of certification for the CEP IAP/DU WRMs.

6. Delivery/Schedule

- 6.1 Submit QAPjP to LMITCO for review and concurrence by February 14, 1997.
- 6.2 Submit Project Management Plan to LMITCO for review and concurrence by February 14, 1997.
- 6.3 Submit Records Management Plan to LMITCO for review and concurrence by February 14, 1997.
- 6.4 Complete CEP IAP/DU WRM characterization, fabrication, and corresponding QA check-point documentation of the IAP/DU WRM set by June 27, 1997.
- 6.5 Complete draft WRM certificates of traceability and transmit to LMITCO contact on or before July 15, 1997.
- 6.6 Complete transportation of CEP IAP/DU WRMs to LMITCO specified facility before July 21, 1997.
- 6.7 Submit Draft CEP IAP/DU WRM Production Plan to LMITCO for review and concurrence by August 18, 1997.

- 6.8 Transmit Final CEP IAP/DU WRM Production Plan and documentation to LMITCO before September 17, 1997.

7. References

- 7.1 *Transuranic Waste Characterization Quality Assurance Program Plan, Revision 0*, CAO-94-1010, April 30, 1995, U.S. Department of Energy, Carlsbad Area Office, National TRU Program Office.
- 7.2 *Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program, Revision 0*, CAO-94-1045, March 1995, U.S. Department of Energy, Carlsbad Area Office.
- 7.3 *Statement of Work, Non-Destructive Assay Performance Demonstration Plan, Phase I Working Reference Material Production, Revision 1.3*, October, 1995, Lockheed Martin Idaho Technologies Company.
- 7.4 *Standard Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Cycle Materials*, ASTM Designation: C 1128-89, American Society for Testing and Materials, Philadelphia, PA.
- 7.5 *Phase I NDA PDP WRM Production Plan*, CST-8-PLA-STD-1-1/Rev2, June 24, 1996 version, Los Alamos National Laboratory.
- 7.6 *Phase II NDA PDP WRM Statement Of Work for Seamless Stainless Steel Tubing Assemblies*, LANL/CST-8, Document # PLA-STD-203/0. November 8, 1995.

B

Los Alamos

NATIONAL LABORATORY

memorandum

Nuclear Materials Processing -
Actinide Process Chemistry
NMT-2

To/MS: R. Marshall, CST-8 G740

Thru: J. D. Williams, NMT-2 Group Ldr.

T. A. Hayes, Team Ldr.

From/MS: T. E. Ricketts, NMT-2 E511

Phone/FAX: 5-4060/FAX 5-1780

Symbol: NMT-2:FY95-394

Date: June 27, 1995

SUBJECT: PLUTONIUM DIOXIDE PREPARATION PROCEDURE AND CHARACTERIZATION RESULTS

I have attached a comprehensive data package which discusses the procedures and operating parameters used to prepare the selected plutonium dioxide item to the agreed upon specifications. I have also included the loss-on-ignition (LOI) and particle size/surface area results as part of the characterization efforts by CST-15 and NMT-2 analytical capability. To complete the data package, I have included a spreadsheet that calculates the mass percent of the individual particles, as well as cumulative, from the particle size distribution analysis.

Due to the dedicated efforts of NMT-2 processing personnel in room 208, we were able to meet the extremely short deadlines associated with this project. Efforts put forth by Charlie Davis, CST-15, and Greg Bird, NMT-2, should also be acknowledged. Their hard work provided almost real-time analysis capability for the samples submitted for LOI and particle size/surface area analysis.

I have enjoyed working with you on this project and if you should have any questions or comments regarding the attached information, please do not hesitate to contact me. I look forward to our future interactions under phase II of this project.

Distribution: w/o attachments

B. McKerley, CST-8 G740

C. Leasure, CST-15 E531

C. Davis, CST-15 E531

G. Bird, NMT-2 E511

M. Romero, NMT-2 E511

Author

PLUTONIUM DIOXIDE PREPARATION

The information presented in this document discusses the procedure and operating parameters used in the processing and characterization of selected plutonium dioxide item (52/PEOR3258). The procedure and parameters for this project were agreed upon in a meeting with R. Marshall, CST-8, on May 22, 1995. This material is going to be used to fabricate standards which will be used to certify NDA instruments measuring waste items destined for WIPP.

All work, associated with the processing of this material, was performed in Room 208, P/S RBJ in glovebox G221.

May 22, 1995:

- Item 52/PEOR3258 was selected for processing and characterization based upon three factors: 1) the purity of material (86.9 weight % Pu); 2) the quantity of material (~1.7 Kgs net weight); and 3) the age of the item (~ 7 yrs old).

May 23, 1995:

- Retrieved selected item from the vault. See attachment I for item status.
- Introduced item in-line.
- Transferred item to glovebox G221.
- The material was initially screened using a 40 mesh sieve - Tyler Equivalent 35. The sieve openings are 0.0165 in./425 μ m.

June 23, 1995

- To assure homogeneity the material was V-blended for approximately 30 minutes.
- The material was then transferred to a fused silica furnace boat for calcination.

The typical composition of a fused silica furnace boat is:

SiO ₂ - 86.7%	Al ₂ O ₃ - 10.4%
Fe ₂ O ₃ - 0.34%	TiO ₂ - 0.05%
CaO + MgO - 0.15%	Na ₂ O + K ₂ O - 0.41%

- The boat and material were placed into a Lindberg furnace (Model 51894) and the following furnace controller program was used for calcination.

<u>Set Pt</u>	<u>Temperature</u>	<u>Time</u>
0	75°C	-
1	200°C	1 Hr
2	400°C	1 Hr
3	600°C	1 Hr
4	800°C	1 Hr
5	980°C	1 Hr
6	980°C	2 Hr
7	700°C	2 Hr
8	500°C	2 Hr
	OFF	

Furnace Started at 3:30 PM and allowed to run overnight.

May 24, 1995:

- Appeared to have a furnace malfunction. See attachment II for printout of furnace temperature profile.

The malfunction was such that the material was calcined between 900 and 960°C for approximately nine hours instead of the programmed 980°C for two hours. After a brief consultation with R. Marshall, it was agreed that the increased time of calcination had no detrimental effect upon the material, perhaps, it even was beneficial considering the intended use of this material.

Evaluation: Ramp-up to 800°C was as anticipated (~5 Hrs).
Ramp-up from 800 to 900°C took ~ 2 Hrs.
Ramp-up from 900 to 950°C took ~3 Hrs.
Ramp-up from 950 to 960°C took ~ 6 Hrs.

Furnace was turned off at 7:20 am after roughly 16 hours in the calcination cycle. Nine of those hours were at 900°C or greater.

- Opened door on furnace to allow rapid heat dissipation. This allowed adequate cooling such that the boat and material was cool enough to be handled in a relatively short period of time (~4 - 6 Hrs).
- Approximately half of the material (roughly 1.0 Kg) was screened using a 80 mesh sieve - Tyler Equivalent 80 and collected in a clean catch pan. The sieve openings are 0.007 in./180µm. The material screened relatively easy which indicated that the majority of the particles were ~200µm or less.

NOTE: The portion of the material selected for the screening was obtained by taking four large scoops of material from different locations in the furnace boat. The material was transferred to the sieve, "mixed" and screened.

- The screened material was then sampled:

* A ten gram sample (PEOR3258-S2) was collected and sent for LOI analysis by G. Bird and C. Davis.

June 23, 1995

- * A three gram sample (PEOR3258-S3) was collected and sent for particle size and surface area analysis by G. Bird and C. Davis.

NOTE: Any material which touched the glovebox floor was treated as sweepings.

- After the material was sampled, 120 gms (net weight) was obtained and packaged to be sent to R. Marshall for use as standard material. The item was created as 52/PEOR3258-STD1.

The conditions of the glovebox in which the handling of the material occurred was:

Atmosphere: Air
Relative Humidity: ~8.0%
Temperature: 77.6°F/24.8°C
Dew Point: 13.3°F/-10.4°C

May 25, 1995:

- Item PEOR3258-STD1 was removed from the glovebox line and sent to the sample room.
- The parent item was sent down to the vault (P/S ST - Standards) with the following comments added to item remarks (see attachment III):
 - * Oxide calcined at greater than 900°C for approximately 9 hours.
 - * Standard material save for Ricketts/Marshall.

RESULTS:

- The ten gram sample (PEOR3258-S2) was split into two five gram aliquots and each aliquot had a LOI analysis. The average LOI for the two aliquots was 0.004%. See attachment IV.
- The three gram sample (PEOR3258-S3) was split, using a riffler, into three ~0.2 gram aliquots. Each aliquot had a particle size and surface area analysis.

* Particle Size

Cut 1 : Spherical Equivalent Mean - $34.1\mu\text{m}$
Cut 2 : Spherical Equivalent Mean - $34.4\mu\text{m}$
Cut 3 : Spherical Equivalent Mean - $32.5\mu\text{m}$

* Surface Area (3 point BET)

Cut 1: 2.45 sq. m./gm
Cut 2: 2.25 sq. m./gm

Due to time constraints, Cut 3 was not able to be analyzed.

See attachment V for detailed particle size and surface area results.

- The spreadsheet in Attachment VI converts the particle size distribution information to a mass percent. As one might expect, there is a relatively large percentage of particles under the $30\mu\text{m}$ size, however, the bulk of the mass consist of particles greater than $30\mu\text{m}$.

Past experiments with similar material (oxide from a nitrate based ion exchange process) indicate that the maximum adsorption after calcination to $\sim 950^{\circ}\text{C}$ in a glovebox of less than $\sim 30 - 40\%$ RH would be $\sim 0.2\text{ mg H}_2\text{O/gm PuO}_2$ or 0.02% LOI.

June 23, 1995

Attachment I

Item Status of PEOR3258 as Retrieved from the

Vault (Acct 700)

WJG
6/23/95
<<< ACCOUNT (722)
<<< MATERIAL TYPE (52)
<<< LOTID 7 PEOR3258
ITEM DOES NOT EXIST
<<< ACCOUNT (722)? 700
<<< MATERIAL TYPE (52)?
<<< LOTID 7?? PEOR3258

B77
GWT - 2241.3
TWT - 2185.6
1755.7

INVENTORY ITEM -----, 5/23/1995

ACCT MT LOT-IDENTIFICATION CONTAINER-ID
700 52 PEOR3258

NM ENRICH ISO NET PKGD WT SEAL #
1524.0 6 6.37 1427.0 1753.5 6 3.4 KG 074047

LOCA SHELF P/S PROJ NMGT SD MEAS IDES COEI AILEV
6190 1 40 345 DOSE C05 C217 C00 C

LAST TRAN # CREATION: PERSON DATE TIME
406333 88602 PRE 8/10/1988 6:10:56

ITEM REMARKS

1 %PU=86.9;NI=430;SI=180;MO=300-3000;CR=250(PPM) UNKNOWN 0/ 0/ 0
<<< ACCOUNT (700)?

June 23, 1995

Attachment I

Item Status of PEOR3258 as Retrieved from the

Vault (Acct 700)

W 6/23/95
ACCOUNT (77 722
MATERIAL TYPE (77 52
LOTID >>> PEOR3258
ITEM DOES NOT EXIST
ACCOUNT (72277 700
MATERIAL TYPE (5277
LOTID >>> PEOR3258

87% GWT - 2241.3
TWT - 2185.6
1765.7

INVENTORY ITEM ----- 5/23/1995
ACCT MT LOT-IDENTIFICATION CONTAINER-ID
700 52 PEOR3258

NM	ENRICH	ISU	NET	PKGD WT	SEAL #
1524.0	6	6.37	1427.0	1753.5 6	3.4 KG 074047

LOCA	SHELF	P/S	PROJ	NMGT	SD	MEAS	IDES	COEI	ATLEV
619D	1	40	345	DOSE		005	0217	000	0

LAST TRAN #	CREATION: PERSON	DATE	TIME
406533	38502 PRE	8/10/1988	6:10:56

ITEM REMARKS
1 %PU=86.8;NT=430;SI=160;MU=300-3000;CR=250(PPM) UNKNOWN 0/ 0/ 0
ACCOUNT (70077

June 23, 1995

Attachment II

Furnace Temperature Profile

OFF VIBRATION MKR
STUNTS

DRY CRACKS

DOOR CLOSED

5/24/95 7:20 AM TURNED OFF MKR

June 23, 1995

Attachment III

Item Status of PEOR3258 as Sent to the

Vault (Acct 700)

6/22/95

ESC...TO EXIT

<<< ACCOUNT ()? 700
<<< MATERIAL TYPE ()? 52
<<< LOTID >>> PEOR3258

INVENTORY ITEM

6/22/1995

ACCT MT LOT-IDENTIFICATION CONTAINER-ID
700 52 PEOR3258

NM	ENRICH	ISO	NET	PKGD	WT	SEAL #
1402.0	G	5.37	1313.0	1513.0	G 0.0	G 010366V

LOCA	SHELF	P/S	PROJ	NMGT	SD	MEAS	IDES	COEI	ATLEV
H09F	I	ST	345	DUSE		B62	C217	C00	C

LAST TRAN # CREATION: PERSON DATE TIME
620239 38602 PRE 8/10/1988 6:10:56

ITEM REMARKS

1. %PU=86.9;NI=430;SI=190;MO=300-3000;CR=250(PPM)	UNKNOWN	0/ 0/ 0
2> OXIDE CALCINED AT GREATER THAN 900C FOR	081579 RNH	6/22/1995
3> APPROXIMATELY NINE HOURS (5/24/95)	081579 RNH	6/22/1995
4> STANDARD MATERIAL SAVE FOR RICKETTS/MARSHALL	081579 RNH	6/22/1995
<<< ACCOUNT (700)?		

980°C
Calcination283-MRD-DR-R00
PAGE 1 OF 1

DETERMINATION OF LOSS-ON-IGNITION OF PLUTONIUM DIOXIDE OR OTHER
PLUTONIUM-CONTAINING POWDERS - DATA RECORD

SAMPLE ID PEOR 3258-S2 DATE 5/25/95
FURNACE CALIBRATION NO. 638783
CALIBRATION EXPIRATION DATE 4/27/96
IGNITION TEMPERATURE 950 °C IGNITION TIME 4 HRS.

ALIQUOT #1
CRUCIBLE NO. 58

ALIQUOT #2
CRUCIBLE NO. 59

SAMPLE WEIGHT & TARE

BEFORE IGNITION (B1T1) 32.3021 g. (B2T2) 32.6708 g.
TARE (T1) 27.2854 g. (T2) 27.6468 g.
INITIAL SAMPLE WEIGHT (B1) 5.0167 g. (B2) 5.0240 g.

SAMPLE WEIGHT & TARE

AFTER IGNITION (A1T1) 32.3020 g. (A2T2) 32.6705 g.
TARE (T1) 27.2854 g. (T2) 27.6468 g.
FINAL SAMPLE WEIGHT (A1) 5.0166 g. (A2) 5.0237 g.

PERCENT LOSS (L1) .00199 % (L2) .00597 %

AVERAGE LOSS ON IGNITION .00398 % ALIQUOT SPREAD .00398 %

OPERATOR SIGNATURE Greg Bird DATE 5/25/95

CHECKED BY Harlan P. Quinn DATE 5/25/95

LAP-TEC PARTICLE ANALYSIS SUMMARY

Ver. 5.20.07

CST-15/NMT-2 PARTICLE SIZE ANALYSIS - C.C.DAVIS/C.D.BIRD
PEOR3258-S3

Sample ID : PEOR3258-S3 CUT 1
CF Code :
Date : 01/03/80
Time : 12:52:49
Spherical Equivalent Mean : 34.1
Standard Deviation : 34.2
Skewness : 0.3
Kurtosis : 7.9
Coeff. of Variance(%) : 100.5
Counts : 12632

Cycles : 19 M.Cycle Time: 1.0 sec.
Scaling : 1.00 Averaged last 5 cycle(s)

Channel Size (microns)	%/Channel	Channel Size (microns)	%/Channel
<0.7	4.7770	25	7.5508
0.7	0.7980	31	6.0979
1.0	1.0411	37	5.9798
1.3	0.7993	44	6.4852
1.8	0.7440	53	5.9660
2.3	1.4872	63	4.6656
3.0	2.3218	75	3.5228
4.0	2.5732	88	3.0044
5.0	3.1904	105	2.1969
6.5	4.6832	125	1.3944
8	6.0118	149	0.6472
10	5.9624	177	0.2581
13	4.9940	210	0.1136
17	5.6922	250	0.0683
20	6.9732		

Charles Davis 5/25/95

1.67

CST-15/NMT-2 PARTICLE SIZE ANALYSIS - C.C.DAVIS/C.D.BIRD
PEOR3258-S3

Sample ID : PEOR3258-S3 CUT 2
CF Code :
Date : 01/03/80
Time : 13:14:04
Spherical Equivalent Mean : 34.4
Standard Deviation : 35.2
Skewness : 0.3
Kurtosis : 8.3
Coeff. of Variance(%) : 102.1
Counts : 12465

Cycles : 20
Scaling : 1.00

M.Cycle Time: 1.0 sec.
Averaged last 5 cycle(s)

Channel Size (microns)	%/Channel	Channel Size (microns)	%/Channel
<0.7	4.9393	25	6.9558
0.7	0.7777	31	6.3888
1.0	1.0566	37	6.1243
1.3	0.8441	44	6.2789
1.8	0.8585	53	5.3691
2.3	1.6496	63	4.9755
3.0	2.3033	75	3.8224
4.0	2.5011	88	2.8794
5.0	3.3432	105	2.1527
6.5	4.5332	125	1.6220
8	5.8849	149	0.7387
10	5.7095	177	0.2593
13	5.2201	210	0.0979
17	5.7926	250	0.1201
20	6.8015		

Charles P. Davis 5/25/95

CST-15/NMT-2 PARTICLE SIZE ANALYSIS - C.C.DAVIS/C.D.BIRD
PEOR3258-S3

Sample ID : PEOR3258-S3 CUT 3
CF Code :
Date : 01/03/90
Time : 13:38:38
Spherical Equivalent Mean : 32.5
Standard Deviation : 32.8
Skewness : 0.3
Kurtosis : 8.0
Coeff. of Variance(%) : 101.0
Counts : 13140

Cycles : 20
Scaling : 1.00

M.Cycle Time: 1.0 sec.
Averaged last 5 cycle(s)

Channel Size (microns)	%/Channel	Channel Size (microns)	%/Channel
<0.7	5.1558	25	7.2609
0.7	0.8090	31	6.7465
1.0	0.9999	37	6.3974
1.3	0.7887	44	6.2628
1.8	0.8273	53	5.1573
2.3	1.7379	63	4.2923
3.0	2.2006	75	3.3680
4.0	2.4354	88	2.7182
5.0	3.3165	105	1.8458
6.5	4.9379	125	1.2385
8	5.9001	149	0.5942
10	5.9371	177	0.2733
12	5.3685	210	0.0521
17	6.2354	250	0.0466
20	7.0961		

4.04

Charles C. Davis 5/25/95

Quantachrome Corporation
NOVA-1000 BET Surface Area Analyzer
Version 2.10 21 April 1993

```

User ID      = 2                      Sample ID      = 59.0
User setup   = USMR_5                 Sample Cell Number = 2
Sample Weight = 0.2332 gm              Sample Volume   = 0.0075 cc
Sample Density = 31.0769 gm/cc
PO Type      = USER                   PO              = 603.85 mmHg

Adsorption Tolerance = 0.1000          Desorption Tolerance = 0.1000
Adsorption Equil Time = 60 sec          Desorption Equil Time = 60 sec
Adsorption Dwell Time = 180 sec         Desorption Dwell Time = 180 sec

Analysis started   : Thu May 25 07:18:41 1995
Analysis finished  : Thu May 25 09:07:22 1995
Elapsed time       : 108.68 minutes
  
```

3 Point BET

Target Relative Pressure	Actual Relative Pressure	Total Volume Adsorbed (cc/g)	BET Transform 1/(W[P0/P - 1])
0.1000	0.1031	0.4436	207.3985
0.2000	0.1946	0.5794	333.5587
0.3000	0.2944	0.7152	466.7227

```

Surface Area      = 0.5793 sq. m.
Specific Surface Area = 2.4456 sq. m./gm

Slope             = 1355.655185
Intercept         = 68.361156
Correlation Coefficient = 0.999953
BET C             = 20.830782
  
```

Single Point BET

Target Relative Pressure	Actual Relative Pressure	Total Volume Adsorbed (cc/g)
0.3000	0.2944	0.7152

```

Surface Area      = 0.5122 sq. m.
Specific Surface Area = 2.1964 sq. m./gm
  
```

Quantachrome Corporation
NOVA-1000 BET Surface Area Analyzer
Version 2.10 21 April 1993

```

User ID      = 2                      Sample ID      = 59.1
User setup   = USER_5                Sample Cell Number = 5
Sample Weight = 0.2106 gm             Sample Volume   = 0.0730 cc
Sample Density = 2.9950 gm/cc
PO Type      = USER                  PO              = 603.85 mmHg

Adsorption Tolerance = 0.1000        Desorption Tolerance = 0.1000
Adsorption Equil Time = 60 sec       Desorption Equil Time = 60 sec
Adsorption Dwell Time = 180 sec      Desorption Dwell Time = 180 sec

Analysis started : Thu May 25 09:23:25 1995
Analysis finished : Thu May 25 11:01:09 1995
Elapsed time     : 97.73 minutes
  
```

3 Point BET

Target Relative Pressure	Actual Relative Pressure	Total Volume Adsorbed (cc/g)	BET Transform 1/(W[P0/P - 1])
0.1000	0.1026	0.4398	207.8966
0.2000	0.1938	0.5725	336.0524
0.3000	0.2927	0.6735	491.6950

```

Surface Area      = 0.4746 sq. m.
Specific Surface Area = 2.2535 sq. m./gm

Slope             = 1493.478759
Intercept         = 51.928420
Correlation Coefficient = 0.999462
BET C             = 29.760335
  
```

Single Point BET

Target Relative Pressure	Actual Relative Pressure	Total Volume Adsorbed (cc/g)
0.3000	0.2927	0.6735

```

Surface Area      = 0.4366 sq. m.
  
```

6/23/95

		PEOR3258-S3 (Cut 1) Mass Percent - Calcined to ~950°C											
Assume: Spherical particles													
P.S. (microns)	% Part	Fract Part	(r)	Volume	Vol Contrib	INDIV MASS %	CUM Vol Contrib	CUM Mass Fraction	CUM Mass %				
0.5	4.777	0.048	0.25	0.065	0.003	0.00	0.003	3.33E-08	0.00				
0.7	0.798	0.008	0.35	0.180	0.001	0.00	0.005	4.86E-08	0.00				
1.0	1.041	0.010	0.50	0.524	0.005	0.00	0.010	1.07E-07	0.00				
1.3	0.799	0.008	0.65	1.150	0.009	0.00	0.019	2.06E-07	0.00				
1.8	0.744	0.007	0.90	3.054	0.023	0.00	0.042	4.49E-07	0.00				
2.3	1.487	0.015	1.15	6.371	0.095	0.00	0.137	1.47E-06	0.00				
3.0	2.322	0.023	1.50	14.137	0.328	0.00	0.465	4.99E-06	0.00				
4.0	2.573	0.026	2.00	33.510	0.862	0.00	1.327	1.42E-05	0.00				
5.0	3.190	0.032	2.50	65.450	2.088	0.00	3.415	3.66E-05	0.00				
6.5	4.683	0.047	3.25	143.793	6.734	0.01	10.149	1.09E-04	0.01				
8.0	6.012	0.060	4.00	268.083	16.117	0.02	26.265	2.82E-04	0.03				
10.0	5.962	0.060	5.00	523.599	31.217	0.03	57.482	6.17E-04	0.06				
13.0	4.994	0.050	6.50	1150.346	57.448	0.06	114.931	1.23E-03	0.12				
17.0	5.692	0.057	8.50	2572.440	146.423	0.16	261.354	2.80E-03	0.28				
20.0	6.973	0.070	10.00	4188.789	292.084	0.31	553.438	5.94E-03	0.59				
25.0	7.551	0.076	12.50	8181.229	617.765	0.66	1171.203	1.26E-02	1.26				
31.0	6.098	0.061	15.50	15598.528	951.198	1.02	2122.401	2.28E-02	2.28				
37.0	5.979	0.060	18.50	26521.843	1585.741	1.70	3708.142	3.98E-02	3.98				
44.0	6.485	0.065	22.00	44602.229	2892.455	3.10	6600.597	7.08E-02	7.08				
53.0	5.966	0.060	26.50	77951.799	4650.604	4.99	11251.201	1.21E-01	12.07				
63.0	4.666	0.047	31.50	130924.276	6108.927	6.55	17360.128	1.86E-01	18.62				
75.0	3.523	0.035	37.50	220893.188	7782.067	8.35	25142.195	2.70E-01	26.97				
88.0	3.004	0.030	44.00	356817.831	10718.808	11.50	35861.002	3.85E-01	38.47				
105.0	2.197	0.022	52.50	606130.907	13316.696	14.28	49177.698	5.28E-01	52.75				
125.0	1.394	0.014	62.50	1022653.646	14255.792	15.29	63433.490	6.80E-01	68.05				
149.0	0.647	0.006	74.50	1732037.686	11206.284	12.02	74639.774	8.01E-01	80.07				
177.0	0.258	0.003	88.50	2903476.605	7490.970	8.04	82130.744	8.81E-01	88.10				
210.0	0.114	0.001	105.00	4849047.252	5527.914	5.93	87658.657	9.40E-01	94.03				
250.0	0.068	0.001	125.00	8181229.167	5563.236	5.97	93221.893	1.00E+00	100.00				
				Total	93221.893	100.00							

PEOR3258-S3 (Cut 2) Mass Percent - Calcined to ~950°C									
Assume: Spherical particles									
P.S. (microns)	% Part	Fract Part	(r)	Volume	Vol Contrib	INDIV MASS %	CUM Vol Contrib	CUM Mass Fraction	CUM Mass %
0.5	4.939	0.049	0.25	0.065	0.003	0.00	0.003	3.19E-08	0.00
0.7	0.777	0.008	0.35	0.180	0.001	0.00	0.005	4.58E-08	0.00
1.0	1.056	0.011	0.50	0.524	0.006	0.00	0.010	1.01E-07	0.00
1.3	0.844	0.008	0.65	1.150	0.010	0.00	0.020	1.97E-07	0.00
1.8	0.858	0.009	0.90	3.054	0.026	0.00	0.046	4.58E-07	0.00
2.3	1.649	0.016	1.15	6.371	0.105	0.00	0.151	1.50E-06	0.00
3.0	2.303	0.023	1.50	14.137	0.326	0.00	0.477	4.75E-06	0.00
4.0	2.501	0.025	2.00	33.510	0.838	0.00	1.315	1.31E-05	0.00
5.0	3.343	0.033	2.50	65.450	2.188	0.00	3.503	3.49E-05	0.00
6.5	4.533	0.045	3.25	143.793	6.518	0.01	10.021	9.98E-05	0.01
8.0	5.885	0.059	4.00	268.083	15.777	0.02	25.798	2.57E-04	0.03
10.0	5.709	0.057	5.00	523.599	29.892	0.03	55.690	5.55E-04	0.06
13.0	5.220	0.052	6.50	1150.346	60.048	0.06	115.738	1.15E-03	0.12
17.0	5.793	0.058	8.50	2572.440	149.021	0.15	264.759	2.64E-03	0.26
20.0	6.801	0.068	10.00	4188.789	284.880	0.28	549.639	5.47E-03	0.55
25.0	6.956	0.070	12.50	8181.229	569.086	0.57	1118.725	1.11E-02	1.11
31.0	6.388	0.064	15.50	15598.528	996.434	0.99	2115.159	2.11E-02	2.11
37.0	6.124	0.061	18.50	26521.843	1624.198	1.62	3739.357	3.72E-02	3.72
44.0	6.279	0.063	22.00	44602.229	2800.574	2.79	6539.931	6.51E-02	6.51
53.0	5.369	0.054	26.50	77951.799	4185.232	4.17	10725.163	1.07E-01	10.68
63.0	4.975	0.050	31.50	130924.276	6513.483	6.49	17238.646	1.72E-01	17.16
75.0	3.822	0.038	37.50	220893.188	8442.538	8.41	25681.183	2.56E-01	25.57
88.0	2.879	0.029	44.00	356817.831	10272.785	10.23	35953.969	3.58E-01	35.80
105.0	2.153	0.022	52.50	606130.907	13049.998	12.99	49003.967	4.88E-01	48.79
125.0	1.622	0.016	62.50	1022653.646	16587.442	16.52	65591.409	6.53E-01	65.31
149.0	0.739	0.007	74.50	1732037.686	12799.758	12.74	78391.168	7.81E-01	78.05
177.0	0.259	0.003	88.50	2903476.605	7520.004	7.49	85911.172	8.55E-01	85.54
210.0	0.097	0.001	105.00	4849047.252	4703.576	4.68	90614.748	9.02E-01	90.22
250.0	0.120	0.001	125.00	8181229.167	9817.475	9.78	100432.223	1.00E+00	100.00
						100.00			
				Total	100432.223				

PEOR3258-S3 (Cut 3) Mass Percent - Calcined to ~950°C									
Assume: Spherical particles									
P.S. (microns)	% Part	Fract Part	(r)	Volume	Vol Contrib	INDIV MASS %	CUM Vol Contrib	CUM Mass Fraction	CUM Mass %
0.5	5.156	0.052	0.25	0.065	0.003	0.00	0.003	4.15E-08	0.00
0.7	0.809	0.008	0.35	0.180	0.001	0.00	0.005	5.93E-08	0.00
1.0	0.999	0.010	0.50	0.524	0.005	0.00	0.010	1.23E-07	0.00
1.3	0.789	0.008	0.65	1.150	0.009	0.00	0.019	2.34E-07	0.00
1.8	0.827	0.008	0.90	3.054	0.025	0.00	0.044	5.43E-07	0.00
2.3	1.738	0.017	1.15	6.371	0.111	0.00	0.155	1.90E-06	0.00
3.0	2.201	0.022	1.50	14.137	0.311	0.00	0.466	5.70E-06	0.00
4.0	2.435	0.024	2.00	33.510	0.816	0.00	1.282	1.57E-05	0.00
5.0	3.317	0.033	2.50	65.450	2.171	0.00	3.453	4.22E-05	0.00
6.5	4.938	0.049	3.25	143.793	7.101	0.01	10.554	1.29E-04	0.01
8.0	5.900	0.059	4.00	268.083	15.817	0.02	26.371	3.22E-04	0.03
10.0	5.937	0.059	5.00	523.599	31.086	0.04	57.457	7.02E-04	0.07
13.0	5.369	0.054	6.50	1150.346	61.762	0.08	119.219	1.46E-03	0.15
17.0	6.235	0.062	8.50	2572.440	160.392	0.20	279.610	3.42E-03	0.34
20.0	7.096	0.071	10.00	4188.789	297.236	0.36	576.847	7.05E-03	0.70
25.0	7.261	0.073	12.50	8181.229	594.039	0.73	1170.886	1.43E-02	1.43
31.0	6.746	0.067	15.50	15598.528	1052.277	1.29	2223.163	2.72E-02	2.72
37.0	6.397	0.064	18.50	26521.843	1696.602	2.07	3919.765	4.79E-02	4.79
44.0	6.263	0.063	22.00	44602.229	2793.438	3.41	6713.203	8.20E-02	8.20
53.0	5.157	0.052	26.50	77951.799	4019.974	4.91	10733.177	1.31E-01	13.11
63.0	4.292	0.043	31.50	130924.276	5619.270	6.87	16352.447	2.00E-01	19.98
75.0	3.368	0.034	37.50	220893.188	7439.683	9.09	23792.129	2.91E-01	29.07
88.0	2.718	0.027	44.00	356817.831	9698.309	11.85	33490.438	4.09E-01	40.92
105.0	1.846	0.018	52.50	606130.907	11189.177	13.67	44679.614	5.46E-01	54.59
125.0	1.239	0.012	62.50	1022653.646	12670.679	15.48	57350.293	7.01E-01	70.07
149.0	0.594	0.006	74.50	1732037.686	10288.304	12.57	67638.597	8.26E-01	82.64
177.0	0.273	0.003	88.50	2903476.605	7926.491	9.68	75565.088	9.23E-01	92.32
210.0	0.052	0.001	105.00	4849047.252	2521.505	3.08	78086.593	9.54E-01	95.40
250.0	0.046	0.000	125.00	8181229.167	3763.365	4.60	81849.958	1.00E+00	100.00
						100.00			
				Total	81849.958				

All numbers as of 6/27/95 (measured)

Pu Assay (Coulometry)	Rep1	Rep1	Rep2	Rep2	Rep3	Rep3	Rep4	Rep4	Rep5	Rep5	Mean	Std. Dev.	%RSD
Total Wt. % Pu	87.86	87.85	87.9	87.84	87.82	87.77	87.77	87.84	87.840	87.78	87.827	0.042	0.048
	avg = 87.855		avg = 87.87		avg = 87.795		avg = 87.805		avg = 87.81				
Pu Isotopic (Mass Spec)	Rep1		Rep2		Rep3		Rep4		Rep5				
At. %													
238	0.0151		0.0141		0.0150		0.0144		0.0144		0.015	4.30E-04	2.946
239	93.7865		93.7880		93.7871		93.7871		93.7864		93.787	6.38E-04	0.001
240	5.9212		5.9207		5.9210		5.9215		5.9219		5.921	4.62E-04	0.008
241	0.2218		0.2221		0.2218		0.2217		0.2219		0.222	1.52E-04	0.068
242	0.0554		0.0551		0.0551		0.0553		0.0555		0.055	1.79E-04	0.324
244	0.0000		0.0000		0.0000		0.0000		0.0000		0.000	-----	-----
total	100.0000		100.0000		100.0000		100.0000		100.0001				
Wt. %													
238	0.0150		0.0140		0.0149		0.0143		0.0143		0.015	4.30E-04	2.966
239	93.7809		93.7824		93.7815		93.7815		93.7809		93.781	6.15E-04	0.001
240	5.9444		5.9439		5.9442		5.9447		5.9451		5.944	4.62E-04	0.008
241	0.2235		0.2239		0.2236		0.2235		0.2237		0.224	1.52E-04	0.068
242	0.0561		0.0558		0.0560		0.0560		0.0560		0.056	1.34E-04	0.240
244	0.0000		0.0000		0.0000		0.0000		0.0000		0.000	-----	-----
total	100.0000		100.0000		100.0000		100.0000		100.0000				
Am content (Mass Spec)	300041378	300041378	300041379	300041379	300041380	300041380	300041381	300041381	300041382	300041382	Mean	Std. Dev.	%RSD
	Rep1	Rep2	Rep1	Rep2	Rep1	Rep2	Rep1	Rep2	Rep1	Rep2			
µg Am/g PuO2	766.2	766.9	766.1	766.9	766.3	766.9	766.9	766.9	766.7	766.7	766.730	0.164	0.021
	avg	766.55	avg	766.55	avg	766.6	avg	766.9	avg	766.7			
µg Am/g Pu	872.4	873.2	872.4	873.2	872.5	873.2	873.2	873.2	872.7	873.0	873.010	0.189	0.022
	avg	872.80	avg	873.2	avg	872.85	avg	873.2	avg	873.0			

[766.730] = not used in average

a	b	c	d	e	f	g	h	i	j	k	l	m	n	o
All numbers as of 6/1/97 (calculated)														
Pu Assay (Coulometry)	Rep1	Rep1	Rep2	Rep2	Rep3	Rep3	Rep4	Rep4	Rep5	Rep5	Mean	Std. Dev.	%RSD	
Total Wt. % Pu		avg = 87.8317		avg = 87.8456		avg = 87.7717		avg = 87.7817		avg = 87.7867	87.804	0.033	0.038	
Pu Isotopic (Mass Spec)	Rep1		Rep2		Rep3		Rep4		Rep5					
At. %														
238	decay	re-norm	decay	re-norm	decay	re-norm	decay	re-norm	decay	re-norm				
238	0.0149	0.0149	0.0139	0.0139	0.0148	0.0148	0.0142	0.0142	0.0142	0.0142	0.01438	4.237E-04	2.946	
239	93.7813	93.8061	93.7828	93.8076	93.7819	93.8067	93.7819	93.8066	93.7812	93.8059	93.80656	6.56E-04	0.001	
240	5.9200	5.9216	5.9195	5.9211	5.9198	5.9214	5.9203	5.9219	5.9207	5.9222	5.92161	4.591E-04	0.008	
241	0.2020	0.2021	0.2023	0.2024	0.2020	0.2021	0.2020	0.2020	0.2021	0.2022	0.20215	1.382E-04	0.068	
242	0.0554	0.0554	0.0551	0.0551	0.0551	0.0551	0.0553	0.0553	0.0555	0.0555	0.05529	1.789E-04	0.324	
244	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.00000	0.000E+00	0.000	
total	99.9736	100.0000	99.9736	100.0000	99.9736	100.0000	99.9736	100.0000	99.9737	100.0000	100.00000	-----	-----	
Wt. %														
238		0.0148		0.0138		0.0147		0.0141		0.0141	0.01432	4.218E-04	2.946	
239		93.7806		93.7821		93.7812		93.7804		93.7810	93.78110	6.673E-04	0.001	
240		5.9448		5.9443		5.9446		5.9451		5.9455	5.94481	4.607E-04	0.008	
241		0.2037		0.2040		0.2036		0.2038		0.2038	0.20379	1.393E-04	0.068	
242		0.0561		0.0558		0.0558		0.0560		0.0562	0.05597	1.811E-04	0.324	
244		0.0000		0.0000		0.0000		0.0000		0.0000	0.00000	0.000E+00	0.000	
total		100.0000		100.0000		100.0000		100.0000		100.0000	100.00000	-----	-----	
Am content (Mass Spec)														
µg Am/g PuO2		938.89140		939.50460		938.82183		939.06279		938.02054	939.062	2.663E-01	0.028	
µg Am/g Pu		1068.96645		1069.46316		1069.61803		1069.77057		1069.67177	1069.502	3.169E-01	0.030	

Isotope	half-life (years)	half-life (days)	decay const	At. Weight
238Pu	87.7	32010.5	2.18537E-05	238.0456
239Pu	2.41E+04	8800150	7.87854E-08	239.0522
240Pu	6.56E+03	2395495	2.89354E-07	240.0538
241Pu	14.35	5237.75	0.000132337	241.0568
242Pu	3.73E+05	136254500	5.08715E-09	242.0587
244Pu	6.00E+07	29200000000	2.37378E-11	244.0642
241Am	432.2	157753	4.39388E-06	241.056823
Rep 1				
Assume 100g PuO2				
		6/27/95	6/1/97	
		9	9	
	238	0.0123	0.0121	
	239	82.3328	82.3282	
	240	5.2193	5.2183	
	241	0.1966	0.1791	
	242	0.0490	0.0490	
	244	0.0000	0.0000	
total Pu =		87.8100	87.7867	

TO: B. MARSHALL
GROUP CST- 8
MAILSTOP G740

DATE: 31-Oct-1995

FROM: CST-3 Sample Management

SUBJECT: Final Chemical Analysis Report and Certification
CST-3 Sample Number: 200016529

Your Sample identification: PEOR3258-STD 1
Sample description: PU OXIDE
Program Code: SA11

Methods Requested: MSAM-S, MSPUIISO, PACPU-S, PSDCA-S, PSMPU-S, RCAM,
RCESS, TECL-S, TEF-S

The chemical analyses listed above have been completed. The attached report package contains the analyses for all requested work.

Analytical Chemistry Report

Sample ID: 200016529 Group: CST- 8 QA: Y Priority: 2
Submission ID: 100013407 Program: SA11 Acct: Y Logged: 05-Jun-1995
User sample ID: PEOR3258-STD 1
Sample Type: AH PLUTONIUM OXIDE
Requester: B. MARSHALL MS: G740 Phone: 7-4087

Sample Description: PU OXIDE
Radioactive Elements: PU239
Radiation: Y
Hazards: N
Special Handling: N

Methods Requested: MSAM-S, MSPUIISO, PACPU-S, PSDCA-S, PSMPU-S, RCAM,
RCESS, TECL-S, TEF-S

Approved by: Apodaca, Lydia 31-Oct-1995 10:58

=====

Analytical Operation: PSDCA-S Task ID: 300038569
Date Completed: 28-Jun-1995 15:09 Replicates: 1 Version: 1

Component	Results			Units
-----	-----	-----	-----	-----
--PSDCA-- ANALYSIS	CBC, RDA			ANALYST(S)
PLATE NUMBER	498606 AND 498607			
ALUMINUM	35	19	19	ug/g
CHROMIUM	130	130	150	ug/g
IRON	99	89	100	ug/g
NICKEL	245	280	285	ug/g
SILICON	205	130	155	ug/g
CALCIUM	1000	770	750	ug/g
LEAD	23	16	15	ug/g
TIN	<5	<5	<5	ug/g
ZINC	<5	<5	<5	ug/g
SILVER	<1	<1	1	ug/g
BISMUTH	3	2	2	ug/g
CADMIUM	<10	<10	<10	ug/g
MAGNESIUM	34	26	26	ug/g
MANGANESE	2	3	4	ug/g
MOLYBDENUM	<20	<20	<20	ug/g
BORON	<5	<5	<5	ug/g
BERYLLIUM	<1	<1	<1	ug/g
COPPER	3	3	4	ug/g
SODIUM	100	50	50	ug/g

Our standards are based on trace impurities in pure PuO₂. Experience has shown that if any one impurity level exceeds 2000 ug/g or the summation of impurity levels for several impurities exceeds 3000 ug/g, results for other impurities may be biased.

Result is based on sample as received and is expressed as ug/g or ug/ml unless indicated otherwise. Result preceded by a < symbol represents our present limit of detection.

Notebook reference: 26648 PG 96

Analyst: Olivares, Jose

Approved by: Olivares, Jose 28-Jun-1995 15:13

Analytical Operation: PSMPU-S

Date Completed: 28-Jun-1995 16:11 Replicates: 1

Task ID: 300038570

Version: 1

Component	Results	Units
ALUMINUM	17 13 16	ug/g
BORON	<8 <8 <8	ug/g
BERYLLIUM	<2 <2 <2	ug/g
CALCIUM	520 <330 420	ug/g
CADMIUM	8.5 9.4 7.7	ug/g
IRON	240 220 190	ug/g
GALLIUM	18 17 17	ug/g
GADOLINIUM	0.6 <0.42 <0.43	ug/g
LITHIUM	8.5 <6 <7	ug/g
MAGNESIUM	34 33 32	ug/g
NICKEL	630 580 490	ug/g
THORIUM	1.1 2.8 0.95	ug/g
ZIRCONIUM	[1]	ug/g

[1] ZIRCONIUM:

URANIUM 238 263 269 416 ug/g

CURIUM 242 1570 1640 1620 ug/g (Intefered by Pu-242)

CURIUM 244 0.10 0.095 2.3 ug/g

CALIFORNIUM 252 4.4 4.2 4.1 ug/g

Notebook reference: LNA12793 PG94-97

Analyst: CB

Approved by: Olivares, Jose 28-Jun-1995 16:48

Analytical Operation: TECL-S

Date Completed: 06-Sep-1995 14:47 Replicates: 3

Task ID: 300038571

Version: 1

Component	Rep 1	Rep 2	Rep 3	Units
CHLORINE	A 31.9	B 23.6	C 51.6	ug/g

Notebook reference: 10733, PP. 117, 120

Analyst: JAK, TRH

Approved by: Marshall, Thomas K. 06-Sep-1995 14:48

Analytical Operation: TEF-S

Date Completed: 06-Sep-1995 14:47 Replicates: 3

Task ID: 300038572

Version: 1

Component	Rep 1	Rep 2	Rep 3	Units
FLUORINE	A 36.3	B 28.3	C 54.6	ug/g

Notebook reference: 10733, PP. 117, 120

Analyst: JAK, TRH

Approved by: Marshall, Thomas K. 06-Sep-1995 14:48

=====

Analytical Operation: PACPU-S Task ID: 300038573
Date Completed: 30-Jun-1995 11:53 Replicates: 5 Version: 1

Component	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Units
PLUTONIUM	87.85 [1]	87.86 [2]	87.79 [3]	87.80 [4]	87.81 [5]	percent

Assay Method: Coulometry

Estimated Precision: 0.1%

[1] PLUTONIUM, Rep 1:

Analyzed five separate dissolutions of the sample. Two analyses were performed for each dissolution and the average for each dissolution is reported. The following results were obtained for Rep 1: 87.86% and 87.85%.

[2] PLUTONIUM, Rep 2:

Analyzed five separate dissolutions of the sample. Two analyses were performed for each dissolution and the average for each dissolution is reported. The following results were obtained for Rep 2: 87.90% and 87.84%.

[3] PLUTONIUM, Rep 3:

Analyzed five separate dissolutions of the sample. Two analyses were performed for each dissolution and the average for each dissolution is reported. The following results were obtained for Rep 3: 87.82% and 87.77%.

[4] PLUTONIUM, Rep 4:

Analyzed five separate dissolutions of the sample. Two analyses were performed for each dissolution and the average for each dissolution is reported. The following results were obtained for Rep 4: 87.77% and 87.84%.

[5] PLUTONIUM, Rep 5:

Analyzed five separate dissolutions of the sample. Two analyses were performed for each dissolution and the average for each dissolution is reported. The following results were obtained for Rep 5: 87.84% and 87.78%.

Notebook references: Rep 1: LANB 9237 PP. 120-121

Rep 2: SAME

Rep 3: SAME

Rep 4: SAME

Rep 5: SAME

Analyst: LMS, EL, WJB

Approved by: Temer, Donald J. 30-Jun-1995 14:04

=====

Analytical Operation: RCAM Task ID: 300040775
Date Completed: 12-Jul-1995 11:55 Replicates: 1 Version: 1

Component	Results	Units
AMERICIUM-241	762 ON 7-10-95	ppm
%RSD +/-	2	
COMMENTS	[1]	

[1] COMMENTS: FIVE CUTS WERE ANALYZED

Analyst: Stalnaker, Nelson D.

Approved by: Stalnaker, Nelson D. 23-Aug-1995 07:39

=====
Analytical Operation: RCESS

Date Completed: 12-Jul-1995 11:55 Replicates: 1

Task ID: 300040776

Version: 1

Component	Result	Units
-----	-----	-----
SURVEY	[1]	
COMMENTS	[2]	

[1] SURVEY:

ALPHA SPECTROSCOPY DID NOT REVEAL ANY ACTIVITY DUE TO CM-244 OR CF-252.
THE DETECTION LIMIT FOR CM-244 IS LESS THAN 0.5 PPM AND THE DETECTION LIMIT FOR
CF-252 IS LESS THAN 0.1 PPM.

[2] COMMENTS: FIVE CUTS WERE ANALYZED

Analyst: Stalnaker, Nelson D.

Approved by: Stalnaker, Nelson D. 23-Aug-1995 07:39

=====
Analytical Operation: MSAM-S

Date Completed: 17-Jul-1995 11:22 Replicates: 2

Task ID: 300041378

Version: 1

Component	Rep 1	Rep 2	Units
-----	-----	-----	-----
AMERICIUM	766.2	766.9	ug/g

Notebook reference: VG-2, CUTS A1 & A2, SEPARATION DATE: 6/26/95, 238PU BY RADIO
CHEMISTRY.

Analyst: CH, LC, PN

Approved by: Callis, Larry 18-Jul-1995 08:52

=====
Analytical Operation: MSAM-S

Date Completed: 17-Jul-1995 11:37 Replicates: 2

Task ID: 300041379

Version: 1

Component	Rep 1	Rep 2	Units
-----	-----	-----	-----
AMERICIUM	771.5	766.9	ug/g

Notebook reference: VG-2, CUTS B1 & B2, SEPARATION DATE: 6/26/95, 238PU BY RADIO
CHEMISTRY.

Analyst: CH, LC, PN

Approved by: Callis, Larry 18-Jul-1995 08:52
=====

Analytical Operation: MSAM-S

Task ID: 300041380

Date Completed: 13-Jul-1995 11:52 Replicates: 2

Version: 1

Component	Rep 1	Rep 2	Units
AMERICIUM	766.3	766.9	ug/g

Notebook reference: VG-2, CUTS C1 & C2, SEPARATION DATE: 6/26/95, 238PU BY RADIO CHEMISTRY.

Analyst: CH, LC, PN

Approved by: Callis, Larry 18-Jul-1995 08:52

Analytical Operation: MSAM-S

Task ID: 300041381

Date Completed: 13-Jul-1995 11:56 Replicates: 2

Version: 1

Component	Rep 1	Rep 2	Units
AMERICIUM	766.9	770.4	ug/g

Notebook reference: VG-2, CUTS D1 & D2, SEPARATION DATE: 6/26/95, 238PU BY RADIO CHEMISTRY.

Analyst: CH, LC, PN

Approved by: Callis, Larry 18-Jul-1995 08:52

Analytical Operation: MSAM-S

Task ID: 300041382

Date Completed: 13-Jul-1995 11:59 Replicates: 2

Version: 1

Component	Rep 1	Rep 2	Units
AMERICIUM	770.0	766.7	ug/g

Notebook reference: VG-2, CUTS E1 & E2, SEPARATION DATE: 6/26/95, 238PU BY RADIO CHEMISTRY.

Analyst: CH, LC, PN

Approved by: Callis, Larry 18-Jul-1995 08:52

Analytical Operation: MSPUIISO

Task ID: 300041383

Date Completed: 13-Jul-1995 11:31 Replicates: 5

Version: 1

Component	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Units
PU-238	0.0151	0.0141	0.0150	0.0144	0.0144	atom percent
PU-239	93.7865	93.7880	93.7871	93.7871	93.7864	atom percent
PU-240	5.9212	5.9207	5.9210	5.9215	5.9219	atom percent
PU-241	0.2218	0.2221	0.2218	0.2217	0.2219	atom percent
PU-242	0.0554	0.0551	0.0551	0.0553	0.0558	atom percent
PU-244	0.0000	0.0000	0.0000	0.0000	0.0000	atom percent
SUM	100.0000	100.0000	100.0000	100.0000	99.9999	atom percent
PU-238	0.0150	0.0140	0.0149	0.0143	0.0143	weight percent
PU-239	93.7609	93.7624	93.7615	93.7615	93.7609	weight percent
PU-240	5.5444	5.9439	5.9442	5.9447	5.9451	weight percent
PU-241	0.2236	0.2239	0.2236	0.2235	0.2237	weight percent

PU-242	0.0561	0.0558	0.0558	0.0560	0.0560	weight percent
PU-244	0.0000	0.0000	0.0000	0.0000	0.0000	weight percent

Component	Average	Units
PU-238	0.0146	atom percent
PU-239	93.7870	atom percent
PU-240	5.9213	atom percent
PU-241	0.2219	atom percent
PU-242	0.0552	atom percent
PU-244	0.0000	atom percent
SUM	100.0000	atom percent
PU-238	0.0145	weight percent
PU-239	93.7614	weight percent
PU-240	5.9445	weight percent
PU-241	0.2237	weight percent
PU-242	0.0559	weight percent
PU-244	0.0000	weight percent

Notebook reference: VG-2

Analyst: CH, LC, PN

Approved by: Callis, Larry 18-Jul-1995 08:52

=====

AmO2 Analyses
Recalc. to 6/1/97

3/23/98

Thermal Ionization Mass Spectrometry (Isotope Dilution)								
Data Run	4/25/97	4/25/97	4/25/97	5/1/97				
Total Am-241	Cut A1	Cut B1	Cut C1	Cut D1	Mean	Std. Dev.	%RSD	
Wt. % Am-241	78.618	79.058	78.556	78.729	78.740	0.224	0.284	

Re-calc to 6/1/97								
Total Am-241	Cut A1	Cut B1	Cut C1	Cut D1	Mean	Std. Dev.	%RSD	
Wt. % Am-241	78.605	79.045	78.543	78.718	78.728	0.224	0.284	

Almost No Change in 37/31 days!!!!!!

Pu in the AmO2								
	5/12/97	5/12/97	5/12/97	5/12/97				
	Cut A1	Cut B1	Cut C1	Cut D1	Mean	Std. Dev.	%RSD	
Total Wt. % Pu	0.403	0.414	0.421	0.421	0.415	0.008	2.049	
At. %	5/15/97	5/15/97	5/15/97	5/15/97				
238	0.0440	0.0459	0.0461	0.0455	0.0454	0.001	2.094	
239	89.4040	89.1441	89.1455	89.0807	89.1936	0.144	0.161	
240	9.9090	10.1479	10.1463	10.2059	10.1023	0.132	1.305	
241	0.4597	0.4757	0.4751	0.4794	0.4725	0.009	1.847	
242	0.1832	0.1864	0.1871	0.1885	0.1863	0.002	1.204	
244	0.0000	0.0000	0.0000	0.0000	0.0000	0.000	0.000	
Total	99.9999	100.0000	100.0001	100.0000	100.0000			
Wt. %	5/15/97	5/15/97	5/15/97	5/15/97				
238	0.0438	0.0457	0.0459	0.0453	0.0452	0.001	2.103	
239	89.3616	89.1007	89.1021	89.0371	89.1504	0.144	0.162	
240	9.9458	10.1855	10.1839	10.2437	10.1397	0.132	1.304	
241	0.4633	0.4795	0.4789	0.4832	0.4762	0.009	1.853	
242	0.1854	0.1887	0.1894	0.1908	0.1886	0.002	1.214	
244	0.0000	0.0000	0.0000	0.0000	0.0000	0.000	0.000	
Total	99.9561	100.0001	100.0002	100.0001	100.0001			

Recalc to 6/1/97											
Pu in the AmO2		Cut A1	Cut B1	Cut C1	Cut D1	Mean	Std. Dev.	%RSD			
Total Wt. % Pu		0.403	0.414	0.421	0.421	0.415	0.008	2.049			
At. %		decay	re-norm	decay	re-norm	decay	re-norm	decay	re-norm		
238		0.0440	0.0440	0.0459	0.0459	0.0461	0.0461	0.0455	0.0455	0.0454	0.0009
239		89.4039	89.4051	89.1440	89.1451	89.1454	89.1464	89.0806	89.0817	89.1946	0.1435
240		9.9090	9.9091	10.1479	10.1480	10.1463	10.1464	10.2058	10.2060	10.1024	0.1318
241		0.4587	0.4587	0.4746	0.4746	0.4740	0.4740	0.4783	0.4783	0.4714	0.0087
242		0.1832	0.1832	0.1864	0.1864	0.1871	0.1871	0.1885	0.1885	0.1863	0.0022
244		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Total		99.9987	100.0000	99.9987	100.0000	99.9988	100.0000	99.9987	100.0000	100.0000	
Wt. %											
238			0.0438		0.0457		0.0459		0.0453	0.0451	0.0009
239			89.3626		89.1017		89.1031		89.0381	89.1514	0.1440
240			9.9459		10.1855		10.1839		10.2437	10.1398	0.1322
241			0.4623		0.4784		0.4778		0.4821	0.4751	0.0088
242			0.1854		0.1887		0.1894		0.1908	0.1886	0.0023
244			0.0000		0.0000		0.0000		0.0000	0.0000	0.0000
Total			100.0000		100.0000		100.0000		100.0000	100.0000	

U in the AmO2		5/16/97	
Total Wt. % U		0.0035	(35 µg/g—compares well to ICP-MS data)
At. %	5/16/97		
233		0.1930	
234		5.3530	
235		43.0924	
236		17.1995	
238		34.1621	
Total		100.0000	
NOTE: Decay corrections for Uranium are not necessary Due to the long half-lives of the isotopes.			
Wt. %	5/16/97		
233		0.1904	
234		5.3044	
235		42.8841	
236		17.1693	
238		34.4319	
Total		99.8097	

	6/1/97	4/25/97	37	
	6/1/97	5/15/97	17	
	6/1/97	5/1/97	31	
Isotope	If-life (yrs)	If-life (day)	decay const.	At. Weight
238Pu	87.7	32010.5	2.17E-05	238.0496
239Pu	2.41E+04	8800150	7.88E-08	239.0522
240Pu	6.56E+03	2395495	2.89E-07	240.0538
241Pu	14.35	5237.75	0.000132	241.0568
242Pu	3.75E+05	1.37E+08	5.06E-09	242.0587
244Pu	8.00E+07	2.92E+10	2.37E-11	244.0642
241Am	432.7	157935.5	4.39E-06	241.0568
237Np	2.14E+06	7.81E+08	8.87E-10	237.0482

Rep 1
Assume 100g AmO2

0.414 g Pu

	5/12/97	6/1/97	days
	g	g	20
238	0.0002	0.0002	
239	0.3689	0.3689	
240	0.0422	0.0422	
241	0.0020	0.0020	
242	0.0008	0.0008	
244	0.0000	0.0000	
total Pu =	0.4140	0.4140	

CEP WRMs
Am Feedstock Anal. Chem Results (200023528)

ICP-MS (Full QA)							
Element	Conc. Limit (µg/g)	Cut 6 (µg/g)	Cut 7 (µg/g)	Cut 8 (µg/g)	Mean	Std. Dev.	%RSD
Fe + Ni	<400	384	359	346	363	19.31	5.32
Fe	—	287	264	259	270	14.93	5.53
Ni	—	97	95	87	93	5.29	5.69
Be	<2	<0.5	<0.5	<0.5			
Mg	<500	58	52	45	52	6.51	12.59
Ca	<1000	1298	1257	1277	1277	20.50	1.61
Np	<100	19976	20201	19150	19776	553.40	2.80
Th	<50	35	36	35	35	0.58	1.63
Ga	<25	3.2	3.3	3	3	0.15	4.82
Al	<50	144	121	102	122	21.03	17.19
U	<500	36	40	37	38	2.08	5.53
U-235	—	18*	20*	18*	18.7*	1.15	6.15
U-238	—	18	20	19	19	1.00	5.26

ICP-MS (Re-analyzed with full QA)							
Element	Conc. Limit (µg/g)	Cut 6 (µg/g)	Cut 7 (µg/g)	Cut 8 (µg/g)	Mean	Std. Dev.	%RSD
Bi	—	576	584	552	571	16.65	2.92
K	—	4821	5640	4124	4862	758.82	15.61
La	—	190	198	180	189	9.02	4.76
Na	—	2981	2223	1438	2214	771.54	34.85
Pb	—	4385	1761	1619	2588	1557.58	60.18
Y	—	43301	45056	42890	43749	1150.40	2.63
Np	—	19590	19631	18718	19313	515.69	2.67

Ion Chromatography		
Element	Conc. Limit (µg/g)	Cut 5 (µg/g)
F	<85	654
Cl	<70	40

Thermal Ionization Mass Spectrometry (Isotope Dilution)							
Total Am-241	Cut A1	Cut B1	Cut C1	Cut D1	Mean	Std. Dev.	%RSD
Wt. % Am-241	78.618	79.058	78.556	78.729	78.740	0.224	0.284
Pu in the AmO2	Cut A1	Cut B1	Cut C1	Cut D1	Mean	Std. Dev.	%RSD
Total Wt. % Pu	0.403	0.414	0.421	0.421	0.415	0.008	2.049
At. %							
238	0.0440	0.0459	0.0461	0.0455	0.045	0.001	2.094
239	89.4040	89.1441	89.1455	89.0807	89.194	0.144	0.161
240	9.9090	10.1479	10.1463	10.2059	10.102	0.132	1.305
241	0.4597	0.4757	0.4751	0.4794	0.472	0.009	1.847
242	0.1832	0.1864	0.1871	0.1885	0.186	0.002	1.204
244	0.0000	0.0000	0.0000	0.0000	0.000	0.000	0.000
Total	99.9999	100.0000	100.0001	100.0000	100.0000		
Wt. %							
238	0.0438	0.0457	0.0459	0.0453	0.0452	0.001	2.103
239	89.3616	89.1007	89.1021	89.0371	89.1504	0.144	0.162
240	9.9458	10.1855	10.1839	10.2437	10.1397	0.132	1.304
241	0.4633	0.4795	0.4789	0.4832	0.4762	0.009	1.853
242	0.1854	0.1887	0.1894	0.1908	0.1886	0.002	1.214
244	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Total	99.9561	100.0001	100.0002	100.0001	100.0001		
U in the AmO2							
Total Wt. % U	0.0035	(35 µg/g—compares well to ICP-MS data)					
At. %							
233	0.1930						
234	5.3530						
235	43.0924						
236	17.1995						
238	34.1621						
Total	100.0000						
Wt. %							
233	0.1904						
234	5.3044						
235	42.8841						
236	17.1893						
238	34.4319						
Total	99.8097						

Total Mass Balance (Wt. %)	
Am241	78.74
Np237	1.93
Y	4.37
Pb	0.17
Na	0.22
Ca	0.13
K	0.49
Bi	0.05
La	0.02
LOI (H2O)	2.19
Pu (total)	0.42
total	88.73
Stoic. Am =	88.28
Stoic. O2 =	11.72
% diff. =	0.50
(Stoic. Am/total constituents less O)	
assume 11.72 % O then total equals	100.45%

Radiochemistry										
Total Am-241	Cut 14a	Cut 14b	Cut 15a	Cut 15b	Cut 16a	Cut 16b	Cut 17a	Cut 17b	Mean	Std. Dev.
Wt. % Am-241 (Gamma Activity)	78.85	78.71	79.27	80.08	78.87	79.24	80.02	79.57	79.33	0.53
Wt. % Am-241 (Alpha Activity)	79.14	78.43	79.62	79.62	79.98	78.87	79.07	79.41	79.27	0.49
Element	Conc. Limit (µg/g)	AmO2***								
Cm-244	<1	<0.02	*** estimated based on half-lives and activity ratio.							
Cf-252	<0.5	<0.003	Activity ratioed to 239,240Pu alpha peak (no actual peaks were observed for 244Cm or 252Cf so the concentrations are estimated)							

Loss On Ignition				
Performed on remaining AmO2 to be used in WRM fabrication (nom. 1.7g)				
	Before (g)	After (g)	**** AmO2 + Pt Dish	
Gross****	14.40158	14.3634		
Tare	12.65575	12.6558		
Net	1.74583	1.70166	Wt. Loss (g)	0.03817
LOI Wt.	2.19			

Analytical Chemistry Report

Sample ID: 200023528 Group: CST-8 QA: N Priority: 1
Submission ID: 100018327 Acct: Y Logged: 15-Apr-1997
User sample ID: FZG45-AMP138-2S2
Sample Type: AH SOLID
Customer Cost Code: MK53
Requester: B. MARSHALL MS: G740 Phone: 7-0409

Sample Description: AM OXIDE
Radioactive Elements: AM241
Radiation: Y
Hazards: N
Special Handling: Y
Final Report Date: 30-Jun-1997

WALK-IN CUSTOMER: N
RECHARGE COST CODE: 8BAN00X41300000000

Methods Requested: IC-ANIONS-S, MSAM-S, MSPUIDS, MSPUIISO, MSU-ISO,
MSUID-S, PSSUR-S, RCESS, TECL-S, TED1, TEF-S

Approved by: System Manager, SQL*LIMS 01-Jul-1997 21:11

Analytical Operation: IC-ANIONS-S Task ID: 300081859
Date Completed: 23-May-1997 16:06 Replicates: 1 Version: 1

WALK-IN QUOTED COST:
WALK-IN CHARGE BALNC: 0
DESCRIPTION: IC-ANIONS:F,CL,SO4,NO3,H04...

Component	Results	Units
FLUORIDE	< 25.39	ug/g
CHLORIDE	1637	ug/g

Notebook reference: ANC 89

Analyst: VMM

Approved by: Sweetin, Deborah 23-May-1997 16:07

Analytical Operation: TED1

Date Completed: 20-May-1997 08:50 Replicates: 1

Task ID: 300081860

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: DISSOLUTION

Component	Result	Units
DISSOLUTION	Y	Y/N

Notebook reference: 4040, P. 141

Analyst: TRH

Approved by: Marshall, Thomas K. 20-May-1997 08:50

Analytical Operation: MSU-ISO

Date Completed: 20-May-1997 14:12 Replicates: 1

Task ID: 300084888

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: MS-MSU ISO

Component	Results	Average	Units
U-233	0.1930	0.1930	atom percent
U-234	5.3530	5.3530	atom percent
U-235	43.0924	43.0924	atom percent
U-236	17.1995	17.1995	atom percent
U-238	34.1621	34.1621	atom percent
SUM	100.0000	100.0000	atom percent
U-233	0.1904	0.1904	weight percent
U-234	5.3044	5.3044	weight percent
U-235	42.8841	42.8841	weight percent
U-236	17.1893	17.1893	weight percent
U-238	34.4319	34.4319	weight percent

Notebook reference: VG-2

Analyst: NMS, CH, JA

Approved by: Callis, Larry 21-May-1997 10:38

Analytical Operation: MSPUIISO

Date Completed: 20-May-1997 14:27 Replicates: 4

Task ID: 300084889

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: MS-MASS SPEC PU ISOTOPIC

Component	Rep 1	Rep 2	Rep 3	Rep 4	Average	Units
-----	-----	-----	-----	-----	-----	-----
PU-238	0.0440	0.0459	0.0461	0.0455	0.0454	atom percent
PU-239	89.4040	89.1441	89.1455	89.0807	89.1936	atom percent
PU-240	9.9090	10.1479	10.1463	10.2059	10.1023	atom percent
PU-241	0.4597	0.4757	0.4751	0.4794	0.4725	atom percent
PU-242	0.1832	0.1864	0.1871	0.1885	0.1863	atom percent
PU-244	0.0000	0.0000	0.0000	0.0000	0.0000	atom percent
SUM	99.9999	100.0000	100.0001	100.0000	100.0000	atom percent
PU-238	0.0438	0.0457	0.0459	0.0453	0.0452	weight percent
PU-239	89.3616	89.1007	89.1021	89.0371	89.1504	weight percent
PU-240	9.9458	10.1855	10.1839	10.2437	10.1397	weight percent
PU-241	0.4633	0.4795	0.4788	0.4832	0.4762	weight percent
PU-242	0.1854	0.1887	0.1894	0.1908	0.1886	weight percent
PU-244	0.0000	0.0000	0.0000	0.0000	0.0000	weight percent

Notebook reference: VG-2

Analyst: NMS, CH, JA

Approved by: Callis, Larry 21-May-1997 10:38

Analytical Operation: MSPUIDS

Task ID: 300084890

Date Completed: 20-May-1997 14:30 Replicates: 4

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: MS-PLUTONIUM IN SOLIDS OR LIQUIDS

Component	Rep 1	Rep 2	Rep 3	Rep 4	Units
-----	-----	-----	-----	-----	-----
PLUTONIUM	0.403	0.414	0.421	0.421	weight percent

Notebook reference: VG-2

Analyst: NMS, CH, JA

Approved by: Callis, Larry 21-May-1997 10:38

Analytical Operation: MSUID-S

Task ID: 300084891

Date Completed: 20-May-1997 14:18 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: MS-URANIUM IN SOLIDS

Component	Result	Units
-----	-----	-----
URANIUM	.0035	weight percent

Notebook reference: VG-2

Analyst: NMS, CH, JA

Approved by: Callis, Larry 21-May-1997 10:38

Analytical Operation: PSSUR-S

Task ID: 300086275

Date Completed: 27-May-1997 00:00 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Result	Units	Component	Result	Units
ALUMINUM	144	ug/g	BERYLLIUM	<0.5	ug/g
BISMUTH	570*	ug/g	CALCIUM	1298	ug/g
IRON	287	ug/g	GALLIUM	3.2	ug/g
POTASSIUM	1743*	ug/g	LANTHANUM	180*	ug/g
MAGNESIUM	58	ug/g	SODIUM	2016*	ug/g
NICKEL	97	ug/g	LEAD	1796*	ug/g
THORIUM	35	ug/g	YTTRIUM	42300*	ug/g
ZIRCONIUM	302*	ug/g	COMMENTS	[1]	
URANIUM 235	18**	ug/g	NEPTUNIUM	19976	ug/g
URANIUM 238	18	ug/g	CUT_ID	CUT-6	

[1] COMMENTS: *LIMITED QA/**SEMI-QUANT

Notebook reference: ANC52 PG 115

Analyst: TMY/AAM/CAB

Approved by: Yoshida, Thomas M 27-May-1997 10:08

Analytical Operation: PSSUR-S

Task ID: 300086276

Date Completed: 27-May-1997 00:00 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Results	Units	Component	Results	Units
ALUMINUM	121	ug/g	BERYLLIUM	<0.5	ug/g
CALCIUM	1257	ug/g	IRON	264	ug/g
GALLIUM	3.3	ug/g	MAGNESIUM	52	ug/g
NICKEL	95	ug/g	THORIUM	36	ug/g
COMMENTS	**SEMI-QUANT		URANIUM 235	20**	ug/g
NEPTUNIUM	20201	ug/g	URANIUM 238	20	ug/g
CUT_ID	CUT-7				

Notebook reference: ANC52 PG 115

Analyst: TMY/AAM/CAB

Approved by: Yoshida, Thomas M 27-May-1997 10:08

Analytical Operation: PSSUR-S

Date Completed: 27-May-1997 00:00 Replicates: 1

Task ID: 300086277

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Results	Units	Component	Results	Units
ALUMINUM	102	ug/g	BERYLLIUM	<0.5	ug/g
CALCIUM	1277	ug/g	IRON	259	ug/g
GALLIUM	3.0	ug/g	MAGNESIUM	45	ug/g
NICKEL	87	ug/g	THORIUM	35	ug/g
COMMENTS	**SEMI-QUANT		URANIUM 235	18**	ug/g
NEPTUNIUM	19150	ug/g	URANIUM 238	19	ug/g
CUT_ID	CUT-8				

Notebook reference: ANC52 PG 115

Analyst: TMY/AAM/CAB

Approved by: Yoshida, Thomas M 27-May-1997 10:08

Analytical Operation: RCESS

Date Completed: 27-May-1997 11:02 Replicates: 1

Task ID: 300086388

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

Component	Results	Units
SURVEY	[1]	
COMMENTS	AW-RC-5 PP91-102	

[1] SURVEY:

sample were received. Several Radiochemical analyses were performed as part of analytical survey on this sample.

(1) The amount of ²⁴¹Am isotope in the sample was determine by the liquid scintillation and gamma ray spectroscopy. The average % of Am in the sample was 79.34 +/- 0.57 % by LSC and 79.40 +/- 0.57 % by gamma ray analysis. The calculations were based on the assumption of all alpha and gamma activities were from Am²⁴¹.

(2) Direct gamma scan of the Am oxide sample could not provide information on other radionuclides in the sample due to high level Am activity. The half-life of ²⁴¹Am isotope is about 5000 times shorter than ²³⁷Np. Based on the gamma analysis, we could not confirm if there is any ²³⁷Np in the sample.

(3) ²⁴⁴Cm and ²⁵²Cf concentrations: Based on alpha spectrometry data, the estimated amount of ²⁴⁴Cm in the sample is less than 0.02 ppm and the amount of ²⁵²Cf in the sample is less than 0.003 ppm.

Analyst: Wong, Amy

Approved by: Wong, Amy 27-May-1997 11:02

Analytical Operation: MSAM-S

Task ID: 300087631

Date Completed: 30-May-1997 14:36 Replicates: 4

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: MS-AMERICIUM IN SOLIDS

Component	Rep 1	Rep 2	Rep 3	Rep 4	Units
AMERICIUM	786180	790580	785560	787290	ug/g

Notebook reference: VG-2

Analyst: NMS, CH, JA

Approved by: Callis, Larry 02-Jun-1997 17:42

Analytical Operation: PSSUR-S

Task ID: 300092946

Date Completed: 19-Jun-1997 00:00 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Results	Units	Component	Results	Units
BISMUTH	576	ug/g	POTASSIUM	4821 [1]	ug/g
LANTHANUM	190	ug/g	SODIUM	2981	ug/g
LEAD	4385	ug/g	YTTRIUM	43301	ug/g
COMMENTS	SEMI-QUANT		NEPTUNIUM	19590	ug/g
CUT_ID	CUT-6				

Notebook reference: ANC52 PG 122

Analyst: TMY/AAM/CAB

Approved by: Yoshida, Thomas M 19-Jun-1997 09:46

Analytical Operation: PSSUR-S

Task ID: 300092947

Date Completed: 19-Jun-1997 00:00 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Results	Units	Component	Results	Units
BISMUTH	584	ug/g	POTASSIUM	5640 [1]	ug/g
LANTHANUM	198	ug/g	SODIUM	2223	ug/g
LEAD	1761	ug/g	YTTRIUM	45056	ug/g
COMMENTS	SEMI-QUANT		NEPTUNIUM	19631	ug/g
CUT_ID	CUT-7				

Notebook reference: ANC52 PG 122

Analyst: TMY/AAM/CAB

Approved by: Yoshida, Thomas M 19-Jun-1997 09:46

Analytical Operation: PSSUR-S

Task ID: 300092948

Date Completed: 19-Jun-1997 00:00 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Results	Units	Component	Results	Units
BISMUTH	552	ug/g	POTASSIUM	4124 [1]	ug/g
LANTHANUM	180	ug/g	SODIUM	1438	ug/g
LEAD	1619	ug/g	YTTRIUM	42890	ug/g
COMMENTS	SEMI-QUANT		NEPTUNIUM	18718	ug/g
CUT_ID	CUT-8				

Notebook reference: ANC52 PG 122

Analyst: TMY/AAM/CAB

Approved by: Yoshida, Thomas M 19-Jun-1997 09:46

Analytical Operation: TEF-S

Task ID: 300093804

Date Completed: 20-Jun-1997 16:44 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: TE-F

Component	Result	Units
-----	-----	-----
FLUORIDE	654	ug/g

Notebook reference: 4070, PP. 142, 143

Analyst: TRH

Approved by: Marshall, Thomas K. 20-Jun-1997 16:54

Analytical Operation: TECL-S

Task ID: 300093805

Date Completed: 20-Jun-1997 16:44 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: TE-CL IN SOLID

Component	Result	Units
-----	-----	-----
CHLORIDE	40	ug/g

Notebook reference: 4070, PP. 142, 143

Analyst: TRH

Approved by: Marshall, Thomas K. 20-Jun-1997 16:54

CEP WRMs

Depleted Uranium Feedstock Anal. Chem Results (200023256)

ICP-MS (Full QA)							
Element	Conc. Limit (µg/g)	Cut 1 (µg/g)	Cut 2 (µg/g)	Cut 4 (µg/g)	Mean	Std.Dev.	%RSD
Al	<200	99	108	102	103	5	4
B	<30	297	307	307	303.666667	5.773502692	1.901263
Be	<1	<0.5	<0.5	<0.5			
Ca	<200	21	23	25	23	2	9
Cd	<2	0.22	0.13	0.31	0.22	0.09	40.91
Fe + Ni + Cr	<250	55.3	57.4	55.9	56.2	1.1	1.9
Fe	----	46	50	49	48	2	4
Ni	----	7.7	5.7	4.9	6.1	1.4	23.6
Cr	----	1.6	1.7	2.0	1.8	0.2	11.8
Li	<1	<0.5	<0.5	<0.5			
Mg	<50	4.1	5.4	4.5	4.7	0.7	14.3
Na	<200	171	175	157	168	1	6
Si	<300	----	----	----			
Pb	<20	7.2	5.5	5.4	6.0	1.0	16.8
W	<350	3.0	2.8	3.1	3.0	0.2	5.1
V	<100	1.6	1.4	1.6	1.5	0.1	7.5

Pyrohydrolysis/Ion Selective Electrode							
Element	Conc. Limit (µg/g)	Cut a	Cut b	Cut c	Mean	Std. Dev.	%RSD
F	<85	4	4	<1	4.0	0.0	0.0
Cl	<70	6	3	3	4.0	1.7	43.3

Thermal Ionization Mass Spectrometry (Isotope Dilution)								
U Assay	Cut 12	Cut 13	Cut 14	Cut 15	Cut 16	Mean	Std. Dev.	%RSD
Total Wt. % U	87.464	87.497	87.496	87.525	87.485	87.493	0.022	0.025
At. %								
233	0.0000	0.0000	0.0000	0.0000	0.0000	0.000	0.000	----
234	0.0008	0.0008	0.0002	0.0014	0.0008	0.001	4.24E-04	53.033
235	0.1925	0.1930	0.1923	0.1922	0.1929	0.193	3.56E-04	0.185
236	0.0035	0.0034	0.0036	0.0029	0.0037	0.003	3.11E-04	9.107
238	99.8031	99.8028	99.8039	99.8036	99.8027	99.803	5.17E-04	0.001
Total	99.9999	100.0000	100.0000	100.0001	100.0001	100.000		
Wt. %								
233	0.0000	0.0000	0.0000	0.0000	0.0000	0.000	0.000	----
234	0.0008	0.0008	0.0002	0.0014	0.0008	0.001	4.24E-04	53.033
235	0.1901	0.1906	0.1899	0.1898	0.1905	0.190	3.56E-04	0.187
236	0.0035	0.0034	0.0036	0.0029	0.0037	0.003	3.11E-04	9.107
238	99.8057	99.8053	99.8064	99.8060	99.8051	99.806	5.24E-04	0.001
Total	100.0001	100.0001	100.0001	100.0001	100.0001	100.000		

Radiochemistry

Element	Conc. Limit (µg/g)	Depleted UO ₂	
Cm-244	<1	< 0.000005*	* estimated based on half-lives using alpha spectrometry
Cf-252	<0.5	< 0.0000007*	

Analytical Chemistry Report

Sample ID: 200023256 Group: CST-8 QA: N Priority: 2
Submission ID: 100018211 Acct: Y Logged: 02-Apr-1997
User sample ID: 0907-43
Sample Type: AH URANIUM OXIDE
Customer Cost Code: MK53
Requester: B. MARSHALL MS: G740 Phone: 7-0409

Sample Description: D-38 OXIDE
Radioactive Elements: U238
Radiation: Y
Hazards: N
Special Handling: N
Final Report Date: 08-Jul-1997

WALK-IN CUSTOMER: N
RECHARGE COST CODE: 8BAN00X41300000000

Methods Requested: MSU-ISO, MSUID-S, PADISS-S, PSSUR-S, RCESS,
TECL-S, TEF-S, XAEPMA

Approved by: System Manager, SQL*LIMS 01-Jul-1997 21:10

Analytical Operation: PADISS-S Task ID: 300079372
Date Completed: 22-Apr-1997 14:37 Replicates: 8 Version: 1

WALK-IN QUOTED COST:
WALK-IN CHARGE BALNC: 0
DESCRIPTION: PA-DISSOLUTIONS OF SOLIDS

Component	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Unit
COMPLETED	Y	Y	Y	Y	Y	Y	Y	Y	Y/N

Notebook references: Rep 1: NONE
Rep 2: SAME
Rep 3: SAME
Rep 4: SAME
Rep 5: SAME
Rep 6: SAME
Rep 7: SAME

Rep 8: SAME

Analyst: EL

Approved by: Temer, Donald J. 22-Apr-1997 14:37

Analytical Operation: MSU-ISO

Task ID: 300081170

Date Completed: 28-Apr-1997 09:54 Replicates: 5

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: MS-MSU ISO

Component	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Units
U-233	0.0000	0.0000	0.0000	0.0000	0.0000	atom percent
U-234	0.0008	0.0008	0.0002	0.0014	0.0008	atom percent
U-235	0.1925	0.1930	0.1923	0.1922	0.1929	atom percent
U-236	0.0035	0.0034	0.0036	0.0029	0.0037	atom percent
U-238	99.8031	99.8028	99.8039	99.8036	99.8027	atom percent
SUM	99.9999	100.0000	100.0000	100.0001	100.0001	atom percent
U-233	0.0000	0.0000	0.0000	0.0000	0.0000	weight percent
U-234	0.0008	0.0008	0.0002	0.0014	0.0008	weight percent
U-235	0.1901	0.1906	0.1899	0.1898	0.1905	weight percent
U-236	0.0035	0.0034	0.0036	0.0029	0.0037	weight percent
U-238	99.8057	99.8053	99.8064	99.8060	99.8051	weight percent

Component	Average	Units
U-233	0.0000	atom percent
U-234	0.0008	atom percent
U-235	0.1926	atom percent
U-236	0.0034	atom percent
U-238	99.8032	atom percent
SUM	100.0000	atom percent
U-233	0.0000	weight percent
U-234	0.0008	weight percent
U-235	0.1902	weight percent
U-236	0.0034	weight percent
U-238	99.8057	weight percent

Notebook reference: VG-2

Analyst: NMS, CH, JA

Approved by: Callis, Larry 02-May-1997 17:12

Analytical Operation: MSUID-S

Task ID: 300081171

Date Completed: 29-Apr-1997 08:20 Replicates: 5

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: MS-URANIUM IN SOLIDS

Component	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Units
URANIUM	87.464	87.497	87.496	87.525	87.485	weight percent

Notebook reference: VG-2

Analyst: NMS, CH, JA

Approved by: Callis, Larry 02-May-1997 17:12

Analytical Operation: XAEPMA

Task ID: 300085086

Date Completed: 22-May-1997 07:23 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: XA-MICROPROBE ANALYSIS

Component	Results	Units
XAEPMA REPORT	SEE REPORT	

Notebook reference: EPMA-4 P 39

Distribution: R. Marshall (original) MS G740

W. Hutchinson

File

Analyst: Hutchinson, William B.

Approved by: Hutchinson, William B. 22-May-1997 07:27

Analytical Operation: PSSUR-S

Task ID: 300086271

Date Completed: 27-May-1997 00:00 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Result	Units	Component	Result	Units
ALUMINUM	99	ug/g	BORON	297	ug/g
BERYLLIUM	<0.5	ug/g	CALCIUM	21	ug/g
CADMIUM	0.22	ug/g	CHROMIUM	1.6	ug/g
IRON	46	ug/g	LITHIUM	<0.5	ug/g

MAGNESIUM	4.1	ug/g	SODIUM	171	ug/g
NICKEL	7.7	ug/g	LEAD	7.2	ug/g
VANADIUM	1.6	ug/g	TUNGSTEN	3.0	ug/g
CUT_ID	CUT-1				

Notebook reference: ANC52 PG 116/ANC6 PG 211/F1994 PG129

Analyst: TMY/AAM/CAB/RDA/PEC/DLG/BJH/DJG/AKS

Approved by: Yoshida, Thomas M 27-May-1997 10:08

Analytical Operation: PSSUR-S

Task ID: 300086272

Date Completed: 27-May-1997 00:00 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Result	Units	Component	Result	Units
ALUMINUM	108	ug/g	BORON	307	ug/g
BERYLLIUM	<0.5	ug/g	CALCIUM	23	ug/g
CADMIUM	0.13	ug/g	CHROMIUM	1.7	ug/g
IRON	50	ug/g	LITHIUM	<0.5	ug/g
MAGNESIUM	5.4	ug/g	SODIUM	175	ug/g
NICKEL	5.7	ug/g	LEAD	5.5	ug/g
VANADIUM	1.4	ug/g	TUNGSTEN	2.8	ug/g
CUT_ID	CUT-2				

Notebook reference: ANC52 PG 116/ANC6 PG 211/F1994 PG129

Analyst: TMY/AAM/CAB/RDA/PEC/DLG/BJH/DJG/AKS

Approved by: Yoshida, Thomas M 27-May-1997 10:08

Analytical Operation: PSSUR-S

Task ID: 300086273

Date Completed: 27-May-1997 00:00 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: PS-ELEMENTAL ANALYSIS IN SOLIDS

Component	Result	Units	Component	Result	Units
ALUMINUM	102	ug/g	BORON	307	ug/g
BERYLLIUM	<0.5	ug/g	CALCIUM	25	ug/g
CADMIUM	0.31	ug/g	CHROMIUM	2.0	ug/g
IRON	49	ug/g	LITHIUM	<0.5	ug/g
MAGNESIUM	4.5	ug/g	SODIUM	157	ug/g
NICKEL	4.9	ug/g	LEAD	5.4	ug/g
VANADIUM	1.6	ug/g	TUNGSTEN	3.1	ug/g

CUT_ID CUT-4

Notebook reference: ANC52 PG 116/ANC6 PG 211/F1994 PG129

Analyst: TMY/AAM/CAB/RDA/PEC/DLG/BJH/DJG/AKS

Approved by: Yoshida, Thomas M 27-May-1997 10:08

Analytical Operation: RCESS

Date Completed: 27-May-1997 11:24 Replicates: 1

Task ID: 300086387

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

Component	Results	Units
SURVEY	[1]	
COMMENTS	AW-RC-5 PP91-102	

[1] SURVEY:

CST#23526 depleted uranium sample was analyzed by radiochemistry team. Three cuts of sample were received (Cuts 9,10,11).

(1) Typical spectra of depleted uranium sample were obtained for this sample. No other noticeable radionuclide was observed by gamma ray analysis.

(2) Based on alpha spectrometry, the estimated ^{244}Cm level in the sample was less than $5\text{E-}6$ ppm and the estimated ^{252}Cf level in the sample was less than $7\text{E-}7$ ppm. The estimated concentrations were calculated based on the half-life, activity ratio of ^{234}U , ^{238}U , ^{244}Cm and ^{252}Cf .

Analyst: Wong, Amy

Approved by: Wong, Amy 27-May-1997 11:28

Analytical Operation: TEF-S

Date Completed: 20-Jun-1997 16:51 Replicates: 1

Task ID: 300093815

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: TE-F

Component	Results	Units
FLUORIDE	4, 4, <1	ug/g

Notebook reference: 4070, PP. 142, 143

Analyst: TRH

Approved by: Marshall, Thomas K. 20-Jun-1997 16:54

Analytical Operation: TECL-S

Task ID: 300093816

Date Completed: 20-Jun-1997 16:51 Replicates: 1

Version: 1

WALK-IN QUOTED COST:

WALK-IN CHARGE BALNC: 0

DESCRIPTION: TE-CL IN SOLID

Component	Results	Units
-----	-----	-----
CHLORIDE	6, 3, 3	ug/g

Notebook reference: 4070, PP. 142, 143

Analyst: TRH

Approved by: Marshall, Thomas K. 20-Jun-1997 16:54

D

Report on Am Samples for INEL Standards

NMT-2

Americium item, FZG45-AMP138-2, 27.0 grams NM, 32.3 grams net, was taken from the vault and samples drawn for particle size determination and standards. The process sheet attached lists the samples and sample weights.

History of the item:

1. Am separation using H₂O₂ precipitation filtrate containing Am²⁴¹ and trace plutonium was sent to OH precipitation.
2. Am hydroxide was desolved in HNO₃ (7M).
3. Am separation from residual Pu by ion exchange.
4. Am in ion exchange eluate precipitated using oxalic acid.
5. Am oxalate calcined to AmO₂.
6. Item screened through 100 mesh screen.
7. Item was sampled and packaged for shipment to Oak Ridge (Isotope Sales Pool).

Processing of Item by NMT-2:

1. The item was introduced into the glovebox line and taken to location G264. The material was contained in a small stainless steel container.
2. The oxide was transferred to a 1 pint container and blended in this container using a rod mill without the rods. The item was blended for 1 hour on 4/3/97.
3. The item was sampled immediately after blending using the an accountability balance accurate to 0.1 gram. Three 2.0 gram samples were taken for standards. One sample, FZG45-AMP138-2S2, was sent to CST-8, Bob Marshall, and the remaining two were placed in the vault as Lot ID's FZG45-AMP138-2S3 and FZG45-AMP138-2S4. A 3 gram sample, FZG45-AMP138-2S1, was provided to CST-15 for particle size determination.

Signed Gregory J. Jullio Date 4/13/97

FZG 45-AMP 138-2

PROCESS SHEET

[illegible]

E

FZG45-AMP138-2S1

Sample ID : CUT # 2
CF Code :
Date : 01/03/80
Time : 13:29:01
Diameter by Volume Mean : 16.7
Standard Deviation : 8.5
Skewness : 0.1
Kurtosis : 3.8
Coeff. of Variance(%) : 51.0
Counts : 44062

Cycles : 20
Scaling : 1.00

M.Cycle Time: 2.0 sec.
Averaged last 5 cycle(s)

Channel Size (microns)	%/Channel	Channel Size (microns)	%/Channel
<0.7	0.0123	25	9.4432
0.7	0.0354	31	4.2566
1.0	0.0865	37	1.4809
1.3	0.1196	44	0.3726
1.8	0.2187	53	0.0724
2.3	0.5969	63	0.0078
3.0	1.1932	75	0.0005
4.0	1.8850	88	0.0000
5.0	3.7011	105	0.0000
6.5	7.0054	125	0.0000
8	11.0746	149	0.0000
10	13.9221	177	0.0000
13	14.9617	210	0.0000
17	15.7722	250	0.0000
20	13.7813

LAB-TEC PARTICLE ANALYSIS SUMMARY

Ver. 5.20.07

FZG45-AMP138-2S1

Sample ID : CUT # 2
CF Code :
Date : 01/03/80
Time : 13:23:32
Spherical Equivalent Mean : 6.1
Standard Deviation : 5.5
Skewness : 0.3
Kurtosis : 6.4
Coeff. of Variance(%) : 89.6
Counts : 43793

Cycles : 20
Scaling : 1.00

M.Cycle Time: 2.0 sec.
Averaged last 5 cycle(s)

Channel Size (microns)	%/Channel	Channel Size (microns)	%/Channel
<0.7	11.0993	25	0.6706
0.7	3.4089	31	0.1758
1.0	4.9924	37	0.0343
1.3	4.0585	44	0.0056
1.8	4.4266	53	0.0007
2.3	7.3006	63	0.0001
3.0	8.7905	75	0.0000
4.0	8.0763	88	0.0000
5.0	9.2564	105	0.0000
6.5	10.4908	125	0.0000
8	10.3009	149	0.0000
10	7.5468	177	0.0000
13	4.7510	210	0.0000
17	2.9809	250	0.0000
20	1.6332

LAB-TEC PARTICLE ANALYSIS SUMMARY

Ver. 5.20.07

FZG45-AMP138-2S1

Sample ID :
CF Code :
Date : 01/03/80
Time : 13:05:22
Diameter by Volume Mean : 14.7
Standard Deviation : 7.9
Skewness : 0.2
Kurtosis : 4.1
Coeff. of Variance(%) : 53.8
Counts : 44688

Cycles : 20
Scaling : 1.00

M.Cycle Time: 2.0 sec.
Averaged last 5 cycle(s)

Channel Size (microns)	%/Channel	Channel Size (microns)	%/Channel
<0.7	0.0122	25	6.6179
0.7	0.0425	31	2.7350
1.0	0.1129	37	0.9020
1.3	0.1646	44	0.2004
1.8	0.3242	53	0.0329
2.3	0.9236	63	0.0045
3.0	1.8112	75	0.0005
4.0	2.7656	88	0.0000
5.0	5.1316	105	0.0000
6.5	9.5716	125	0.0000
8	13.8209	149	0.0000
10	15.2693	177	0.0000
13	14.6603	210	0.0000
17	14.0332	250	0.0000
20	10.8630

LAB-TEC PARTICLE ANALYSIS SUMMARY

Ver. 5.20.07

FZG45-AMP138-2S1

Sample ID :
CF Code :
Date : 01/03/80
Time : 12:59:51
Spherical Equivalent Mean : 5.7
Standard Deviation : 4.8
Skewness : 0.2
Kurtosis : 7.0
Coeff. of Variance(%) : 84.6
Counts : 44406

Cycles : 20
Scaling : 1.00

M.Cycle Time: 2.0 sec.
Averaged last 5 cycle(s)

Channel Size (microns)	%/Channel	Channel Size (microns)	%/Channel
<0.7	8.3469	25	0.3859
0.7	3.1013	31	0.0945
1.0	5.1506	37	0.0168
1.3	4.5005	44	0.0024
1.8	5.0798	53	0.0002
2.3	8.5120	63	0.0000
3.0	10.4549	75	0.0000
4.0	9.4943	88	0.0000
5.0	10.2237	105	0.0000
6.5	10.9499	125	0.0000
8	10.0163	149	0.0000
10	6.7283	177	0.0000
13	3.8034	210	0.0000
17	2.0963	250	0.0000
20	1.0420		

CST-8

Microanalysis

EPMA/SEM

Sample Description.

A small portion of U oxide powder was submitted for particle sizing (ID # 0907-43).

Analysis and results.

A few tens of milligrams of the powder was sonicated for about 20 minutes in distilled water and then transferred to a graphite planchet for sizing. Optical examination revealed some agglomeration and that only a small fraction of the particles were less than 1 micron in diameter. Based on these observations optical sizing was selected. Ten frames were collected and stored (see a typical example in Fig. 1). Each of the 10 images were loaded into the particle sizing software (NIH Image), a binary image created and then sized. When completed over 10,000 particles were sized. The average area was $98 \mu\text{m}^2$. Assuming spherical particles this would translate to an average diameter of 11 microns. The range of areas sized was 2 to $1460 \mu\text{m}^2$. A lower limit of 2 microns in diameter was set based on the attainable resolution.

Fig. 1. Photomicrograph showing the grey scale and binary images from a typical region.

Fig. 2.

DU Oxide Part. Size dist.

mean = 11μ
 largest = 43μ
 Spec
 $< 100\mu$
 No more
 than 2%
 $\geq 250\mu$

Fig. 2.

*assumes spherical particles

Fig. 3.

DU Oxide Part-Size dist.

Fig. 3.

G

JUNE 5, 1997

ITEM = 241AM58MG

DATA FAXED TO G. BECKER 6/7/97

FULFILLS 6/2/97 CEP SOW NEUTRON

COUNT REQUIREMENT, PIZ. Rfm/6-8-97

Instrument: Shuffler Neutron Counter						
Date Assayed: June 4, 1997						
Assay Mode: Passive						
	<u>TIME</u> sec	<u>TOTAL</u>	<u>R+A</u>	<u>A</u>	<u>R</u>	<u>R c/s</u>
BACKGROUND						
RUN 1	400	10958	1254	38	1216	3
RUN 2	1000	27594	3718	95	3623	3.6
RUN 3	4000	109377	14413	385	14028	3.5
AmO2						
RUN 1	400	59901	2612	1118	1494	3.7
RUN 2	1000	148648	6146	2875	3271	3.3
RUN 3	4000	596740	24785	11230	13555	3.4

H

September 24, 1997

Project Nos. 5061.06.0201 and 5061.06.0203
Subcontract No. B45370007-8N
Document No. AL-7286

Dr. Robert Marshall
Los Alamos National Laboratory
P.O. Box 1663, MS G740
Los Alamos, New Mexico 87545

Transmittal of the Letter Reports, "Pressurization of the PDP DE-Plutonium/Americium Matrix Cylinder Due to the Buildup of Hydrogen, Oxygen, and Helium Gases" and "Maximum Internal Allowable (Design) and Failure (Yield) Stresses on Stainless Steel and Zirconium Test Cylinders"

Dear Dr. Marshall:

Please find enclosed for your review the letter report entitled "Pressurization of the PDP DE-Plutonium/Americium Matrix Cylinder Due to the Buildup of Hydrogen, Oxygen, and Helium Gases," which summarizes the results of calculations performed by Benchmark Environmental Corporation (Benchmark). The calculation results estimate the pressure buildup in the Performance Demonstration Program (PDP) diatomaceous earth (DE)-plutonium/amerium matrix cylinders that is likely to occur over the course of the PDP DE-plutonium/amerium matrix cylinder experiment. This letter report also considers the radiolytic depletion of the residual water trapped in the test matrix.

Also enclosed is the letter report entitled "Maximum Internal Allowable (Design) and Failure (Yield) Stresses on Stainless Steel and Zirconium Test Cylinders," which documents the results of Benchmark's evaluation of the maximum internal allowable (design) pressures and the expected failure (yield) pressures for the cylinders used in the above-noted experiment. As seen in the attached letter of certification, this evaluation was reviewed by an independent registered professional engineer. This letter report incorporates your comments on the prior report (dated July 22, 1997).

Based upon the evaluations presented in the attached letter reports, the pressure inside the test cylinder rises to reach 22.6 psig after the experiment has proceeded for 27.9 years. This pressure increase is primarily due to the radiolysis of the residual water in the PDP DE-plutonium/amerium matrix. The residual water is completely consumed at 27.9 years. After the residual water has been consumed, the pressure inside the test cylinder continues to rise very slowly as a result of the helium gas formed by the decay of alpha-emitting radionuclides. The pressure inside the inner stainless steel cylinders will reach the material's maximum design and failure (yield) pressures within 83 and 176 years, respectively. The pressure inside the inner zirconium cylinders will reach the material's maximum design and failure (yield) pressures within 494 and 1,022 years, respectively. These values represent the length of time the experiment may run before the cylinders begin to experience plastic deformation, resulting in bulging and subsequent failure. The values presented above are affected by the assumptions that were made in the attached evaluations. No credit was given to fatigue, creep, corrosion, or hydrogen embrittlement impacting the cylinder's operating life; or to the outer cylinders providing extra strength or resistance to the internal cylinders.

Dr. Robert Marshall

-2-

September 24, 1997

Please contact Sinisa Djordjevic or Monika Kinker at (505) 262-2694 with any comments or concerns you may have regarding the enclosed.

Sincerely,

Anthony Drypolcher
Project Manager

Sinisa Djordjevic
Task Leader

AD/TK/PBS

Enclosures

cc with enclosures:

Greg Becker, LMITCO
William F. Lyon, Applied Sciences Laboratory, Albuquerque
Sinisa Djordjevic, Benchmark, Albuquerque
Monika Kinker, Benchmark, Albuquerque
Terry Kraus, Benchmark, Albuquerque
Central Files, Benchmark, Albuquerque

cc without enclosures:

Pete Ayala, LANL, BUS2, MS P274
Kenneth Hargis, LANL, EM/WM, MS J552

PRESSURIZATION OF THE PDP DE-PLUTONIUM/AMERICIUM MATRIX CYLINDER DUE TO THE BUILDUP OF HYDROGEN, OXYGEN, AND HELIUM GASES

September 24, 1997

Project No. B45370007-8N

Sinisa Djordjevic
Chemical Engineer

Terry Kraus
Health Physicist

Benchmark Environmental Corporation
4501 Indian School Road NE, Suite 105
Albuquerque, New Mexico 87110

This report summarizes the results of calculations (Benchmark 1997a and 1997b) made by Benchmark Environmental Corporation to estimate the gas pressure build-up that occurs, over time, in the PDP DE-Plutonium (Pu)/Americium (Am) test cylinders as the radionuclides in the waste matrix undergo radioactive decay. This paper investigates two gas generation processes that result in the pressurization of sealed waste containers. The processes are (a) the formation of hydrogen and oxygen gas by the radiolysis of water and (b) the formation of helium gas by radionuclides that decay via alpha emission. It is assumed that the radiolytically generated hydrogen and oxygen gas do not recombine to form water. Therefore, this report presents the worst case pressure buildup scenario.

The first gas generation process investigated in this analysis was the radiolysis of water by alpha particles resulting in the formation of hydrogen and oxygen gas. The waste matrix used in this analysis was contained in an air-tight test cylinder, and consisted of 65 g of a Pu blend (i.e., 73.70 g PuO₂) and 6.5x10⁻² g of ²⁴¹Am intermixed within 128 g of diatomaceous earth (DE) (LANL 1997). Table 1 indicates the isotopic composition of the Pu blend, by weight, and the decay heat of the plutonium isotopes. The effective (total) decay heat of the Pu blend is calculated in the fourth column of Table 1 by multiplying the weight percent of each plutonium isotope by the isotope's decay heat and then summing the decay heat contributions of each isotope to derive the total decay heat of the Pu blend. The decay heat of ²⁴¹Am is 1.16x10⁻¹ W/g (NRC 1997). The total decay heat of the Pu/Am mixture is calculated to be 0.16 W using Equation 1:

$$\begin{aligned} \text{Pu / Am decay heat} = & \text{Pu blend decay heat} \times \text{Pu blend mass} + \\ & {}^{241}\text{Am decay heat} \times {}^{241}\text{Am mass} \end{aligned} \quad (1)$$

where

$$\begin{aligned} \text{Pu blend decay heat} &= 2.34 \times 10^{-3} \text{ W/g} \\ \text{Pu blend mass} &= 65 \text{ g} \\ {}^{241}\text{Am decay heat} &= 1.16 \times 10^{-1} \text{ W/g} \\ {}^{241}\text{Am mass} &= 6.52 \times 10^{-2} \text{ g} \end{aligned}$$

Table 1. Isotopic Composition and Decay Heat of Pu Blend

Radionuclide	Weight Fraction ^a	Decay Heat (W/g) ^b	Decay Heat Contribution (W/g) (Column 2 x Column 3)
Pu-238	1.46×10^{-4}	5.73×10^{-1}	8.3658×10^{-5}
Pu-239	9.37722×10^{-1}	1.95×10^{-3}	1.8286×10^{-3}
Pu-240	5.9460×10^{-2}	7.16×10^{-3}	4.2573×10^{-4}
Pu-241	2.093×10^{-3}	3.31×10^{-3}	6.9278×10^{-6}
Pu-242	5.78×10^{-4}	1.17×10^{-4}	6.7626×10^{-8}
Totals	1.0	-	2.34E-3

^a LANL 1997.^b NRC 1997.

Although DE is included in the chemical lists for the TRUPACT-II content codes, DE is not considered to generate gas as a result of radiolysis, and therefore, the oxygen incorporated into the constituents comprising DE is not considered to be a source of radiolytically generated oxygen gas. Residual water trapped within the PuO₂ and the DE serves as the source of hydrogen and oxygen. The residual water content of PuO₂ is 0.2 percent by weight and the residual moisture content of DE is 0.11 percent by weight (LANL 1997). It was necessary to quantitate the amount of water in the waste matrix in order to determine the rate of hydrogen and oxygen gas formation. The weighted average residual water content of the waste matrix was calculated to be 1.43×10^{-3} g H₂O per g waste matrix (i.e., 0.143 percent by weight) using Equation 2:

$$\text{Wtd. av. water content} = \left(\frac{0.002 \text{ g H}_2\text{O}}{\text{g PuO}_2} \times 73.70 \text{ g PuO}_2 + \frac{0.0011 \text{ g H}_2\text{O}}{\text{g DE}} \times 128 \text{ g DE} \right) / 201.7 \text{ g test matrix} \quad (2)$$

where

Wtd av. water content = weighted average number of grams of H₂O per gram of waste matrix

and all other terms are as previously defined.

The total amount of residual water trapped in the test matrix was determined to be 0.29 g, or 0.016 moles. It was necessary to determine the total void volume in the test cylinder before the pressure buildup in the test cylinder could be evaluated. The inside volume of the test cylinder was calculated to be 19.243 in³ (315.3 cm³) using Equation 3:

$$\text{Volume} = \pi r^2 h \quad (3)$$

where:

$$\begin{aligned}\pi &= 3.141 \\ r &= \text{test cylinder radius, 0.855 in INEEL 1997} \\ h &= \text{test cylinder height, 8.379 in INEEL 1997}\end{aligned}$$

The volume of the waste matrix placed in each test cylinder was 315 cm³ (LANL 1997), leaving a head-space (void) volume of 315.3 cm³ - 315.0 cm³ = 0.3 cm³ in each test cylinder. However, the waste matrix is porous and also has a void volume that must be considered. Therefore, it was necessary to calculate the density of DE to determine the true volume occupied by the DE. Table 2 lists the weight fraction, the normalized weight fraction that takes into account the residual water in the DE matrix, the density, and the volume contribution of each constituent comprising DE. The density of the DE was calculated from Equation 4 (Butcher and Mendenhall 1993):

$$\begin{aligned}\text{Density (solid)} = \rho (DE) &= \frac{1}{V_{DE}} = \frac{1}{\sum \frac{w_i}{\rho_i}} \\ \frac{1}{\frac{w_{SiO_2}}{\rho_{SiO_2}}} + \frac{1}{\frac{w_{Fe_2O_3}}{\rho_{Fe_2O_3}}} + \frac{1}{\frac{w_{CaO}}{\rho_{CaO}}} + \frac{1}{\frac{w_{Na_2O}}{\rho_{Na_2O}}} + \frac{1}{\frac{w_{Al_2O_3}}{\rho_{Al_2O_3}}} &= \frac{1}{0.423}\end{aligned}\quad (4)$$

where

$$\begin{aligned}\rho(DE) &= \text{the calculated density of DE (2.364 g/cm}^3\text{)} \\ V_{DE} &= \text{the volume occupied by DE} \\ w_i &= \text{the normalized weight fraction, listed in Table 2, of the } i\text{th constituent comprising the DE (g/g DE)} \\ \rho_i &= \text{the crystalline density, listed in Table 2, of the } i\text{th constituent comprising the DE (g/cm}^3\text{)}\end{aligned}$$

Table 2. Composition of DE (LANL 1997)

Constituent	Weight Fraction	Normalized Weight Fraction, w_i (g i/g DE)	Crystalline Density, Y_i (g i/cm ³ i) ^a	Volume Contribution w_i/Y_i (cm ³ I/g DE)
SiO ₂	8.77 x 10 ⁻¹	9.04 x 10 ⁻¹	2.3	3.93 x 10 ⁻¹
Fe ₂ O ₃	1.31 x 10 ⁻²	1.4 x 10 ⁻²	5.24	2.7 x 10 ⁻³
CaO	3.5 x 10 ⁻³	4.0 x 10 ⁻³	3.32	1.2 x 10 ⁻³
Na ₂ O	3.2 x 10 ⁻²	3.3 x 10 ⁻²	2.27	1.45 x 10 ⁻²
Al ₂ O ₃	4.4 x 10 ⁻²	4.5 x 10 ⁻²	4.0	1.12 x 10 ⁻²
Totals	9.696 x 10 ⁻¹	1.000	-	4.23 x 10 ⁻¹

^a Weast 1978.

The density of the solid waste matrix was calculated to be 3.33 g/cm³ using Equation 5:

$$\text{Density (solid)} = \rho(\text{solid waste matrix}) = \frac{1}{V_{DE}} = \frac{1}{\sum \frac{w_i}{\rho_i}} = \frac{1}{\frac{w_{PuO_2}}{\rho_{PuO_2}} + \frac{w_{DE}}{\rho_{DE}}} \quad (5)$$

where

$$\begin{aligned} \rho(\text{solid waste matrix}) &= \text{the calculated density of the solid waste matrix (i.e., DE plus PuO}_2\text{/Am source material) (3.33 g/cm}^3\text{)} \\ w_{PuO_2} &= \text{the mass of PuO}_2\text{ per gram of waste matrix (73.70 g PuO}_2\text{/201.70 g waste matrix = 0.365 g Pu/g waste matrix)} \\ \rho_{PuO_2} &= \text{the density of PuO}_2\text{ (11.46 g/cm}^3\text{)} \\ w_{DE} &= \text{the mass of DE per gram of waste matrix (128 g DE/201.70 g waste matrix = 0.635 g DE/g waste matrix)} \\ \rho_{DE} &= \text{the density of DE (2.364 g/cm}^3\text{)} \end{aligned}$$

Equation 6 is used to calculate the porosity of the waste matrix (Butcher and Mendenhall 1993):

$$\text{Porosity} = 1 - \frac{\text{actual density}}{\text{solid matrix density}} = 0.808 \quad (6)$$

where

$$\begin{aligned} \text{actual density} &= \text{actual density of the waste matrix [(73.70 g PuO}_2\text{ + 128 g DE)/315 cm}^3\text{ = 0.640 g/cm}^3\text{]} \\ \text{solid matrix density} &= \text{solid matrix density of the waste matrix (3.33 g/cm}^3\text{)} \end{aligned}$$

The pore void volume of the waste matrix was calculated using Equation 7:

$$\text{Waste matrix pore void volume} = \text{porosity} \times \text{total volume} \quad (7)$$

where

$$\begin{aligned} \text{Waste matrix pore void volume} &= 254.5 \text{ cm}^3 \\ \text{porosity} &= \text{porosity of the waste matrix (0.808)} \\ \text{total volume} &= \text{total volume of the waste matrix (315 cm}^3\text{)} \end{aligned}$$

Therefore, the total void volume in the test cylinder was determined to be 254.8 cm³ by summing the head-space volume (0.3 cm³) and the pore void volume (254.5 cm³).

The effectiveness at which alpha particles decompose water (i.e., radiolysis) is described by the G-value. The effective G-value for hydrogen formation is calculated using Equation 8 (Table A2-5, NRC 1997):

$$G_{\text{eff}}(H) = G(H - \text{water radiolysis}) \times \text{wt. frac. water in sample} \times \text{fraction of alpha energy} \quad (8)$$

where:

$G_{\text{eff}}(H)$ = effective G-value for hydrogen formation (7.32×10^{-4} molecules H_2 /100 eV alpha energy)

$G(H\text{-water radiolysis})$ = 1.6 molecules H_2 formed/100 eV alpha energy

wt. frac. water in sample = the fraction of water in the waste matrix (1.43×10^{-3})

fraction of alpha energy = the fraction of alpha energy that escapes a 30 μm PuO_2 particle (0.32) (Table A2-5, NRC 1997)

The effective G-value for oxygen formation is calculated using Equation 9 (NRC 1997):

$$G_{\text{eff}}(O) = G(O - \text{water radiolysis}) \times \text{wt. frac. water in sample} \times \text{fraction of alpha energy} \quad (9)$$

where:

$G_{\text{eff}}(O)$ = effective G-value for oxygen formation (3.66×10^{-4} molecules O_2 /100 eV alpha energy)

$G(O\text{-water radiolysis})$ = 0.8 molecules O_2 formed/100 eV alpha energy

and all other terms are as previously defined.

The net gas generation rate is calculated using Equation 10:

$$G_{\text{eff}}(\text{net gas}) = G_{\text{eff}}(H) + G_{\text{eff}}(O) = 1.10 \times 10^{-3} \text{ molecules / 100 eV} \quad (10)$$

where all terms are as previously defined.

Equation 11 (NRC 1977) is used to calculate the gas generation rate in the waste matrix due the radiolysis of water by alpha particles:

$$C_g = \frac{Q_i G_{\text{eff}}}{N_A 1.602 \times 10^{-19} \frac{\text{W sec}}{\text{eV}}} \quad (11)$$

where

C_g = gas generation rate in the test cylinder due to radiolysis (1.82×10^{-11} mole/sec)

Q_i = decay heat (0.16 W)

G_{eff} = effective net G-value (1.10×10^{-3} molecules/100 eV)

N_A = Avogadro's number (6.0225×10^{23} molecules/mole)

Equation 12 (BEC 1997a) is used to calculate the test cylinder pressure, at time t , due to the hydrogen and oxygen formed by the radiolysis of residual water in the waste matrix:

$$P_{(t)} = \frac{P_o V + C_g t R T}{V} \times K \quad (12)$$

where

- $P_{(t)}$ = the test cylinder pressure (psi) at time t
- P_o = initial test cylinder (ambient) pressure at Los Alamos, New Mexico (0.7755 atm)
- V = void volume in test cylinder (254.8 cm³)
- C_g = gas generation rate (1.82x10⁻¹¹ mole/sec)
- t = elapsed time (sec)
- R = Gas Law Constant (82.057 atm cm³/mol K) (Weast 1978)
- T = absolute temperature of test cylinder (298.15 K)
- K = conversion factor (14.696 psi/atm) (Weast 1978)

The pressure profile of the test cylinder was calculated, using Equation 12, from the time the experiment started (time zero) to 78.5 years. The results from Equation 12, in units of psi, absolute, were converted to psi, gauge (psig) by subtracting the ambient pressure (11.4 psi) at Los Alamos, New Mexico. The calculated pressure profile (psig versus time) of the test cylinder is graphed in Figure 1. Inspection of Figure 1 reveals that the pressurization of the test cylinder that results from the radiolysis of water, represented by the solid line, reaches a maximum value of 22.6 psig at 27.9 years. The pressure profile becomes flat after 27.9 years because the total amount of residual water (i.e., 0.016 moles) trapped in the test matrix has been consumed by radiolysis at this point in time.

The second gas generation process investigated in this analysis considered the pressure buildup in the test cylinder due to the formation of helium gas. Helium gas is formed by alpha-emitting radionuclides because the alpha particles will eventually acquire two electrons from the surrounding matter to form neutral helium atoms. It is assumed that one helium atom is formed each time an alpha-emitting radionuclide undergoes radioactive decay. Table 3 lists the characteristics of the radionuclides comprising the Pu/Am source material.

The mass of each plutonium isotope in 65 g of the Pu-Am alpha source material is calculated in the sixth column of Table 3 by dividing the weight percent by 100 to get the mass of each isotope in one gram of source material and then multiplying the mass per gram by 65 to derive the mass of each radionuclide per 65 g of source material. The mass of the individual plutonium isotopes comprising the source material was calculated from Equation 13:

$$Mass_i = \frac{weight\ percent_i}{100} \times 65\ g \quad (13)$$

where

- $Mass_i$ = mass of the i th radionuclide (g)
- $weight\ percent_i$ = weight percent of the i th radionuclide (from Table 3)
- 100 = conversion factor to convert percentage values to fractional values
- 65 g = mass of the Pu/Am source material.

The alpha-specific activity of each radionuclide comprising the source material is taken from the LANL RAD_CODE Table (LANL 1995) and is listed in Table 3. The alpha activity (Ci) of each radionuclide comprising the source material is calculated using Equation 14:

$$Alpha\ Activity_i = mass_i \times \alpha - specific\ activity_i \quad (14)$$

AR-7230

7

Figure 1. Matrix Cylinder Gas Pressure Buildup (psig vs. time)

where

Alpha Activity_i = the alpha radioactivity (Ci) of the i th radionuclide comprising the source material

mass_i = the mass of the i th radionuclide comprising the source material (g)

$\alpha\text{-specific activity}_i$ = the α -specific activity of the i th radionuclide comprising the source material (Ci/g)

Table 3. Characteristics of Pu/Am Source Material

Radio-nuclide	α -Specific Activity (Ci/g) ^a	Half-life (yr) ^a	Decay Constant (s ⁻¹)	Weight Percent per Pu ^b	Mass per 65 g Pu (g)	α Activity per 65 g Pu (Ci)
Pu238	1.7119×10^1	8.77×10^1	2.51×10^{-10}	1.46×10^{-2}	9.49×10^{-3}	1.624×10^{-1}
Pu239	6.204×10^{-2}	2.41×10^4	9.12×10^{-13}	9.377×10^1	6.095×10^1	3.781
Pu240	2.2696×10^{-1}	6.56×10^3	3.35×10^{-12}	5.946	3.865	8.772×10^{-1}
Pu241	2.48×10^{-3}	1.44×10^1	1.53×10^{-9}	2.093×10^{-1}	1.360×10^{-1}	3.37×10^{-4}
Pu242	3.926×10^{-3}	3.76×10^5	5.84×10^{-14}	5.78×10^{-2}	3.757×10^{-2}	1.47×10^{-4}
Am241	3.428	4.33×10^2	5.08×10^{-11}	1.0×10^{-1}	6.52×10^{-2}	2.234×10^{-1}

^a LANL 1995.

^b LANL 1997.

The number of alpha decays (transformations) each radionuclide undergoes during a time interval (time integrated decay) is calculated from Equation 15:

$$\text{Number of decays}_i = C \frac{A_{\alpha,i}}{\lambda_i} (1 - e^{-\lambda_i t}) \quad (15)$$

where

$\text{Number of decays}_i$ = the number of transformations the i th radionuclide undergoes during the time interval

C = the constant 3.7×10^{10} decays/sec (dps) per curie, used to convert Ci to dps

$A_{\alpha,i}$ = the alpha activity of the i th radionuclide at the beginning of the interval (Ci)

λ_i = the decay constant of the i th radionuclide (sec⁻¹)

e = base of the natural log

t = length of the interval (sec)

The number of alpha decays, as calculated above, is equivalent to the number of atoms of helium formed during the time interval in question. The number of moles of helium atoms formed during a time interval is calculated using Equation 16:

$$\text{moles He} = \frac{\text{atoms of He}}{6.0225 \times 10^{23} \frac{\text{atoms}}{\text{mole}}} \quad (16)$$

where

moles He = the number of moles of helium formed during the specified time interval
 6.0225×10^{23} atoms/mole = Avogadro's number

The Ideal Gas Law (Equation 17) is used to determine the pressure created by the helium formed by the decay of the *i*th radionuclide:

$$P = \frac{nRT}{V} \times K \quad (17)$$

where

P = the calculated gas pressure created by the helium (psi)
n = the number of moles of helium formed by the radioactive decay of the *i*th radionuclide
R = the Gas Law constant (82.057 atm cm³/mole K) (Weast 1978)
T = the absolute temperature of the test cylinder (298.15 K)
V = the void volume in the test cylinder (254.8 cm³)
K = conversion factor (14.696 psi/atm) (Weast 1978)

The gas pressure created by the helium, formed from the radioactive decay of the *i*th radionuclide comprising the source material, was calculated for time intervals ranging from 10 days to 78.5 years. The total gas pressure created by the radioactive decay of the radionuclides comprising the source material is calculated by summing the pressures created by the helium from the decay of each radionuclide. The results from Equation 17 were converted into units of psig as described above.

Figure 1 graphically presents the buildup of pressure (psig) in the test cylinder versus time for the following cases: (a) helium plus atmospheric pressure; (b) hydrogen and oxygen plus atmospheric pressure; and (c) helium, hydrogen, and oxygen plus atmospheric pressure. Inspection of Figure 1 indicates that the test cylinder pressure profile is dominated by the radiolysis gas generation process until all of the residual water in the test matrix has been consumed by radiolysis after the experiment has proceeded for 27.9 years. The pressure profile continues to slowly rise, after all the residual water has been consumed, as a result of the pressurization created by the buildup of helium gas, formed by the decay of alpha emitting radionuclides. The pressurization created by the buildup of helium gas is negligible when compared to the pressurization created by the buildup of hydrogen and oxygen gas, formed by radiolysis. The test cylinder reaches a pressure of 23.6 psig at 78.5 years.

REFERENCES

- BEC, 1997a. *PDP-DE-Plutonium/Americium Matrix Cylinder Gas Pressure Build-Up*, Benchmark Environmental Corporation. Albuquerque, New Mexico.
- BEC, 1997b. *PDP-DE-Plutonium/Americium Matrix Cylinder Helium Pressure Build-Up*, Benchmark Environmental Corporation. Albuquerque, New Mexico.
- Butcher and Mendenhall, 1993. *A Summary of the Models Used for the Mechanical Response of Disposal Rooms in the Waste Isolation Pilot Plan with regard to Compliance with 40 CFR 191, Subpart B*, SAND92-0427, Sandia National Laboratories, Albuquerque, New Mexico
- INEEL, 1997. Fax from Greg Becker, March 31, 1997, Idaho National Engineering and Environmental Laboratory, Idaho Falls, Idaho.
- LANL, 1995. RAD_CODE Table, Los Alamos National Laboratory, Los Alamos, New Mexico.
- LANL, 1997. Fax from Dr. Robert Marshall, March 28, 1997, Los Alamos National Laboratory, Los Alamos, New Mexico.
- NRC, 1997. *Safety Analysis Report for the TRUPACT-II Shipping Package*, Revision 16, NRC Docket No. 9218, U.S. Nuclear Regulatory Commission, Washington, D.C.
- Weast, R. C., 1978. *Handbook of Chemistry and Physics*, 58th edition, CRC Press, West Palm Beach, Florida.

MAXIMUM INTERNAL ALLOWABLE (DESIGN) AND FAILURE (YIELD) STRESSES ON STAINLESS STEEL AND ZIRCONIUM TEST CYLINDERS

September 24, 1997

Project No. B45370007-8N

**Monika Kinker
Materials Engineer**

**John Kinker, P.E.
Environmental Engineer**

**Benchmark Environmental Corporation
4501 Indian School Road NE, Suite 105
Albuquerque, New Mexico 87110**

1.0 INTRODUCTION

Benchmark Environmental Corporation (Benchmark) performed an evaluation of the maximum internal allowable (design) operating pressures and the expected failure (yield) pressures for the cylinders used in the diatomaceous earth (DE)-plutonium (Pu)/americium (Am) matrix gas cylinder experiment. The evaluation was based on information supplied to Benchmark by the Los Alamos National Laboratory Inorganic Elemental Analysis Group, which is attached for reference. The evaluated cylinders are seamless stainless steel and seamless zirconium cylinders with flat endcaps welded onto each cylinder's ends. Inner and outer cylinders of the same material were designed and assembled so that the inner cylinder would fit snugly within the outer cylinder. Benchmark performed this evaluation, which (as seen in the attached letter of certification) was reviewed by an independent registered professional engineer, to determine: 1) the maximum design internal pressures the cylinders are expected to withstand, and 2) the internal pressure at which the cylinders are expected to fail. The methodology used and the results are presented herein.

2.0 CALCULATION

The attached calculation note, "Allowable Stresses of PDP DE-Pu/Am Matrix Test Cylinders," evaluates the maximum design and yield pressures using standard analytical techniques for designing thin-walled pressure vessels, which are broken down into the pressure on the cylindrical shell and the pressure on the endcaps. The internal pressure on the cylindrical shell was determined using hoop stress analysis for thin-walled cylindrical vessels, while internal pressure on the endcaps was determined for uniformly-loaded, flat circular plates. Dimensional tolerances were incorporated so that the most limiting internal pressure would result. Applicable safety factors for hoop stress and endcap stress were based on and applied to the yield strength of each material type to obtain the maximum design pressures. No safety factor was used to determine yield pressures.

This evaluation used the following input data and assumptions:

- Yield strengths of 30,000 psig for the stainless steel AISI type 304 and 66,000 psig for the zirconium UNS R60802
- Weld joint efficiencies were not considered (cylinder tubes are seamless and endcaps are electron-beam, tungsten-inert gas, or laser welded with 100 percent weld penetration required)
- High temperature effects, including creep, were not considered (the test was assumed to be conducted at ambient temperature)
- Corrosion was not considered (DE matrix is not expected to contribute to corrosion of the assemblies)
- Hydrogen embrittlement was not considered (radioactivity levels are not expected to be sufficient enough to contribute to hydrogen embrittlement of the tubing assemblies)
- Fatigue of the material (failure of the material due to repeated application of stress below the material's yield strength) was not considered
- Contribution of outer cylinder to strength of inner cylinder was not considered

3.0 RESULTS

Table 3-1 lists the allowable (design) and maximum (yield) internal pressures calculated for the tubes and endcaps for both the inner and outer cylinders for each material. Table 3-1 also shows maximum design internal pressures for the inner stainless steel and zirconium assemblies of 25 and 149 psig, respectively. These values represent the maximum operating pressures at or below which the cylinders are expected to operate continuously without failing. Above this value, the cylinders have an increasing probability of plastic deformation and subsequent mechanical failure that approaches 100 percent at the yield pressure. The pressures at which the inner stainless steel and zirconium cylinders will exceed their yield strengths are 53 and 308 psig, respectively. Table 3-1 shows the weakest component for both the stainless steel and zirconium cylinders as being the top endcap, with the zirconium cylinders having six times greater strength than the stainless steel cylinders.

4.0 DISCUSSION

Figure 4-1 shows a stress-strain diagram, which illustrates the response (stress) of a metal to an applied force (strain). Below the yield strength, the bonds between the atoms are stretched but not broken. Thus, the assembly experiences elastic, or recoverable, deformation as the internal pressure increases, so that the material returns to its original shape once the applied force is removed. This elastic deformation accounts for the linear relationship between the stress experienced by the material and the applied force. At the yield strength of the material, plastic deformation begins to take place so that the component permanently deforms from its original shape.

**Table 3-1. Design and Failure (Yield) Internal Pressure
on Tubing and Top and Bottom Endcaps**

Material Type	Cylinder Pressure (psig)		Top Endcap Pressure (psig)		Bottom Endcap Pressure (psig)	
	design	yield	design	yield	design	yield
<i>Stainless Steel</i>						
Inner Tube	826	1,364	25	53	288	596
Outer Tube	1,161	1,915	38	78	257	533
<i>Zirconium</i>						
Inner Tube	2,043	3,371	149	308	183	380
Outer Tube	2,530	4,175	130	269	216	447

PART II CONTROLLING THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MATERIALS

Figure 4-1. Stress-Strain Diagram for an Aluminum Alloy (Askeland 1985)

The tensile strength is the stress obtained at the highest applied force and thus is the maximum stress on the stress-strain curve. The tensile strength is the easiest value to obtain and is therefore the most reported for most materials, but is less useful than the yield strength in the design and manufacturing of materials.

As the pressure increases, the material eventually fails. The breaking strength is the value at which the material fails. However, in the case of the evaluated assemblies, the yield strength is considered the failure strength, because the internal pressure on the assemblies is modeled to rise continuously. No pressure relief is expected because the assemblies are welded shut; thus, the applied force will continue to increase, and eventually, the material will fail.

In the case of the evaluated assemblies, failure will most likely occur at or near the welded region of the endcaps. Generally, the high temperatures associated with welding causes the physical properties of the localized region of the weld to change, resulting in the weld region to potentially be the weakest region of the assembly. This potential should be minimized by the types of welding specified (i.e., electron beam, etc.), which exhibit high cooling rates and may preclude major or serious alteration of the material's microstructure. An additional consideration is the location of the welded zone in proximity to increased mechanical strain.

The top endcaps of the stainless steel cylinders have a groove machined near the perimeter to allow for deflection and an interference fit into the outer cylinder. The reduced thickness of the grooved area of the endcap resides in the area of highest stress; therefore, this thickness was used as the dimension of the top inner and outer endcaps in determining the maximum design and yield pressures for the stainless steel cylinders.

BENCHMARK

ENVIRONMENTAL CORPORATION

CALCULATION NOTE

Date: July 9, 1997 Project Number: 5061.06.0203 Sheet No. 1 of 3
Prepared By: Monika Kinker Checked By: [Signature] Check Date: 7-8-97
Calculation Title: Allowable Stresses of PDP DE-Pu/Am Matrix Test Cylinders

Purpose:

The purpose of this calculation is to evaluate the internal design stresses on stainless steel and zirconium cylinders used in the Performance Demonstration Project (PDP) diatomaceous earth (DE) -Plutonium (Pu)/Americium(Am) Matrix Gas Cylinder project. More specifically, this calculation estimates the maximum internal allowable pressure, and the pressure that would result in failure of the cylindrical structures and the endcaps of the cylinders.

Approach:

The evaluation of the maximum allowable internal pressure is broken down into pressure on the cylindrical shell and the pressure on the endcaps. The internal design pressure for the shell is obtained by the following equation for hoop stress in thin-walled cylindrical vessels:

$$\sigma = \frac{pd}{2t} \rightarrow p = \frac{2t\sigma}{d}; \quad \sigma_{allow} = \frac{\sigma}{n} \quad \begin{array}{l} \text{(Azbel et al 1982)} \\ \text{(Askeland 1985)} \end{array}$$

where p = internal pressure (psi)
 d = inside diameter (in.)
 t = thickness (in.)
 σ = circumferential or hoop stress (psi)
 n = safety factor

The safety factor η_y for yield strength is recommended by Azbel *et al.* as 1.65.

The design of the endcaps for the stainless steel and zirconium cylindrical vessels being reviewed in this calc note is similar to what Azbel *et al.* calls a flat head closure. The maximum internal pressure is obtained by the following equation for a uniformly loaded flat circular plate that is clamped at the edges (due to circumferential weld):

$$\sigma = 0.49p(rs)^2 \rightarrow p = \frac{\sigma(s/r)^2}{0.49} \quad \text{(Azbel et al 1982)}$$

where s = thickness of plate (in.)
 r = radius of plate (in.)

Under the same conditions, the maximum internal pressure at the edge of the endcaps is:

$$\sigma = 0.75p(rs)^2 \rightarrow p = \frac{\sigma(s/r)^2}{0.75}$$

Azbel *et al.* recommends smaller allowable stresses for flat heads than for cylindrical shells, with $\eta_y = 2.07$.

BENCHMARK

ENVIRONMENTAL CORPORATION

CALCULATION NOTE

Date: July 9, 1997 Project Number: 5061.06.0203 Sheet No. 2 of 3
Prepared By: Monika Kinker Checked By: [Signature] Check Date: 7-8-97
Calculation Title: Allowable Stresses of PDP DE-Pu/Am Matrix Test Cylinders

Input Data:

Input data on material specifications (wall thickness, diameter, etc.) are obtained from the attached statements of work.

The properties for the stainless steel and zirconium tubing and endcaps are as follows:

Material	Specification	Yield σ (psi)
Stainless Steel	SAE type 30404 or AISI type 304	30,000 ^a
Zirconium	ASTM Designation B353-91: R60001, R60802, or R60804 UNS	66,000 ^b

Source: ^a Askeland, 1985
^b Rust, 1979

Assumptions:

The following assumptions are made in this calculation:

- Cylinder tubes are seamless
- Endcaps are electron-beam, tungsten-inert gas, or laser welded with 100% penetration and certification of He leak testing provided to client
- Cylinders will not be heated above ambient
- Thickness allowances for corrosion are ignored
- Hydrogen embrittlement will not occur

Computation:

All computations are performed in the attached spreadsheet "Pressure Calculation." All equations used to calculate hoop stress and stress on the flat circular endcaps are as described earlier. The smallest dimension in the endcap design was used to provide a conservative estimate. Dimensional tolerances for the stainless steel and zirconium tubes were incorporated in the hoop stress and center and edge endplate stress equations so that the most limiting (smallest) internal pressure would result.

Results:

The attached spreadsheet shows the allowable and maximum (based on yield strength of material) internal pressures on the cylinder and endcaps for both the inner and outer tube for each material. Table 1 lists results of the attached calculations.

As seen in the attached spreadsheet, the pressure on the edge of the endcaps was the most limiting value, most likely because for this type of shape (cylindrical vessel with flat end) the stress is concentrated at the edge of the end plate.

BENCHMARK

ENVIRONMENTAL CORPORATION

CALCULATION NOTE

Date: July 9, 1997 Project Number: 5061.06.0203 Sheet No. 3 of 3
Prepared By: Monika Kinker Checked By: J. B. K. Check Date: 7-8-97
Calculation Title: Allowable Stresses of PDP DE-Pu/Am Matrix Test Cylinders

Table 1. Maximum Internal Allowable and Yield Pressures

Material Type	Maximum Cylinder Design pressure (psi)		Maximum Top Endcap Design Pressure at Edge (psi)		Maximum Bottom Endcap Design Pressure at Edge (psi)	
	allowable	yield	allowable	yield	allowable	yield
Stainless Steel						
Inner Tube	826	1364	25	53	288	596
Outer Tube	1161	1915	38	78	257	533
Zirconium Tubing						
Inner Tube	2043	3371	149	308	183	380
Outer Tube	2530	4175	130	269	216	447

As seen above, the limiting design pressure for both the stainless steel and zirconium tubings is the top endcap. The entire thickness of the bottom endcaps for both material types, which had machined insets on the inner and outer bottom endcaps, and the entire thickness of the zirconium top endcaps, which had no machined insets, was used in the evaluation of maximum internal pressure; however, the top endcaps for the stainless steel tubing had deep grooves near the outer edge to assist in the insertion of the endcaps into the tubing, with the inner tubing endcap machined to provide an interference fit inside the inner tubing and the outer tubing endcap machined to provide a clearance fit inside the outer tubing. Because the groove was so close to the edge of the endcap and is therefore in the area of stress concentration, both from the strain induced by the interference fit of the end cap and by deflection caused by internal pressure, the groove was considered for the evaluation of maximum internal pressure at the edge of the endcap. Therefore, the effective thickness of the endcap was equated to the original thickness minus the groove depth.

References:

Askeland, Donald. 1985. The Science and Engineering of Materials. Boston, Massachusetts, PWS Publishers.

Azbel, David S. *et al.* 1982. Chemical and Process Equipment Design. Ann Arbor, Michigan, Ann Arbor Science Publishers.

LANL. 1995/6. Statements of Work for the tubing and endcap components and assemblies. Los Alamos, New Mexico, Los Alamos National Laboratory.

Rust, James H. 1979. Nuclear Power Plant Engineering. Atlanta, Georgia, Haralson Publishing Company.

Pressure Calculation

	A	B	C	D	E	F	G	H	I
1	Hoop Stress on Cylinder								
2	P = $\frac{2 \cdot t \cdot \text{sig}(a)}{d}$			t =	smallest thickness of cylinder wall (in.)				
3	d			sig =	maximum design stress (psi)				
4				sig(a) =	allowable stress (psi)				
5				d =	largest inside diameter (in.)				
6				values resulting in smallest P used					
7									
8	Material	sig	sig(a)	t	d	P(max)	P(allow)		
9		(psi)	(psi)	(in.)	(in.)	(psi)	(psi)		
10	SS								
11	Inner	30000	18182	0.04	1.76	1364	826		
12	Outer	30000	18182	0.06	1.88	1915	1161		
13	Zirc								
14	Inner	66000	40000	0.045	1.762	3371	2043		
15	Outer	66000	40000	0.06	1.897	4175	2530		
16									
17	Uniformly Loaded Clamped Plate Stress								
18	P(ctr)=		$\text{sig}(a) \cdot (s/r)^2$		s=	smallest thickness of plate (in.)			
19			0.49		r=	largest radius of plate (in.)			
20					values resulting in smallest P used				
21	P(edge)=		$\text{sig}(a) \cdot (s/r)^2$						
22			0.75						
23									
24						maximum		allowable	
25		sig	sig(a)	s	r	P(ctr)	P(edge)	P(ctr)	P(edge)
26	Material	(psi)	(psi)	(in.)	(in.)	(psi)	(psi)	(psi)	(psi)
27	SS								
28	Inner								
29	bottom	30000	14493	0.105	0.860	913	596	441	288
30	top	30000	14493	0.031	0.855	80	53	39	25
31	Outer								
32	bottom	30000	14493	0.105	0.910	815	533	394	257
33	top	30000	14493	0.040	0.904	120	78	58	38
34	Zirc								
35	Inner								
36	bottom	66000	31884.06	0.056	0.853	581	380	281	183
37	top	66000	31884.06	0.050	0.845	472	308	228	149
38	Outer								
39	bottom	66000	31884.06	0.065	0.913	683	447	330	216
40	top	66000	31884.06	0.050	0.905	411	269	199	130

Handwritten signature and date: 1-5-97

CEP WRMs

IAP WRMs

WRM ID	CEP001	CEP002	CEP003	CEP004	CEP005	CEP006	CEP007	CEP008	CEP009	CEP010
Blend ID	CEP001B	CEP002	CEP003	CEP004	CEP005	CEP006	CEP007	CEP008	CEP009	CEP010

Americium

200023528 (Cut #)	36	37	38	39	40	41	42	43	44	45
AmO ₂ (g)	0.01320	0.01249	0.06614	0.06682	0.11450	0.11575	0.06905	0.06568	0.13138	0.12679
Am* (g) in the AmO ₂ in the blend	0.01039	0.00983	0.05207	0.05261	0.09014	0.09113	0.05436	0.05171	0.10343	0.09982
Am* (g) in the PuO ₂ in the blend	9.120E-05	8.413E-05	2.125E-03	2.147E-03	2.130E-03	2.128E-03	4.227E-04	4.419E-04	-----	-----
Total Am* (g) in the blend	0.01048	0.00992	0.05419	0.05475	0.09227	0.09325	0.05478	0.05215	0.10343	0.09982
% hold-up	0.052	0.051	0.063	0.028	0.034	0.022	0.029	0.033	0.034	0.043

Total Am* in WRM (g)	0.010478	0.009912	0.054161	0.054737	0.092241	0.093234	0.054766	0.052133	0.103396	0.099775
----------------------	----------	----------	----------	----------	----------	----------	----------	----------	----------	----------

Plutonium

200023717 (Cut #)	9	10	11	12	13	14	15	16	-----	-----
PuO ₂ (g)	0.09721	0.08968	2.26525	2.28825	2.27046	2.26803	0.45051	0.47090	-----	-----
Pu* (g) in the PuO ₂ in the blend	0.08535	0.07874	1.98898	2.00918	1.99355	1.99142	0.39557	0.41347	-----	-----
% hold-up	0.052	0.051	0.063	0.028	0.034	0.022	0.029	0.033	-----	-----

Total Pu* in WRM (g)	0.08531	0.07870	1.98773	2.00861	1.99288	1.99098	0.39545	0.41333	-----	-----
----------------------	---------	---------	---------	---------	---------	---------	---------	---------	-------	-------

DE (g) in WRM	134.57	134.49	134.00	133.99	133.99	133.98	134.49	134.49	134.49	134.50
---------------	--------	--------	--------	--------	--------	--------	--------	--------	--------	--------

Shipping Drum	1	2	1	2	1	2	1	2	1	2
---------------	---	---	---	---	---	---	---	---	---	---

1 = 16-55-96 017

2 = 16-55-96 015

* Decay Corrected to 6/1/97

Am assay = 78.727 % on 6/1/97

Pu assay = 87.804 % on 6/1/97

Am from decay of Pu (in PuO₂) = 1069 µg Am/g Pu on 6/1/97

7

CEP WORMs

DU WRMs						
WRM ID	CEP011	CEP012	CEP013	CEP014	CEP015	CEP016
Blend ID	CEP011a	CEP012	CEP013	CEP014	CEP015	CEP016
Uranium						
200023256 (Cut #)	26	27	28	29	30	31
UO ₂ (g)	570.61	1010.37	1094.58	1097.25	1111.82	1084.47
U (g)	499.24	884.00	957.68	960.01	972.76	948.84
% hold-up	0.013	0.007	0.008	0.053	0.012	0.018
U in WRM (g)	499.18	883.94	957.60	959.51	972.64	948.66
DE (g) in WRM	113.49	41.95	30.14	30.48	30.71	30.62
Shipping Drum	1	2	1	2	1	2
1 = 16-55-96 017						
2 = 16-55-96 015						
Note: The U values are not decay corrected due to the relatively long half-lives of the U isotopes.						

K

Waste Item Measurements for CEP Uranium Waste Items (7/97)

STANDARD STD#2 67.4 mg DU
 STD#1 168.5 mg DU

Count		63.32 keV, 234Th						
DATE	Time (s)	ID	SPEC	CT	cpm	ERR	%ERR	cpm/mg DU
7/11/97	30000	STD#2	DUSTD2	1484	2.968	188	12.67	0.044
7/16/96	30000	STD#2	DUSTD2b	1401	2.802	187	13.35	0.042
7/14/97	10000	STD#1	DUSTD1	1331	7.986	110	8.26	0.047
7/16/97	10000	STD#1	DUSTD1b	1120	6.72	110	9.82	0.040
AVG								0.043
STD								0.003
%RSD								7.55

63.32 keV, 234Th										
DATE	Time (s)	ID	SPEC	CT	cpm	ERR	%ERR	mg DU in waste	Total U	% Hold-Up*
7/11/97	10000	CEP011a	CEP11-W	1170	7.02	109	9.32	162.42	499.01	0.033
7/11/97	10000	CEP012	CEP12-W	432	2.592	107	24.77	59.97	884.00	0.007
7/14/97	10000	CEP013	CEP13-W	546	3.276	107	19.60	75.80	957.68	0.008
7/15/97	10000	CEP014	CEP14-W	3664	21.984	121	3.30	508.64	960.01	0.053
7/15/97	10000	CEP015	CEP15-W	871	5.226	108	12.40	120.91	972.76	0.012
7/11/97	10000	CEP016	CEP16-W	1259	7.554	113	8.98	174.78	948.84	0.018

Waste Item Measurements for CEP Am Waste Items (6/97)									
STANDARD	Am241=	27.015	µCi or	7.873	µg	4/27/97	51.00	days	
	Am241 =	27.009	µCi or	7.879	ng	6/17/97			
	Count			Am241		Counting			
DATE	Time (s)	ID	SPEC	ROI	ERR	%ERR	µCi	cpm/µCi	
6/17/97	1200	4/97 High STD	STD0617a	98430	353	0.36	27.009	182.22	
6/17/97	1200	4/97 High STD	STD0617b	103697	369	0.36	27.009	191.97	
6/17/97	1200	4/97 High STD	STD0617c	102594	359	0.35	27.009	189.93	
6/18/97	1200	4/97 High STD	STD0618a	102946	359	0.35	27.009	190.58	
							AVG, cpm/µCi	188.67	
							STD	3.80	
Blending Bottles							%RSD	2.01	
	Count			Am241		Counting			Total
DATE	Time (s)	ID	SPEC	ROI	ERR	%ERR	µCi	µg	Am, mg
6/17/97	1200	CEP001-Pro	CEP001-Pro	148216	427	0.29	39.28	11.45	58.0
6/17/97	1200	CEP001-B	CEP001-B	70866	299	0.42	18.78	5.47	10.5
6/17/97	1200	CEP002	CEP002-W	66096	290	0.44	17.52	5.10	9.9
6/17/97	1200	CEP003	CEP003-W	444003	740	0.17	117.67	34.29	54.2
6/17/97	1200	CEP004	CEP004-W	198572	496	0.25	52.62	15.34	54.8
6/17/97	1200	CEP005	CEP005-W	408645	712	0.17	108.29	31.56	92.3
6/17/97	1200	CEP006	CEP006-W	263178	571	0.22	69.74	20.33	93.3
6/17/97	1200	CEP007	CEP007-W	209350	508	0.24	55.48	16.17	54.8
6/18/97	1200	CEP008	CEP008-W	222826	525	0.24	59.05	17.21	52.2
6/18/97	1200	CEP009	CEP009-W	458186	751	0.16	121.42	35.39	103.4
6/18/97	1200	CEP010	CEP010-W	550938	825	0.15	146.00	42.55	99.8
6/18/97	1200	CEP005-Vial	CEP005-V	109957	373	0.34	29.14	8.49	92.3
									0.009

Decay Corr. for Hold-up

Total Am in the <u>Blends</u>	CEP001	CEP002	CEP003	CEP004	CEP005	CEP006	CEP007	CEP008	CEP009	CEP010
grams as of 6/1/97	0.010500	0.009930	0.054490	0.055050	0.092570	0.093550	0.054840	0.052210	0.103430	0.099820
grams as of 6/17/97	0.010499	0.009929	0.054486	0.055046	0.092563	0.093543	0.054836	0.052206	0.103423	0.099813
grams as of 6/18/97	0.010499	0.009929	0.054486	0.055046	0.092563	0.093543	0.054836	0.052206	0.103422	0.099813
μ g Am in hold-up	5.47	5.1	34.29	15.34	31.56	20.33	16.17	17.21	35.39	42.55
% Hold-up	0.052	0.051	0.063	0.028	0.034	0.022	0.029	0.033	0.034	0.043

Isotope	half-life (years)	half-life (days)	decay const.	At. Weight
241Am	432.7	157935.5	4.39E-06	241.0568
	35598	35582	16	
	35599	35582	17	

L

Gamma-Ray Measurement, 600 seconds													
59.5 keV 241Am													
WRM#	Time (s)	Side	Am, mg	Counts	±	L + R	±	(L + R)/mg	±	L+R/mg - mea	2 sigma	Statistical Difference from the mean?	L/R
CEP001	600	L	10.4778	11750	121	23787	173	2270	16	27	351	no	0.98
CEP001	600	R	10.4778	12037	123								
CEP002	600	L	9.9121	10808	118	21597	164	2179	17	64	351	no	1.00
CEP002	600	R	9.9121	10789	118								
CEP003	600	L	54.1621	59368	270	119288	383	2202	7	41	350	no	0.99
CEP003	600	R	54.1621	59920	271								
CEP004	600	L	54.7379	54107	259	107893	368	1971	7	272	350	no	1.01
CEP004	600	R	54.7379	53786	259								
CEP005	600	L	92.2422	102536	353	205095	499	2223	5	20	350	no	1.00
CEP005	600	R	92.2422	102559	353								
CEP006	600	L	93.2348	98057	345	190381	482	2042	5	201	350	no	1.06
CEP006	600	R	93.2348	92324	338								
CEP007	600	L	54.7680	68923	272	137330	388	2507	7	264	350	no	1.01
CEP007	600	R	54.7680	68407	277								
CEP008	600	L	52.1327	58825	267	117228	377	2249	7	5	350	no	1.01
CEP008	600	R	52.1327	58401	266								
CEP009	600	L	103.3964	117609	378	233310	530	2256	5	13	350	no	1.02
CEP009	600	R	103.3964	115701	373								
CEP010	600	L	99.7750	126938	391	252894	552	2533	6	289	350	no	1.01
CEP010	600	R	99.7750	125756	390								

Mean 2243
Std. Dev. 175
%RSD 8

Gamma-Ray Measurement, 600 seconds													
129 keV 239Pu													
WRM#	Time (s)	Side	Pu, g	Counts	±	L + R	±	(L + R)/g	±	L+R/g - mean	2 sigma	Statistical Difference from the mean?	L/R
CEP001	600	L	0.0854	391	29	750	41	8788	480	1355	1600	no	1.09
CEP001	600	R	0.0854	359	29								
CEP002	600	L	0.0788	284	27	545	39	6920	494	510	1617	no	1.09
CEP002	600	R	0.0788	281	28								
CEP003	600	L	1.9880	7181	97	14318	137	7201	69	230	1287	no	1.01
CEP003	600	R	1.9880	7135	97								
CEP004	600	L	2.0089	7087	96	14001	135	6970	67	481	1287	no	1.03
CEP004	600	R	2.0089	6914	95								
CEP005	600	L	1.9934	7159	89	14445	140	7247	70	184	1287	no	0.98
CEP005	600	R	1.9934	7286	89								
CEP006	600	L	1.9915	7067	98	13666	139	7013	70	418	1287	no	1.02
CEP006	600	R	1.9915	6899	98								
CEP007	600	L	0.3957	1447	51	2906	72	7343	182	88	1330	no	0.99
CEP007	600	R	0.3957	1459	51								
CEP008	600	L	0.4136	1681	52	3295	73	7987	178	538	1327	no	1.02
CEP008	600	R	0.4136	1634	51								
CEP009	600	L											
CEP009	600	R											
CEP010	600	L											
CEP010	600	R											

Mean 7431
Std. Dev. 640
%RSD 9

CEP ²⁴¹Am WRMs Homogeneity Measurements

					Gamma-Ray Measurement, 600 seconds, 7/11/97, 7/15/97, 7/17/97							
					59.5 keV 241Am				129 keV 239Pu			
WRM #	Time (s)	Side	Am, mg	Pu, g	count	+/-	L/R	ct/mg	count	+/-	L/R	ct/g
CEP001	600	L1	10.48	0.09	11750	121	1.000	1112	391	29		4134
CEP001	600	R1			12037	123			359	29		
CEP001	600	L2			11545	119			363	29		
CEP001	600	R2			11857	121			330	29		
CEP001	1800	L3			12009				322			
CEP001	7200*	CEN			10911				307		0.976	3642
CEP001	7200*	L4			11512				315			
CEP002	600	L1	9.92	0.08	10808	116	1.002	1089	284	27	1.088	3460
CEP002	600	R1			10789	116			261	28		
CEP003	600	L1	54.21	1.99	59368	270	0.991	1100	7181	97	1.006	3598
CEP003	600	R1			59920	271			7135	97		
CEP004	600	L1	54.76	2.01	54107	259	1.006	985	7087	96	1.025	3484
CEP004	600	R1			53786	259			6914	95		
CEP005	600	L1	92.29	1.99	102536	353	1.000	1111	7159	99	0.983	3622
CEP005	600	R1			102559	353			7286	99		
CEP006	600	L1	93.27	1.99	98057	345	1.045	1021	7067	98	1.004	3537
CEP006	600	R1			92324	336			6899	98		
CEP006	600	R2			95400	341			7172	90		
CEP007	600	L1	54.79	0.40	68923	272	1.021	1198	1447	51	1.035	3855
CEP007	600	R1			68407	277			1459	51		
CEP007	600	L2			63690	276			1656	53		
CEP007	600	R2			61498	271			1539	51		
CEP008	600	L1	52.16	0.41	58825	267	1.011	1128	1661	52	1.016	3883
CEP008	600	R1			58401	266			1634	51		
CEP008	600	L2			58637	266			1565	50		
CEP008	600	R2			58180	258			1607	52		
CEP008	600	L3			60003	267			1630	51		
CEP008	600	R3			58923	261			1539	52		
CEP009	600	L1	103.45		117609	376	1.018	1118				
CEP009	600	L2			115701	373						
CEP009	600	R1			114137	372						
CEP009	600	R2			114976	373						
CEP010	600	L1	99.83		126938	391	0.999	1189				
CEP010	600	R1			125756	390						
CEP010	600	L2			110417	362						
CEP010	600	R2			111864	363						
					AVG	1105			AVG**	3635		
					STD	65			STD	158		
					%RSD	5.89			%RSD	4.35		

*The counts in 59.5-keV and 129-keV peaks were expressed as 600 s counts.

** The short counts (600, 1800 s) on CEP001 WRM were not included in the average of normalized 129-keV peak area due to poor peak shape and counting statistics.

M

Gamma-Ray Measurement, 600 seconds 186 keV 235U															
WRM#	Time (s)	Segment	U, g	Counts	±	1+2+3	±	(1+2+3)/g	±	1+2+3/g - mean	2 sigma	Statistical Difference from the mean?	Segments		
													1/2	1/3	2/3
CEP011	7200	1	499.18	13464	648	40104	1122	80	2	29	29	no?	0.98	1.04	1.06
CEP011	7200	2	499.18	13680	648										
CEP011	7200	3	499.18	12960	648										
CEP012	7200	1	883.94	12744	720	41472	1247	47	1	4	29	no	0.85	0.93	1.09
CEP012	7200	2	883.94	14976	720										
CEP012	7200	3	883.94	13752	720										
CEP013	7200	1	957.60	14616	720	42192	1247	44	1	7	29	no	1.00	1.13	1.14
CEP013	7200	2	957.60	14688	720										
CEP013	7200	3	957.60	12888	720										
CEP014	7200	1	959.51	14184	720	45000	1247	47	1	4	29	no	0.85	1.01	1.19
CEP014	7200	2	959.51	16776	720										
CEP014	7200	3	959.51	14040	720										
CEP015	7200	1	972.64	12960	720	40824	1247	42	1	9	29	no	0.88	0.98	1.11
CEP015	7200	2	972.64	14688	720										
CEP015	7200	3	972.64	13176	720										
CEP016	7200	1	948.66	14904	720	45360	1247	48	1	4	29	no	0.99	0.97	0.98
CEP016	7200	2	948.66	15048	720										
CEP016	7200	3	948.66	15408	720										

Mean	51
Std. Dev.	14
%RSD	28

Count time 2 h,
segm width 2"

*** U-235 LONG REPORT/G501 ***
Uncorrected Counts/sec

		88 keV PEAK	136 keV PEAK	✓ 186 keV PEAK	279 keV PEAK
SEGMENT # 1	OPEN	133.37	1973.88	15.67	1115.08
	+/-	0.82	2.19	0.47	1.64
	CLOSED	751.98	0.49	0.40	0.03
	+/-	1.32	0.07	0.05	0.03
SEGMENT # 2	OPEN	673.59	136.21	7.14	242.09
	+/-	1.29	0.62	0.32	0.72
	CLOSED	753.80	0.64	1.22 -	-0.05
	+/-	1.32	0.08	0.07	0.05
SEGMENT # 3	OPEN	716.68	7.10	4.86	122.77
	+/-	1.31	0.26	0.23	0.52
	CLOSED	754.46	0.53	1.87 -	0.12
	+/-	1.32	0.09	0.09	0.06
SEGMENT # 4	OPEN	718.84	7.16	4.72	107.12
	+/-	1.31	0.25	0.23	0.49
	CLOSED	754.13	0.75	1.90 -	0.26
	+/-	1.32	0.09	0.09	0.06
SEGMENT # 5	OPEN	722.92	6.27	4.55	100.59
	+/-	1.31	0.24	0.23	0.47
	CLOSED	754.72	0.72	1.80 -	0.32
	+/-	1.32	0.09	0.09	0.06
SEGMENT # 6	OPEN	490.35	1639.95	10.61	1121.88
	+/-	1.19	1.90	0.41	1.54
	CLOSED	756.89	0.32	1.21 -	0.19
	+/-	1.32	0.08	0.07	0.04
SEGMENT # 7	OPEN	476.29	1624.08	11.63	1193.95
	+/-	1.19	1.90	0.42	1.59
	CLOSED	758.78	0.60	0.34	0.06
	+/-	1.32	0.07	0.05	0.03

*** Uranium 235 Report/G501 ***

Date & Time: 10-Jul-97 18:40
Operator ID: FH
Sample ID: CEP011
Isotope: U-235
Enrichment: 0.19
Parameter File Number: 14
Corrected Counts: 33.0258 +/- 0.7855 cps
Isotope Weight: 33.0258 +/- 0.7855 grams
Total SNM: 17381.9785 +/- 413.3983 grams
*** ASSAY IS SUSPECT ***

*** U-235 LONG REPORT/G501 ***
Uncorrected Counts/sec

		88 keV PEAK	136 keV PEAK	186 keV PEAK	279 keV PEAK
SEGMENT # 1	OPEN	135.33	1987.08	15.96	1127.27
	+/-	0.82	2.19	0.47	1.64
	CLOSED	767.54	0.47	0.28	0.03
	+/-	1.32	0.07	0.06	0.03
SEGMENT # 2	OPEN	739.52	27.95	3.58	29.63
	+/-	1.32	0.32	0.22	0.27
	CLOSED	768.60	0.60	1.28	-0.03
	+/-	1.32	0.08	0.08	0.05
SEGMENT # 3	OPEN	755.79	3.42	2.73	9.81
	+/-	1.32	0.16	0.16	0.17
	CLOSED	768.59	0.96	1.77-	0.24
	+/-	1.32	0.10	0.10	0.07
SEGMENT # 4	OPEN	757.28	3.70	2.81	10.00
	+/-	1.32	0.16	0.16	0.17
	CLOSED	766.15	0.95	2.08 -	0.20
	+/-	1.32	0.10	0.10	0.07
SEGMENT # 5	OPEN	752.72	3.50	2.73	10.29
	+/-	1.32	0.16	0.16	0.18
	CLOSED	768.08	0.61	1.91-	0.32
	+/-	1.32	0.09	0.10	0.07
SEGMENT # 6	OPEN	513.86	1640.23	10.74	1091.10
	+/-	1.20	1.89	0.40	1.51
	CLOSED	767.56	0.59	1.02 -	-0.01
	+/-	1.32	0.08	0.08	0.05
SEGMENT # 7	OPEN	485.62	1616.60	11.44	1197.19
	+/-	1.19	1.89	0.42	1.59
	CLOSED	770.93	0.62	0.34	-0.04
	+/-	1.32	0.07	0.06	0.04

*** Uranium 235 Report/G501 ***

Date & Time: 11-Jul-97 11:33
 Operator ID: CG
 Sample ID: CEP012
 Isotope: U-235
 Enrichment: 0.19
 Parameter File Number: 14
 Corrected Counts: 47.1131 +/- 1.2297 cps
 Isotope Weight: 47.1131 +/- 1.2297 grams
 Total SNM: 24796.3906 +/- 647.2253 grams
 *** ASSAY IS SUSPECT ***

*** U-235 LONG REPORT/G501 ***
Uncorrected Counts/sec

		88 keV PEAK	136 keV PEAK	186 keV PEAK	279 keV PEAK
SEGMENT # 1	OPEN	141.60	2042.12	15.09	1159.54
	+/-	0.83	2.21	0.47	1.66
	CLOSED	769.37	0.52	0.36	0.08
	+/-	1.32	0.07	0.06	0.03
SEGMENT # 2	OPEN	748.41	16.77	3.35	17.85
	+/-	1.31	0.27	0.19	0.22
	CLOSED	767.64	0.84	1.42	0.22
	+/-	1.32	0.08	0.08	0.06
SEGMENT # 3	OPEN	756.25	3.01	2.84	7.52
	+/-	1.31	0.16	0.15	0.16
	CLOSED	768.25	0.63	-2.03	0.13
	+/-	1.32	0.09	0.10	0.07
SEGMENT # 4	OPEN	755.09	3.37	2.66	6.68
	+/-	1.31	0.16	0.15	0.15
	CLOSED	768.23	0.87	-2.04	0.19
	+/-	1.32	0.10	0.10	0.07
SEGMENT # 5	OPEN	756.87	3.36	2.83	6.89
	+/-	1.31	0.15	0.15	0.15
	CLOSED	766.28	1.03	-1.79	0.27
	+/-	1.32	0.09	0.10	0.07
SEGMENT # 6	OPEN	519.54	1607.64	10.57	1068.92
	+/-	1.21	1.87	0.39	1.50
	CLOSED	768.93	0.76	1.22	0.16
	+/-	1.32	0.08	0.08	0.05
SEGMENT # 7	OPEN	491.23	1604.36	11.76	1188.52
	+/-	1.19	1.88	0.42	1.58
	CLOSED	770.61	0.52	0.52	0.06
	+/-	1.32	0.07	0.06	0.04

*** Uranium 235 Report/G501 ***

Date & Time: 15-Jul-97 15:13
Operator ID: CG'
Sample ID: CEP013
Isotope: U-235
Enrichment: 0.19
Parameter File Number: 14
Corrected Counts: 52.5802 +/- 1.3252 cps
Isotope Weight: 52.5802 +/- 1.3252 grams
Total SNM: 27673.7695 +/- 697.4525 grams
*** ASSAY IS SUSPECT ***

*** U-235 LONG REPORT/G501 ***
Uncorrected Counts/sec

		88 keV PEAK	136 keV PEAK	186 keV PEAK	279 keV PEAK
SEGMENT # 1	OPEN	132.59	1984.18	14.71	1124.26
	+/-	0.82	2.19	0.47	1.64
	CLOSED	763.45	0.44	0.46	0.07
	+/-	1.32	0.07	0.05	0.03
SEGMENT # 2	OPEN	652.39	634.75	8.08	435.24
	+/-	1.28	1.19	0.32	0.95
	CLOSED	765.46	0.70	1.33	0.13
	+/-	1.32	0.08	0.08	0.05
SEGMENT # 3	OPEN	752.59	3.63	3.13	7.72
	+/-	1.32	0.16	0.16	0.16
	CLOSED	761.24	0.78	1.97	0.22
	+/-	1.32	0.09	0.10	0.07
SEGMENT # 4	OPEN	754.20	3.60	3.00	6.24
	+/-	1.32	0.16	0.15	0.15
	CLOSED	763.92	0.88	2.33	0.29
	+/-	1.32	0.09	0.10	0.07
SEGMENT # 5	OPEN	754.93	3.51	2.66	5.40
	+/-	1.32	0.16	0.15	0.14
	CLOSED	767.73	0.98	1.95	0.23
	+/-	1.32	0.10	0.10	0.07
SEGMENT # 6	OPEN	512.77	1638.61	10.87	1093.23
	+/-	1.20	1.89	0.40	1.52
	CLOSED	764.45	0.66	1.10	0.20
	+/-	1.32	0.08	0.08	0.06
SEGMENT # 7	OPEN	481.95	1606.93	10.62	1192.66
	+/-	1.19	1.88	0.42	1.59
	CLOSED	768.85	0.46	0.37	0.10
	+/-	1.33	0.07	0.06	0.04

*** Uranium 235 Report/G501 ***

Date & Time: 11-Jul-97 15:57
 Operator ID: CG
 Sample ID: CEP014
 Isotope: U-235
 Enrichment: 0.19
 Parameter File Number: 14
 Corrected Counts: 49.5422 +/- 1.2456 cps
 Isotope Weight: 49.5422 +/- 1.2456 grams
 Total SNM: 26074.8398 +/- 655.5772 grams
 *** ASSAY IS SUSPECT ***

*** U-235 LONG REPORT/G501 ***

Uncorrected Counts/sec

		88 keV PEAK	136 keV PEAK	186 keV PEAK	279 keV PEAK
SEGMENT # 1	OPEN	144.42	2054.76	13.66	1167.39
	+/-	0.83	2.21	0.47	1.65
	CLOSED	784.24	0.46	0.46	0.04
	+/-	1.32	0.07	0.06	0.03
SEGMENT # 2	OPEN	760.58	14.81	3.09	14.30
	+/-	1.32	0.26	0.19	0.20
	CLOSED	776.04	0.71	1.27	0.22
	+/-	1.32	0.09	0.08	0.06
SEGMENT # 3	OPEN	766.46	3.00	2.74	7.34
	+/-	1.32	0.15	0.15	0.16
	CLOSED	771.82	0.74	1.80	0.30
	+/-	1.32	0.09	0.10	0.07
SEGMENT # 4	OPEN	764.80	2.92	2.77	5.31
	+/-	1.32	0.15	0.15	0.14
	CLOSED	769.84	0.71	2.04	0.13
	+/-	1.32	0.10	0.10	0.07
SEGMENT # 5	OPEN	761.71	2.71	2.53	5.38
	+/-	1.32	0.15	0.14	0.14
	CLOSED	773.69	0.82	1.83	0.26
	+/-	1.32	0.09	0.10	0.07
SEGMENT # 6	OPEN	521.02	1638.98	10.82	1091.50
	+/-	1.20	1.89	0.40	1.51
	CLOSED	772.11	0.68	1.18	0.12
	+/-	1.32	0.08	0.08	0.05
SEGMENT # 7	OPEN	491.28	1619.24	11.56	1199.87
	+/-	1.19	1.89	0.42	1.59
	CLOSED	777.95	0.56	0.46	0.09
	+/-	1.32	0.07	0.06	0.04

*** Uranium 235 Report/G501 ***

Date & Time: 14-Jul-97 11:04

Operator ID: CG

Sample ID: CEP015

Isotope: U-235

Enrichment: 0.19

Parameter File Number: 14

Corrected Counts: 50.0062 +/- 1.3173 cps

Isotope Weight: 50.0062 +/- 1.3173 grams

Total SNM: 26319.0527 +/- 693.3214 grams

*** ASSAY IS SUSPECT ***

*** U-235 LONG REPORT/G501 ***
Uncorrected Counts/sec

		88 keV PEAK	136 keV PEAK	186 keV PEAK	279 keV PEAK
EGMENT # 1	OPEN	141.74	2046.15	15.24	1162.07
	+/-	0.83	2.21	0.47	1.66
	CLOSED	772.28	0.54	0.38	0.07
	+/-	1.32	0.07	0.05	0.03
EGMENT # 2	OPEN	704.14	307.23	5.27	223.89
	+/-	1.30	0.85	0.28	0.69
	CLOSED	770.19	0.49	1.13	0.14
	+/-	1.32	0.08	0.08	0.06
EGMENT # 3	OPEN	763.40	2.94	2.66	6.90
	+/-	1.32	0.15	0.15	0.15
	CLOSED	769.90	0.83	2.07	0.22
	+/-	1.32	0.09	0.10	0.07
EGMENT # 4	OPEN	757.57	3.35	2.83	7.34
	+/-	1.31	0.15	0.15	0.15
	CLOSED	768.35	0.72	2.09	0.25
	+/-	1.32	0.10	0.10	0.07
EGMENT # 5	OPEN	762.27	2.92	2.90	5.70
	+/-	1.32	0.15	0.15	0.15
	CLOSED	771.57	1.02	2.14	0.20
	+/-	1.32	0.09	0.10	0.07
EGMENT # 6	OPEN	523.21	1609.97	10.13	1069.48
	+/-	1.21	1.87	0.39	1.50
	CLOSED	771.04	0.50	1.25	0.16
	+/-	1.32	0.08	0.08	0.05
EGMENT # 7	OPEN	494.30	1607.72	11.90	1186.61
	+/-	1.19	1.88	0.42	1.58
	CLOSED	774.99	0.36	0.29	0.08
	+/-	1.32	0.07	0.06	0.04

*** Uranium 235 Report/G501 ***

Date & Time: 15-Jul-97 12:53
Operator ID: CG
Sample ID: CEP016
Isotope: U-235
Enrichment: 0.19
Parameter File Number: 14
Corrected Counts: 51.0776 +/- 1.2678 cps
Isotope Weight: 51.0776 +/- 1.2678 grams
Total SNM: 26882.9473 +/- 667.2374 grams
*** ASSAY IS SUSPECT ***

Z

LOS ALAMOS
NATIONAL LABORATORY
SHIPPING MANIFEST #
310958

IMPORTANT: SEE INSTRUCTIONS FOR COMPLETING THE SHIPPING MANIFEST.
THIS FORM MUST BE TYPED. REPRODUCE A COPY FOR YOUR FILES.

From Phillip Noll		Telephone Number (505) 667-4970		Date 7/22/97	
Group CST-8		Mail Stop G740		Laboratory's Reference # (e.g., Purchase Order #) None	
Return Material Authorization Number 				Consignee's Reference Number 	
Consignee's Name, Complete Street Address, Zip Code <i>(Include all destinations on international shipments)</i> INEEL/LMITCO RWMC MS4202 Scoville, Idaho 83415 ATTN: Cliff Stanley				Delivery Date Requested 	
Telephone Number: <u>208-526-5637</u> Fax Number: <u>208-526-2033</u> <small>Required for RAM/CLASSIFIED shipments</small>				Cost Code / Program Code / Cost Acct / Wk Pkg K40 8BAN/MSSG/0000/0000	
Carrier				☐ PPD ☐ Collect	
Carrier Waybill Number 					
Pieces		Weight		Kind of Package	

ALL PACKAGES AND CORRESPONDENCE MUST BE IDENTIFIED WITH LABORATORY REFERENCE NUMBER

Item No.	Quantity	Complete Description (Manufacturer, model number, serial number)	Property Number	Line Value
1	1	RQ Radioactive Material N.O.S. 7, UN2982, 238Pu, 239Pu, 240Pu, 241Pu, 242Pu, 241Am, U238, Normal Form Solid, Chemical Form Elemental, 3.91TBq. Radioactive Yellow II, TI , DOT 6M Type B Container 16-55-96 \$/\$N015, 741 #AUA-JSG-10. TID #B26332		
2	1	RQ Radioactive Material, N.O.S., 7 UN2982, 238Pu, 239Pu, 240Pu, 241Pu, 242Pu, 242Am U238, Normal Form Solid, Chemical Form Elemental, 3.91 TBq, Radioactive Yellow II, TI , DOT 6M Type B Container 16-55-96\$/\$N017, 741 #AUA-JSG-10, TID #B26333		
NON-WEAPONS RELATED, PERMANENT TRANSFER				

Reason for Shipment/Justification for Premium Transportation Support of inter-job standards measurement program	Total Value
---	----------------------------

HAZARDOUS MATERIAL EMERGENCY RESPONSE CONTACTS			
Name	Group	Office Telephone	After Hour Telephone
1. EMO		505-667-6211	
2. David Martinez	CST-3	505-667-4989	473-1324
3. Phillip Noll	CST-8	505-667-4970	466-6507

WARNING: IT IS A FEDERAL OFFENSE TO VIOLATE SHIPPING REGULATIONS FOR HAZARDOUS MATERIALS, WASTE, OR SUBSTANCES.

Check all that apply

☐ Return of Loan # _____ ☒ Permanent Transfer # _____ ☐ Repair ☐ Loan, # _____ How Long? _____ (1 year MAXIMUM) ☐ Return No Replacement ☐ Government Furnished Property ☐ FRAGILE	☐ Security Classification of this SM Document _____ ☐ Security Classification of this Material _____ ☐ Export (Country of Origin _____) ☐ Hazardous/Radioactive Material HMTF# _____ RMTF# _____ ☐ Material Safety Data Sheet (MSDS) ☐ RUSH ☐ Other: _____
---	---

Pick-up Date 7/25/97	From P. Noll	Group CST-8	Site 3	Building/Drop Point 29/05S	Telephone Number 7-4970
Authorized Signature		Property Administrator Signature		Buyer Signature 	
Type Name, Title, and Z Number Robert Marshall, TSM, 083739		Type Name, and Z Number Kerry Coffelt, 093102		Type Name, and Z Number 	
Export License 		TDO Checked By 		Packed By 	Date Shipped

Items in Drum 16-55-96 017					
ID	Amt. Am (g)	Amt. Pu (g)	Amt. U (g)	Gamma Dose Direct Meas. (at Contact) mR/hr.	Alpha/Beta-Gamma Swipe mR/hr.
CEP001	0.01048	0.08531	-----	3.1	NDA
CEP003	0.05416	1.98773	-----	15	NDA
CEP005	0.09224	1.99288	-----	24	NDA
CEP007	0.05477	0.39545	-----	16	NDA
CEP009	0.10340	-----	-----	27	NDA
CEP011	-----	-----	499.18	0.4	NDA
CEP013	-----	-----	957.60	0.6	NDA
CEP015	-----	-----	972.64	0.7	NDA

Items in Drum 16-55-96 015					
ID	Amt. Am (g)	Amt. Pu (g)	Amt. U (g)	Gamma Dose Direct Meas. (at Contact) mR/hr.	Alpha/Beta-Gamma Swipe mR/hr.
CEP002	0.00991	0.07870	-----	3.0	NDA
CEP004	0.05474	2.00861	-----	15	NDA
CEP006	0.09323	1.99098	-----	23	NDA
CEP008	0.05213	0.41333	-----	14	NDA
CEP010	0.09978	-----	-----	28	NDA
CEP012	-----	-----	883.94	0.7	NDA
CEP014	-----	-----	959.51	0.7	NDA
CEP016	-----	-----	948.66	0.7	NDA

NDA = No Detectable Activity

HPAL ANALYSIS REPORT FORM

FILE: 97011453

SAMPLE DESCRIPTION ANALYSIS REQUESTED RCT
HPAL 1 at CMR

Login Comments: CEP-PROJECT

Sample Date: 7/22/97

TA: 3

Nucon

LLD (cpm): Alpha = 3 Beta= 8

Name: Kenny Espinosa

TA: 3

BLDG: 29

Analyst: Michael Jensen

MS: G749

Sample ID #	Alpha Activity dpm	2*sigma cpm	Beta Activity dpm	2*sigma cpm
1	NDA	NDA	NDA	NDA
2	NDA	NDA	NDA	NDA
3	NDA	NDA	NDA	NDA
4	NDA	NDA	NDA	NDA
5	NDA	NDA	NDA	NDA
6	NDA	NDA	NDA	NDA
7	NDA	NDA	NDA	NDA
8	NDA	NDA	NDA	NDA
9	NDA	NDA	NDA	NDA
10	NDA	NDA	NDA	NDA
11	NDA	NDA	NDA	NDA
12	NDA	NDA	NDA	NDA
13	NDA	NDA	NDA	NDA
14	NDA	NDA	NDA	NDA
15	NDA	NDA	NDA	NDA
16	NDA	NDA	NDA	NDA

ESH-1 SMEAR SURVEY FORM

SAMPLE DESCRIPTION

Sample Date/Time: 7-22-97 No. Of Samples: 16
 TA: 3 Bldg: 29 CMA W-5/W-3
 RCT: K ESPINOZA Z Number: 96185
 Phone/Fax: 5-1607

PURPOSE OF SURVEY

- ☐ ROUTINE ☐ PRE-JOB ☐ POST-JOB ☐ HOT-JOB
☐ ITEM RELEASE ☒ OFFSITE SHIPMENT ☐ ONSITE SHIPMENT
☐ NON-ROUTINE/OTHER: _____

ADDITIONAL INFORMATION

- ☐ Occurrence No.: _____
☐ Incident No.: _____
☐ RWP No.: _____

SAMPLE TRACKING NUMBER

ESH-1 SAMPLE TRACKING

INSTRUMENTATION

TYPE	HSE No.	CAL DUE	% EFF	BKC

Smear No.	Location	Alpha*	Beta*	Gamma*
1	CEP 13			
2	CEP 14			
3	CEP 15			
4	CEP 16			
5	CEP 11			
6	CEP 12			
7	CEP 01			
8	CEP 02			
9	CEP 03			
10	CEP 04			
11	CEP 05			
12	CEP 06			
13	CEP 07			
14	CEP 08			
15	CEP 09			

Smear No.	Location	Alpha*	Beta*	Gamma*
16	CEP 10			
17	N/A			
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30

Page 1

[illegible]

[illegible]

Page 4

[illegible]

Page 6

Phil Noll SA uncertainty Conditioned on the SAi values
COMPLETE CASE SA = **0.06833**

$SA = F \cdot \{ \text{SUM}(F_i \cdot SA_i) \} + F_{am} \cdot SA_{am}$ (241 term has BR factor)

$V(SA) = (F^2) \cdot V(\text{SUM}) + (\text{SUM}^2) \cdot V(F) + (F_{am}^2) \cdot V(SA_{am}) + (SA_{am}^2) \cdot V(F_{am})$

$V(\text{SUM}) = \text{SUM}(V(F_i \cdot SA_i)) + 2 \text{SUM}(SA_{ij} \text{COV}_{ij})$

$V(F_i \cdot SA_i) = (F_i^2) \cdot V(SA_i) + (SA_i^2) \cdot V(F_i)$

$SA_{ij} \text{COV}_{ij} = SA_i \cdot SA_j \cdot \text{RHO} \cdot \text{SD}(F_i) \cdot \text{SD}(F_j) = SA_i \cdot SA_j \cdot \text{COV}_{ij}$

Set all Var(SAi) values to 0.0

case %RSD
 pu
 238 0.098952514
 239 0.002382143
 240 0.014096329
 241 0.749791452
 242 0.500268001
 am 0.04
 % RSD are from 2 yrs of data
 and not from the original data

	Value	F*SA	s.d.	%rsd		V(Fi*SAj)	SD(term)	%rsd
f-Pu	0.87804		0.0003300000	0.0376				
f-238	0.0001432	0.002454178	0.0000001417	0.0990		5.90E-12	2.43E-06	0.00355
SA-238	17.138114		0.0000000000	0.0000				
f-239	0.937811	0.058217753	0.0000223400	0.0024		1.92E-12	1.39E-06	0.00203
SA-239	0.062078343		0.0000000000	0.0000				
f-240	0.0594481	0.013500721	0.0000083800	0.0141		3.62E-12	1.90E-06	0.00279
SA-240	0.22710097		0.0000000000	0.0000				
f-241	0.0020379	5.16425E-06	0.0000152800	0.7498	Inc. BR	1.50E-15	3.87E-08	0.00006
SA-241	103.4329		0.0000000000	0.0000				
alphaBR24	0.0000245							
f-242	0.0005597	2.21619E-06	0.0000028000	0.5003		1.23E-16	1.11E-08	0.00002
SA-242	0.003959599		0.0000000000	0.0000				
f-am	0.000938486	0.003222295	0.0000001679	0.0179		3.32E-13	5.77E-07	0.00084
SA-am	3.434201727		0.0000000000	0.0000				
Sum-noAm		0.074180032						
Am term		0.003222295						

0.000939
 0.003225

COV	SDi	SDj	RHO	COVij	SAIJCOVij
8-9	0.000004	0.000006	1	2.58E-11	2.74488E-11
8-0	0.000004	0.000005	1	2.15E-11	8.36798E-11
8-1	0.000004	0.000002	1	8.60E-12	3.73496E-13
8-2	0.000004	0.000001	1	4.30E-12	2.91798E-13
9-0	0.000006	0.000005	1	3.00E-11	4.22942E-13
9-1	0.000006	0.000002	1	1.20E-11	1.88776E-15
9-2	0.000006	0.000001	1	6.00E-12	1.47483E-15
0-1	0.000005	0.000002	1	1.00E-11	5.75498E-15

Pu SA-unc (Phil Noll) (2)

0-2	0.000005	0.000001	1	5.00E-12		4.49614E-15
1-2	0.000002	0.000001	1	2.00E-12	Inc. BR	5.06821E-15

$V(\text{SUM}) = \text{SUM}(V(F_i \cdot \text{SA}_i)) + 2\text{SUM}(\text{COV}(i,j))$ 2.35915E-10

$V(\text{SA}) = (F^2) \cdot V(\text{SUM}) + (\text{SUM}^2) \cdot V(F) + (\text{Fam}^2) \cdot V(\text{SAam}) + (\text{SAam}^2) \cdot V(\text{Fam})$ 7.815E-10
SD(SA) 0.0000280
%rsd 0.0413

SA Uncertainty			
		SD	%RSD
Approximate 95% Confidence Bound is 2 SD or 2%RSD		5.59E-05	0.083
Significant Digits: 1 or 2 maximum.			
	1 sig.digit	0.0002	0.003
	2 sig.digits	0.00022	0.0032

ACTIVITY = SA * W = Specific Activity * Weight

SA units are "curies/(g PuO2)"

Activity = SA * (g PuO2) and has units "curies"

Then $\text{var}(A) = (\text{SA}^2) \cdot \text{var}(\text{PuO2}) + (\text{PuO2}^2) \cdot \text{var}(\text{SA})$
and $\% \text{RSD}(A) = \sqrt{\{\% \text{RSD}(\text{SA})\}^2 + \{\% \text{RSD}(W)\}^2}$

SA(Pu)	=	0.065133036	curies "from Pu"/gPuO2	
V(SA(Pu))		7.81121E-10		
SA		0.068355986	curies "from Pu and Am"/g PuO2	
V(SA)		7.81454E-10		
			%RSD(SA) =	0.041

Wgt(PuO2) varies

V(PuO2) varies with amount

Fraction(Pu 0.87804

*Note: These values assume that the specific activity values for the particular isotopes are fixed.
No uncertainties were associated with the isotopic specific activity values.

				Holdup	gU	Holdup	DE	DE	DUO2	Blend	Blend lost to	Blend + Bottle 1
			g U	gU	in WRM	UO2	initial	removed	initially added	total mixed	homogeneity ck	
WRM#	Blend#	gUO2				g	g	g	g	g	(ignore)	g
CEP011	CEP011	570.66000		0.000000	0.000000	0.000000	100.990000	6.650600	571.281200	665.620600	0.150000	739.000000
CEP011B	CEP011B	570.61000	499.243807	0.162420	499.081387	0.185638	113.476166	0.000000	570.413834	683.890000	0.000000	
CEP012	CEP012	1010.37000	884.003024	0.061880	883.941145	0.070725	47.290000	0.000000	1139.190000	1186.480000	0.010000	1255.800000
CEP013	CEP013	1094.58000	957.680879	0.075800	957.605079	0.086636	47.330000	15.833100	1143.560000	1175.056900	0.100000	1244.360000
CEP014	CEP014	1097.25000	960.016943	0.508640	959.508303	0.581349	68.800000	36.978600	1145.320000	1177.141400	0.000000	1246.950000
CEP015	CEP015	1111.82000	972.764673	0.120910	972.643763	0.138194	47.300000	15.751600	1142.150000	1173.698400	0.000000	1243.060000
CEP016	CEP016	1084.47000	948.835337	0.174780	948.660557	0.199765	47.330000	15.058700	1142.960000	1175.231300	0.000000	1248.200000
Variance of the fraction of U in UO2			9.68E-09	g2/g2	From UO2 Assay, 5 meas.)							
Mean of the fraction of U in UO2			8.7493E-01	g/g								
Balances -- %uncertainty (kind of:			Std. Dev.	Variance								
Balances	15233	10	0.0004857	2.35904E-07								
		200	0.00715	5.11225E-05								
	16834	500	0.02952	0.00087143								
		1000	0.05785	0.003346623	nges to only random error effect 3rd decimal place of Var(U)							
	13045	100	0.009034	8.16132E-05								
		30	0.0014235	2.02635E-06								
As Per B. Marshall: Use t-value with 4 d.f. (2.776) to get 95% C.I. instead of the generic 2.0												
This makes sense from thinking that the variance in the fraction of U in UO2 is the term of importance for g UO2												
Homogeneity: expressed as a standard deviation of gDUO2/gBlend, see write up												

Page 2

[illegible]

Page 4

Page 5

Phil Noll
 COMPLETE CASE SA uncertainty Conditioned on the SAI values
 SA = 0.06773

$$SA = F \cdot \{ \text{SUM}(F_i \cdot SAI_i) \}$$

$$V(SA) = (F^2) \cdot V(\text{SUM}) + (\text{SUM}^2) \cdot V(F)$$

$$V(\text{SUM}) = \text{SUM}(V(F_i \cdot SAI_i)) + 2 \text{SUM}(SA_{ij} \text{COV}_{ij})$$

$$V(F_i \cdot SAI_i) = (F_i^2) \cdot V(SAI_i) + (SA_i^2) \cdot V(F_i)$$

$$SA_{ij} \text{COV}_{ij} = SAI_i \cdot SA_j \cdot \text{RHO} \cdot \text{SD}(F_i) \cdot \text{SD}(F_j) = SAI_i \cdot SA_j \cdot \text{COV}_{ij}$$

Set all Var(SAI) values to 0.0

	Value	F*SA	s.d.	%rsd
f-U	0.87493		0.000220	0.0251
f-234	0.00001	6.2E-08	0.000004	42.4000
SA-234	6.23E-03		0.000000	0.0000
f-235	0.0019	4.1E-09	0.000004	0.1874
SA-235	2.16E-06		0.000000	0.0000
f-236	0.00003	1.9E-09	0.000003	10.3667
SA-236	6.47E-05		0.000000	0.0000
f-238	0.99806	3.4E-07	0.000005	0.0005
SA-238	3.36E-07		0.000000	0.0000
f-233	0	0	0.000000	0.0000
SA-233	9.64E-03		0.000000	0.0000
Sum		4E-07		

COV	SDi	SDj	RHO	COVij
8-9	0.000004	0.000006	1	2.58E-11
8-0	0.000004	0.000005	1	2.15E-11
8-1	0.000004	0.000002	1	8.60E-12
8-2	0.000004	0.000001	1	4.30E-12
9-0	0.000006	0.000005	1	3.00E-11
9-1	0.000006	0.000002	1	1.20E-11
9-2	0.000006	0.000001	1	6.00E-12
0-1	0.000005	0.000002	1	1.00E-11

case %RSD
 pu

238	6.673
239	0.0014
240	0.0106
241	0.4142
242	0.838
am	0.04

% are from 1995 948 data, not from
 the observed data

V(Fi*SAj)	SD(term)	%rsd
6.97E-16	2.64E-08	6.53437
5.93E-23	7.70E-12	0.00191
4.05E-20	2.01E-10	0.04981
3.11E-24	1.76E-12	0.00044
0.00E+00	0.00E+00	0.00000
6.97E-16		

SAijCOVij
3.47482E-19
8.66508E-18
1.80149E-20
2.58233E-16
4.19947E-21
8.7308E-24
1.25151E-19
2.17718E-22

U SA-unc (Phil Noll)

0-2	0.000005	0.000001	1	5.00E-12	3.12086E-18
1-2	0.000002	0.000001	1	2.00E-12	6.72786E-19
					2.71187E-16

$V(\text{SUM}) = \text{SUM}(V(F_i \cdot SA_i)) + 2\text{SUM}(\text{COV}(i,j))$ 1.23953E-15

$V(SA) = (F^2) \cdot V(\text{SUM}) + (\text{SUM}^2) \cdot V(F)$	9.488718E-16
$SD(SA)$	0.0000000
%rsd	7.6235

SA Uncertainty			
		SD	%RSD
Approximate 95% Confidence Bound is 2 SD or 2%RSD		6.16E-08	15.247
Significant Digits: 1 or 2 maximum.		1 sig.digit	0.0002
		2 sig.digits	0.00022
			0.0032

ACTIVITY = SA * W = Specific Activity * Weight

SA units are "curies/(g U)"

Activity = SA * (g U) and has units "curies"

Then $\text{var}(A) = (SA^2) \cdot \text{var}(U) + (U^2) \cdot \text{var}(SA)$
 and $\%RSD(A) = \sqrt{(\%RSD(SA))^2 + (\%RSD(W))^2}$

SA	3.535E-07 curies "from U"/g UO2		
V(SA)	9.489E-16	%RSD(SA) =	8.713

Wgt(UO2) varies
 V(UO2) = varies with amount

*Note: These values assume that the specific activity values for the particular isotopes are fixed.
 No uncertainties were associated with the isotopic specific activity values.

P

	Total Alpha Activity in Curies				
	6/1/97	6/1/98	6/1/99	6/1/00	6/1/01
CEP001	0.04231	0.04228	0.04225	0.04221	0.04218
CEP002	0.03988	0.03985	0.03982	0.03978	0.03975
CEP003	0.33345	0.33376	0.33405	0.33430	0.33452
CEP004	0.33698	0.33730	0.33758	0.33783	0.33806
CEP005	0.46461	0.46471	0.46479	0.46483	0.46485
CEP006	0.46788	0.46798	0.46805	0.46808	0.46809
CEP007	0.21742	0.21724	0.21705	0.21686	0.21666
CEP008	0.20970	0.20954	0.20937	0.20920	0.20902
CEP009	0.35508	0.35451	0.35395	0.35338	0.35281
CEP010	0.34265	0.34210	0.34155	0.34100	0.34046

	half-life (days)	decay const.	At. Weight	S.A. (Ci/g)	S.A. (mCi/g)
238Pu	32010.5000000	0.0000217	238.0495530	17.1381141	17138.1140570
239Pu	8800150.0000000	0.0000001	239.0521570	0.0620783	62.0783431
240Pu	2395495.0000000	0.0000003	240.0538070	0.2271010	227.1009706
241Pu	5237.7500000	0.0001323	241.0568450	0.0025341	2.5341056
242Pu	136254500.0000000	0.0000000	242.0587370	0.0039596	3.9595993
241Am	157753.0000000	0.0000044	241.0568230	3.4342017	3434.2017270

$$\lambda_1/(\lambda_2 - \lambda_1) = -1.0343425$$

CEP001

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0000122	0.2093592	0.0000121	0.2077110	0.0000120	0.2060758	0.0000119	0.2044535	0.0000118	0.2028440
*239Pu	0.0800044	4.9665390	0.0800021	4.9663963	0.0799998	4.9662535	0.0799975	4.9661107	0.0799952	4.9659679
*240Pu	0.0050715	1.1517426	0.0050710	1.1516209	0.0050704	1.1514993	0.0050699	1.1513777	0.0050694	1.1512561
*241Pu	0.0001739	0.0004406	0.0001657	0.0004198	0.0001578	0.0004000	0.0001504	0.0003811	0.0001433	0.0003632
*242Pu	0.0000477	0.0001891	0.0000477	0.0001891	0.0000477	0.0001891	0.0000477	0.0001891	0.0000477	0.0001891
*241Am in PuO2	0.0000912	0.3133331	0.0000993	0.3409619	0.0001069	0.3672200	0.0001142	0.3921719	0.0001211	0.4158796
*241Am in the AmO2	0.0103865	35.6692023	0.0103698	35.6120431	0.0103532	35.5549756	0.0103366	35.4979994	0.0103200	35.4411146
total Pu =	0.0853097		0.0852986		0.0852878		0.0852774		0.0852674	
total S.A. (Ci) =		0.0423108		0.0422793		0.0422466		0.0422127		0.0421776

* less the 0.052% hold up

CEP002

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0000113	0.1931465	0.0000112	0.1916260	0.0000111	0.1901174	0.0000110	0.1886207	0.0000109	0.1871358
*239Pu	0.0738077	4.5818594	0.0738056	4.5817277	0.0738035	4.5815960	0.0738013	4.5814643	0.0737992	4.5813325
*240Pu	0.0046787	1.0625350	0.0046782	1.0624228	0.0046777	1.0623106	0.0046772	1.0621984	0.0046767	1.0620863
*241Pu	0.0001604	0.0004064	0.0001528	0.0003873	0.0001456	0.0003690	0.0001388	0.0003516	0.0001322	0.0003350
*242Pu	0.0000441	0.0001744	0.0000440	0.0001744	0.0000440	0.0001744	0.0000440	0.0001744	0.0000440	0.0001744
*241Am in PuO2	0.0000842	0.2890636	0.0000916	0.3145524	0.0000986	0.3387766	0.0001054	0.3617959	0.0001117	0.3836672
*241Am in the AmO2	0.0098279	33.7510873	0.0098122	33.6970019	0.0097965	33.6430031	0.0097808	33.5890909	0.0097651	33.5352651
total Pu =	0.0787021		0.0786918		0.0786819		0.0786723		0.0786631	
total S.A. (Ci) =		0.0398783		0.0398479		0.0398163		0.0397837		0.0397500

* less the 0.051% hold up

CEP003

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0002846	4.8782271	0.0002824	4.8398234	0.0002802	4.8017220	0.0002780	4.7639207	0.0002758	4.7264169
*239Pu	1.8641073	115.7206910	1.8640537	115.7173642	1.8640001	115.7140374	1.8639465	115.7107108	1.8638929	115.7073842
*240Pu	0.1181663	26.8356764	0.1181538	26.8328423	0.1181413	26.8300086	0.1181288	26.8271751	0.1181164	26.8243419
*241Pu	0.0040508	0.0102651	0.0038598	0.0097810	0.0036778	0.0093198	0.0035043	0.0088803	0.0033391	0.0084616
*242Pu	0.0011125	0.0044052	0.0011125	0.0044052	0.0011125	0.0044051	0.0011125	0.0044051	0.0011125	0.0044051
*241Am in PuO2	0.0021259	7.3006596	0.0023133	7.9444113	0.0024915	8.5562237	0.0026608	9.1376054	0.0028216	9.6899939
*241Am in the AmO2	0.0520361	178.7025744	0.0519527	178.4162072	0.0518695	178.1302988	0.0517864	177.8448487	0.0517034	177.5598560
total Pu =	1.9877215		1.9874622		1.9872119		1.9869702		1.9867367	
total S.A. (Ci) =		0.3334525		0.3337648		0.3340460		0.3342975		0.3345209

* less the 0.063% hold up

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0002876	4.9294872	0.0002854	4.8906799	0.0002831	4.8521782	0.0002809	4.8139796	0.0002787	4.7760817
*239Pu	1.8836985	116.9368832	1.8836444	116.9335214	1.8835902	116.9301596	1.8835361	116.9267980	1.8834819	116.9234365
*240Pu	0.1194082	27.1177120	0.1193956	27.1148481	0.1193830	27.1119845	0.1193703	27.1091213	0.1193577	27.1062583
*241Pu	0.0040934	0.0103730	0.0039003	0.0098838	0.0037164	0.0094178	0.0035412	0.0089737	0.0033742	0.0085505
*242Pu	0.0011242	0.0044515	0.0011242	0.0044515	0.0011242	0.0044514	0.0011242	0.0044514	0.0011242	0.0044514
*241Am in PuO ₂	0.0021482	7.3773899	0.0023376	8.0279074	0.0025177	8.6461498	0.0026887	9.2336418	0.0028513	9.7918358
*241Am in the AmO ₂	0.0525896	180.6032574	0.0525053	180.3138443	0.0524212	180.0248951	0.0523372	179.7364089	0.0522533	179.4483850
total Pu =	2.0086119		2.0083498		2.0080969		2.0078527		2.0076167	
total S.A. (Ci) =		0.3369796		0.3372951		0.3375792		0.3378334		0.3380590
* less the 0.028% hold up										

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0002854	4.8908579	0.0002831	4.8523547	0.0002809	4.8141547	0.0002787	4.7762555	0.0002765	4.7386546
*239Pu	1.8689380	116.0205765	1.8688843	116.0172410	1.8688306	116.0139056	1.8687768	116.0105703	1.8687231	116.0072352
*240Pu	0.1184725	26.9052202	0.1184600	26.9023788	0.1184475	26.8995376	0.1184350	26.8966968	0.1184225	26.8938563
*241Pu	0.0040613	0.0102917	0.0038698	0.0098064	0.0036873	0.0093440	0.0035134	0.0089034	0.0033477	0.0084835
*242Pu	0.0011154	0.0044166	0.0011154	0.0044166	0.0011154	0.0044166	0.0011154	0.0044166	0.0011154	0.0044165
*241Am in PuO ₂	0.0021314	7.3195786	0.0023193	7.9649985	0.0024979	8.5783963	0.0026677	9.1612846	0.0028289	9.7151046
*241Am in the AmO ₂	0.0901106	309.4580571	0.0899662	308.9621569	0.0898221	308.4670514	0.0896781	307.9727393	0.0895344	307.4792192
total Pu =	1.9928726		1.9926126		1.9923617		1.9921193		1.9918852	
total S.A. (Ci) =		0.4646090		0.4647134		0.4647868		0.4648309		0.4648470
* less the 0.034% hold up										

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0002851	4.8862306	0.0002829	4.8477639	0.0002806	4.8096000	0.0002784	4.7717366	0.0002762	4.7341713
*239Pu	1.8671694	115.9107837	1.8671157	115.9074513	1.8670621	115.9041191	1.8670084	115.9007870	1.8669547	115.8974550
*240Pu	0.1183604	26.8797590	0.1183479	26.8769203	0.1183354	26.8740818	0.1183229	26.8712437	0.1183104	26.8684058
*241Pu	0.0040574	0.0102820	0.0038661	0.0097971	0.0036838	0.0093351	0.0035101	0.0088949	0.0033446	0.0084755
*242Pu	0.0011144	0.0044124	0.0011144	0.0044124	0.0011144	0.0044124	0.0011143	0.0044124	0.0011143	0.0044124
*241Am in PuO ₂	0.0021280	7.3079813	0.0023158	7.9527980	0.0024942	8.5656228	0.0026638	9.1479670	0.0028249	9.7012704
*241Am in the AmO ₂	0.0911070	312.8798167	0.0909610	312.3784332	0.0908152	311.8778532	0.0906697	311.3780753	0.0905244	310.8790983
total Pu =	1.9909867		1.9907269		1.9904762		1.9902341		1.9900003	
total S.A. (Ci) =		0.4678793		0.4679776		0.4680450		0.4680831		0.4680933
* less the 0.022% hold up										

CEP007

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0000566	0.9704971	0.0000562	0.9628569	0.0000557	0.9552769	0.0000553	0.9477565	0.0000549	0.9402953
*239Pu	0.3708564	23.0221522	0.3708458	23.0214903	0.3708351	23.0208285	0.3708244	23.0201667	0.3708138	23.0195049
*240Pu	0.0235087	5.3388468	0.0235062	5.3382829	0.0235037	5.3377192	0.0235012	5.3371555	0.0234988	5.3365918
*241Pu	0.0008059	0.0020422	0.0007679	0.0019459	0.0007317	0.0018541	0.0006972	0.0017667	0.0006643	0.0016834
*242Pu	0.0002213	0.0008764	0.0002213	0.0008764	0.0002213	0.0008764	0.0002213	0.0008764	0.0002213	0.0008764
*241Am in PuO2	0.0004229	1.4524372	0.0004602	1.5805091	0.0004957	1.7022267	0.0005293	1.8178902	0.0005613	1.9277857
*241Am in the AmO2	0.0543448	186.6308927	0.0542577	186.3318205	0.0541707	186.0332276	0.0540839	185.7351131	0.0539973	185.4374764
total Pu =	0.3954490		0.3953974		0.3953476		0.3952995		0.3952530	
total S.A. (Ci) =		0.2174177		0.2172378		0.2170520		0.2168607		0.2166642

* less the 0.029% hold up

CEP008

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0000592	1.0143878	0.0000587	1.0064021	0.0000583	0.9984792	0.0000578	0.9906187	0.0000573	0.9828201
*239Pu	0.3876279	24.0632952	0.3876167	24.0626034	0.3876056	24.0619117	0.3875944	24.0612199	0.3875833	24.0605282
*240Pu	0.0245718	5.5802883	0.0245692	5.5796989	0.0245666	5.5791097	0.0245641	5.5785205	0.0245615	5.5779313
*241Pu	0.0008423	0.0021346	0.0008026	0.0020339	0.0007648	0.0019380	0.0007287	0.0018466	0.0006943	0.0017595
*242Pu	0.0002313	0.0009160	0.0002313	0.0009160	0.0002313	0.0009160	0.0002313	0.0009160	0.0002313	0.0009160
*241Am in PuO2	0.0004421	1.5181198	0.0004810	1.6519835	0.0005181	1.7792056	0.0005533	1.9000999	0.0005867	2.0149652
*241Am in the AmO2	0.0516906	177.5160886	0.0516078	177.2316227	0.0515251	176.9476126	0.0514425	176.6640577	0.0513601	176.3809572
total Pu =	0.4133326		0.4132786		0.4132266		0.4131763		0.4131278	
total S.A. (Ci) =		0.2096952		0.2095353		0.2093692		0.2091973		0.2090199

* less the 0.033% hold up

CEP009

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*239Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*240Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*241Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*242Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*241Am in PuO2	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*241Am in the AmO2	0.1033961	355.0830652	0.1032304	354.5140518	0.1030650	353.9459503	0.1028998	353.3787592	0.1027349	352.8124770
total Pu =	0.0000000		0.0000000		0.0000000		0.0000000		0.0000000	
total S.A. (Ci) =		0.3550831		0.3545141		0.3539460		0.3533788		0.3528125

* less the 0.034% hold up

CEP010

	6/1/97		6/1/98		6/1/99		6/1/00		6/1/01	
	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)	grams	S.A. (mCi)
*238Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*239Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*240Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*241Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*242Pu	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*241Am in PuO ₂	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000
*241Am in the AmO ₂	0.0997754	342.6488510	0.0996155	342.0997632	0.0994559	341.5515553	0.0992965	341.0042260	0.0991374	340.4577737
total Pu =	0.0000000		0.0000000		0.0000000		0.0000000		0.0000000	
total S.A. (Ci) =		0.3426489		0.3420998		0.3415516		0.3410042		0.3404578
* less the 0.043% hold up										

Q

<u>Diatomaceous Earth Weighings</u>					
<i>Balance: 13045 (Certified)</i>					
<i>Check Wts: Cenco Class S (Certified)</i>					
Standard Wt.	Number of Measurements	Mean	Std. Dev.	% Uncert.	
30 g	15	29.99853	5.16E-04	0.009	
50 g	3	50.001	0.002	0.014	
100 g	20	99.993	0.004	0.018	

<u>Diatomaceous Earth Adjustment Weighings</u>					
<i>Balance: 015233 (Certified)</i>					
<i>Check Wts: Not Certified</i>					
Standard Wt.	Number of Measurements	Mean	Std. Dev.	% Uncert.	
10 g	15	10.00094	1.46E-05	0.010	
200 g	12	200.0143	4.93E-05	0.007	

<u>PuO₂ and AmO₂ Weighings</u>					
<i>Balance: 013599 (Certified)</i>					
<i>Check Wts: 15082 (Certified)</i>					
Standard Wt.	Number of Measurements	Mean	Std. Dev.	% Uncert.	
10 mg	5	0.01001	4.47E-06	0.204	
50 mg	5	0.05002	5.48E-06	0.062	
100 mg	10	0.10002	6.75E-06	0.032	
1 g	6	1.00001	5.16E-06	0.003	
2 g	10	2.00003	6.75E-06	0.002	

<u>UO₂ Weighings</u>					
<i>Balance: 016834 (Certified)</i>					
<i>Check Wts: 4482, 3648 (Certified)</i>					
Standard Wt.	Number of Measurements	Mean	Std. Dev.	% Uncert.	
500 g	41	499.954	5.91E-03	0.012	
1 kg	38	999.899	6.49E-03	0.012	

Count	Mean	s.d.	bias	t	bias + 2 s.d.	% uncert.
-------	------	------	------	---	---------------	-----------

Calibration Certificate

WEIGHT SET

File No.: 15082

Mfg.: Troemner
Serial No.: 23424
Other ID: NA

Certified: April 13, 1995
Expires: April 12, 1997

Calibration of the above weights was achieved in a controlled environment through the use of standards traceable to national standards.

Item Received: ☒ In tolerance
 ☐ Out of tolerance
Item Returned: ☒ In tolerance
 ☐ Limited

Values reported are those that would be observed in comparing the weights to standards of density 8.0 g/cc in local air of density 0.923 mg/cc. The values reported under True Mass correction are compensated for local environment and correspond to values which would be observed in a vacuum.

Estimated uncertainties in the reported values do not exceed:

1.5 ppm for weights 100 g to 10 g
0.01 mg for weights 5 g to 1 g
0.005 mg for weights 500 mg to 1 mg

It is expected that this item will remain within the stated uncertainty for the duration of the calibration interval, provided it is used in accordance with the operational conditions specified by the manufacturer, and provided it is not subjected to any physical damage.

Calibrated by: George perrault *[Signature]*

Reviewed by: Bernadette Salazar *[Signature]*

Copy to: Adrian Lovell, CST-08, G740
ESH-9 file

Calibration Certificate

BALANCE

File No. 013559

Sartorius R200D

Property No. 901559

Location: TA-03 Bldg. CMR Rm. 5129

Certified: July 15, 1997

Expires: July 15, 1998

This instrument was calibrated on-site using equipment traceable to national standards. Under normal operating conditions, this item is expected to remain within the stated uncertainty for the duration of calibration interval.

Calibration Results

Instrument Received

☒ In Tolerance

☐ Other

Instrument Returned

☒ In Tolerance

☐ Other

It's recommended that the balance be calibrated using the internal calibration weight when there's a requirement for high accuracy measurements.

Tolerance

+/- 0.00010 grams : 40 gram range

+/- 0.0004 grams : 200 gram range

Calibrated by: Michael A. Salazar

Reviewed by: Bernadette L. Salazar

Copy to: Donna Hofmann, CST-08, Mail Stop G740
ESH-09 File

Calibration Certificate

WEIGHT SET

Manufacturer Cenco

Serial No. NA

File No. 18535

UNOFFICIAL
COPY

Certified: November 06, 1997

Expires: November 06, 1999

This weight set was calibrated in a controlled environment using equipment traceable to national standards. Under normal operating conditions, this item is expected to remain within the stated uncertainty for the duration of calibration interval.

Values reported are those that would be observed in comparing the weights to standards of density 8.0 g/cc in local air of density .924 mg/cc.

Calibration Results

Instrument Received

☒ In Tolerance

☐ Other

Instrument Returned

☒ In Tolerance

☐ Other

Designation	Correction	Uncertainty
100 g	+ 1.27	+/- 0.30 mg
50	+ 0.39	0.15
30	+ 0.30	0.09
20	- 0.22	0.06
5	+ 0.28	0.02
3	- 0.02	0.02
500 mg	- 0.080	0.01
300	- 0.005	0.01
100	+ 0.031	0.01
30	+ 0.020	0.01

SET NOT COMPLETE

Calibrated by: George Perrault

Reviewed by: Bernadette L. Salazar

Copy to: Donna Hofmann, CST-08, Mail Stop G740
ESH-09 File

Calibration Certificate

BALANCE

Sartorius LC-620-D

Property No. 839672

Location: TA 03 Bldg. CMR Room 5165

File No. 013045

Certified: March 31, 1997

Expires: March 31, 1998

This instrument was calibrated on-site using equipment traceable to national standards. Under normal operating conditions, this item is expected to remain within the stated uncertainty for the duration of calibration interval.

Calibration Results

Instrument Received

☒ In Tolerance

☐ Other

Instrument Returned

☒ In Tolerance

☐ Other

Tolerance

+/- 0.003 grams : 60 gram range

+/- 0.03 grams : 600 gram range

Calibrated by: Christopher J. Pelchat

Reviewed by: Bernadette L. Salazar

Copy to: Adrian P. Lovell, CST-08, Mail Stop G740
ESH-09 File

Calibration Certificate

500 G WEIGHT
Manufacturer Troemner
Serial No. N/A
Other Id. N/A

File No. 4482

Certified: June 12, 1997
Expires: June 12, 1999

This instrument was calibrated in a controlled environment using equipment traceable to national standards. Under normal operating conditions, this item is expected to remain within the stated uncertainty for the duration of calibration interval.

Values reported are those that would be observed in comparing the weights to standards of density 8.0 g/cc in local air of density .924 mg/cc.

Calibration Results

Instrument Received

☒ In Tolerance

☐ Other

Instrument Returned

☒ In Tolerance

☐ Other

Nominal
500.0 g

Apparent Mass
500.00126 g

Uncertainty
+/- 2.3 mg

Calibrated by: Christopher J. Pelchat

Reviewed by: Bernadette L. Salazar

Copy to: Donna Hofmann, CST-08, Mail Stop G740
ESH-09 File

Calibration Certificate

1 KG WEIGHT
Manufacturer Troemner
Serial No. N/A
Other Id. N/A

File No. 3648

Certified: June 12, 1997
Expires: June 12, 1999

This instrument was calibrated in a controlled environment using equipment traceable to national standards. Under normal operating conditions, this item is expected to remain within the stated uncertainty for the duration of calibration interval.

Values reported are those that would be observed in comparing the weights to standards of density 8.0 g/cc in local air of density .924 mg/cc.

Calibration Results

Instrument Received

☒ In Tolerance

☐ Other

Instrument Returned

☒ In Tolerance

☐ Other

Nominal
1000.0 g

Apparent Mass
1000.00154 g

Uncertainty
+/- 4.5 mg

Calibrated by: Christopher J. Pelchat

Reviewed by: Bernadette L. Salazar

Copy to: Donna Hofmann, CST-08, Mail Stop G740
ESH-09 File

Calibration Certificate

BALANCE

File No. 016834

Arbor 1600

Property No. 452574

Location: TA-03 Bldg. CMR Rm. 5122

Certified: September 6, 1996

Expires: September 6, 1997

This instrument was calibrated on-site using equipment traceable to national standards. Under normal operating conditions, this item is expected to remain within the stated uncertainty for the duration of calibration interval.

Calibration Results

Instrument Received

☒ In Tolerance

☐ Other

Instrument Returned

☒ In Tolerance

☐ Other

Tolerance

+/- 0.05 grams

Calibrated by: Michael A. Salazar

Reviewed by: Bernadette L. Salazar

Copy to: Adrian P. Lovell, CST-08, Mail Stop G740
ESH-09 File

Calibration Certificate

Balance
Sartorius BP210D
Serial: N/A
Prop. No: N/A
Other ID: TA-03 Bldg CMR Rm 5127A

File No. 015233

Certified: July 02, 1997
Expires: July 02, 1998

Calibration of the above item(s) was achieved in a controlled environment through the use of equipment traceable to national standards. It is expected that for the duration of the calibration interval and under normal operational conditions, this item will remain within the tolerance limits specified.

Calibration	Item Received: ☒ In Tolerance	☐ Other
Information:	Item Returned: ☒ In Tolerance	☐ Other

Tolerance: +/- 0.0005 grams

Calibration data is available upon request.

Calibrated by: Pelchat, Christopher J.

Reviewed by: Salazar, Bernadette L.

Copy to: DONNA HOFMANN, CST-08, MS G740
ESH-09 File

National Bureau of Standards

Certificate

Standard Reference Material 4322

Alpha-Particle-Solution Standard

Radionuclide	Americium-241
Source identification	SRM 4322
Source description	Liquid in 5-mL flame-sealed glass ampoule
Source mass	Approximately 5.2 grams
Source composition	Americium-241 in 1-molar nitric acid
Reference time	1200 EST, 1 November 1986
Radioactivity concentration	38.66 Bq g ⁻¹
Overall uncertainty	0.81 percent (1)*
Alpha-particle-emitting impurities (Activities at reference time)	None detected (2)
Measuring instrument	NBS "0.1π"α defined-solid-angle counter with scintillation detector
Half life	432.2 ± 0.5 years (3)

This Standard Reference Material was prepared in the Center for Radiation Research, Ionizing Radiation Division, Radioactivity Group, Dale D. Hoppes, Group Leader.

Gaithersburg, MD 20899
November, 1986

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

*Notes on back

NOTES

- (1) Individual uncertainties have the significance of one standard deviation of the mean, or an approximation thereof. The combined uncertainty is the individual uncertainties shown below added in quadrature. The overall uncertainty is taken to be three times the combined uncertainty.

<u>Source of uncertainty</u>	<u>Uncertainty (%)</u>
a) alpha-particle-emission-rate measurements	0.16
b) gravimetric measurements	0.05
c) deadtime	0.05
d) background	0.03
e) detection efficiency	0.10
f) count-rate-vs-energy extrapolation to zero energy	0.15
g) half life	0.00
h) alpha-particle-emitting impurities	0.10
Combined uncertainty	<u>0.27</u>
	* 3
Overall uncertainty	<u>0.81</u>

- (2) The limit of detection for alpha-particle-emitting impurities is $0.004 \text{ } \alpha\text{s}^{-1}\text{g}^{-1}$ for energies greater than 5.60 MeV.

The material was also examined for photon-emitting impurities and none were found. The limit of detection for photon-emitting impurities is $0.002 \text{ } \gamma\text{s}^{-1}\text{g}^{-1}$ for energies between 90 and 1900 keV.

- (3) NCRP Report No. 58, Second Edition (1985) p. 365.

For further information call Larry Lucas at (301) 975-5546.

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 948

Plutonium Isotopic Standard

This Standard Reference Material (SRM) is certified as an isotopic standard for use in isotopic measurements of plutonium. SRM 948 consists of approximately 250 mg of plutonium in the form of plutonium sulfate tetrahydrate packaged in a glass microbottle.

	<u>^{238}Pu</u>	<u>^{239}Pu</u>	<u>^{240}Pu</u>	<u>^{241}Pu</u>	<u>^{242}Pu</u>
Atom Percent*	0.010	91.736	7.922	0.299	0.0330
	± 0.001	± 0.010	± 0.010	± 0.001	± 0.0003

*As of January 1, 1982, refer to Table 1 for quarterly decay-adjusted values.

The plutonium isotopic distribution was determined by thermal ionization mass spectrometry at the National Bureau of Standards (NBS) on samples from which americium and uranium were removed. Because high-purity plutonium isotopes have not been used to prepare known synthetic mixtures, the accuracy is dependent on uranium and plutonium exhibiting similar behavior. The observed mass spectrometer data were corrected for mass discrimination effects using data from the analysis of uranium isotopic SRM's that had been analyzed under similar conditions. In addition, the value for ^{238}Pu was checked by alpha-count of ^{238}Pu , using the known value for ^{239}Pu as a ratio check.

SRM 948 contains uranium and americium isotopes, including growing-in daughters of plutonium that are isobaric with the plutonium isotopes. In addition, there may be radiation damage to the glass bottle and teflon cap liner resulting in small glass slivers. Therefore, in its use, a chemical separation that provides a purified plutonium fraction is essential to the attainment of high accuracy.

Measurements leading to the certification of this SRM were made in the Inorganic Analytical Research Division by E.L. Garner, L.A. Machlan, and W.R. Shields.

The technical and support aspects involved in the revision of this Certificate were coordinated through the Office of Standard Reference Materials by T.E. Gills.

August 19, 1982
Washington, D.C. 20234
(Revision of Certificate
dated 12-3-71)

George A. Urano, Chief
Office of Standard Reference Materials

(over)

The decay-adjusted values for the plutonium isotopic composition, in atom percent, are tabulated below in Table I. The half-life values, in years, used for the decay-adjustment are: ^{238}Pu , 87.74; ^{239}Pu , 24,119; ^{240}Pu , 6,560; ^{241}Pu , 14.34; and ^{242}Pu , 387,000.

Table I
Decay-Adjusted Plutonium Isotopic Composition
Atom Percent

Date	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
January 1, 1982	0.010	91.736	7.922	0.299	0.0330
April 1, 1982	.010	91.739	7.922	.295	.0330
July 1, 1982	.010	91.743	7.923	.292	.0330
October 1, 1982	.010	91.746	7.923	.288	.0330
January 1, 1983	.010	91.749	7.923	.285	.0330
April 1, 1983	.010	91.753	7.923	.281	.0330
July 1, 1983	.010	91.756	7.923	.278	.0330
October 1, 1983	.010	91.759	7.923	.274	.0330
January 1, 1984	.010	91.762	7.923	.271	.0330
April 1, 1984	.010	91.765	7.923	.268	.0330
July 1, 1984	.010	91.769	7.924	.265	.0330
October 1, 1984	.010	91.772	7.924	.262	.0330
January 1, 1985	.010	91.775	7.924	.258	.0330
April 1, 1985	.010	91.778	7.924	.255	.0330
July 1, 1985	.010	91.781	7.924	.252	.0330
October 1, 1985	.010	91.784	7.924	.249	.0330
January 1, 1986	.010	91.787	7.924	.246	.0330
April 1, 1986	.010	91.789	7.924	.243	.0330
July 1, 1986	.010	91.792	7.924	.240	.0330
October 1, 1986	.010	91.795	7.925	.238	.0330
95% Confidence Limit:	±0.001	±0.010	±0.010	±0.001	±0.0003

Certificate

Standard Reference Material U-005

Uranium Isotopic Standard

	^{234}U	^{235}U	^{236}U	^{238}U
Atom percent	0.00218 ± 0.00004	0.4895 ± 0.0005	0.00466 ± 0.00005	99.504 ± 0.01
Weight percent	0.00214	0.4833	0.00462	99.510

The material consists of highly purified oxide, U_3O_8 . The atomic weight of the material is calculated to be 238.036 using the nuclidic masses 234.0409; 235.0439; 236.0457 and 238.0508.

The values for ^{234}U and ^{236}U were calculated from measurements at the National Bureau of Standards. The samples were spiked with high-purity ^{233}U to approximate the ^{234}U concentration, the ratios ^{233}U to ^{234}U and ^{233}U to ^{236}U were measured on a triple-filament equipped surface ionization mass spectrometer with ion-multiplier amplifier circuits.

The values for ^{235}U and ^{238}U were calculated from measurements of the ^{235}U to ^{238}U ratio made at the National Bureau of Standards on a triple-filament, surface ionization mass spectrometer equipped with dc amplifier circuits. The observed ratios were corrected for mass discrimination effects by intercomparison with synthetic mixtures prepared at the 0.5 percent ^{235}U level from high-purity ^{235}U and ^{238}U .

The limits indicated for the isotopic concentrations are at least as large as the 95-percent confidence limits for a single determination, and include terms for inhomogeneities in the material as well as analytical error. The ^{235}U to ^{238}U ratio for this standard, 0.004919, is known to at least 0.1 percent.

Mass spectrometry measurements at NBS were made by E. L. Garner on solutions prepared by L. A. Machlan.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of W. R. Shields.

The technical and support aspects in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. L. Hague.

Washington, D. C. 20234
April 21, 1969

W. Wayne Meinke, Chief
Office of Standard Reference Materials

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 949f

Plutonium Metal

(In cooperation with the University of California Los Alamos
National Laboratory, Los Alamos, New Mexico)

This Standard Reference Material is certified for plutonium content and is primarily intended for use in the chemical assay of plutonium. SRM 949f consists of approximately 0.5 gram of plutonium metal that has been sealed in a glass tube under a reduced-pressure argon atmosphere. Each unit is identified by tube number and sample mass.

Plutonium assay, percent _____ 99.99 (A's of October, 1980,
[99.90-100]^a See Table 2.)

Tube No. _____

Mass _____

^aThe uncertainty of the certified value is expressed at the 99% one-sided confidence interval

The plutonium assays were performed by the Los Alamos National Laboratory with collaborative analysis performed at the Department of Energy, New Brunswick Laboratory. The Los Alamos National Laboratory used two titrimetry methods utilizing both photometric and potentiometric end points [1,2,3,4]. The New Brunswick Laboratory used a controlled-potential coulometric method [5,6]. The certified value is based on the results from these two laboratories using three different methods. However, the confidence interval on the certified value is a one-sided 99% confidence interval on the mean of the potassium dichromate titrimetric method, which is 99.98-100, expanded by the bound on the systematic error ± 0.08 . The 99% confidence interval is based upon the potassium dichromate method because adequate systematic error bounds are available. The mean value for the potassium dichromate titrimetric method is 99.999 with a standard error on the mean of ± 0.006 based on 16 determinations.

The americium resulting from the decay of 14.35 years plutonium-241 was approximately 21 $\mu\text{g/g}$ as of October 1980. The total of all detected impurities is approximately $115 \pm 65 \mu\text{g/g}$. 99.9995

The technical and support aspects leading to certification and issuance of this Standard Reference Material was coordinated through the Office of Standard Reference Materials by T. E. Gills.

The statistical assessment of the data for the certification of this SRM was performed by W. Liggett of the Statistical Engineering Division of the National Bureau of Standards.

Supplemental Information

Isotopic Analysis

Plutonium isotopic distribution was determined by thermal ionization mass spectrometry at the Los Alamos National Laboratory. The values given in Table I are *not certified*, but given for information only.

Table I

<u>Isotope</u>	<u>Isotope Mass</u>	<u>Average Atom % on October 8, 1980</u>
^{238}Pu	238.04942	0.0040
^{239}Pu	239.05208	97.121
^{240}Pu	240.05382	2.804
^{241}Pu	241.05669	0.065
^{242}Pu	242.05864	0.006

Calculated Atomic Wt. 239.082

Notice and Warnings to Users

SRM 949f may contain small pieces of Pu metal that may have become separated from the larger pieces in the tube. It is recommended that the tube be carefully rinsed with 3 M hydrochloric acid during the transfer.

The mass of each sample was determined with an overall accuracy of ± 0.05 mg. The assigned mass for each unit, however, has not been corrected for the buoyancy of the dry argon atmosphere in which the plutonium metal was weighed and packaged. The computed atmosphere buoyancy correction factor for dry argon at 592 mm Hg and 20 °C is 0.99991 for plutonium metal (having a density of 19.7 g/cm³) relative to brass weights. The value given on this certificate for the mass of the plutonium may be multiplied by this factor to obtain the "true" in vacuo mass.

SRM 949f had a radioactivity of approximately 0.07 Ci per unit, as of October, 1980. The material is believed to be stable and free of oxidation; however, the metal should be inspected before use for signs of oxidation. If oxidation has occurred, the material should be acid-etched, dried, and reweighed before use.

The decay-adjusted values for plutonium in 949f for a 5-year period are shown in Table 2. The half-life values, in years, used for the decay adjustments are ^{238}Pu , 87.74; ^{239}Pu , 24119; ^{240}Pu , 6560; ^{241}Pu , 14.35; and ^{242}Pu , 387,000.

Table 2

Plutonium Assay, Percent

<u>1970</u>	<u>1981</u>	<u>1982</u>	<u>1983</u>	<u>1984</u>
99.99	99.98	99.98	99.97	99.97

References

1. C. E. Caldwell, L. S. Grille, F. J. Miner, and N. E. Moody, *Anal. Chem.* **34**, 346, 1962.
2. G. R. Waterbury and C. S. Metz, Los Alamos Scientific Lab Report, LA 3141, 1965.
3. K. S. Bergstresser, Los Alamos Scientific Lab Report, LA 2859, 1963.
4. C. E. Pietri and J. A. Baglio, *Talanta*, **6**, 159-166, 1960.
5. M. K. Holland, J. R. Weiss, and C. E. Pietri, *Anal. Chem.* **50**, 236-240 (1978).
6. D. D. Jackson, R. M. Hollen, F. R. Roensch, and J. E. Rein, *Anal. Chim. Acta* **117** 205-15 (1980).

N. Stip... / 2.2F9-D 836... / 6740 / 065-1

National Bureau of Standards

Certificate

Standard Reference Material 4906H-C Radioactivity Standard

Radionuclide	Plutonium-238 ^{(1)*}
Source identification	4906H-C-9
Activity	1.674×10^3 Bq
Reference time	1200 EST October 1, 1987
Overall uncertainty	1.3 percent ⁽²⁾
Radionuclide impurities (Activity ratio at reference time)	$^{241}\text{Am}/^{238}\text{Pu}: 6.5 \times 10^{-5}$ ⁽³⁾ $^{240}\text{Pu}/^{238}\text{Pu}: <4.0 \times 10^{-5}$ $^{239}\text{Pu}/^{238}\text{Pu}: <2.4 \times 10^{-5}$
Half life	87.74 ± 0.04 years ⁽⁴⁾
Measuring instrument	"0.8 π " α counter ⁽⁵⁾

This Standard Reference Material was prepared in the Center for Radiation Research, Ionizing Radiation Division, Radioactivity Group, Dale D. Hoppes, Group Leader.

Gaithersburg, MD 20899
November, 1987

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

*Notes on back

NOTES

- (1) A practically weightless source electroplated onto a 0.025-cm-thick platinum foil 0.6 cm in diameter which is cemented to a stainless-steel disk 2.54 cm in diameter and 0.16-cm thick.
- (2) The overall uncertainty was formed by taking three times the quadratic combination of the standard deviations of the mean, or approximations thereof, for the following:

a) one standard deviation of the mean	0.10 percent
b) efficiency calibration	0.15 percent
c) count-rate vs. energy	
extrapolation to zero energy	0.40 percent
d) system live time	0.05 percent
- (3) An impurity search was performed at NBS using a Ge detector.
- (4) NCRP Report No. 58, Second Edition, February 1985, p. 505.
- (5) Defined-solid-angle counter with scintillation detector.

For further information contact J.M.R. Hutchinson at (301) 975-5543 or S.J. Bright at (301) 975-5542.

4906H-C

Tracer Solution for Environmental Analysis & Disequilibrium Studies.

Product Description & Measurement Certificate

Description	Principal radionuclide:	americium 241 (Am-241)	Product code: AMP10040
	Daughter Nuclide:	Np-237	Batch Number: 95/241/49
Measurement	Reference date:	06 January 1987	
	Radioactive concentration Am-241	4.711E+05 becquerels per gram of solution	
	which is equivalent to	1.273E+01 microcuries per gram of solution	
	Mass of solution	5.36 grams	
	Volume of solution	5.01 millilitres	
	Total activity of Am-241	2.53E+06 becquerels	
	which is equivalent to	6.82E+01 microcuries	
Accuracy	Method of measurement (see reverse of this certificate)		
	Random uncertainty is: ± 0.1% Systematic uncertainty: ± 0.4%		
	Overall uncertainty in the radioactive concentration quoted above: ± 0.8%		
	Overall uncertainty is defined on the reverse of this certificate.		
Radionuclidic Purity	Any radioactive impurities measured are listed below, expressed as percentages of the activity of the principle radionuclide at the reference date .		
	Am-241 100%		
Isotopic Purity	The elemental composition, expressed as atom per cent at the reference date .		
	Not Measured		
Chemical Composition	Calculated weight of Am-241, 1.99E-05 grams, as 2M HNO3 solution in a flame sealed glass vial. This Tracer solutions has been produced 'carrier free'.		
Physical Data	Recommended half life of americium 241: 4.327E+02 years		
	Principle energies of alpha emissions (MeV): 5.443 12.8%, 5.486 85.2%		
	Branching ratio for alpha emission: 100.0%		
	Calculated specific activity of americium 241: 1.268E+05 Bq per microgram Am-241.		
Remarks	For safety information and notes to ensure correct usage by all persons handling this radioactive Tracer solution please read the instructions accompanying the package.		
	AEA Technology operates a quality management system which has been independently audited and approved to ISO 9001.		
	Intercomparison of measured alpha activity concentration with NPL established agreement to within 0.2%. Reference, AERE R 13291 October 1988		

Approved
Signatory

Roger Wiltshire

Project Ref. AE3066

Prepared and characterised at AEA Technology UK, world wide distribution by Isotrak, QSA Amersham International.

Methods of measurement

The activity concentration of this batch of Tracer solutions has been measured by alpha counting accurately known weight aliquots prepared as thin sources, using a pair of absolute low geometry alpha counters. The counting efficiency of the alpha counters [ca 1:700] have been determined by accurate measurement of both the source to collimator distance and collimator aperture area. Background and dead time corrections have been applied when necessary.

The alpha particle energy spectrum data has been collected using a high resolution solid state detector operating under vacuum. The reported contribution of source activity of each nuclide has been assessed by integrating the channel contents over the expected energy range for each nuclide. Corrections for co-incident alpha particle energies and low energy tailing have been applied when necessary.

Measured radionuclidic and isotopic data may be decay corrected to the reference date shown.

S.I. Units of radioactivity

The S.I. unit of radioactivity is the becquerel

1 becquerel [Bq]	=	1 nuclear transformation per second, therefore
1 curie [Ci]	=	3.7E+7 becquerels exactly

Useful conversion factors are:

1 microcurie [μ Ci]	=	3.7E+4 Bq	=	37 kilobecquerels [kBq]
1 millicurie [mCi]	=	3.7E+7 Bq	=	37 megabecquerels [MBq]
1 kilobecquerel [kBq]	=	27.027 nanocuries [nCi]		
1 megabecquerel [MBq]	=	27.027 microcuries [μ Ci]		

Overall uncertainty

The reported uncertainties have been estimated in accordance with the recommendations of the NAMAS document, NIS 3003, 'The Expression of Uncertainty and Confidence in Measurement for Calibrations' Edition 8 May 1995. The overall uncertainty has been calculated by combining the systematic and random uncertainties, in quadrature, and multiplying the resulting sum by the coverage factor $k=2$, providing a level of confidence of approximately 95%.

References

In presenting and calculating the data a number of sources of data have used. Where possible and appropriate, data has been abstracted from the IAEA Technical Report Series, No 261, 1986 'Decay Data of the Transactinium Nuclides'.

Additional half life and decay data has been abstracted from The 8th Edition of Table of Radio Isotopes, 1986, ISBN No. 0 471-84909-X, and 'Catalogue of Alpha Particles from Radioactive Decay', W. Westmeier, A. Merklin, ISSN 0344-8402, 1985.

Atomic mass data has been taken from the 68th Edition of the Handbook of Chemistry and Physics.

One year	=	365.2422 days
Avagadro's No	=	6.02252E+23

TRACEABILITY TO NIST

Amersham Corporation
2636 S. Clearbrook Drive
Arlington Heights, IL 6000
tel (708) 593-6300
fax (708) 593-8091

Traceability is the ability to relate the accuracy of measurement of radionuclides to the National Institute of Standards and Technology (NIST). Traceability is achieved by participation in a Measurements Assurance Program linked to NIST and production of certified materials in accordance with a quality assurance program.

Amersham participates in measurement assurance programs conducted by NIST in cooperation with the Nuclear Energy Institute (NEI, formerly USCEA). Additionally, our production facilities and measurement laboratories operate under routinely audited quality assurance programs.

Therefore, Amersham certified standardized products meet or exceed, the NRC requirements for measurements traceable to NIST.

278004C

Certificate of calibration of mixed radionuclide gamma-ray reference solution

M 27533

CALIBRATION
No 0146

Description

Product code: QCY.44

Solution number: R4/305/8

This mixed radionuclide gamma-ray reference standard consists of a solution in 4M HCl of the nine radionuclides listed below.

Measurement and Accuracy

Reference time: 1200 GMT on 1 November 1994

Mass of solution: 5.2477 grams Density: 1.064 g/ml at 20°C

Parent radionuclide	Gamma-ray energy (keV)	Gamma-rays per second per gram of solution	Random uncertainty	Systematic uncertainty	Overall uncertainty
Cadmium-109	88.03	641	± 0.3 %	± 3.4 %	± 3.7 %
Cobalt-57	122.1	584	± 0.2 %	± 1.6 %	± 1.8 %
Cerium-139	165.9	686	± 0.3 %	± 1.4 %	± 1.7 %
Mercury-203	279.2	1983	± 0.3 %	± 1.9 %	± 2.2 %
Tin-113	391.7	2092	± 0.1 %	± 4.5 %	± 4.6 %
Strontium-85	514.0	4027	± 0.5 %	± 1.5 %	± 2.0 %
Caesium-137	661.7	2464	± 0.2 %	± 1.9 %	± 2.1 %
Yttrium-88	898.0	6217	± 0.2 %	± 3.5 %	± 3.7 %
Cobalt-60	1173	3361	± 0.2 %	± 0.4 %	± 0.6 %
Cobalt-60	1333	3364	± 0.2 %	± 0.4 %	± 0.6 %
Yttrium-88	1836	6589	± 0.2 %	± 3.4 %	± 3.6 %

Purity

At the reference time the solution also contained the following impurities:

Iron-55 equal to 8.5 becquerels (0.23 nanocuries) per gram.

Remarks

Further details, including composition of the solution, methods of measurement, decay scheme assumptions, decay tables and definitions of uncertainties, are given in the data sheet accompanying this certificate.

This product meets the quality assurance requirements of NRC Regulatory Guide 4.15 for achieving explicit NIST traceability as defined in NCRP58 (1985).

Approved
Signatory

W. F. Case

W F Case

Page 1 of 1

Amersham

This certificate is issued in accordance with the conditions of accreditation granted by the National Measurement Accreditation Service, which has assessed the measurement capability of the laboratory and its traceability to recognised national standards and to the units of measurement realised at the corresponding national standards laboratory. Copyright of this certificate is owned jointly by the British Crown and the issuing laboratory and may not be reproduced other than in full except with the prior written approval of the Head of NAMAS and the issuing laboratory.

TRACEABILITY TO NIST

Amersham Corporation
2636 S. Clearbrook Drive
Arlington Heights, IL 60005
tel (708) 593-6300
fax (708) 593-8091

Amersham

Traceability is the ability to relate the accuracy of measurement of radionuclides to the National Institute of Standards and Technology (NIST). Traceability is achieved by participation in a Measurements Assurance Program linked to NIST and production of certified materials in accordance with a quality assurance program.

Amersham participates in measurement assurance programs conducted by NIST in cooperation with the Nuclear Energy Institute (NEI, formerly USCEA). Additionally, our production facilities and measurement laboratories operate under routinely audited quality assurance programs.

Therefore, Amersham certified standardized products meet or exceed, the NRC requirements for measurements traceable to NIST.

278004C

Listing of SRM Certificates

Page 1:

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 89

Lead-Barium Glass

This Standard Reference Material (SRM) is in the form of a powder and is intended for use in evaluating chemical and instrumental methods of analyses. The SRM slowly absorbs carbon dioxide and water. Therefore, the analyst must determine the loss on ignition at 900 to 1000 °C and correct all determinations for the difference between the value found and the original value of 0.32 weight percent. The recommended values are given below.

[All results are based on a sample dried for one hour at 105 to 110 °C]

Analyst*	SiO ₂	PbO	K ₂ O	Na ₂ O	BaO	TiO ₂	ZrO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	P ₂ O ₅	MnO	As ₂ O ₃	As ₂ O ₅	SO ₃	Cl	Loss on Ignition
1	65.36	17.50	8.38	5.73	1.41	0.011	0.003	0.048	0.181	0.21	0.029	0.22	0.089	0.39	0.03	0.031	0.051	0.30
2	65.37	17.51	8.41	5.68	1.39	.020	.006	.051	.178	.208	.032	.243	.088	.348	.032	.031	.054	.37
3	65.28	17.36	8.20	5.64	1.49	.011	---	.044	.130	.21	.04	.22	---	---	---	---	---	.26
4	65.41	17.38	8.30	5.74	1.45	.006	---	.047	.149	.203	.033	.216	.087	.332	---	.041	.05	.20
5	65.38	17.38	8.19	5.99	1.42	.02	---	.050	.12	.14	.06	.29	.060	.36	.07	---	---	.36
6	65.28	17.41	8.40	5.62	1.41	---	---	.052	.148	.23	.032	.20	---	---	---	---	---	.30
7	65.26	17.51	8.38	5.72	1.39	---	---	.049	.18	.18	.09	.24	.081	.383	---	---	---	.46
Averages	65.33	17.44	8.32	5.69	1.42	.014	.005	.049	.155	.197	.033	.233	.081	.362	.044	.034	.052	.32
Recommended percentages	65.35	17.50	8.40	5.70	1.40	0.01	0.005	0.049	0.18	0.21	0.03	0.23	0.088	0.36	0.03	0.03	0.05	0.32

¹ Omitted from average.

² The recommended value listed for a constituent is the present best estimate of the "true" value. The recommended values, except for Cl, are given as the oxide on an equivalent weight basis and assume stoichiometry in the form of the oxide listed.

* List of Analysts

- H.B. Knowles, Bureau of Standards,
- M.O. Lamar, Norton Co., Worcester, MA.
- P.E. Corbin, Corning Glass Works, Corning, N.Y.
- R.H. Lardin, Pittsburgh Plate Glass Co., Creighton, PA

- D.W. Moore, 3d, Libbey-Owens-Ford Glass Co., Charleston, W.V.
- R.W. Goodwin, Glass Technology Laboratory, Incandescent Lamp Department of General Electric Co., Cleveland, Ohio.
- The Sharp-Schuriz Co., Lancaster, Ohio

Gaithersburg, MD 20899
December 20, 1990
(Editorial Revision of certificate dated 8-27-32)

William P. Reed, Acting Chief
Standard Reference Materials Program

Listing of SRM Certificates

Page 1:

U. S. Department of Commerce
Malcolm Baldrige
Secretary
National Bureau of Standards
Ernest Ammer, Director

National Bureau of Standards

Certificate

Standard Reference Material 91

Opal Glass Powder

This Standard Reference Material is intended for use in checking chemical methods of analysis and for calibrating optical emission and x-ray spectrometric methods of analysis.

<u>Constituent</u>	<u>Percent by Weight</u>
SiO ₂	67.5
CaO	10.49
Na ₂ O	8.47
Al ₂ O ₃	6.01
F	5.73
K ₂ O	3.24
As ₂ O ₅	0.10
As ₂ O ₃	0.09
PbO	0.10
ZnO	0.08
Fe ₂ O ₃	0.079
P ₂ O ₅	0.023
TiO ₂	0.019
Cl	0.015
ZrO ₂	0.009

Values are based on drying the sample for one hour at 105 to 110°C. The determinations of silica and fluorine followed the fusion of a 0.5-g sample with sodium or potassium carbonate according to Hoffman and Lundell, B.S. Jour. Research 1, 581 (1930). The other constituents were determined by methods described by Lundell and Hoffman, B.S. Jour. Research 1, 91 (1928) and by Lundell and Knowles, J. Am. Ceram. Soc. 10, 829 (1927).

Washington, D.C. 20234
October 28, 1982
(Revision of Certificate
dated 6-15-81)

(over)

George A. Uriano, Chief
Office of Standard Reference Materials

Page 2:

Present day procedures should follow the ASTM Standard Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass, C169.

MnO and MgO were both reported to be about 0.008%. No value is given for loss on ignition because reliable results could not be obtained.

Laboratories that contributed to the certification of this glass were located at:

National Bureau of Standards, Washington, D.C.
Newburgh Steel Works, Cleveland, Ohio
Booth, Garrett, & Blair, Philadelphia, Pa.
Libby-Owens Sheet Glass Co., Charleston, W. Va.
Corning Glass Works, Corning, N.Y.

Listing of SRM Certificates

Page 1:

U. S. Department of Commerce
Malcolm Baldrige
Secretary
National Bureau of Standards
Eugene Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 92

Soda-Lime Glass Powder

(Low Boron)

This Standard Reference Material (SRM) is intended for use in checking chemical methods of determining the content of B_2O_3 in glass.

B_2O_3 0.70 $\pm$ 0.03 percent by weight

The certified value is the best estimate of the "true" value based on the results of a cooperative analytical program. The certified value is not expected to deviate from the "true" value by more than the stated uncertainty. A remeasurement made in 1939 confirmed that the certified value had not changed due to aging of the powder.

This SRM should be dried for one hour at 105 °C before making an analysis. The determination of B_2O_3 was made by the Chapin distillation method including the recovery from the residue and correction by careful blank determinations.

The overall direction and coordination of the cooperative analytical program were performed by H. B. Knowles of the Analytical Chemistry Division of the National Bureau of Standards. The laboratories that contributed to the certification of this glass were located at:

National Bureau of Standards, Washington, D.C.

Norton Company, Worcester, Mass.

Booth, Garrett, & Blair, Philadelphia, Penn.

Sharp-Schurtz Company, Lancaster, Ohio

For information only, this soda-lime glass has, in addition to the certified B_2O_3 , the following approximate composition:

SiO_2	(75.0) percent by weight
Na_2O	(13.1)
CaO	(8.3)
R_2O_3	(1.5)
K_2O	(0.6)
ZnO	(0.2)
MgO	(0.1)
Loss on Ignition	(0.42)

Washington, D.C. 20234
March 11, 1982
(Revision of Certificates
dated 4-22-30 & 4-30-71)

George A. Uriano, Chief
Office of Standard Reference Materials

Listing of SRM Certificates

Page 1:

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 98b

Plastic Clay

This Standard Reference Material (SRM) is intended for use in the determination of constituent elements in clay or material of similar matrix. SRM 98b is powdered clay that was air-dried, ball-milled, and blended to ensure homogeneity.

The certified constituent elements of SRM 98b are given below in Table 1. The certified values are based on measurements made using two or more independent reliable methods or techniques. Non-certified values for constituent elements are given in Table 2 as additional information on the composition. The non-certified values should not be used for calibration or quality control. All values are based on samples that were dried for 2 hours in a conventional oven at 140 °C and a minimum sample size of 250 mg.

Table 1

Certified Values for Constituent Elements

Element ¹	Content, Wt. % ²	Element	Content, Wt. %
Aluminum ^{c,d,g}	14.30 ± 0.20	Manganese ^{b,g}	0.0116 ± 0.0005
Calcium ^{b,d,f}	0.0759 ± 0.0035	Potassium ^{b,c,f,g,i}	2.81 ± 0.07
Chromium ^{c,g}	0.0119 ± 0.0005	Silicon ^{c,i}	26.65 ± 0.16
Iron ^{c,g}	1.18 ± 0.01	Sodium ^{b,d,g}	0.1496 ± 0.0066
Lithium ^{d,f}	0.0215 ± 0.0003	Strontium ^{d,f,g}	0.0189 ± 0.0008
Magnesium ^{b,c}	0.358 ± 0.012	Titanium ^{b,g,i}	0.809 ± 0.012

¹ Methods/Techniques

a Colorimetry (o-phenanthroline)

b DC Plasma Spectrometry

c Flame Atomic Absorption Spectrometry

d Flame Emission Spectrometry

e Gravimetry

f Isotope Dilution Mass Spectrometry

g Instrumental Neutron Activation Analysis

h Spectrophotometry

i X-ray Fluorescence

² The certified value is a weighted mean of results from two or more analytical techniques. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel (NBS Journal of Research 87, 1982, pp. 377-385). The uncertainty is the sum, in quadrature, of the half-width of a 95% expected tolerance interval and an allowance for systematic error among the methods used. The interval whose endpoints are the certified value minus and plus the uncertainty, respectively, will cover the concentration in a minimum sample size of 250 mg of this SRM for at least 95% of the samples with 95% confidence.

Gaithersburg, MD 20899
April 21, 1988

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(over)

Page 2:

Table 2

Non-certified Values for Constituent Elements

<u>Element</u>	<u>Content, Wt%</u>	<u>Element</u>	<u>Content, Wt%</u>
Barium	(0.07)	Rubidium	(0.018)
Phosphorus	(0.03)	Zinc	(0.011)
		Zirconium	(0.022)
<u>Element</u>	<u>Content, µg/g</u>	<u>Element</u>	<u>Content, µg/g</u>
Antimony	(1.6)	Hafnium	(7.2)
Cesium	(16.5)	Scandium	(22)
Cobalt	(16.3)	Thorium	(21)
Europium	(1.3)		

Loss on Ignition (7.5 wt.%)

Loss on ignition was obtained by igniting sample for two hours at 1100 °C after sample was dried for two hours at 140 °C.

Source and Preparation

The plastic clay for SRM 98b was donated to NBS by F.J. Flanagan and J.W. Hasterman of the United States Geological Survey, Reston, Virginia. Approximately 220 kg of plastic clay was collected from the underclay of the Clarion coal bed at the Harbison-Walker Refractories Co. plant at Clearfield, Clearfield County, PA. The collected clay was air-dried and processed by the same method used to prepare USGS rock standards (USGS Bulletin 1582, Flanagan 1986). After processing, the sample was delivered to NBS, where it was again mixed in a 0.3 cubic meter "V" blender for approximately 45 minutes. After blending the clay was placed in polyethylene lined aluminum pails and subsequently bottled.

Homogeneity testing was performed using x-ray fluorescence and instrumental activation analysis on samples randomly selected samples from cans of bulk material. There were no significant differences between samples for any of the measured elements.

Chemical analyses were performed in the following laboratories:

- National Bureau of Standards, Center for Analytical Chemistry, E.S. Beary, D.A. Becker, W.A. Bowman III, T.A. Butler, K.A. Brletic, J.W. Gramlich, D. Mo, J.R. Moody, and T.C. Rains.
- Mineral Constitution Laboratory, Pennsylvania State University, University Park, Pennsylvania, J.B. Bodkin.
- Engelhard Corporation, Specialty Chemical Division, Edison, New Jersey, B.P. Scibek.
- Construction Technology Laboratories, Inc., Skokie, Illinois, H.M. Kanare.

The statistical analysis and evaluation of the data for certification was performed by K.R. Eberhardt and S.B. Schiller of the Statistical Engineering Division and R.L. Watters, Jr. of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Listing of SRM Certificates

Page 1:

National Institute of Standards & Technology

Certificate

Standard Reference Material 278

Obsidian Rock

This Standard Reference Material (SRM) is intended for use in evaluating the accuracy of analytical methods and instruments used in the analysis of geological type materials. SRM 278 is a finely powdered obsidian rock, which was obtained from Clear Lake, Newberry Crater, Oregon.

Certified Values of Constituents

The concentrations of the constituents were determined by methods that are widely used in the field of geological analysis and have a demonstrated accuracy. The values given are "certified" values, i.e., those values that were determined by either a definitive method, reference method, or by two or more independent methods, and "information" values that were determined by single or non-reference methods. The certified values are given in Table 1.

Table 1. Certified Values of Constituents

<u>Constituent¹</u>	<u>Content², wt %</u>	<u>Constituent¹</u>	<u>Content², mg/Kg</u>
Al ₂ O ₃ ^d	14.15 ± 0.15	Cu ^e	5.9 ± 0.2
CaO ^a	0.983 ± 0.002	Ni ^e	3.6 ± 0.3
FeO ^j	1.36 ± 0.02	Rb ^{aa}	127.5 ± 0.3
Fe ₂ O ₃ ^u	2.04 ± 0.02	Sr ^e	63.5 ± 0.1
(Total Fe as Fe ₂ O ₃)			
K ₂ O ^{add}	4.16 ± 0.02	Th ^{ef}	12.4 ± 0.3
MnO ^{aj}	0.052 ± 0.002	Ti ^e	0.54 ± 0.04
Na ₂ O ^{add}	4.84 ± 0.05	U ^a	4.58 ± 0.04
P ₂ O ₅ ^{ba}	0.036 ± 0.003	Pb ^e	16.4 ± 0.2
SiO ₂	73.05 ± 0.13		
TiO ₂ ^{ba}	0.245 ± 0.007		

¹Methods of Analysis: codes are identified in table 2.

²The estimated uncertainties of the certified values are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods and material variability of 250 mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate value or any technical data presented in this certificate.

Gaithersburg, MD 20899
March 27, 1992
(Revision of certificate dated 8-19-81)

William P. Reed, Chief
Standard Reference Materials Program

(over)

Page 2:

The overall direction and coordination of the technical measurements leading to certification were performed in the Inorganic Analytical Research Division by E.L. Garner.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by T.E. Gills. Revision of this certificate was coordinated through the Standard Reference Materials Program by J.S. Kanc.

The constituents given in Table 2 are *not certified* but are included for information only.

Table 2. Information Values

<u>Constituent¹</u>	<u>Content, wt %</u>	<u>Constituent¹</u>	<u>Content, mg/Kg</u>
C (Total Carbon) ^f	(0.05)	Ba ^f	(1140)
CO ₂ ^d	(0.01)	B ^g	(25)
F ^b	(0.05)	Ce ^f	(62.2)
MgO ^d	(0.23)	Co ^f	(1.5)
		Cr ^f	(6.1)
		Cs ^f	(5.5)
		Eu ^f	(0.84)
		Gd ^g	(5.3)
		Hf ^f	(8.4)
		Lu ^f	(0.73)
		Sb ^f	(1.5)
		Sc ^f	(5.1)
		Sm ^g	(5.7)
		Ta ^f	(1.2)
		Tb ^f	(1.0)
		Yb ^f	(4.5)
		Zn ^f	(55)

¹Methods of Analysis:

^aAtomic Absorption

^bColorimetry

^cEmission Spectrometry

^dGravimetry

^eIsotope Dilution Mass Spectrometry

^fNeutron Activation Analysis

^gPrompt-gamma Activation Analysis

^hSpecific Ion Electrode Potentiometry

ⁱTitrimetry

^jVolumetry

Page 3:

Supplemental Information

Preparation

The material was processed by the Colorado School of Mines, Colorado. Approximately 350 lbs of obsidian rock were crushed, ground, and sieved to <200 mesh. The material was mixed in a cone blender to ensure homogeneity. For homogeneity, testing and certification, samples were randomly chosen and analyzed for both major and minor constituents. The inhomogeneity of this material is considered to be $\leq 2\%$ relative.

Analysis

SRM 278, a natural glass, is hygroscopic and contains water that cannot be driven off by drying at low temperatures. This material will pick up additional water on exposure to the atmosphere. Thus, exposure time should be kept to a minimum. Furthermore, the sample should be ignited to a constant weight in a muffle furnace or over a small flame at a temperature between 350-600 °C prior to analysis. This procedure will ensure the accurate and precise determination of SiO_2 , K_2O , Na_2O , Al_2O_3 and possibly other major constituents.

The analysts and laboratories cooperating in the analytical program for certification were:

- M.J. Blackman, E.L. Garner, J.W. Gramlich, L.A. Machlan, L.J. Moore, and R. Zeisler of the Inorganic Analytical Research Division, National Institute of Standards & Technology.
- J.B. Bodkin, J.C. DeVine, and N.H. Suhr of the Mineral Constitution Laboratories, The Pennsylvania State University, University Park, Pennsylvania.
- S.S. Goldrich of the Department of Geology, Northern Illinois University, DeKalb, Illinois.
- M.D. Glascock, C.C. Graham, J.R. Vogt, University of Missouri, Columbia, Missouri.

Document attributes:

SRM# = 278

REVISION DATE = 1992-03-27

1
S

NBS-948 Analyses

Instrument: VG-2 Total Evaporation Method Decay Corrected to 6/1/97

Date	Weight %				
	238	239	240	241	242
3/28/95	0.0108	91.8295	7.9571	0.1447	0.0342
4/13/95	0.0127	91.8282	7.9590	0.1431	0.0339
4/14/95	0.0123	91.8288	7.9576	0.1441	0.0340
4/25/95	0.0121	91.8290	7.9575	0.1449	0.0335
4/26/95	0.0121	91.8281	7.9584	0.1446	0.0339
5/5/95	0.0131	91.8252	7.9578	0.1475	0.0334
5/16/95	0.0121	91.8294	7.9582	0.1441	0.0338
5/19/95	0.0142	91.8272	7.9595	0.1434	0.0336
5/24/95	0.0109	91.8312	7.9577	0.1445	0.0337
5/25/95	0.0133	91.8280	7.9585	0.1442	0.0339
6/1/95	0.0121	91.8304	7.9577	0.1445	0.0336
6/16/95	0.0110	91.8304	7.9593	0.1445	0.0336
6/20/95	0.0109	91.8311	7.9572	0.1455	0.0341
6/27/95	0.0110	91.8288	7.9578	0.1473	0.0338
6/28/95	0.0118	91.8292	7.9570	0.1470	0.0338
7/10/95	0.0117	91.8292	7.9575	0.1471	0.0336
7/11/95	0.0134	91.8290	7.9567	0.1467	0.0333
7/11/95	0.0119	91.8272	7.9595	0.1467	0.0339
7/11/95	0.0167	91.8207	7.9591	0.1481	0.0345
7/11/95	0.0113	91.8283	7.9582	0.1474	0.0340
7/11/95	0.0138	91.8277	7.9576	0.1463	0.0338
<hr/>					
Mean	0.0123	91.8284	7.9580	0.1455	0.0338
Std. Dev.	0.0014	0.0022	0.0008	0.0015	0.0003
% RSD	11.4844	0.0024	0.0105	1.0503	0.8279
Cert. (6/1/97)	0.0088	91.8576	7.9563	0.1438	0.0335
±	0.0010	0.0100	0.0100	0.0010	0.0030
<hr/>					
% Deviation	28.44689	-0.03184	0.021895	1.221252	1.004675

NBS-948 Analyses

Instrument: VG-2 Total Evaporation Method Decay Corrected to 6/1/97

Date	Weight %				
	238	239	240	241	242
3/6/97	0.0090	91.8515	7.9591	0.1439	0.0340
3/11/97	0.0101	91.8496	7.9604	0.1437	0.0338
3/12/97	0.0099	91.8501	7.9599	0.1440	0.0337
3/14/97	0.0092	91.8525	7.9591	0.1433	0.0337
3/25/97	0.0091	91.8499	7.9617	0.1438	0.0335
3/26/97	0.0099	91.8516	7.9596	0.1433	0.0337
3/27/97	0.0098	91.8521	7.9598	0.1427	0.0337
3/28/97	0.0105	91.8500	7.9608	0.1433	0.0335
4/8/97	0.0098	91.8519	7.9591	0.1436	0.0339
4/9/97	0.0149	91.8465	7.9596	0.1435	0.0339
4/17/97	0.0111	91.8489	7.9612	0.1439	0.0336
4/24/97	0.0128	91.8485	7.9604	0.1436	0.0336
4/25/97	0.0094	91.8526	7.9601	0.1428	0.0340
4/30/97	0.0124	91.8484	7.9606	0.1438	0.0338
5/1/97	0.0136	91.8468	7.9604	0.1443	0.0340
5/2/97	0.0096	91.8520	7.9597	0.1441	0.0338
5/5/97	0.0103	91.8498	7.9615	0.1436	0.0339
5/8/97	0.0098	91.8507	7.9616	0.1437	0.0336
5/12/97	0.0104	91.8510	7.9611	0.1435	0.0334
5/15/97	0.0097	91.8518	7.9604	0.1438	0.0338
<hr/>					
Mean	0.0106	91.8503	7.9603	0.1436	0.0338
Std. Dev.	0.0016	0.0018	0.0008	0.0004	0.0002
% RSD	15.1927	0.0020	0.0104	0.2709	0.5283
Cert. (6/1/97)	0.0088	91.8576	7.9563	0.1438	0.0335
±	0.0010	0.0100	0.0100	0.0010	0.0030
<hr/>					
% Deviation	16.6151	-0.00799	0.050214	-0.09649	0.827802

NBS-U005 Analyses

Instrument: VG-2 Total Evaporation Method Decay Corrected to 6/1/97

Date	Weight %				
	233	234	235	236	238
12/13/95	0.0000	0.0023	0.4839	0.0049	99.5089
2/1/96	0.0000	0.0024	0.4834	0.0053	99.5089
9/13/96	0.0000	0.0022	0.4837	0.0048	99.5093
10/11/96	0.0000	0.0027	0.4835	0.0053	99.5085
10/22/96	0.0000	0.0025	0.4841	0.0049	99.5085
11/21/96	0.0000	0.0027	0.4847	0.0055	99.5072
4/21/97	0.0000	0.0021	0.4841	0.0049	99.5089
Mean	0.0000	0.0024	0.4840	0.0050	99.5086
Std. Dev.	0.0000	0.0002	0.0004	0.0003	0.0007
% RSD	0.0000	9.6928	0.0900	5.3763	0.0007
Cert. (6/1/97)	0.0000	0.0021	0.4833	0.0046	99.5099
±	0.0000	0.0000	0.0005	0.0001	0.0010
% Deviation	0.000	11.533	0.135	8.788	-0.001

T

**Radiochemical Analysis on Working Reference Materials (WRMs)
for Capability Evaluation Project (CEP)
(May – August of 1997)**

A Summary Report

by

Amy Wong

CMR Analytical Chemistry Group, NMT-1

May 1998

Radiochemical Analysis on Working Reference Materials (WRMs) for Capability Evaluation Project (CEP)

Radiochemical Analysis of Starting Materials ($^{241}\text{AmO}_2$ and depleted uranium oxide):

Radiochemistry team received four cuts of ^{241}Am solutions (CST#200023528, 17 to 25 μg each in 10-mL diluted acid) and three cuts of depleted uranium solutions (CST#200023526, 150 mg each in 10 mL diluted acid) from the Trace Element and Pu Assay teams. All the initial aliquots were performed gravimetrically. The radiochemistry analyses were performed volumetrically using calibrated pipettes.

Determination of ^{244}Cm and ^{252}Cf

Alpha spectroscopy analysis was performed to estimate the amounts of ^{244}Cm and ^{252}Cf levels in the starting materials (^{241}Am and depleted oxide) for the CEP WRMs. If there is detectable amount ($\mu\text{g/g}$ level) of ^{244}Cm and ^{252}Cf in these starting materials, it will have adverse effects on the neutron and gamma based measurements of the WRMs.

Alpha spectroscopy was performed in an Oxford alpha spectroscopy system (OASIS) using surface barrier silicon detectors. The alpha spectrometers were energy calibrated with a source prepared from weapons grade Pu and ^{244}Cm . The energy calibrations were validated with an NIST ^{238}Pu source (SRM 4906-C-9). The concentration levels of ^{244}Cm and ^{252}Cf in the ^{241}Am and depleted uranium starting materials were estimated based on the ratio of the measured region of interests (ROIs) or alpha activities, and the half-life of each isotope. This method provides a rough upper limit estimate of ^{244}Cm and ^{252}Cf concentrations.

No actual peaks were observed in the 5.7 to 7.5 MeV region so an integrated background area (5 counts) in this energy region was used to bound ^{244}Cm and ^{252}Cf concentrations. For AmO_2 , the concentration of ^{244}Cm was less than 0.02 ppm and ^{252}Cf was less than 0.003 ppm. For depleted uranium oxide, the concentration of ^{244}Cm was less than 0.001 ppm and ^{252}Cf was less than 0.001 ppm.

^{241}Am Concentration in AmO_2

In additions, the concentration of ^{241}Am in the AmO_2 starting material (CST#200023528) was determined by liquid scintillation analysis and gamma-ray spectrometry. The liquid scintillation counter (Packard Tri-Carb 2200C) was calibrated using an NIST traceable Radiochemistry Primary plutonium oxide standard (PEOR3258-STD1) The NaI based

gamma-ray spectrometer was calibrated using an NIST traceable ^{241}Am standard solution (Amersham, AMP10040, batch 95/241/49).

The amount of ^{241}Am isotope within the sample was determined by radiochemical methods. Duplicated analyses were performed on each cut of sample. The total alpha activity was determined by liquid scintillation counting and ^{241}Am -gamma activity was determined by gamma spectrometry.

Cut	Expected, μg	LSC, μg^*	%Am	Gamma, μg	%Am
14a	21.09	16.69	79.14	16.63	78.85
14b	21.09	16.54	78.43	16.60	78.71
15a	17.37	13.83	79.62	13.77	79.27
15b	17.37	13.83	79.62	13.91	80.08
16a	24.33	19.46	79.98	19.19	78.87
16b	24.33	19.19	78.87	19.28	79.24
17a	17.82	14.09	79.07	14.26	80.02
17b	17.82	14.15	79.41	14.18	79.57
		AVG	79.27 ± 0.49	AVG	79.33 ± 0.53
		%RSD	0.62	%RSD	0.66

* Assume all alpha activity was due to ^{241}Am .

Excellent agreement in americium concentration was obtained between LSC and gamma spectroscopy results.

Other gamma emitting radionuclides.

To determine if there were other gamma-emitting radionuclides present in the ^{241}Am and depleted uranium samples, gamma-ray analysis was performed on these samples. A high purity germanium detector was energy calibrated using an Amersham QCY.44 (NIST traceable) mixed gamma radionuclide reference solution. No noticeable peaks from other gamma-emitting radionuclides were detected by the HPGe gamma-ray measurements under the extremely high level of ^{241}Am -gamma background.

DE-UO₂ Blending Verification Measurements:

Prior to loading different mass range of diatomaceous earth (DE) and depleted uranium oxide blend in the WRM, the homogeneity of these blends was verified by non-destructive gamma-ray analysis. Two different mass ranges were evaluated which included 570 and 1100 g of depleted uranium oxide in the blend.

Approximately 100 g, weighed blend sample was packed into a pre-marked, constant fill height 125 mL Nalgene polycarbonate straight side jars. Six jars of samples were obtained from different portions of the blend. These vials were bagged out from the glovebox with one layer of plastic bags. Each sample jar was held in place with a wooden ring and was counted on a high purity germanium detector at 8.5-cm distance from the detector surface.

The measurement results are shown in Tables I to III. The normalized gamma-ray peak areas at 63.3, 187, 772, and 1009-keV (from the daughter nuclides of ²³⁵U, ²³⁸U) are used to evaluate the homogeneity of the blend.

TABLE I. Blending Homogeneity of CEP011 (~570 g UO₂ in 113 g DE)

Cut #	normalized counts per 1200 second (ct/g/1200s)			
	63.3 keV	186.7 keV	772.4 keV	1009 keV
1	218.4	105.6	41.5	89.4
2	223.7	104.7	41.3	86.7
3	217.8	104.9	42.6	86.5
4	223.6	105.0	41.2	86.4
5	220.8	106.7	41.7	86.3
6	216.4	101.7	41.4	87.2
AVG	220.1	104.8	41.6	87.1
STD	3.1	1.7	0.5	1.2
% RSD at 95% CL	1.47	1.67	1.31	1.44

TABLE II. Blending Homogeneity of CEP013 (~1090 g UO₂ in 32 g DE)

Cut #	normalized counts per 1200 second (ct/g/1200s)			
	63.3 keV	186.7 keV	772.4 keV	1009 keV
1	214.1	107.7	49.4	102.3
2	215.8	105.2	48.5	103.0
3	221.3	108.0	49.1	104.5
4	209.7	106.9	46.7	103.5
5	212.1	105.0	47.9	100.4
6	211.5	103.6	47.8	104.6
AVG	214.1	106.1	48.2	103.0
STD	4.1	1.7	1.0	1.6
% RSD at 95% CL	2.02	1.71	2.13	1.60

TABLE III. Blending Homogeneity of CEP014 (~1090 g UO₂ in 32 g DE)

Cut #	normalized counts per 1200 second (ct/g/1200s)			
	63.3 keV	186.7 keV	772.4 keV	1009 keV
1	336.3	169.9	73.7	158.1
2	333.3	164.2	74.0	157.2
3	338.1	169.1	73.9	157.5
4	329.1	164.4	73.2	153.9
5	341.7	165.9	72.9	154.9
6	341.1	165.5	74.1	154.1
AVG	336.6	166.5	73.6	155.9
STD	4.8	2.4	0.5	1.8
% RSD at 95% CL	1.50	1.54	0.71	1.24

Based on the data in Tables I to III, all the blends were very homogeneous. The uniformity of these blends had met the statement of work requirements.

DE-AmO₂/PuO₂ Blending Verification Measurements:

Prior to loading plutonium oxide and americium oxide diatomaceous earth blend in the WRM, the homogeneity of a blending material was verified by non-destructive gamma-ray analysis. Due to relatively high radiation dose rate, only one set of blending was evaluated.

Approximately 6.3 g of weighed blending mix was packed into a pre-marked, constant fill height 125 mL Nalgene polycarbonate straight side jars. Six jars of samples were obtained from different portions of the blend. These vials were bagged out from the glovebox with two layer of plastic bags. Each sample jar was held in place with a 2-inch roll of tape on a rotating table and was counted on a high purity germanium detector at 99-cm distance from the detector surface. Due to high activity, the detector surface was covered with 4 layers of lead tape to reduce the dead time to ~14 %.

TABLE IV. Blending Homogeneity of CEP005 (~0.11 g of ²⁴¹AmO₂ + ~2.3 g PuO₂ + 134 g DE)

Cut #	normalized counts per 300 s (ct/g/300s)
	59.5 keV
1	116194
2	116860
3	114348
4	119827
5	113739
6	116562
AVG	116255
STD	2154
% RSD at 95% CL	1.94

From Table IV data table, the uniformity of this americium-plutonium oxide-DE blend had met the statement of work requirements.

²⁴¹Am Count Rate Assessment.

Approximately 100 µg and 200 µg of ²⁴¹Am oxide solutions were aliquoted onto health physics smears. These smears were counted at various distance in front of a high purity germanium detector. Table VI showed by increasing distance from 30 cm to 100 cm, the count rate was decreased by 90 %. These data were used to assess the personnel exposure for ALARA purpose.

TABLE IV

100 µg Am				200 µg Am			
Description	per 60 s	dead time	% at 30 cm	Description	per 60 s	dead time	% at 30 cm
BKG	150			BKG	150		
At detector surface	NA	100 %		At surface	NA	100 %	
30 cm	231919	39 %		30 cm	454702	39 %	
70 cm	43275	14 %	0.19	70 cm	87707	14 %	0.19
100 cm	22086	10 %	0.10	100 cm	43609	10 %	0.10

Determination of ²⁴¹Am in the ²⁴¹Am-Pu-DE Waste Items by Comparative Method

Preparation of ²⁴¹Am Waste Item Standards

The same set of ²⁴¹Am NDA Waste Item Standards was used to calibrate the gamma-ray measurements. The standards were prepared for the NDA PDP Phase II Standard Preparation Project.

The simulated waste container was prepared into a standard by taping four cloth swipes spiked with ²⁴¹Am standard solution. These four cloths were arranged symmetrically around the inner wall of the blending bottle. The waste item calibration standard was prepared using the same constituents of an actual waste item; a clean blending bottle, kraft papers, Q-tips, paper funnel, and kimwipes.

The amount of activity in the high-level standard (labeled as NDA High STD, 4/97) was 27.009 µCi or 7.879 µg ²⁴¹Am (as of 6/17/97). This standards was prepared from a pure ²⁴¹Am standardized solution (CST#200001684, see RC-AW-5, p.43). The CST ²⁴¹Am solution was standardized using calibrated liquid scintillation and gamma counters. The calibrations of these instruments were performed using NIST traceable standards.

Counting Conditions

The amount of ^{241}Am in each of the waste items was determined by gamma-ray spectrometry using comparative method. The count rate of the ^{241}Am 59.5- keV peak acquired for a waste item was compared to the total number of 59.5-keV counts for the standard. The waste items and standard bottles were all triply bagged in order to prevent possible contamination. Each item was counted once for 1200 seconds at 45-cm distance from the detector.

Standard Measurements

The high-level americium-241 standard (NDA High STD, 4/97) was counted four times during the waste item measurements (6/17-6/18/97). The count rate is around 100,000 counts. The relative standard deviation for the four counts was $\pm 2.0\%$.

Counting statistics of the waste item measurements

Each waste item was measured once in a fixed counting geometry similar to the standard. The counting statistics of the waste item measurements ranged from 0.15 to 0.44 %. The 59.5-keV count rate (cpm) in each waste item was compared to the average normalized count rate of the waste item standards and converted to μg of ^{241}Am .

Amount of americium left in the waste item

TABLE V. Waste Item Measurements for CEP Am Waste Items (6/97): Blending Bottles (count time 1200 s)								
		Am241		Counting	Amt of Am241 in Waste Item		Total in blend	
DATE	ID	ROI	ERR	%ERR	μCi	μg	Am, mg	%left in waste
6/17/97	CEP001-Pro	148216	427	0.29	39.28	11.45	58.0	0.020
6/17/97	CEP001-B	70866	299	0.42	18.78	5.47	10.5	0.052
6/17/97	CEP002	66096	290	0.44	17.52	5.10	9.9	0.051
6/17/97	CEP003	444003	740	0.17	117.67	34.29	54.2	0.063
6/17/97	CEP004	198572	496	0.25	52.62	15.34	54.8	0.028
6/17/97	CEP005	408645	712	0.17	108.29	31.56	92.3	0.034
6/17/97	CEP006	263178	571	0.22	69.74	20.33	93.3	0.022
6/17/97	CEP007	209350	508	0.24	55.48	16.17	54.8	0.030
6/18/97	CEP008	222826	525	0.24	59.05	17.21	52.2	0.033
6/18/97	CEP009	458186	751	0.16	121.42	35.39	103.4	0.034
6/18/97	CEP010	550938	825	0.15	146.00	42.55	99.8	0.043
6/18/97	CEP005-Vial	109957	373	0.34	29.14	8.49	92.3	0.009

Note: CEP001-Pro = CEP001, prototype; CEP001-B = CEP001, WRM; CEP005-Vial = CEP005, homogeneous measurement vials

Determination of Depleted Uranium in the DU-DE Waste Items by Comparative Method

Preparation of Depleted Uranium Standards

Depleted uranium (DU) waste item standards were prepared from highly characterized depleted uranium oxide solution. A 4.8147 g of DU oxide (87.493% U) was dissolved in 25 mL volumetric flask with 8 N HNO_3 .

The simulated waste item was prepared into a standard by taping four cloth swipes spiked with depleted uranium standard solution. These four cloths were arranged symmetrically around the inner wall of the blending bottle. The waste item calibration standard was prepared using the same constituents of an actual waste item; a clean blending bottle, kraft papers, Q-tips, paper funnel, and kimwipes.

Two waste item standards were prepared. The amount of depleted uranium in the DU Standard#1 was 168.5 mg and in the DU Standard#2 was 67.40 mg.

Counting Conditions

The amount of depleted uranium in each of the waste items was determined by gamma-ray spectrometry using comparative method. Both ^{238}U and ^{235}U have long half-life which do not have good gamma-ray signatures at low concentration. Therefore the daughter radionuclide of ^{238}U , ^{234}Th (half-life = 24.1 days) is used to quantify the amount of ^{238}U left in the waste items.

The count rate of ^{234}Th 63.3-keV peak acquired for a waste item was compared to the total number of 63.3-keV counts for the standard. The waste items and standard bottles were all double bagged in order to prevent possible contamination. Each waste item was counted once for 10000 seconds at 25-cm distance from the detector.

Standard Measurements

Each of the DU waste item standards was counted twice times during the waste item measurements (7/11/97-7/16/97). The DU Standard #1 had a total of ~1200 counts in 10000 seconds, and the DU standard #2 had a total of ~1400 counts in 30000 seconds. The overall counts per minute per mg of DU (cpm/mg DU) was 0.043 cpm/mg $\pm$ 0.003 cpm/mg (% RSD = 6.8 %).

Counting statistics of the waste item measurements

Each waste item was measured once for 10,000 seconds. The counting statistics of the waste item measurements ranged from 3.3 to 24.8 %. The count rate (cpm) at 63.3 keV of each waste item was compared to the average normalized count rate of the waste item standards and converted to mg of depleted uranium.

Amount of depleted uranium left in the waste item

All the measurements showed that less than 0.05 % of depleted uranium was retained in all the waste items. Although the counting statistics were poor for all the measurements, it was satisfied and met the criteria of the Statement of Work.

**TABLE VI. Waste Item Measurements for CEP Depleted Uranium Waste Items (7/97):
Blending Bottles (count time 10000 s)**

		63.32 keV, ^{234}Th			Amt of DU in waste item	Total in blend	
DATE	ID	Total counts	ERR	%ERR	mg DU	DU, g	% in Waste
7/11/97	CEP11a	1170	109	9.3	162.4	499.1	0.033
7/11/97	CEP12	432	107	24.8	60.0	884.0	0.007
7/14/97	CEP13	546	107	19.6	75.8	957.7	0.008
7/15/97	CEP14	3664	121	3.3	508.6	960.0	0.053
7/15/97	CEP15	871	108	12.4	120.9	972.8	0.012
7/11/97	CEP16	1259	113	9.0	174.8	948.8	0.018

Homogeneity Evaluation of ^{239}Pu - ^{241}Am -DE Content within Each Working Reference Material:

CEP ^{239}Pu - ^{241}Am -DE working reference materials (WRMs) were analyzed by non-destructive gamma-ray analysis on a HPGe detector. The purpose of this measurement was to provide a verification of the homogeneity of the Pu or/and Am content within each WRM. The homogeneity of each WRM is verified by comparing the count rate of ^{239}Pu or/and ^{241}Am peaks for the left and right segments of each WRM.

Counting Geometry

A portable HPGe photon detector was shielded with lead brick with the detector surface facing horizontally. A few lead bricks were used to create a window of 5 cm wide and 56 cm away from the detector surface. The WRM was placed horizontally inside of the lead bricks with the left or right side exposed the window opening.

Each side of WRM was counted for 600 seconds except CEP001. The net areas of gamma-ray peaks at 59.5 keV (^{241}Am), and 129 keV (^{239}Pu) were recorded.

Method of Data Analysis:

The net areas of each gamma ray peak obtained from the left and right sides of each WRM were compared, and the deviation from the average net area was calculated. The table below showed the count ratio obtained from the left to right side of each WRM. The average left to right ratios for ^{241}Am and ^{239}Pu peaks were within 5% except for low mass Pu WRM (CEP002). This data (TABLE VI) showed that the blending materials were homogeneous distributed within each WRM.

TABLE VI. CEP ²⁴¹Am WRMs Homogeneity Measurements

					Gamma-Ray Measurement, 600 seconds, 7/11/97, 7/15/97, 7/17/97							
					59.5 keV ²⁴¹ Am				129 keV ²³⁹ Pu			
WRM #	Time (s)	Side	Am, mg	Pu, g	count	+/-	L/R	ct/mg	count	+/-	L/R	ct/g
CEP001	600	L1	10.48	0.09	11750	121	1.000	1112	391	29		4134
CEP001	600	R1			12037	123			359	29		
CEP001	600	L2			11545	119			363	29		
CEP001	600	R2			11857	121			330	29		
CEP001	1800	L3			12009				322			
CEP001	7200*	CEN			10911				307		0.976	3642
CEP001	7200*	L4			11512				315			
CEP002	600	L1	9.92	0.08	10808	116	1.002	1089	284	27	1.088	3460
CEP002	600	R1			10789	116			261	28		
CEP003	600	L1	54.21	1.99	59368	270	0.991	1100	7181	97	1.006	3598
CEP003	600	R1			59920	271			7135	97		
CEP004	600	L1	54.76	2.01	54107	259	1.006	985	7087	96	1.025	3484
CEP004	600	R1			53786	259			6914	95		
CEP005	600	L1	92.29	1.99	102536	353	1.000	1111	7159	99	0.983	3622
CEP005	600	R1			102559	353			7286	99		
CEP006	600	L1	93.27	1.99	98057	345	1.045	1021	7067	98	1.004	3537
CEP006	600	R1			92324	336			6899	98		
CEP006	600	R2			95400	341			7172	90		
CEP007	600	L1	54.79	0.40	68923	272	1.021	1198	1447	51	1.035	3855
CEP007	600	R1			68407	277			1459	51		
CEP007	600	L2			63690	276			1656	53		
CEP007	600	R2			61498	271			1539	51		
CEP008	600	L1	52.16	0.41	58825	267	1.011	1128	1661	52	1.016	3883
CEP008	600	R1			58401	266			1634	51		
CEP008	600	L2			58637	266			1565	50		
CEP008	600	R2			58180	258			1607	52		
CEP008	600	L3			60003	267			1630	51		
CEP008	600	R3			58923	261			1539	52		
CEP009	600	L1	103.45		117609	376	1.018	1118
CEP009	600	L2			115701	373						
CEP009	600	R1			114137	372						
CEP009	600	R2			114976	373						
CEP010	600	L1	99.83		126938	391	0.999	1189				
CEP010	600	R1			125756	390						
CEP010	600	L2			110417	362						
CEP010	600	R2			111864	363						
					AVG	1105			AVG*	3635		
					STD	65			*			
					%RSD	5.89			STD	158		
									%RSD	4.35		

*The counts in 59.5-keV and 129-keV peaks were expressed as 600 s counts.

** The short counts (600, 1800 s) on CEP001 WRM were not included in the average of normalized 129-keV peak area due to poor peak shape and counting statistics.

Verification of ²³⁹Pu and ²⁴¹Am Content of the Working Reference Materials:

Table VI also showed the normalized ²⁴¹Am and ²³⁹Pu count rates (ct/g) for each WRM. The average counts per gram of ²⁴¹Am and ²³⁹Pu were within 6 %. CEP001 was not included in ²³⁹Pu calculation due to low Pu mass.

U

The calculations of interest for this appendix are the uncertainties associated with the measurements of the activity and the grams of Pu and Am in each of the Working Reference Material(s) or "WRM".

Activity Calculations

Activity, in curies, has been defined as:

$$\text{Activity} = \text{Specific Activity} * \text{Weight}$$

$$\text{or: } A = SA * W$$

So, the variance of Activity can be written as:

$$\text{Var}(A) = SA^2 * \text{Var}(W) + W^2 * \text{Var}(SA) + \text{Var}(SA) * \text{Var}(W)$$

Specific Activity can be written as:

$$SA = F_{Pu} * \sum_i F_i * SA_i + F_{Am} * SA_{Am}, \text{ where}$$

$$i = Pu_{238}, Pu_{239}, Pu_{240}, Pu_{241}, Pu_{242}$$

$$F_i = \text{Isotope Fraction}$$

$$SA_i = \text{Isotope Specific Activity}$$

The branching ratio was included with the 241, but not any others.

Variance of the Specific Activity is then:

$$\begin{aligned} \text{Var}(SA) \cong F_{Pu}^2 * \text{Var}\left(\sum_i F_i SA_i\right) + \left(\sum_i F_i SA_i\right)^2 * \text{Var}(F_{Pu}) \\ + F_{Am}^2 * \text{Var}(SA_{Am}) + SA_{Am}^2 * \text{Var}(F_{Am}) \end{aligned}$$

$$\text{Var}\left(\sum_i F_i SA_i\right) = \sum_i \left(\text{Var}(F_i SA_i)\right) + 2 \sum_{i \neq j} \text{Cov}(F_i SA_i, F_j SA_j)$$

$$\text{Var}(F_i SA_i) = F_i^2 \text{Var}(SA_i) + SA_i^2 \text{Var}(F_i) + \text{Var}(SA_i) * \text{Var}(F_i)$$

and

$$\text{Cov}(F_i SA_i, F_j SA_j) \cong \rho * SA_i \sqrt{\text{Var}(F_i)} * SA_j \sqrt{\text{Var}(F_j)}$$

since

$$\rho(x, y) = \frac{\text{Cov}(x, y)}{\sqrt{\text{Var}(x) * \text{Var}(y)}}$$

It is assumed that SA_i and SA_j are constants, so their variance is 0. Since $\rho \leq 1$, we can get a maximum covariance value to use in deriving the variance of the Specific Activity by setting $\rho = 1$. Using values supplied, all terms can be calculated to derive the variance for the activity due to Plutonium or Americium.

In the activity calculations it is assumed that all Americium is in the form of AmO_2 . So the Am from the Plutonium was added to that of the AmO_2 to do the calculations.

Gram Value Uncertainties

Each WRM has a measured amount of PuO_2 put into the container that makes up the WRM. The PuO_2 is weighed and then transferred into the container for the WRM. Any PuO_2 not transferred is considered in a loss term which is a count measurement calibrated to grams of Am. The "net" transferred grams of PuO_2 is used to determine the amount of Pu and Am in a WRM. Using information on the fraction of Pu in PuO_2 and the fraction of Am in the PuO_2 , the grams of Am and Pu are calculated. As equations this information is presented as:

$$Pu = PuO_{2(net)} * \bar{F}_{Pu}$$

$$Am = PuO_{2(net)} * \bar{F}_{Am}$$

and:

$$\begin{aligned} PuO_{2(net)} &= PuO_{2(meas)} - PuO_{2(loss)} \\ &= PuO_{2(meas)} - \frac{Am_{(loss)}}{\bar{F}_{Am}} \end{aligned}$$

Where:

Pu = grams of PU in the WRM

Am = grams of Am in the WRM

$\bar{F}_{Pu}$ = fraction of Pu in the PuO_2

$\bar{F}_{Am}$ = fraction of Am in the PuO_2

$Am_{(loss)}$ = amount of Am lost during transfer

$PuO_{2(meas)}$ = amount of PuO_2 measured before transfer

$PuO_{2(net)}$ = amount of PuO_2 after loss is subtracted

The uncertainty associated with the grams of Pu in each WRM.

The variance of the Pu value, where $Pu = PuO_{2(net)} * \bar{F}_{Pu}$, can be calculated as:

$$Var[Pu] = (PuO_{2(net)})^2 * Var[\bar{F}_{Pu}] + (\bar{F}_{Pu})^2 * Var[PuO_{2(net)}] + Var[\bar{F}_{Pu}] * Var[PuO_{2(net)}]$$

where $Var[x]$ is the variance for variable "x".

$PuO_{2(net)}$ is the measured amount of PuO_2 after the loss has been subtracted. The uncertainty in this value, " $Var[PuO_{2(net)}]$ ", includes measurement uncertainty from the balance and uncertainty in the calculation of the loss of PuO_2 .

$\bar{F}_{Pu}$ is the relative amount of Pu in the PuO_2 . The determination of the values for $\bar{F}_{Pu}$ and $Var[\bar{F}_{Pu}]$ is discussed elsewhere in the document.

All values except $Var[PuO_{2(net)}]$ come directly from current measurements and historical values (meaning values for these variables are available from existing data). $Var[PuO_{2(net)}]$ can be found from the equation:

$$Var[PuO_{2(net)}] = Var[PuO_{2(meas)}] + Var\left[\frac{Am_{(loss)}}{\bar{F}_{Am}}\right]$$

Here $Var[PuO_{2(meas)}]$ is the balance uncertainty and $Var\left[\frac{Am_{(loss)}}{\bar{F}_{Am}}\right]$ is the uncertainty for the loss term. As discussed elsewhere in the document there are replicate measurements from which $Var[PuO_{2(meas)}]$ can be estimated. Loss comes from a count measurement for Am, so this term includes systematic uncertainties due to the calibration of a Am standard to counts and the uncertainty of deriving the fraction of Am in the PuO_2 . The determination of the values for $\bar{F}_{Am}$ and $Var[\bar{F}_{Am}]$ is discussed elsewhere in the document. $Var\left[\frac{Am_{(loss)}}{\bar{F}_{Am}}\right]$ can be approximated as:

$$Var\left[\frac{Am_{(loss)}}{\bar{F}_{Am}}\right] \cong \left(\frac{Am_{(loss)}}{\bar{F}_{Am}}\right)^2 * \left(\frac{Var[Am_{(loss)}]}{(Am_{(loss)})^2} + \frac{Var[\bar{F}_{Am}]}{(\bar{F}_{Am})^2}\right)$$

This can be rearranged to appear as relative standard deviations (RSD):

$$\frac{Var\left[\frac{Am_{(loss)}}{\bar{F}_{Am}}\right]}{\left(\frac{Am_{(loss)}}{\bar{F}_{Am}}\right)^2} \cong \frac{Var[Am_{(loss)}]}{(Am_{(loss)})^2} + \frac{Var[\bar{F}_{Am}]}{(\bar{F}_{Am})^2}$$

or:

$$[RSD_{loss}]^2 \cong [RSD_{Am_{(loss)}}]^2 + [RSD_{assay}]^2$$

Looking at the value for the RSD for the Am assay:

$$[RSD_{assay}]^2 = \frac{Var[\bar{F}_{Am}]}{(\bar{F}_{Am})^2}$$

$$= \frac{1.35 * 10^{-14}}{(8.23 * 10^{-4})^2} = 2.0 * 10^{-8}$$

The values used in the equations presented in this appendix are discussed briefly in the last section of this appendix.

The value for the RSD of the Am measurement is a function of the amount of Am in the measurement. At present, a lower bound for this value is of interest. From Appendix ??? (the listing of values) the counting statistics for the Am measurements will be used to estimate the RSD for the Am measurements. The nominally 30 mg WRMs have RSD values for Am measurements ranging from 2.0% to 10%. For the WRMs nominally close to 300 mg and 3000 mg the uncertainty is considered no greater than 4.4%. It is assumed that 2.0% is the lower bound for the RSD of the Am measurements, so:

$$[RSD_{Am(meas)}]^2 = \frac{Var[Am_{(loss)}]}{(Am_{(loss)})^2}$$

$$\geq (0.02)^2 = 4 * 10^{-4}$$

Since RSD_{assay} will be much less than $RSD_{Am(meas)}$, the value of the RSD_{assay} will not affect the final result and will not be included in the calculation. So:

$$Var\left[\frac{Am_{(loss)}}{\bar{F}_{Am}}\right] \equiv \left(\frac{Am_{(loss)}}{\bar{F}_{Am}}\right)^2 * \left(\frac{Var[Am_{(loss)}]}{(Am_{(loss)})^2}\right)$$

or:

$$Var\left[\frac{Am_{(loss)}}{\bar{F}_{Am}}\right] \equiv [RSD_{Am_{(loss)}}]^2 * \left(\frac{Am_{(loss)}}{\bar{F}_{Am}}\right)^2$$

so:

$$\begin{aligned}
Var[PuO_2] &= Var[PuO_{2(meas)}] + Var\left[\frac{Am_{(loss)}}{\bar{F}_{Am}}\right] \\
&= Var[PuO_{2(meas)}] + [RSD_{Am_{(loss)}}]^2 * \left(\frac{Am_{(loss)}}{\bar{F}_{Am}}\right)^2 \\
&= Var[PuO_{2(meas)}] + \left(\frac{RSD_{Am_{(loss)}} * Am_{(loss)}}{\bar{F}_{Am}}\right)^2
\end{aligned}$$

The values for $RSD_{Am_{(meas)}}$ vary with the specific measurement of Am. In this application, for the WRMs nominally close to 300 mg and 3000 mg the uncertainty is considered no greater than 4.4%. For the nominally 30 mg WRMs the counting statistics values are used which range from 2.0% to 10%.

The uncertainty associated with the grams of Am from PuO₂ in each WRM.

Having discussed the values for calculating the variance of the grams of Pu, all the values to calculate the variance for the grams of Am from PuO₂ have also been discussed. Since:

$$Am = PuO_{2(net)} * \bar{F}_{Am},$$

The variance of Am is calculated as:

$$Var[Am_{(meas)}] = (PuO_{2(net)})^2 * Var[\bar{F}_{Am}] + (\bar{F}_{Am})^2 * Var[PuO_{2(net)}] + Var[\bar{F}_{Am}] * Var[PuO_{2(net)}]$$

Including Systematic Uncertainty in the uncertainty in Pu and Am from PuO₂ values

It is assumed that errors in developing a calibration for Am (which is needed to measure the amount of PuO₂ loss) show up as a bias in the net amount of PuO₂. The size of the bias changes when PuO₂ is converted to grams of Pu using the fraction of Pu in PuO₂. We assume that with the bias, a 95% confidence interval on the amount of Pu would be the minimum and maximum of:

$$(PuO_2 \pm Bias) * \bar{F}_{Pu} \pm 2 * \sqrt{Var[Pu]}$$

This has the effect of putting a 95% confidence interval on the value of the PuO₂ measurement plus the bias. This should yield a conservative or upper bound estimate of the true 95% confidence interval.

Similarly, for Americium, a 95% confidence interval for the Am measurement is the minimum and maximum of:

$$(PuO_2 \pm Bias) * \bar{F}_{Am} \pm 2 * \sqrt{Var[Am]}$$

The estimated maximum bias varies with the amount of material in the WRM. For all WRMs the random uncertainty in the calibration is expected to be less than 1.6%. For the WRMs nominally 30 mg, the systematic error in the calibration is expected to be less than 15%. Total bias for a nominal 30 mg WRM is expected to be less than 15%+2*1.6% = 18% (to two significant digits). Similarly, for the nominal 300 mg WRMs, total bias is expected to be less than 9% + 2*1.6% = 12%. The nominal 3000mg WRMs are expected to have a total bias less than 6% + 2*1.6% = 9%. These values are used in the equation above.

There is also a bias that develops in the fraction of Am in PuO₂ measurement. The ²⁴¹Am term relates to the ²⁴³Am spike and there is uncertainty in this spike. There is no hard data on the size of this uncertainty. From comparisons with other measurements and expert opinion it is felt that the ²⁴³Am concentration has a bias of 0.25% or less. It is assumed that the value of $\bar{F}_{Am}$ has a bias of 0.25% or less with at least 95% confidence.

For the grams of Am value, this means that the uncertainty for this value is at least 0.25% since $Am = PuO_{2(net)} * \bar{F}_{Am}$ and the uncertainty on $\bar{F}_{Am}$ is 0.25%. This is an additive error and adding 0.25% error onto the error already found would roughly give the total uncertainty. Adding this uncertainty value to the uncertainty already discussed was accomplished by finding the maximum total uncertainty using $\bar{F}_{Am} + 0.25\%$, then finding the minimum total uncertainty using $\bar{F}_{Am} - 0.25\%$. Here, total uncertainty is defined by the equation above for the 95% confidence interval. The difference between these two, the maximum and the minimum total uncertainties, gives an uncertainty bound when this 0.25% bias in $\bar{F}_{Am}$ is included. It is believed this uncertainty bound is at least 95%.

For the uncertainty in the grams of Pu, the same process is done as for Am. Using the equation above for the 95% confidence interval for the Pu measurements, the maximum total uncertainty using $\bar{F}_{Am} + 0.25\%$, and the minimum total uncertainty using $\bar{F}_{Am} - 0.25\%$ are found. The

difference in these two values gives an uncertainty bound that is at least 95%. The added effect of this bias is much less for Pu than for Am.

Total Grams of Am From AmO₂ and PuO₂

The total Americium in the standard comes from Plutonium oxide and Americium oxide or:

$$Am_{net} = Am_{PuO_2} + Am_{AmO_2}$$

$$Am_{AmO_2} = AmO_2 * \bar{F}_{Am_{AmO_2}} \quad \text{where } \bar{F}_{Am_{AmO_2}} \text{ is the fraction of Am in } AmO_2$$

So :

$$Am_{net} = PuO_2 * \bar{F}_{Am_{PuO_2}} - Am_{loss_{PuO_2}} + AmO_2 * \bar{F}_{Am_{AmO_2}} - Am_{loss_{AmO_2}}$$

$$Var(Am_{net}) = Var(PuO_2 * \bar{F}_{Am_{PuO_2}} - Am_{loss_{PuO_2}}) + Var(AmO_2 * \bar{F}_{Am_{AmO_2}}) + Var(Am_{loss_{AmO_2}})$$

We have previously dealt with the variance of the Am from PuO₂, so now we look at the variance of the Am from AmO₂, which we write as:

$$Var(Am_{AmO_2}) = AmO_2^2 * Var(\bar{F}_{Am}) + \bar{F}_{Am}^2 * Var(AmO_2) + Var(\bar{F}_{Am}) * Var(AmO_2).$$

All these terms are available from the data.

Looking at the loss term, from the data we have a relative standard deviation term for the Am loss term, so we use $Var(Am_{loss_{AmO_2}}) = (RSD_{Am} * Am_{Loss_{AmO_2}})^2$. Note that uncertainty of the homogeneity of the AmO₂ and PuO₂ mixture add uncertainty to the Am loss term. We can write this in an equation as:

$$Loss_{Am} = P_{AmO_2} * Am_{Loss_{total}}, \quad \text{where } P_{AmO_2} \text{ is the proportion of Am from } AmO_2$$

So :

$$Var(Loss_{Am}) = P_{AmO_2}^2 * Var(Am_{Loss_{total}}) + Var(P_{AmO_2}) * Am_{Loss_{total}}^2 + Var(Am_{Loss_{total}}) * Var(P_{AmO_2})$$

We assume that the mixture is homogeneous so that there is no uncertainty in the proportion of Am from AmO₂. This last equation, when assuming homogeneity, is then the same equation above with the RSD term.

More Details

The initial measurements were made in June 1995. All assay and isotopic measurements were made current to 6/1/97.

The balance variability values came from check weighings done during the measurements. The PuO₂ assay and isotopic values and uncertainties are long term uncertainties from months of checks. The AmO₂ assay and isotopic values and uncertainties came from four replicates.

$PuO_{2(meas)}, AmO_{2(meas)}$ = varies with each WRM

$Var[PuO_{2(meas)} \text{ or } AmO_{2(meas)}]$ = 5.883 E-10g for <0.10g
 =1.258 E-09g for <1.0 and >0.10g
 =1.323 E-09g for >1.0g

Mean of the fraction of Pu in PuO₂ =0.87804 g/g

Variance of the fraction of Pu in PuO₂ =9.868E-08 g²/g²

Mean of the fraction of Am in PuO₂ = 1.35E-14 g/g

Variance of the fraction of Am in PuO₂ =9.3907 E-04 g²/g²

Mean of the fraction of Am in AmO₂ =0.78727 g/g

Variance of the fraction of Am in AmO₂ =1.25E-06 g²/g²

Mean of the fraction of Pu in AmO₂ is set at 0.

Variance of the fraction of Pu in AmO₂ is set at 0.

$Am_{(loss)}$ = varies with each WRM

$RSD_{Am(meas)}$ =4.4% or RSD from counts, whichever is larger

Bias of $\bar{F}_{Am}$ = 0.25%

Bias of Am calibration = 6.8% for 6000 cpm and above
 =7% for below 6000 cpm and above 1500 cpm

The calculations of interest for this appendix are the uncertainties associated with the measurements of the activity and the grams of U in each of the Working Reference Material(s) or "WRM".

Activity Calculations

Activity has units curies and is defined as:

$$Activity = Specific\ Activity * Weight$$

or: $A = SA * W$

So, the variance of Activity can be written as:

$$Var(A) = SA^2 * Var(W) + W^2 * Var(SA) + Var(SA) * Var(W)$$

Specific Activity can be written as:

$$SA = \sum_i F_i * SA_i, \text{ where}$$

$$i = U_{233}, U_{234}, U_{235}, U_{236}, U_{238}$$

$$F_i = \text{Isotope Fraction}$$

$$SA_i = \text{Isotope Specific Activity}$$

Variance of the Specific Activity is then:

$$Var\left(\sum_i F_i SA_i\right) = \sum_i (Var(F_i SA_i)) + 2 \sum_{i \neq j} Cov(F_i SA_i, F_j SA_j)$$

$$Var(F_i SA_i) = F_i^2 Var(SA_i) + SA_i^2 Var(F_i) + Var(SA_i) * Var(F_i)$$

and

$$Cov(F_i SA_i, F_j SA_j) \cong \rho * SA_i \sqrt{Var(F_i)} * SA_j \sqrt{Var(F_j)}$$

since

$$\rho(x, y) = \frac{Cov(x, y)}{\sqrt{Var(x) * Var(y)}}$$

It is assumed that SA_i and SA_j are constants, so their variance is 0. Since $\rho \leq 1$, we can get a maximum covariance value to use in deriving the variance of the Specific Activity by setting $\rho = 1$. Using values supplied, all terms can be calculated to derive the variance for the activity due to Uranium.

Specific Activity is given in terms of curies per gram of Uranium oxide. The weight term is then weight of Uranium oxide.

U Gram Value Uncertainties

We are interested in the gram of Uranium in each “working reference material” or WRM and the uncertainty associated with this gram value. We have developed equations to fit the procedures by which the WRMs were developed. Some knowledge of the procedures used to prepare the WRM’s is assumed as no explanations are given below. A couple WRM’s has procedures that were slightly different from the rest and so had terms that were defined using different weighings or may had an added term. These changes account for minute changes in the final answer, so the work below gives the information needed to determine if the uncertainties were derived in a reasonable fashion.

The amount of Uranium in each tube that is an WRM is:

$$U_{Tube} = gDUO_{2\ REM} * \ddot{F}_U - gU_{Holdup}$$

where :

U_{Tube} = grams of U in the Tube

$gDUO_{2\ REM}$ = grams of DUO_2 removed from the mixing bottle

$\ddot{F}_U$ = Fraction of Uranium in DUO_2

gU_{Holdup} = grams of U in Holdup

So :

$$\begin{aligned} Var(U_{Tube}) &= Var(gDUO_{2\ REM} * \ddot{F}_U) + Var(gU_{Holdup}) \\ &\equiv Var(gDUO_{2\ REM}) * (\ddot{F}_U)^2 + (gDUO_{2\ REM})^2 * Var(\ddot{F}_U) + Var(gU_{Holdup}) \end{aligned}$$

Note : $\ddot{F}_U$, $Var(\ddot{F}_U)$ come from five U assays

: gU_{Holdup} , $Var(gU_{Holdup})$ are supplied with the holdup measurements

Continuing :

$$gDUO_{2\ REM} = gBlend_{Rem} * P$$

where :

$$P = \frac{gDUO_2 \text{ put in blend}}{gBlend} = \text{proportion of } DUO_2 \text{ in Blend}$$

$$Var(gDUO_{2\ REM}) \equiv Var(gBlend_{Rem}) * P^2 + Var(P) * (gBlend_{Rem})^2$$

$$gBlend_{Rem} = gBlendandBottle_{beforeloading} - gBlendandBottle_{afterloading}$$

$$\begin{aligned} Var(gBlend_{Rem}) &\leq Var(gBlendandBottle_{beforeloading}) + Var(gBlendandBottle_{afterloading}) \\ &\quad + 2\sqrt{Var(gBlendandBottle_{beforeloading}) * Var(gBlendandBottle_{afterloading})} \end{aligned}$$

where :

$Var(gBlendandBottle_{beforeloading})$, $Var(gBlendandBottle_{afterloading})$ are balance uncertainties

Writing $P = \frac{X}{Y}$,

$$\text{Var}(P) \leq \left(\frac{X}{Y}\right)^2 * \left(\left(\frac{\text{Var}(X)}{X^2}\right) + \frac{\text{Var}(Y)}{Y^2} + 2 \frac{\sqrt{\text{Var}(X)\text{Var}(Y)}}{XY} \right) + \text{Homogeneity}$$

where : Homogeneity is a Variance term

Here we are using $\rho(x, y) = \frac{\text{Cov}(x, y)}{\sqrt{\text{Var}(x)\text{Var}(y)}}$ and $-1 \leq \rho \leq 1$

Note :

$\text{Var}(X) = \text{Var}(g\text{DUO}_2 \text{ put in blend}) = \text{Balance uncertainty}$

$$\begin{aligned} \text{Var}(Y) &= \text{Var}(g\text{Blend}) = \text{Var}(g\text{DE}_{\text{initial}} - g\text{DE}_{\text{removed}} + g\text{DUO}_2) \\ &\leq \text{Var}(g\text{DE}_{\text{initial}}) + \text{Var}(g\text{DE}_{\text{removed}}) + \text{Var}(g\text{DUO}_2) + 2\sqrt{\text{Var}(g\text{DE}_{\text{initial}})\text{Var}(g\text{DE}_{\text{removed}})} + 0 \end{aligned}$$

where DE is simply a filler

$\text{Var}(g\text{DE}_{\text{initial}})$, $\text{Var}(g\text{DE}_{\text{removed}})$, $\text{Var}(g\text{DUO}_2)$ are balance uncertainties

Balance uncertainties come from multiple weighings of standards throughout the procedure. There are anywhere from 12 to 40 weighings of a given standard on a given balance. Uncertainties include a systematic and random error term. An approximate 95% uncertainty for the balances was defined as systematic plus two standard deviations of the random error. For the propagation of error work, one half of this 95% uncertainty bound was used as one standard deviation. This overestimates the true error, though these errors add very little to the final uncertainty, so we believe this conservative estimate is adequate.

The homogeneity estimates come from looking at six subsamples of the blend. Counts are measured at five keV values for each of the six subsamples. To be conservative, the KeV value that gives the largest distribution of counts, as measured by the standard deviation of the counts, is used for the homogeneity value. The counts per gram of blend are transformed into gDUO2 per gram of blend and expressed as a variance term. This variance term is added directly to the variance for the proportion of DUO2 in the blend. This again is conservative in that it tends to over estimate the true variance. This term is more important than the balance uncertainties, but still is a small variance term for the final uncertainty. Again, this amount of conservativeness in defining the error seems adequate.

More Details

The balance variability values came from check weighings done during the measurements.

The UO₂ assay and isotopic values and uncertainties came from five replicates.

$UO_{2(\text{meas})}$ = varies with each WRM

$\text{Var}[UO_{2(\text{meas})}]$ = Varies with balance and range of mass

Mean of the fraction of U in UO₂ = 0.87493 g/g

Variance of the fraction of U in UO₂ = 9.68-09 g²/g²

$U_{(\text{loss})}$ = varies with each WRM

$RSDU_{(\text{meas})}$ = varies with each WRM

Bias of $\bar{F} = 0. \%$

Bias of U calibration = 0% for 6000 cpm and above

v

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP001

PuO_2 - AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity plutonium dioxide and americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Pu and Am content, Pu isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
PuO_2 in WRM	0.09716 g	$\pm 0.00012 \text{ g}^{(a)}$
Pu Assay	0.87804 g Pu/g PuO_2	$\pm 0.00036 \text{ g}^{(b)}$
Pu in WRM	0.08531 g	$\pm 0.00011 \text{ g}^{(c)}$
AmO_2 in WRM ^(d)	0.01319 g	$\pm 0.00009 \text{ g}^{(e)}$
Am Assay1 ^(f)	1069.5 μg Am/g PuO_2	$\pm 2.8 \mu\text{g}^{(g)}$
Am Assay2 ^(h)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(i)}$
Am in WRM ^(j)	0.010478 g	$\pm 0.00007 \text{ g}^{(k)}$
Isotopic Weight Fraction		
^{238}Pu	0.000143	$\pm 0.000018^{(l)}$
^{239}Pu	0.937811	$\pm 0.000028^{(l)}$
^{240}Pu	0.059448	$\pm 0.000010^{(l)}$
^{241}Pu	0.002038	$\pm 0.000019^{(l)}$
^{242}Pu	0.000560	$\pm 0.000003^{(l)}$
Total α Activity in WRM	42.31 mCi	$\pm 0.23 \text{ mCi}^{(m)}$
	1.56E+09 Bq	$\pm 8.54\text{E}+06 \text{ Bq}^{(m)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Pu Assay	Controlled Potential Coulometry	NBL CRM 126
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
Pu Isotopic	Total Evaporation Mass Spec.	NBS SRM 948
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Amount of AmO_2 weighed for the WRM (does not contain the amount of Am from the decay of ^{241}Pu in the PuO_2).
- (e) Above weighing and assay terms and correction for transfer loss
- (f) The amount of Am contained within the PuO_2 from grow-in.
- (g) Long term precision, weighing precision and bias
- (h) The amount of Am contained within the AmO_2
- (i) Long term precision, weighing precision and bias
- (j) Total amount of Am in the WRM from both the AmO_2 and the PuO_2
- (k) Above weighing terms, Am assay terms, and corrections for transfer loss
- (l) Long term precision and bias
- (m) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP002

PuO_2 - AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity plutonium dioxide and americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Pu and Am content, Pu isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

Component	Quantity or Value	95 % CI
PuO_2 in WRM	0.08963 g	$\pm 0.00012 \text{ g}^{(a)}$
Pu Assay	0.87804 g Pu/g PuO_2	$\pm 0.00036 \text{ g}^{(b)}$
Pu in WRM	0.07870 g	$\pm 0.00010 \text{ g}^{(c)}$
AmO_2 in WRM ^(d)	0.01248 g	$\pm 0.00008 \text{ g}^{(e)}$
Am Assay1 ^(f)	1069.5 μg Am/g PuO_2	$\pm 2.8 \mu\text{g}^{(g)}$
Am Assay2 ^(h)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(i)}$
Am in WRM ^(j)	0.00991 g	$\pm 0.00007 \text{ g}^{(k)}$
Isotopic Weight Fraction		
^{238}Pu	0.000143	$\pm 0.000018^{(l)}$
^{239}Pu	0.937811	$\pm 0.000028^{(l)}$
^{240}Pu	0.059448	$\pm 0.000010^{(l)}$
^{241}Pu	0.002038	$\pm 0.000019^{(l)}$
^{242}Pu	0.000560	$\pm 0.000003^{(l)}$
Total α Activity in WRM	39.88 mCi	$\pm 0.23 \text{ mCi}^{(m)}$
	1.48E+09 Bq	$\pm 8.37\text{E}+06 \text{ Bq}^{(m)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Pu Assay	Controlled Potential Coulometry	NBL CRM 126
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
Pu Isotopic	Total Evaporation Mass Spec.	NBS SRM 948
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Amount of AmO_2 weighed for the WRM (does not contain the amount of Am from the decay of ^{241}Pu in the PuO_2).
- (e) Above weighing and assay terms and correction for transfer loss
- (f) The amount of Am contained within the PuO_2 from grow-in.
- (g) Long term precision, weighing precision and bias
- (h) The amount of Am contained within the AmO_2
- (i) Long term precision, weighing precision and bias
- (j) Total amount of Am in the WRM from both the AmO_2 and the PuO_2
- (k) Above weighing terms, Am assay terms, and corrections for transfer loss
- (l) Long term precision and bias
- (m) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP003

PuO_2 - AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity plutonium dioxide and americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Pu and Am content, Pu isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
PuO_2 in WRM	2.26382 g	$\pm 0.00236 \text{ g}^{(a)}$
Pu Assay	0.87804 g Pu/g PuO_2	$\pm 0.00036 \text{ g}^{(b)}$
Pu in WRM	1.98772 g	$\pm 0.00207 \text{ g}^{(c)}$
AmO_2 in WRM ^(d)	0.06610 g	$\pm 0.00027 \text{ g}^{(e)}$
Am Assay1 ^(f)	1069.5 μg Am/g PuO_2	$\pm 2.8 \mu\text{g}^{(g)}$
Am Assay2 ^(h)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(i)}$
Am in WRM ^(j)	0.04516 g	$\pm 0.00021 \text{ g}^{(k)}$
Isotopic Weight Fraction		
^{238}Pu	0.000143	$\pm 0.000018^{(l)}$
^{239}Pu	0.937811	$\pm 0.000028^{(l)}$
^{240}Pu	0.059448	$\pm 0.000010^{(l)}$
^{241}Pu	0.002038	$\pm 0.000019^{(l)}$
^{242}Pu	0.000560	$\pm 0.000003^{(l)}$
Total α Activity in WRM	333.45 mCi	$\pm 0.75 \text{ mCi}^{(m)}$
	$1.23\text{E}+10 \text{ Bq}$	$\pm 2.78\text{E}+07 \text{ Bq}^{(m)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Pu Assay	Controlled Potential Coulometry	NBL CRM 126
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
Pu Isotopic	Total Evaporation Mass Spec.	NBS SRM 948
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Amount of AmO_2 weighed for the WRM (does not contain the amount of Am from the decay of ^{241}Pu in the PuO_2).
- (e) Above weighing and assay terms and correction for transfer loss
- (f) The amount of Am contained within the PuO_2 from grow-in.
- (g) Long term precision, weighing precision and bias
- (h) The amount of Am contained within the AmO_2
- (i) Long term precision, weighing precision and bias
- (j) Total amount of Am in the WRM from both the AmO_2 and the PuO_2
- (k) Above weighing terms, Am assay terms, and corrections for transfer loss
- (l) Long term precision and bias
- (m) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP004

PuO_2 - AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity plutonium dioxide and americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Pu and Am content, Pu isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
PuO_2 in WRM	2.28761 g	$\pm 0.00232 \text{ g}^{(a)}$
Pu Assay	0.87804 g Pu/g PuO_2	$\pm 0.00036 \text{ g}^{(b)}$
Pu in WRM	2.00861 g	$\pm 0.00204 \text{ g}^{(c)}$
AmO_2 in WRM ^(d)	0.06680 g	$\pm 0.00027 \text{ g}^{(e)}$
Am Assay1 ^(f)	1069.5 μg Am/g PuO_2	$\pm 2.8 \mu\text{g}^{(g)}$
Am Assay2 ^(h)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(i)}$
Am in WRM ^(j)	0.05474 g	$\pm 0.00022 \text{ g}^{(k)}$
Isotopic Weight Fraction		
^{238}Pu	0.000143	$\pm 0.000018^{(l)}$
^{239}Pu	0.937811	$\pm 0.000028^{(l)}$
^{240}Pu	0.059448	$\pm 0.000010^{(l)}$
^{241}Pu	0.002038	$\pm 0.000019^{(l)}$
^{242}Pu	0.000560	$\pm 0.000003^{(l)}$
Total α Activity in WRM	336.98 mCi	$\pm 0.76 \text{ mCi}^{(m)}$
	$1.25\text{E}+10 \text{ Bq}$	$\pm 2.80\text{E}+07 \text{ Bq}^{(m)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Pu Assay	Controlled Potential Coulometry	NBL CRM 126
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
Pu Isotopic	Total Evaporation Mass Spec.	NBS SRM 948
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Amount of AmO_2 weighed for the WRM (does not contain the amount of Am from the decay of ^{241}Pu in the PuO_2).
- (e) Above weighing and assay terms and correction for transfer loss
- (f) The amount of Am contained within the PuO_2 from grow-in.
- (g) Long term precision, weighing precision and bias
- (h) The amount of Am contained within the AmO_2
- (i) Long term precision, weighing precision and bias
- (j) Total amount of Am in the WRM from both the AmO_2 and the PuO_2
- (k) Above weighing terms, Am assay terms, and corrections for transfer loss
- (l) Long term precision and bias
- (m) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP005

PuO_2 - AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity plutonium dioxide and americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Pu and Am content, Pu isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
PuO_2 in WRM	2.26968 g	$\pm 0.00231 \text{ g}^{(a)}$
Pu Assay	0.87804 g Pu/g PuO_2	$\pm 0.00036 \text{ g}^{(b)}$
Pu in WRM	1.99287 g	$\pm 0.00203 \text{ g}^{(c)}$
AmO_2 in WRM ^(d)	0.11446 g	$\pm 0.00046 \text{ g}^{(e)}$
Am Assay1 ^(f)	1069.5 μg Am/g PuO_2	$\pm 2.8 \mu\text{g}^{(g)}$
Am Assay2 ^(h)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(i)}$
Am in WRM ^(j)	0.09224 g	$\pm 0.00037 \text{ g}^{(k)}$
Isotopic Weight Fraction		
^{238}Pu	0.000143	$\pm 0.000018^{(l)}$
^{239}Pu	0.937811	$\pm 0.000028^{(l)}$
^{240}Pu	0.059448	$\pm 0.000010^{(l)}$
^{241}Pu	0.002038	$\pm 0.000019^{(l)}$
^{242}Pu	0.000560	$\pm 0.000003^{(l)}$
Total α Activity in WRM	464.61 mCi	$\pm 1.26 \text{ mCi}^{(m)}$
	1.72E+10 Bq	$\pm 4.67\text{E}+07 \text{ Bq}^{(m)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Pu Assay	Controlled Potential Coulometry	NBL CRM 126
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
Pu Isotopic	Total Evaporation Mass Spec.	NBS SRM 948
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Amount of AmO_2 weighed for the WRM (does not contain the amount of Am from the decay of ^{241}Pu in the PuO_2).
- (e) Above weighing and assay terms and correction for transfer loss
- (f) The amount of Am contained within the PuO_2 from grow-in.
- (g) Long term precision, weighing precision and bias
- (h) The amount of Am contained within the AmO_2
- (i) Long term precision, weighing precision and bias
- (j) Total amount of Am in the WRM from both the AmO_2 and the PuO_2
- (k) Above weighing terms, Am assay terms, and corrections for transfer loss
- (l) Long term precision and bias
- (m) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP006

PuO_2 - AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity plutonium dioxide and americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Pu and Am content, Pu isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
PuO_2 in WRM	2.26754 g	$\pm 0.00229 \text{ g}^{(a)}$
Pu Assay	0.87804 g Pu/g PuO_2	$\pm 0.00036 \text{ g}^{(b)}$
Pu in WRM	1.99099 g	$\pm 0.00201 \text{ g}^{(c)}$
AmO_2 in WRM ^(d)	0.11573 g	$\pm 0.00046 \text{ g}^{(e)}$
Am Assay1 ^(f)	1069.5 $\mu\text{g Am/g PuO}_2$	$\pm 2.8 \mu\text{g}^{(g)}$
Am Assay2 ^(h)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(i)}$
Am in WRM ^(j)	0.09324 g	$\pm 0.00037 \text{ g}^{(k)}$
Isotopic Weight Fraction		
^{238}Pu	0.000143	$\pm 0.000018^{(l)}$
^{239}Pu	0.937811	$\pm 0.000028^{(l)}$
^{240}Pu	0.059448	$\pm 0.000010^{(l)}$
^{241}Pu	0.002038	$\pm 0.000019^{(l)}$
^{242}Pu	0.000560	$\pm 0.000003^{(l)}$
Total α Activity in WRM	467.88 mCi	$\pm 1.28 \text{ mCi}^{(m)}$
	$1.73\text{E}+10 \text{ Bq}$	$\pm 4.72\text{E}+07 \text{ Bq}^{(m)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Pu Assay	Controlled Potential Coulometry	NBL CRM 126
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
Pu Isotopic	Total Evaporation Mass Spec.	NBS SRM 948
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Amount of AmO_2 weighed for the WRM (does not contain the amount of Am from the decay of ^{241}Pu in the PuO_2).
- (e) Above weighing and assay terms and correction for transfer loss
- (f) The amount of Am contained within the PuO_2 from grow-in.
- (g) Long term precision, weighing precision and bias
- (h) The amount of Am contained within the AmO_2
- (i) Long term precision, weighing precision and bias
- (j) Total amount of Am in the WRM from both the AmO_2 and the PuO_2
- (k) Above weighing terms, Am assay terms, and corrections for transfer loss
- (l) Long term precision and bias
- (m) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP007

PuO_2 - AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity plutonium dioxide and americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Pu and Am content, Pu isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

Component	Quantity or Value	95 % CI
PuO_2 in WRM	0.45038 g	$\pm 0.00047 \text{ g}^{(a)}$
Pu Assay	0.87804 g Pu/g PuO_2	$\pm 0.00036 \text{ g}^{(b)}$
Pu in WRM	0.39545 g	$\pm 0.00041 \text{ g}^{(c)}$
AmO_2 in WRM ^(d)	0.06903 g	$\pm 0.00028 \text{ g}^{(e)}$
Am Assay1 ^(f)	1069.5 μg Am/g PuO_2	$\pm 2.8 \mu\text{g}^{(g)}$
Am Assay2 ^(h)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(i)}$
Am in WRM ^(j)	0.05477 g	$\pm 0.00022 \text{ g}^{(k)}$
Isotopic Weight Fraction		
^{238}Pu	0.000143	$\pm 0.000018^{(l)}$
^{239}Pu	0.937811	$\pm 0.000028^{(l)}$
^{240}Pu	0.059448	$\pm 0.000010^{(l)}$
^{241}Pu	0.002038	$\pm 0.000019^{(l)}$
^{242}Pu	0.000560	$\pm 0.000003^{(l)}$
Total α Activity in WRM	217.42 mCi	$\pm 0.76 \text{ mCi}^{(m)}$
	8.04E+9 Bq	$\pm 2.81\text{E}+07 \text{ Bq}^{(m)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Pu Assay	Controlled Potential Coulometry	NBL CRM 126
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
Pu Isotopic	Total Evaporation Mass Spec.	NBS SRM 948
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Amount of AmO₂ weighed for the WRM (does not contain the amount of Am from the decay of ²⁴¹Pu in the PuO₂).
- (e) Above weighing and assay terms and correction for transfer loss
- (f) The amount of Am contained within the PuO₂ from grow-in.
- (g) Long term precision, weighing precision and bias
- (h) The amount of Am contained within the AmO₂
- (i) Long term precision, weighing precision and bias
- (j) Total amount of Am in the WRM from both the AmO₂ and the PuO₂
- (k) Above weighing terms, Am assay terms, and corrections for transfer loss
- (l) Long term precision and bias
- (m) Based on propagated uncertainties on the Pu mass, ²⁴¹Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP008

PuO_2 - AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity plutonium dioxide and americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Pu and Am content, Pu isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
PuO_2 in WRM	0.47074 g	$\pm 0.00049 \text{ g}^{(a)}$
Pu Assay	0.87804 g Pu/g PuO_2	$\pm 0.00036 \text{ g}^{(b)}$
Pu in WRM	0.41333 g	$\pm 0.00043 \text{ g}^{(c)}$
AmO_2 in WRM ^(d)	0.06566 g	$\pm 0.00027 \text{ g}^{(e)}$
Am Assay1 ^(f)	1069.5 μg Am/g PuO_2	$\pm 2.8 \mu\text{g}^{(g)}$
Am Assay2 ^(h)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(i)}$
Am in WRM ^(j)	0.05213 g	$\pm 0.00021 \text{ g}^{(k)}$
Isotopic Weight Fraction		
^{238}Pu	0.000143	$\pm 0.000018^{(l)}$
^{239}Pu	0.937811	$\pm 0.000028^{(l)}$
^{240}Pu	0.059448	$\pm 0.000010^{(l)}$
^{241}Pu	0.002038	$\pm 0.000019^{(l)}$
^{242}Pu	0.000560	$\pm 0.000003^{(l)}$
Total α Activity in WRM	209.70 mCi	$\pm 0.73 \text{ mCi}^{(m)}$
	7.76E+9 Bq	$\pm 2.68\text{E}+07 \text{ Bq}^{(m)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Pu Assay	Controlled Potential Coulometry	NBL CRM 126
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
Pu Isotopic	Total Evaporation Mass Spec.	NBS SRM 948
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Amount of AmO_2 weighed for the WRM (does not contain the amount of Am from the decay of ^{241}Pu in the PuO_2).
- (e) Above weighing and assay terms and correction for transfer loss
- (f) The amount of Am contained within the PuO_2 from grow-in.
- (g) Long term precision, weighing precision and bias
- (h) The amount of Am contained within the AmO_2
- (i) Long term precision, weighing precision and bias
- (j) Total amount of Am in the WRM from both the AmO_2 and the PuO_2
- (k) Above weighing terms, Am assay terms, and corrections for transfer loss
- (l) Long term precision and bias
- (m) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP009

AmO_2 -Diatomaceous Earth

This Working Reference Material (WRM) contains high purity americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Am content, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
AmO_2 in WRM ^(a)	0.13133 g	$\pm 0.00053 \text{ g}^{(b)}$
Am Assay ^(c)	0.78727 g Am/g AmO_2	$\pm 0.00352 \text{ g}^{(d)}$
Am in WRM ^(e)	0.10340 g	$\pm 0.00042 \text{ g}^{(f)}$
Total α Activity in WRM	355.08 mCi	$\pm 1.43 \text{ mCi}^{(g)}$
	$1.31\text{E}+10 \text{ Bq}$	$\pm 5.28\text{E}+07 \text{ Bq}^{(g)}$

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
α Activity	Calculated from Am mass and isotope α specific activity	

$T_{1/2}$ value is from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic mass used is from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Amount of AmO_2 weighed for the WRM; Balance precision, buoyancy, standard weight sets and correction for transfer loss
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Long term precision and bias
- (e) Total amount of Am in the WRM from the AmO_2
- (f) Above weighing terms, Am assay terms, and corrections for transfer loss
- (g) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP010

AmO₂-Diatomaceous Earth

This Working Reference Material (WRM) contains high purity americium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the Am content, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
AmO ₂ in WRM ^(a)	0.12674 g	±0.00051 g ^(b)
Am Assay ^(c)	0.78727 g Am/g AmO ₂	±0.00352 g ^(d)
Am in WRM ^(e)	0.099775 g	±0.00040 g ^(f)
Total α Activity in WRM	342.65 mCi	±1.38 mCi ^(g)
	1.27E+10 Bq	±5.10E+07 Bq ^(g)

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
Am Assay	Isotope Dilution Mass Spec.	NIST SRM 4322B
α Activity	Calculated from Am mass and isotope α specific activity	

$T_{1/2}$ value is from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic mass used is from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Amount of AmO_2 weighed for the WRM; Balance precision, buoyancy, standard weight sets and correction for transfer loss
- (b) Long term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Long term precision and bias
- (e) Total amount of Am in the WRM from the AmO_2
- (f) Above weighing terms, Am assay terms, and corrections for transfer loss
- (g) Based on propagated uncertainties on the Pu mass, ^{241}Am mass and Pu isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP011

UO₂-Diatomaceous Earth

This Working Reference Material (WRM) contains high purity uranium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the U content, U isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

Component	Quantity or Value	95 % CI
UO ₂ in WRM	570.58 g	±0.64 g ^(a)
U Assay	0.87493 g Pu/g PuO ₂	±0.00012 g ^(b)
U in WRM	499.22 g	±0.56 g ^(c)
Isotopic Weight Fraction		
²³³ U	0.000000	±0.000000 ^(d)
²³⁴ U	0.000080	±0.000005 ^(d)
²³⁵ U	0.001902	±0.000004 ^(d)
²³⁶ U	0.000034	±0.000004 ^(d)
²³⁸ U	0.998057	±0.000007 ^(d)
Total α Activity in WRM	17.65 mCi	±0.05 mCi ^(e)
	6.530E+08 Bq	±1.803E+06 Bq ^(e)

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
U Assay	Isotope Dilution Mass Spec.	NBS U005
U Isotopic	Total Evaporation Mass Spec.	NBS U005
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Short term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Short term precision and bias
- (e) Based on propagated uncertainties on the U mass and U isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP012

UO₂-Diatomaceous Earth

This Working Reference Material (WRM) contains high purity uranium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the U content, U isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
UO ₂ in WRM	1010.30 g	±0.61 g ^(a)
U Assay	0.87493 g Pu/g PuO ₂	±0.00012 g ^(b)
U in WRM	883.94 g	±0.55 g ^(c)
Isotopic Weight Fraction		
²³³ U	0.000000	±0.000000 ^(d)
²³⁴ U	0.000080	±0.000005 ^(d)
²³⁵ U	0.001902	±0.000004 ^(d)
²³⁶ U	0.000034	±0.000004 ^(d)
²³⁸ U	0.998057	±0.000007 ^(d)
Total α Activity in WRM	31.25 mCi	±0.08 mCi ^(e)
	1.156E+10 Bq	±2.998E+06 Bq ^(e)

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
U Assay	Isotope Dilution Mass Spec.	NBS U005
U Isotopic	Total Evaporation Mass Spec.	NBS U005
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Short term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Short term precision and bias
- (e) Based on propagated uncertainties on the U mass and U isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP013

UO₂-Diatomaceous Earth

This Working Reference Material (WRM) contains high purity uranium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the U content, U isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
UO ₂ in WRM	1094.49 g	±1.10 g ^(a)
U Assay	0.87493 g Pu/g PuO ₂	±0.00012 g ^(b)
U in WRM	957.60 g	±0.96 g ^(c)
Isotopic Weight Fraction		
²³³ U	0.000000	±0.000000 ^(d)
²³⁴ U	0.000080	±0.000005 ^(d)
²³⁵ U	0.001902	±0.000004 ^(d)
²³⁶ U	0.000034	±0.000004 ^(d)
²³⁸ U	0.998057	±0.000007 ^(d)
Total α Activity in WRM	33.85 mCi	±0.09 mCi ^(e)
	1.253E+09 Bq	±3.399E+06 Bq ^(e)

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
U Assay	Isotope Dilution Mass Spec.	NBS U005
U Isotopic	Total Evaporation Mass Spec.	NBS U005
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Short term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Short term precision and bias
- (e) Based on propagated uncertainties on the U mass and U isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP014

UO₂-Diatomaceous Earth

This Working Reference Material (WRM) contains high purity uranium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the U content, U isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
UO ₂ in WRM	1096.66 g	±1.10 g ^(a)
U Assay	0.87493 g Pu/g PuO ₂	±0.00012 g ^(b)
U in WRM	959.50 g	±0.96 g ^(c)
Isotopic Weight Fraction		
²³³ U	0.000000	±0.000000 ^(d)
²³⁴ U	0.000080	±0.000005 ^(d)
²³⁵ U	0.001902	±0.000004 ^(d)
²³⁶ U	0.000034	±0.000004 ^(d)
²³⁸ U	0.998057	±0.000007 ^(d)
Total α Activity in WRM	33.92 mCi	±0.09 mCi ^(e)
	1.255E+09 Bq	±3.406E+06 Bq ^(e)

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
U Assay	Isotope Dilution Mass Spec.	NBS U005
U Isotopic	Total Evaporation Mass Spec.	NBS U005
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Short term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Short term precision and bias
- (e) Based on propagated uncertainties on the U mass and U isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP015

UO₂-Diatomaceous Earth

This Working Reference Material (WRM) contains high purity uranium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the U content, U isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
UO ₂ in WRM	1111.68 g	±1.12 g ^(a)
U Assay	0.87493 g Pu/g PuO ₂	±0.00012 g ^(b)
U in WRM	972.64 g	±0.98 g ^(c)
Isotopic Weight Fraction		
²³³ U	0.000000	±0.000000 ^(d)
²³⁴ U	0.000080	±0.000005 ^(d)
²³⁵ U	0.001902	±0.000004 ^(d)
²³⁶ U	0.000034	±0.000004 ^(d)
²³⁸ U	0.998057	±0.000007 ^(d)
Total α Activity in WRM	34.39 mCi	±0.09 mCi ^(e)
	1.272E+09 Bq	±3.452E+06 Bq ^(e)

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
U Assay	Isotope Dilution Mass Spec.	NBS U005
U Isotopic	Total Evaporation Mass Spec.	NBS U005
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Short term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Short term precision and bias
- (e) Based on propagated uncertainties on the U mass and U isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.

Los Alamos National Laboratory

Certificate of Content and Traceability

Department of Energy/Idaho National Engineering and Environmental Laboratory
Working Reference Material # CEP016

UO₂-Diatomaceous Earth

This Working Reference Material (WRM) contains high purity uranium dioxide dispersed in diatomaceous earth. Quantitative information and uncertainties on the nuclear content of this WRM are listed below. Complete information regarding the U content, U isotopic ratios, chemical composition, elemental impurities, containment, and WRM fabrication procedure is described in the NDA CEP WRM Production Plan, LACP-98-155, and in files retained at the Idaho National Engineering and Environmental Laboratory by the Lockheed Martin Idaho Technology Co. Transuranic Waste Department and at the Los Alamos National Laboratory Analytical Chemistry Group.

NUCLEAR MATERIAL CONTENT

The nuclear material content and total alpha activity for this WRM are listed below. Also listed are overall uncertainty estimates at 95% confidence intervals (CI) for each component.

<u>Component</u>	<u>Quantity or Value</u>	<u>95 % CI</u>
UO ₂ in WRM	1084.27 g	±1.09 g ^(a)
U Assay	0.87493 g Pu/g PuO ₂	±0.00012 g ^(b)
U in WRM	948.66 g	±0.95 g ^(c)
Isotopic Weight Fraction		
²³³ U	0.000000	±0.000000 ^(d)
²³⁴ U	0.000080	±0.000005 ^(d)
²³⁵ U	0.001902	±0.000004 ^(d)
²³⁶ U	0.000034	±0.000004 ^(d)
²³⁸ U	0.998057	±0.000007 ^(d)
Total α Activity in WRM	33.54 mCi	±0.09 mCi ^(e)
	1.241E+09 Bq	±3.366E+06 Bq ^(e)

All values decay corrected to 6/1/97.

Bill J. McKerley, Group Leader
Analytical Chemistry, NMT-1

Robert S. Marshall, Project Manager
NDA CEP WRM Production

MEASUREMENT METHOD AND TRACEABILITY

The nuclear contents of this WRM were characterized and qualified using the following methods with traceability to the National Institute of Standards and Technology (formerly NBS) or New Brunswick Laboratory (NBL) standards.

<u>Measurement</u>	<u>Measurement Method</u>	<u>Reference Material</u>
Weighing	5 place analytical balance	NIST traceable weights
U Assay	Isotope Dilution Mass Spec.	NBS U005
U Isotopic	Total Evaporation Mass Spec.	NBS U005
α Activity	Calculated from Pu and Am mass and isotope α specific activities.	

$T_{1/2}$ values are from: Firestone, Table of Isotopes, Vol. II, 8th ed., John Wiley & Sons, New York, (1996). Atomic masses used are from: Audi & Wapstra, The 1995 Update to the Atomic Mass Evaluation, Nuclear Physics, A595, p.409, (1995).

NOTES

Random and systematic error terms were combined and reported as 95% confidence intervals. Error terms and other notes are listed below:

- (a) Balance precision, bouyancy, standard weight sets
- (b) Short term precision, weighing precision and bias
- (c) Above weighing and assay terms and correction for transfer loss
- (d) Short term precision and bias
- (e) Based on propagated uncertainties on the U mass and U isotopic uncertainties listed above. It is recommended that all facilities participating in the NDA CEP program use the half-lives listed above to preclude facility to facility error terms introduced by using different half-life values.