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Ages, Reactor Modelling, and Sources of Material: Further Discussion
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High- Precision Plutonium Isotopic Compositions Measured on Los Alamos National Laboratory's General's Tanks Samples: Bearing on Model Ages, Reactor Modelling, and Sources of Material: Further Discussion of Chronometry

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Abstract: In this study, we re-analyzed late-1940's, Manhattan Project era Plutonium-rich sludge samples recovered from the "General's Tanks" located within the nation's oldest Plutonium processing facility, Technical Area 21. These samples were initially characterized by lower accuracy, and lower precision mass spectrometric techniques. We report here information that was previously not discernable: the two tanks contain isotopically distinct Pu not only for the major (i.e., ^{240}Pu, ^{239}Pu) but trace (^{238}Pu,^{241}Pu, ^{242}Pu) isotopes. Revised isotopics slightly changed the calculated ^{241}Am-^{241}Pu model ages and interpretations.

Background. The two "General's Tanks" found at the Los Alamos National Laboratory (LANL) Technical Area 21 (TA-21) are boxcar-size steel storage tanks, buried underground, that were used from approximately 1945-47 (Emelity, 1996) to store Pu laden process materials for possible later recovery. Both of these tanks were in operation during the very early post-WWII period. We have not researched which reactors were used to produce the Pu found in the tanks under the scope of this study, but this information may be available. We chose this location for study because TA-21 is one of the nation's earliest Plutonium chemistry research areas and thus an excellent "test bed" for making forensics measurements on site debris (Spencer and Roback, 2013). The entire TA-21 site has been largely remediated and most site material has been removed, thus these tanks provided one of the few key pieces of onsite physical evidence with which one could identify the purpose, timing, or scope of this Manhattan-Project era facility.

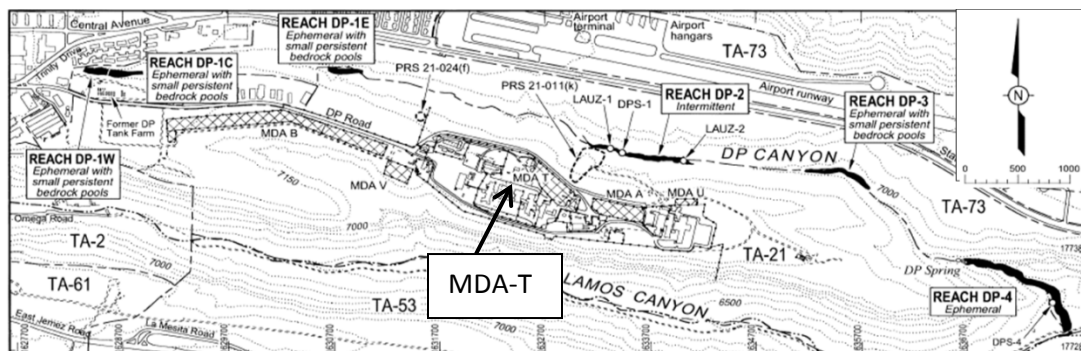Figure 1, TA-21 site in Los Alamos. The location for the General's Tanks was within MDA-T, directly above the box label.

TA-21's origins were during the wartime Manhattan Project as a means to relocate the most hazardous Pu facilities to a purpose-built facility away from the center of the Los Alamos and to decommission the highly hazardous earlier laboratory locations which were constructed in due haste due to the

importance of concluding the mission. The distinct complex of laboratory and support facilities at TA-21 was the locus of research and development related to plutonium and uranium processing. Such efforts were central to the processing of Pu into weapons material in our earliest postwar nuclear devices.

General's Tanks (GT) Detailed Description

The General's Tanks, known as the East and West tanks, are each 50,000 gallon steel tanks that were used for the storage of low level retrievable waste streams derived from plutonium reprocessing and recovery operation carried out at LANL from 1945 to 1947. The tanks received basic supernatant solutions from which no further plutonium recovery was practical, but which contained levels deemed too large to discard at that time. These residual solutions were reported to contain approximately 1 mg/L (10^{-3} g/L) of plutonium (LASL, 1947). The final recovery processes for the bulk (over 90%) of solutions were generated by one of 2 processes. The first process involved solutions that were treated with hydrogen peroxide and sodium hydroxide that resulted in solutions sent to the west tank that contained little or no calcium and magnesium. The second involved a final treatment with ammonium nitrate and ammonium hydroxide to precipitate an ammonium hydroxide precipitate as a plutonium carrier and was sent to the east tank. The resulting supernatant solutions were high in calcium and magnesium.

As the availability of plutonium increased in the following years the recovery of plutonium from the General's Tanks was deemed uneconomical. The tanks were sealed and buried in place. In 1974, the tanks were partially excavated; the soil and concrete were removed from the top of the tanks and holes about 16-inch diameter were cut in the top of each tank. It was determined that an estimated 40,000 gallons of liquid and solid material was held in the west tank from the sodium hydroxide process and 9,000 gallons was in the east tank from the ammonium hydroxide process (Rogers, 1977). It was also recognized that the wastes in the tanks had separated and that solid sludge material had collected in the bottoms of the tanks. The sludge was overlain by a layer of supernatant water that comprised the bulk of the mass in the tanks. The holes in the tanks remained open for 4 or 5 years, and the water in the west tank was pumped periodically as rainwater collected in the tank. The tanks were sealed and buried in 1985 to prevent further rainwater infiltration (Emelity, 1996).

Thus, this is a classic location where we could test our ability to retrospectively determine the plutonium isotopics and parent-progeny relationships with state of the art methods to identify this unique material against well-established historic records. Uranium-plutonium parent-daughter isotopic pairs or the [241]Am-[241]Pu pair can potentially be used to determine the age of these materials since last chemical separation and thus back-calculate initial Pu isotopic compositions. But a limit of our earlier work was that the [241]Pu isotope was near, and [242]Pu was below our method detection limits. Thus, there were large uncertainties on [241]Pu, limiting our ability to reactor-model these data or date samples using the [241]Am-[241]Pu chronometer, which we hypothesized to be very resistant to open system behavior in this environment (Paton et al, 2004). To further refine our predictive reactor modelling capability and minimize age-related uncertainties, we have re-measured the plutonium isotopic compositions in the General's Tanks to more accurately characterize the minor isotopes [238]Pu (used in reactor modelling calculations), [241]Pu (bearing on [241]Pu-[241]Am ages) and [242]Pu (bearing on reactor modelling). To provide

better confidence in the ^{238}Pu measurement, which is significantly, impacted by abundance sensitivity and ^{238}U interference issues when using TIMS, we also measured ^{238}Pu activity using alpha spectrometry.

Procedure: Fresh splits were obtained from previously digested large volume samples that were processed and archived from an earlier study (Spencer and Roback,2013), thus enabling us to recalculate earlier Pu isotopic compositions and ages without re-analyzing daughter elements. Splits were dried, converted to the nitrate form, and Pu was separated on anion exchange columns using Lewatit anion resin and high purity acids. Am and major matrix constituents including Fe and alkali metals were eluted with 7M HNO_3. Residual Am and matrix constituents were eluted, and U retained on the column by switching from 7M HNO_3 to a short wash of 10M HCl. Pu was then reduced and eluted from the columns in 10M HCl:0.2M HI. Initial separations were done on 7/8/2014. Due to residual U and Fe being present on all samples (high ^{238}Pu due to a ^{238}U interference was measured on the mass spectrometer), this process was then repeated (7/23/2014) to further purify Pu and re-measure to obtain optimal ^{238}Pu and ^{241}Pu isotopic compositions. To provide quality control, two splits of a similar standard, NIST traceable, Certified Reference Material CRM-126 (nominally 2% ^{240}Pu) and a blank were processed alongside the samples and analyzed. Although CRM126a was not specifically co-analyzed, it is routinely analyzed by our laboratory. Results for this standard are included as well.

Measurements were made on an IsotopX IsoprobeT mass spectrometer equipped with a Daly detector located behind a WARP energy filter to remove peak tailing from the large ^{239}Pu beam and minimize other low energy ions. Ion beams of approximately 1.0-1.2E^6 counts per second on the major isotope (^{239}Pu) were obtained in order to obtain adequate count rates on minor isotopes while not overloading and tripping the Daly detector. A peak hopping routine was used that obtained baselines at the beginning of ten ratio blocks. The peak sequence was 238-239-240-241-242-244. Separate backgrounds were taken at half masses on the side of each peak with longer counting times for the minor 238-241-242-244 isotopes. Nine analyses by ion counting and eight by the total evaporation technique (Callis and Abernathy, 1991) were made on CRM126 in order to estimate uncertainties, including instrumental fractionation corrections, using the ^{240}Pu/^{239}Pu on a quasi- "known" of similar isotopic composition, although we note that the minor isotopes on CRM126, including ^{240}Pu, have not been certified but were reported for information only as "nominal" concentrations.

To correct for instrumental fractionation of the minor isotopes in the unknowns, each of the unknowns was analyzed by total evaporation on a VG354 mass spectrometer. The CRM126 measured by total evaporation was compared to the certificate value (not a certified value) to make a minor correction for bias for the unknowns. The ion counting values in the unknowns were then corrected for instrumental fractionation by comparing the ^{240}Pu/^{239}Pu measured on the samples via ion counting to the bias corrected total evaporation value and these fractionation corrections applied to the minor isotope ratios.

We observed that in some analyses, there were negative values of ^{244}Pu/^{239}Pu, possibly due to irregularities in baseline measurements at high masses. We found that by correcting the ^{244}Pu/^{239}Pu to zero and applying this correction to ^{242}Pu/^{239}Pu, we reduced the error and raised the ratio slightly and with closer agreement to the nominal values on the certificate. Subsequent to the measurements made

in this study we replaced the amplifier cards on the Faraday cups and analyzed two CRM126 standards using Total Evaporation Analysis with the ^{242}Pu and ^{241}Pu measured on 10^{12} resistors. These results were in agreement with the small correction and when this "negative count" correction was applied to ^{241}Pu/^{239}Pu, brought the Faraday vs. Daly measurements for both ^{241}Pu and ^{242}Pu into closer agreement. It is these data we report here. We note that the best way to decide on a final, high confidence set of ratios would be by a round robin using several independent laboratories.

Because of a discrepancy between the certificate and measured value of ^{238}Pu/^{239}Pu on CRM126, the samples and standard were analyzed for ^{238}Pu independently by alpha counting in order to obtain a high confidence measurement by an independent technique (Bortels and Callaers, 1987; Bortels et al, et al, 1995) not constrained by the TIMS problems of abundance sensitivity or isobaric interferences. Those results are shown in Table 2. Table 1 shows the certificate value (corrected to the time of the analyses) and uncertainty in parentheses; the TIMS and alpha counting results are in agreement. The expanded uncertainties reported in this report for all the samples and the standards are ISO GUM compliant at coverage factor, of k=2. Note, however, that the certified values for the minor Pu isotopes were not certified but reported as "nominal" value on the certificate with no reported uncertainties. The measurements on that standard were followed by communication with NBL, we were lead to believe that these results are unprecedented in terms of accuracy, precision and uncertainty for the minor isotopes on this standard (Richard Essex, New Brunswick Laboratory, personal communication).

Blank corrections were not necessary for isotopic compositions because the sample: process blank ratio was in all cases ~100,000 and of a similar isotopic composition as the unknowns. A total process blank measured with these samples resulted in a blank of 100 picograms with an isotopic composition almost entirely of ^{239}Pu.

Discussion

The primary separations were sufficient to obtain good results on the pure CRM126 standard and in addition, good analyses results were obtained for the ^{241}Pu/^{239}Pu and ^{242}Pu/^{239}Pu ratios on the unknown tank samples. The ^{238}Pu data measured on re-separated tank samples produced consistent results with lower standard deviations and no signs of an isobaric interference from ^{238}U. Minor isotope ratios were extremely low in the tank samples as expected, but the Isoprobe T was capable of measuring background corrected count rates of 1- 10 cps, therefore meaningful results were obtained on all of the minor isotopes.

One result that surprised us was the CRM126 ^{238}Pu/^{239}Pu was approximately 50% higher than the age-corrected value on the Certificate of Analysis (Appendix 1). We note, however, that the minor Pu isotopes (including ^{240}Pu) were not certified due to their low abundances and listed on the 1986 certificate "for information purposes only". To further independently test the ^{238}Pu isotopic result, sample splits for two standards (CRM126 and 126a) and the unknown were analyzed by an advanced alpha counting peak deconvolution technique that eliminates a potential ^{241}Am interference; statistically identical results to our ^{238}Pu/^{239}Pu ratios were obtained. This comparison is shown on Table 2. In addition, we had analyzed CRM126 as an unknown in October 2013 by total evaporation analysis and

our calculations of the ^{234}U-^{238}Pu, ^{235}U-^{239}Pu, and ^{236}U-^{240}Pu are all concordant using the higher values. We request a re-valuation of the ^{238}Pu values for CRM126 by NBL and for subsequent internal quality control charts. Also, it is important to note that the certified ^{238}Pu concentration in CRM126a as determined by both alpha spectrometry and TIMS agreed with the certified values.

Table 1. TIMS results on Pu isotopics. For the CRM126 standard, the measured uncertainty for ^{238}Pu/^{239}Pu is in parentheses but the certificate does not report ratio uncertainties or any uncertainties on isotopes other than ^{239}Pu. All data were corrected to the date of the beginning of the measurement campaign, 15 July, 2014. Uncertainties calculated using GUM Workbench 2.4.1

Atom ratio	GT-W1 4948-1	GT-W2 4948-2	GT-E3 4942-3	GT-E4 4942-4	CRM-126 certificate values, age corrected (ratios are "nominal" on certificate)	CRM126 (as measured, ion counting)	CRM 126 by Total Evaporation (n=2, corrected to time of other analyses)
^{238}Pu/^{239}Pu	0.0000164	0.0000160	0.0000147	0.0000152	0.000013	0.0000191	.0000202
Uncertainty (k=2)	0.0000009	0.0000014	0.0000013	0.0000013		0.0000002	.0000003
^{240}Pu/^{239}Pu	0.016288	0.016259	0.015624	0.015694	0.020959	0.020959	.021037
Uncertainty (k=2)	.0000430	0.000043	0.000043	0.000043		0.000042	.000158
^{241}Pu/^{239}Pu	.0000167	0.0000167	0.0000155	0.0000152	0.0000039	0.000042	.000043
Uncertainty (k=2)	.0000005	0.0000002	0.0000004	0.0000002		0.0000003	.000001
^{242}Pu/^{239}Pu	0.0000045	0.0000044	0.0000039	0.0000042	0.000010	0.00000995	.0000096
Uncertainty (k=2)	0.0000004	0.0000003	0.0000003	0.0000004		0.0000002	.0000022

Table 2. TIMS vs. Alpha Counting Results on samples and CRM126, units are ppm of total Pu. (Overall Uncertainties calculated using GUM Workbench, version 2.4.1,coverage factor, k=2)

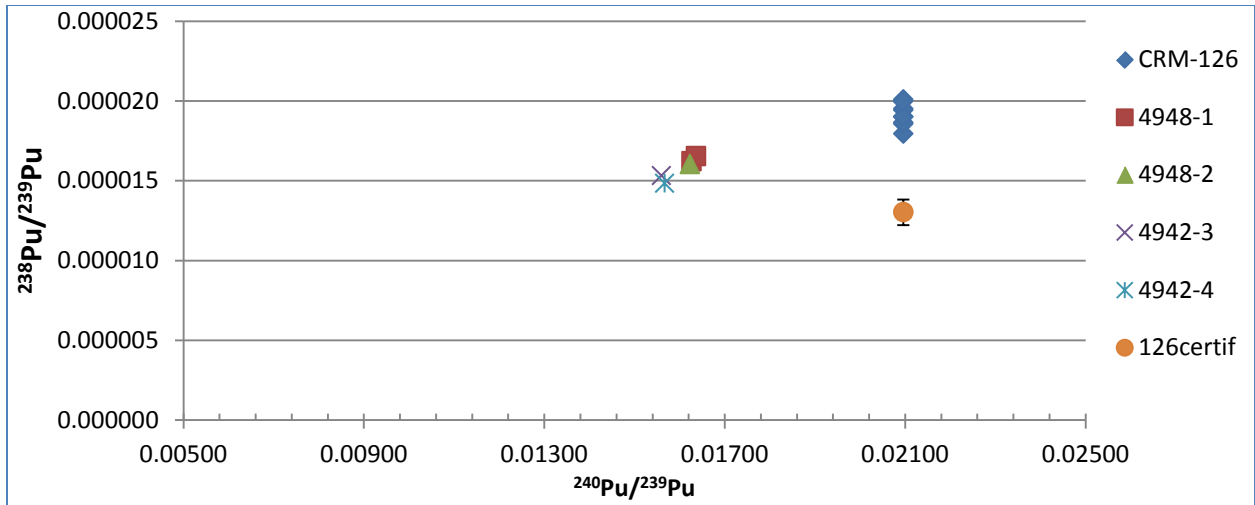| Sample	GT-W1 4948-1	GT-W2 4948-2	GT-E3 4942-3	GT-E4 4942-4	Measured CRM126	Measured CRM126a	**CRM126a certificate**
^{238}Pu TIMS	16.1 (0.3)	16.5 (0.3)	14.5 (0.5)	14.9 (0.2)	18.7 (0.2)	117(8)	112(0.3)
^{238}Pu alpha	16.8 (2.7)	16.8 (2.9)	15.8 (2.0)	16.0 (2.6)	20.0 (2.6)	121(16)	

Figure 1. ^{238}Pu/^{239}Pu vs. ^{240}Pu/^{239}Pu, including measurements on CRM126. Uncertainty limits are the size of the symbols.
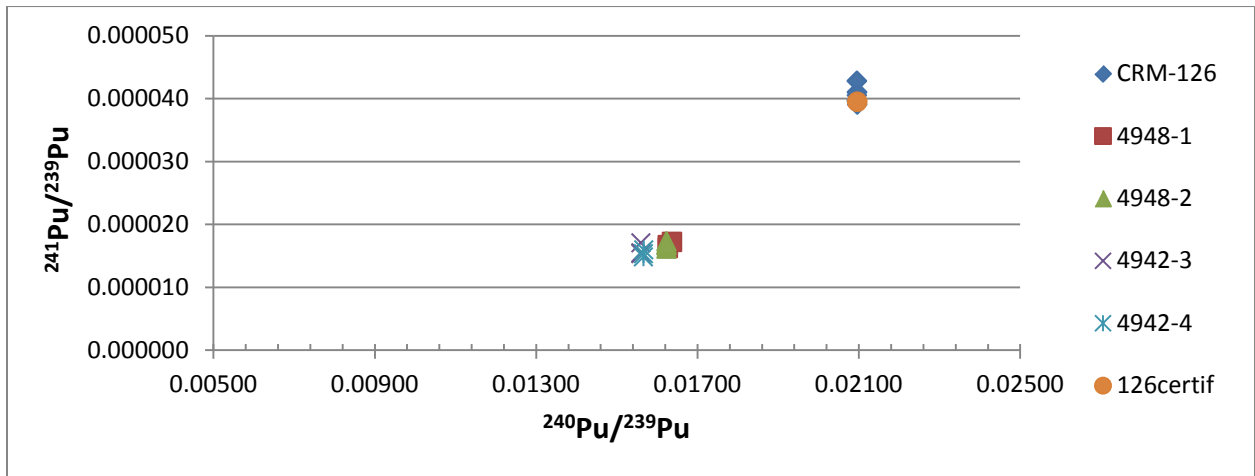

Figure 2. ^{241}Pu/^{239}Pu vs. ^{240}Pu/^{239}Pu, including measurements on CRM126
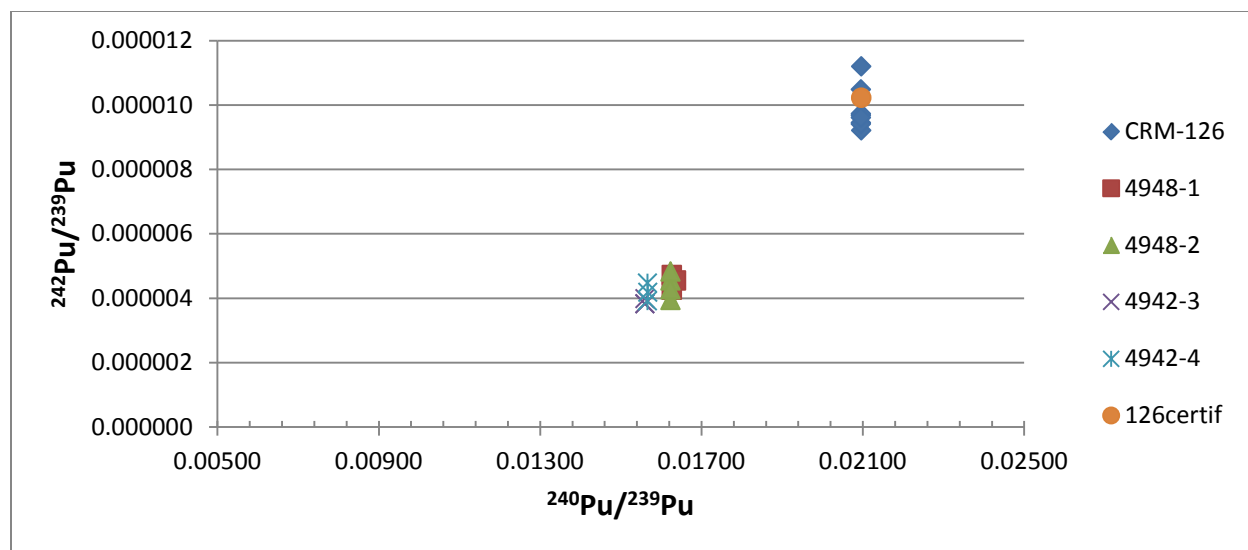
Figure 3. $^{242}Pu/^{239}Pu$ vs. $^{240}Pu/^{239}Pu$, including measurements on CRM126

Improved isotopic measurements and potential implications for forensic dating.

1. Minor and trace isotopic compositions
   The new measurements for $^{241}Pu/^{239}Pu$ were somewhat lower than those initially measured on a less advanced mass spectrometer (a Sector-54 without a WARP filter). In addition, the earlier measurements could not produce valid $^{238}Pu/^{239}Pu$ and $^{242}Pu/^{239}Pu$ as the measurements were below the minimum limits of detection of the Sector 54. Thus, these measurements on the General's Tank samples should be considered valuable since the values measured are at or above the quantitation limits for the minor isotopes. There also is a small but significant difference between the Pu isotopics in the two tanks best seen in the more precise $^{240}Pu/^{239}Pu$ measurements. We have not investigated the meaning of this difference.
   Our measurements of $^{238}Pu/^{239}Pu$ on CRM126 were lower than the age-corrected value from the certificate. We note, however, that none of the minor isotopes on the CRM 126 certificate were certified measurements. To our knowledge (Richard Essex, NBL, personal communication) no one else has measured this material by a similar high precision technique although our laboratory has measured CRM 126 by traditional total evaporation. The ^{238}Pu levels by the total evaporation technique had too much variation to use as a test, so we analyzed this material by an enhanced alpha counting technique, which resulted in statistically indistinguishable ^{238}Pu content to our measurements. We therefore consider both the CRM126 $^{238}Pu/^{239}Pu$ and our General's Tanks ratios to be reliable. Also the measured CRM 126a results for this isotope by both the techniques were in good agreement with the certified values.

2. Implications for model ages
   Our initial measurements on tank remains (Spencer and Roback, 2013) produced Pu-U model ages that were concordant but significantly younger than the known ages available from historical records. We hypothesized the Pu-U model ages were too young due to preferential

loss of relatively soluble U daughters when the tanks were pumped of their liquid contents in 1974. A rough calculation based on measured U activity in water removed from the tanks in 2009 (Spencer and Roback, in prep) indicates that sludge remains may have lost roughly 10-30% of its U to overlying water. In contrast to the Pu-U model ages, the ^{241}Pu-^{241}Am model ages were, within error, consistent with the historic record but with significant uncertainties, which more detailed uncertainty analysis (Gum Workbench 2.4.1.402, Metrodata) shows is largely due to the substantial uncertainties in the initial minor Pu isotope measurements and to some degree, the Pu assay. The general agreement between Pu-Am model ages and the historic record is consistent with the Pu and Am pair remained largely immobile in the residual sludge, i.e., the solid phases. The new data with slightly (and statistically significantly) lower ^{241}Pu/^{239}Pu , when used to recalculate ages, results in slightly older Pu-Am model ages (especially in the East tank) relative to the known ages and with smaller uncertainties; both original and revised model ages are shown in Table 3.

This model age discrepancy could result from several possible scenarios including preferential Pu loss relative to Am (not likely, given the extremely low concentrations of Pu measured in overlying water during the 2009 measurements (Spencer and Roback, in prep) or excess Am from original processing. Our preferred explanation is that there was excess initial Am in the tanks. The tanks were used to store effluent produced during Pu processing and purification. Newly produced Pu would have its maximum ^{241}Pu content, so initial purification processes to remove trace elements and daughter products would result in the maximum amount of ^{241}Am removed from a given amount of purified Pu. Thus, the tanks would be sensitive to high rates of Am production and disposal early in the material's history.

Table 3. Original and revised Pu-Am and Pu-U model ages. Tanks were filled from 1945-47 from historical records (Emility, 1996). Uncertainties from GUM (ver 2.4.1).

Sample	GT-W1	GT-W2	GT-E3	GT-E4
Original Pu-Am model age	1945 (7)	1947 (7)	1944 (7)	1944 (7)
Revised Pu-Am model ages	1943 (1)	1943(0.7)	1941.5(0.8)	1939.6(0.5)
Pu-U model ages	1955	1966	1962	1955

Acknowledgements

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New Brunswick Laboratory
Certified Reference Materials
Certificate of Analysis
CRM No. 126

Plutonium Metal
(Plutonium Assay and Isotopic Standard)
(In cooperation with the University of California
Los Alamos National Laboratory, Los Alamos, New Mexico)

Plutonium	. . . . 99.962 ± 0.018 Wt. %* (999.62 ± 0.18 g·kg^{-1})
Plutonium-239	97.925 ± 0.001 At. %*
Relative Atomic Weight.	. . 239.073*

*As of October 1, 1985. Refer to Table I for Semi-Annual Decay-Adjusted Values.

This Certified Reference Material (CRM) is an assay and isotopic standard primarily for use in the analysis of plutonium materials in process intermediate or finished product forms. Each unit of CRM No. 126 consists of a single piece of plutonium metal, approximately 1 gram, that has been sealed in a glass tube under a reduced-pressure argon atmosphere. NOTE: *the tube and its outer plastic containment should be handled under proper radiologically-controlled conditions at all times.*

The statistical uncertainty assigned to *each* certified value is the individual 95% confidence interval for the mean of the respective assay and isotopic measurements. The uncertainty assigned to the assay value is derived from random measurement variations, sample-to-sample variations, and the uncertainty associated with the correction factor measured using quality control standards of NBS SRM 949f. In addition to random measurement variations, the uncertainty assigned to the isotopic value includes the uncertainty on the mass discrimination correction extrapolated from NBL CRM No. 128.

Preparation and packaging of CRM No. 126 were carried out by the Los Alamos National Laboratory, under the direction of the CHM-1 Group headed by P. T. Cunningham. For certification, sample dissolutions and assay measurements were performed by C. G. Cacic, NBL; isotopic measurements were performed by D. W. Crawford

January 1, 1986
Argonne, Illinois

Carleton D. Bingham
Director

(Over)

and M. A. Legel, NBL. Characterization studies and impurity measurements were performed by CHM-1, Los Alamos. Additional impurity measurements were performed by J. A. Carter and associates, Oak Ridge National Laboratory. Technical assistance was provided by J. E. Rein and J. W. Dahlby, Los Alamos. Statistical assessment of the data for certification was performed by M. D. Soriano, NBL. Overall direction and coordination of the preparation, certification and issuance of the CRM were provided by N. M. Trahey, NBL.

The plutonium metal was prepared according to specifications, nibbled into single, 1-gram pieces, sealed in Pyrex glass tubes under argon atmosphere and stored in air. After one year, those tubes which did not exhibit oxide formation were judged to be completely sealed and were then shipped to NBL for packaging and certification. CRM units were selected according to a statistical sampling plan and cleaned by electrolytic cleaning or mechanical filing. The clean metal pieces were weighed, dissolved in acid media, subsampled and purified for plutonium assay and isotopic analyses. The plutonium content of each subsample was determined by the NBL controlled-potential coulometric method verified with NBS SRM 949f; plutonium isotopic composition for masses 238-242 was determined by thermal ionization mass spectrometry verified with NBL CRM No. 128.[1] Only the atom % ^{239}Pu and relative atomic weight are certified due to extremely low levels of ^{238}Pu and ^{242}Pu in CRM No. 126. Isotopic dilution mass spectrometry using NBS SRM 996 ^{244}Pu spike was used as an independent method verification of the CRM No. 126 plutonium assay. Metallic impurity content (excluding ^{241}Am, ^{237}Np, and uranium) was determined by emission spectrography and spectrophotometry and is estimated to be 78 μg/g metal. Uranium and ^{241}Am were determined using isotope dilution mass spectrometry. The uranium content is estimated to be 103 μg/g metal as of October 1, 1985, with a calculated ingrowth of 31 μg/g metal per year and the ^{241}Am content is estimated to be 37 μg/g metal as of October 1, 1985, with a calculated ingrowth of 8 μg/g metal per year. ^{237}Np was determined by radiochemistry and is estimated to be 22 μg/g metal as of October 1, 1985, with a calculated ingrowth of <1 μg/g metal per year. These noncertified impurity values are provided for information only.

CRM No. 126 had a radioactivity of 3.04 × 10^9 Bq (0.082 Ci) per unit as of October 1, 1985, which is dominated by ^{239}Pu and ^{241}Pu.

Table I provides the decay-adjusted values of the certified assay content, ^{239}Pu isotopic content and relative atomic weight of CRM No. 126 at semi-annual intervals for a five-year period. In addition, the noncertified isotopic values are listed for information only. The half-life values (in years) used for the decay calculation are as follows: ^{238}Pu — 87.74; ^{239}Pu — 24,119; ^{240}Pu — 6,564; ^{241}Pu — 14.35; ^{242}Pu — 376,000.

TABLE I
CRM No. 126 Semi-Annual Decay-Adjusted Values
(Assay in Wt. %; Isotopic Composition in At. %)

Date	Certified Plutonium	^{238}Pu	Certified ^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu	Certified RAW
October 1, 1985	99.962	0.0016	97.925	2.0570	0.0156	0.0010	239.073
April 1, 1986	99.960	0.0015	97.925	2.0569	0.0152	0.0010	239.073
October 1, 1986	99.958	0.0015	97.926	2.0569	0.0148	0.0010	239.073
April 1, 1987	99.956	0.0015	97.926	2.0568	0.0145	0.0010	239.073
October 1, 1987	99.954	0.0015	97.927	2.0567	0.0141	0.0010	239.073
April 1, 1988	99.952	0.0015	97.927	2.0567	0.0138	0.0010	239.073
October 1, 1988	99.950	0.0015	97.927	2.0566	0.0135	0.0010	239.073
April 1, 1989	99.949	0.0015	97.928	2.0565	0.0132	0.0010	239.073
October 1, 1989	99.947	0.0015	97.928	2.0564	0.0128	0.0010	239.073
April 1, 1990	99.945	0.0015	97.929	2.0564	0.0125	0.0010	239.073
October 1, 1990	99.943	0.0015	97.929	2.0563	0.0122	0.0010	239.073
95% Confidence Interval for Certified Values (expressed as % of value):							
	±0.018	—	±0.001	—			