U.S. Department of Energy

A Documentary History of the United States' First Plutonium Isotopic Reference Materials

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NBL Program Office
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ABSTRACT

The New Brunswick Laboratory (NBL) was founded in 1949 by the U.S. Atomic Energy Commission to assist in the development of uranium (and in 1959 plutonium) analytical measurement methods. Over the years, the laboratory developed expertise and analytical methods relating to many aspects of the nuclear fuel cycle and weapons materials. The National Bureau of Standards (re-named NIST) served (as NIST still serves) as the nation's source for Standard Reference Materials (SRMs) and is the certifying authority and the national metrology institute for the United States. Beginning in the early 1950's, the need for uranium and then plutonium primary reference materials became apparent. NBS, NBL and various AEC laboratories collaborated on identifying reference materials needs, procuring base materials, developing the necessary chemical and handling techniques, studying chemical forms for suitability and storage as reference materials, and certifying the chemical and physical properties of the produced SRMs. Beginning in 1956, there were a series of communications between the AEC and NBS identifying nuclear reference materials needs and priorities, including a plutonium isotopic standard. The first plutonium isotopic standard produced was SRM 948, which was of weapons-grade isotopic composition. As commercial reactors and fuel reprocessing developed through the early 1960's, the need for two new isotopic materials containing higher amounts of ^{240}Pu became apparent. This need lead to the certification of SRMs 946 and 947. This report compiles the information and data regarding the production and certification of the three plutonium isotopic reference materials, and discusses plans for the future of these and other materials.

INTRODUCTION

The information in this report came from the New Brunswick Laboratory (NBL) Reference Materials Program files. Most of the documents cited in this report were transferred to NBL during the 1980's from the National Bureau of Standards (NBS), now NIST, when the nuclear reference material program was transferred from NBS to NBL. Some information came from publicly-available reports.

The three plutonium isotopic reference materials discussed in this report were originally named by NBS as SRM's 946, 947 and 948 and were re-named CRM‟s 136, 137 and 138 in the 1980's when NBS formally transferred nuclear reference materials responsibilities and stocks to NBL. These materials are still available for sale from NBL and correspond as:

SRM 946 = CRM 136
SRM 947 = CRM 137
SRM 948 = CRM 138

SRMs 946 and 947 share a common history in that they came from similar sources (commercial power reactor spent fuel) and were processed, purified, manipulated and certified by essentially identical methods at the same facility at roughly the same time as part of a single effort to produce two plutonium isotopic reference materials. SRM 948 pre-dates the other two materials by approximately 5 years, and likely originated in a different manner, although many of the processing steps were likely similar or identical since all three are plutonium sulfate tetrahydrate materials.

There is much more surviving information on the origin and processing of SRM's 946 and 947 than for 948. A chronological listing of information taken from the NBL/NBS files follows the summary section below.

SUMMARY DATA FOR SRMs 946, 947 and 948

A. The SRM 946 plutonium came from reprocessed fuel from the Yankee Atomic Electric Company reactor located in Rowe, Massachusetts. The reactor was a 167 MW pressurized water reactor which began operation in 1960. The spent-fuel reprocessing likely took place at the Nuclear Fuel Service (NFS) West Valley, NY reprocessing plant sometime in the late 1960's, utilizing the PUREX process. A plutonium solution from NFS was transferred to Richland, Washington and converted to low-fired oxide for intermediate storage. As part of a project to produce two plutonium isotopic standards (the other being SRM 947), the oxide was dissolved, ion exchanged as the nitrate form sometime between March and April 1970 to remove americium and impurities, converted to sulfate, and formed into plutonium sulfate tetrahydrate crystals. Literature related to age-dating plutonium suggests that the ion exchange, while perhaps not quantitative, left only traces of americium. The provisional certificate values of 1971 (see below) were officially adopted in the NBS Certificate of Analysis dated August 18, 1982 with values valid as of January 1, 1982. The provisional atom percent values below were decayed to January 1, 1982 using the half-lives listed on the 1982 Certificate of Analysis.

SRM 946 Provisional Certificate (values valid as of Oct 19, 1971) dated Dec 3, 1971:

SRM 946	238	239	240	241	242
Atom %:	0.247	83.128	12.069	3.991	0.565
95% CI:	0.007	0.015	0.015	0.005	0.003
Weight %:	0.246	83.052	12.109	4.021	0.572

There were impurity measurements made on both the originating oxides and the resultant plutonium sulfate tetrahydrate materials. The results are summarized below.

Impurity Measurements on the Yankee Oxide (SRM 946 base material):

Pu Assay:	86.42% Pu
C:	225 ug/g
Fe:	95 ug/g
Ca:	20 ug/g

Note: The Pu assay value was not corrected. The chemical assay was performed on 4-11-69. There is no other information on the Yankee oxide measurements.

Impurity Measurements of SRM 946 as Pu Sulfate Tetrahydrate

(DC Arc on two 100 mg samples by 8 mm Crater; 1970, Richland):

Element	µg/g	Element	µg/g
Ag	n.d.	Li	n.d.
Al	<2	Mg	<5
As	n.d.	Mn	n.d.
B	n.d.	Mo	n.d.
Ba	n.d.	Na	7
Be	n.d.	Ni	<10
Bi	n.d.	P	n.d.
Ca	<1	Pb	5
Cd	n.d.	Sb	n.d.
Co	n.d.	Si	<5
Cr	<0.5	Sn	5
Cu	2	Tl	n.d.
Fe	<10	V	<20
In	<1	Zn	<50
K	n.d.		

n.d. = not detected.

B. The SRM 947 plutonium came from reprocessed fuel from the Commonwealth Edison Dresden reactor #1 located near Morris, Illinois. The reactor was a 200 MW boiling water reactor and on July 4, 1960 became the first operating commercial reactor. The spent-fuel reprocessing likely took place at the Nuclear Fuel Service West Valley plant sometime in the late 1960's, utilizing the PUREX process. The solution was transferred to Richland, Washington and converted to low-fired oxide for intermediate storage. As part of a project to produce two plutonium isotopic standards (the other being SRM 946), the oxide was dissolved, ion exchanged in the nitrate form in September 1970 to remove americium and impurities, converted to sulfate, and formed into plutonium sulfate tetrahydrate crystals. Literature related to age-dating plutonium suggests that the ion exchange, while perhaps not quantitative, left only traces of americium. The provisional certificate values of 1971 (see below) were officially adopted in the NBS Certificate of Analysis dated August 18, 1982 with values valid as of January 1, 1982. The provisional atom percent values below were decayed to January 1, 1982 for the final NBS Certificate of Analysis dated 1982, using the half-lives listed on the 1982 Certificate.

SRM 947 Provisional Certificate (values valid as of Oct 13, 1971) dated Dec 3, 1971:

SRM 947	238	239	240	241	242
Atom %:	0.296	75.696	18.288	4.540	1.180
95% CI:	0.006	0.022	0.022	0.006	0.004
Weight %:	0.294	75.600	18.341	4.572	1.193

There were impurity measurements made on both the originating oxides and the resultant plutonium sulfate tetrahydrate materials. The results are summarized below.

Impurity Measurements on the Dresden Oxide (SRM 947 base material):

Pu Assay:	86.43% Pu
Ca:	5 ppm
Mo:	1 ppm

Note: Pu assay not corrected for bias. Analyzed for chemical assay on 9-11-68.

Impurity Measurements of SRM 947 as Pu Sulfate Tetrahydrate

(DC Arc on two 100 mg samples by 8 mm Crater; February 27, 1971, Richland):

Element	µg/g	Element	µg/g
Ag	0.1.	Li	n.d.
Al	<2	Mg	<5
As	n.d.	Mn	n.d.
B	n.d.	Mo	n.d.
Ba	n.d.	Na	6
Be	n.d.	Ni	<10
Bi	n.d.	P	n.d.
Ca	5	Pb	3.5
Cd	n.d.	Sb	n.d.
Co	<10.	Si	<5
Cr	<2.5	Sn	7.5
Cu	2	Tl	n.d.
Fe	<10	V	<20
In	n.d.	Zn	<50
K	n.d.		

C. SRM 948 came from an unknown source, likely a Hanford or Savannah River reactor used to produce weapons grade plutonium, at an unknown time. It's likely the SRM was converted from high-purity metal to the sulfate tetrahydrate form sometime in 1963, and the bottling was completed in early 1964. The original certification measurements (including a nine laboratory inter-comparison) did not include Pu-238. The final NBS certified isotopic values are given as:

Certificate of Analysis for SRM 948 (values valid for July 12, 1966) dated Aug 8, 1966:

SRM 948	238	239	240	241	242
Atom %:	0.011	91.471	7.911	0.628	0.0330
95% CI:	0.002	0.010	0.010	0.002	0.0003
Weight %:	0.011	91.382	7.941	0.633	0.0334

At the time of bottling at NBL, each microbottle contained 526 mg ± 5 mg of plutonium sulfate tetrahydrate.

DOCUMENTARY DATA ON THE PLUTONIUM ISOTOPIC STANDARDS

SRM 948

The earliest mention of the need for plutonium isotopic standards is in a series of letters between the AEC and NBS dating from 1956. There is evidence of a number of studies in the 1963-1964 timeframe to decide on a suitable chemical form for a plutonium isotopic standard, including investigation of the stabilities/stoichiometries of a sulfate, sulfate tetrahydrate and a dicesium tetrachloride double salt. The sulfate tetrahydrate was chosen as suitably stable to provide for long-term storage and use as an isotopic standards.

There was an effort to characterize the isotopic composition of the weapons-grade material (SRM 948) by interlaboratory exercise. There is a Dec 22, 1964 letter from the AEC to NBS indicating that the production of SRM 948 was a joint AEC-NBS effort with a start date of September 1963. In this letter, there is a mention that the material used was "prepared by an AEC contractor", but no other information on the origin of the material. All of the certificates in the records state that the material "was prepared from high-purity metal by the New Brunswick Laboratory".

The records contain data from 9 labs reporting Pu isotopic data w/ decayed values apparently to June 1, 1964**.** The labs were: NBS; DuPont –Savannah River; Dow – Rocky Flats; GE – Hanford; Los Alamos Scientific Laboratory; NBL; GE – Knolls Atomic Power Lab; Argonne National Lab; Euratom (no specific lab identified). The average values for the material were reported in a table listed as "Summary of First Reported Values Plutonium Isotopic Standard 948" as:

SRM 948	**239**	**240**	**241**	**242**
Atom % Avg:	91.3946	7.8829	0.6896	0.0330
1 sigma:	0.044	0.039	0.0046	0.0022

No Pu-238 values were measured. It does not appear that the multiple laboratory data was used for certification measurements, but rather as verification.

In 1964, NBS issued a provisional certificate of analysis with the following data and date:

Provisional certification for SRM 948 (values valid June 1, 1964); dated Aug 7, 1964:

SRM 948	**239**	**240**	**241**	**242**
Atom %:	91.365	7.907	0.695	0.0330
95% CI:	0.010	0.010	0.002	0.0003

It appears there were five samples analyzed to yield the provisional values, and these were purified via ion exchange in the nitrate form between June 4 and June 10, 1964 prior to mass spectrometric analysis at NBS. It appears there were also measurements made earlier, on April 23, 1964, without Am separation, which yielded significantly different ^{239}Pu/^{241}Pu ratios. The mass spectrometrist (believed to be W. Shields) reported in an April 1964 memo "The Pu^{239}/Pu^{241} ratio is meaningless because the Am^{241} has not been removed". The April 1964 ^{239}Pu/^{241}Pu ratio measurements ranged between 122.8 – 128.3; while the June 1964 measurements of the ion exchanged samples yielded corrected ^{239}Pu/^{241}Pu measurements of 11.554. There is very good agreement for the two sets of measurements for the ^{239}Pu/^{240}Pu ratio for the purified and unpurified samples (11.5543 ± 0.0064 one sigma for purified and 11.5564 ± 0.0022 one sigma for not purified).

At a later date, NBS performed additional analyses to characterize the ^{238}Pu content, and also due to in-growth re-calculated the ^{241}Pu content. This caused slight changes to the ^{239}Pu and ^{240}Pu abundances. The samples were ion exchanged prior to mass spectrometric analyses at NBS. A letter dated Aug 23, 1966 stated "SRM 948 … recertified to include the small amount of Pu-238 present but not previously requested by your office. To maintain the desired tolerances, the Pu-241 was also redetermined because of uncertainties in the half-life of Pu-241".

Certificate of Analysis for SRM 948 (values valid for July 12, 1966) dated Aug 8, 1966:

SRM 948	238	239	240	241	242
Atom %:	0.011	91.471	7.911	0.628	0.0330
95% CI:	0.002	0.010	0.010	0.002	0.0003
Weight %:	0.011	91.382	7.941	0.633	0.0334

It is clear that bottling and sampling of SRM 948 occurred at NBL, though there is no date on the packaging and sampling report. The report describes a bottling operation which occurred at NBL, in which a total of 467 microbottles were filled with 526 mg ± 5 mg of $Pu(SO_4)_2 \cdot 4H_2O$. Five samples (1 mg Pu complex) were taken periodically during the bottling process and shipped to NBS for homogeneity testing. After an extensive filling and wiping protocol, the filled, capped bottles were placed into a poly heat-sealed sleeve and then removed from the glove box and placed into a red mailing carton which was taped shut with scotch tape. Then each into a plastic bag, each into a 2.5" long pipe that had been capped, the other cap then placed on (both caps containing a piece of plastic foam on the interior). A standard sample label was marked with the lot number and attached to the middle of the pipe with scotch tape. A piece of tape was placed on the pipe opposite the standard label which was marked as "Pu-239; 16 mc, 14 Feb '4". The pipes were placed in metal cans w/ approximately 60 pipes per can and then shipped to NBS. At NBS, each end of the pipe was dipped in "Seal-Stop". It's clear that NBL performed some mass spectrometric measurements accompanied by an Am-Pu ion exchange separation.

There is no other information regarding the origin of the plutonium, though some assumptions can be made. It's likely that the material did not originate from a commercial reactor (as did SRM's 946 and 947) because of the isotopic composition (weapons-grade) and the fact that re-processing of commercial fuels at the West Valley Nuclear Fuel Services plant did not begin until 1966. It is logical then that the source of the SRM 948 plutonium was either a Hanford or Savannah River reactor, of which both sites were producing weapons grade plutonium from 1944 (Hanford) and 1955 (Savannah River).

SRM 946 and 947

SRM 946 and 947 originated from requests for higher-containing ^{240}Pu isotopic standards more indicative of commercial power reactors. The production of the materials was performed as one project, sequentially, and occurred in Richland, Washington. The project was originally overseen by Battelle Northwest and later by WADCO (a subsidiary of Westinghouse). These two companies were the operators of the Hanford Engineering Development Laboratory in Richland during the time of the project (1970 – 1971 timeframe).

Both materials originated in commercial reactor spent fuels, SRM 946 from a reprocessed solution made from irradiated Yankee Rowe reactor fuel and SRM 947 from a reprocessed solution made from irradiated Dresden reactor fuel.

Below is a chronology of the project pieced together from Battelle and WADCO status reports and other communications.

FEBRUARY 5, 1968:

In a letter from AEC to NBS requesting NBS certify plutonium standards of 12% and 20% ^{240}Pu NBS detailed their project priorities and requested AEC fund the project in order to move it to a higher priority. The AEC was requesting the two new standards because "the plutonium presently being produced in nuclear power reactor fuel has a considerably higher concentration of Pu-240:

Dresden:	20% Pu-240
Yankee:	12-13% Pu-240

And further "the value of the large quantities of reclaimed plutonium from power reactors is highly dependent on the isotopic ratios". Additionally, the AEC indicated that "plutonium sulfate tetrahydrate will be prepared from plutonium actually recovered from fuel elements discharged from the Yankee and Dresden reactors".

It is worth nothing that during the 1960's there was great interest in commercial reactor construction and operation, and three reprocessing facilities were planned and built to handle the subsequent spent fuel. Nuclear Fuel Service's West Valley reprocessing plant was the only one actually operated. The other two, GE's Morris, Illinois reprocessing plant and Allied Chemical's Barnwell, SC site were built but never operated.

JANUARY 21, 1969:

From NBS form "Research and Technology Resume" a proposal to produce new SRM's similar to SRM 948: "Plutonium from use of the Yankee and Dresden reactors is available, and Battelle-Northwest has submitted a proposal for processing the material into a suitable sulfate form".

JULY 31, 1969:

Same NBS form as entry above, different date: "Material has been obtained from fuel elements from the Yankee and Dresden reactors… and approximately 1 kg of material is stored at Battelle-Northwest".

MARCH 2, 1970:

Letter from AEC to NBS:
Refers to the sources of the materials as "using material from the 'Dresden' and 'Yankee' fuels".

Analytical data attached to letter:
Yankee Oxide: 86.42% Pu; C=225 ppm; Fe 95 ppm; Ca 20 ppm; Al, Cr,Pb, Si,Sn, Zn=1-12 ppm
all others:Ag, As, B, Be, Bi, Cd, Cu, Ge, K, Li, Mg, Mn, Mo, Na, Ni P, Ti, Tl, Vare less than values ranging from 1 ppm – 100 ppm.
Note: Pu assay not corrected for bias. Analyzed for chemical assay on **4-11-69**.

Isotopics (no date given):

238	239	240	241	242
0.224	82.606	12.053	4.534	0.533

Dresden Oxide: 86.43% Pu; Ca 50 ppm; Mo 1 ppm; Pb, Si, Sn, Zn = 1 to 16 ppm. All others (same as above) less than values ranging from 1-100.
Note: Pu assay not corrected for bias. Analyzed for chemical assay on **9-11-68**.

Isotopics (no date given):

238	239	240	241	242
0.347	74.846	18.239	5.399	1.169

MARCH 7, 1970

Status report from Battelle Northwest titled "Preparation of Pu Sulfate Tetrahydrate Isotopic Standards":

"As you will recall from our last progress report, we are preparing the Pu isotope standards as Pu sulfate tetrahydrate from low fired Pu oxide powder. Initially two Pu nitrate solutions were used; one with a 240-Pu isotopic content of about 12% and the other of about 18%. Pu from the "Dresden" and "Yankee" fuels are being used for these standards. These supplies had been converted to low fired oxide to permit easier storage for the past year".

"On March 3, we started processing the Pu oxide from the 'Yankee' fuel (~12% ^{240}Pu) for the preparation of the Pu sulfate tetrahydrate isotopic standards. 189 g of Pu oxide were dissolved and are being processed through ion exchange purification".

MAY 21, 1970

Status report from Battelle Northwest titled "Preparation of Pu Sulfate Tetrahydrate Isotopic Standards":

"The preparation of the plutonium sulfate tetrahydrate crystals, however, has not been straightforward … C.E. Pietri (of NBL) …was consulted for advice and for clarification of some details in his report."

[Editorial comment: Battelle was experiencing yield problems, only recovering ~50% of the Pu in the crystallization process. C. Pietri, who helped develop the method and studied the stability of the tetrahydrate form provided assistance to Battelle.]

JULY 1, 1970: Work, personnel, and facilities were transferred from Battelle Northwest to WADCO.

SEPTEMBER 1, 1970:

Status report from WADCO titled "STATUS REPORT – PREPARATION OF PLUONIUM SULFATE TETRAHYDRATE ISOTOPIC STANDARDS".

"The preparation of 285 g Pu sulfate tetrahydrate crystals, which appear to be of satisfactory quality, was completed for the Pu with the ^{240}Pu content of about 12%".

"Processing of the batch of plutonium with the ^{240}Pu content of about 18% is expected to start in September. …and is expected to be completed by January 1971".

NOVEMBER 4, 1970:

Status report from WADCO titled "PREPARATION OF PLUTONIUM SULFATE TETRAHYDRATE ISOTOPIC STANDARDS"

"The ion-exchange purification of a 169 gram batch of 'Dresden' plutonium (~18 wt% Pu-240) has been completed. The nitrate solution from the purification step has been converted to the sulfate form required to prepare the crystalline plutonium sulfate tetrahydrate product desired".

"Preliminary assays were made on duplicate samples taken from the first batch (~12% Pu-240) of Pu sulfate tetrahydrate salt prepared. The results are tabulated in the attached tables indicate the material is of satisfactory quality …the rather close agreement with the theoretical values based on true stoichiometric proportions is gratifying".

Data from Nov 4, 1970 Status Report on SRM 946 base material as sulfate tetrahydrate:

SRM 946	Sample 1	Sample 2	Theoretical
Pu Wt%:	47.33	47.18	47.524
Sulfate Wt%:	38.2	36.9*	38.162
Water Wt%:	14.223	14.194	14.314

*This value is low due to losses occurring while performing the unfamiliar analysis which was done in a hot cell using remote manipulators.

Isotopic values from Nov 4, 1970 Status Report

SRM 946	238	239	240	241	242
Atom %:	0.242	82.73	12.08	4.36	0.581
95% CI:	0.004	0.07	0.06	0.02	0.006

Single aliquant after purification.

JANUARY 6, 1971

Status report from WADCO:

"The prep of 314 g of Pu sulfate tetrahydrate crystals from the purified 'Dresden' Pu (18 wt% Pu-240) was completed in December, 1970".

JUNE 4, 1971

Status report from WADCO:

"Packaging of 496 individual lots of … [SRM 946]… was completed during the last week of April. …480 lots were shipped to NBS on May 6, 1971. Remaining 16 lots were retained here for additional work on the chemical and isotopic assay of the material".

"After meticulously thorough decontamination of the glove box facilities used for …[previous material]… 533 lots of crystals prepared from…'Dresden' material…were packaged during the last week of May".

NBS SRM 946 Provisional Certificate (values valid as of Oct 19, 1971) dated Dec 3, 1971:

SRM 946	238	239	240	241	242
Atom %:	0.247	83.128	12.069	3.991	0.565
95% CI:	0.007	0.015	0.015	0.005	0.003
Weight %:	0.246	83.052	12.109	4.021	0.572

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Atom %:	0.296	75.696	18.288	4.540	1.180
95% CI:	0.006	0.022	0.022	0.006	0.004
Weight %:	0.294	75.600	18.341	4.572	1.193

Am-241 Content and Dating SRM 946 and 947

It's clear that the SRM 946 bulk material was purified by ion exchange sometime between March and April 1970 and the SRM 947 bulk material was ion exchanged between September and November 1970. The effectiveness of the removal of Am from the Pu by the technique is not clear as there is no indication that Am measurements were performed on any of the materials either before and/or after the purification process. Additionally, while its clear the reprocessing of the spent fuel took place sometime after 1965, the material was then converted to oxide, then dissolved as sulfate, and then crystallized to form the final sulfate tetrahydrate; there is no way of knowing the Am content throughout the process.

The original Yankee and Dresden spent fuel would have undergone reprocessing via the PUREX process in use at the Nuclear Fuel Services West Valley facility sometime between 1966 and late 1969. The two reactors which were the source of the plutonium went on-line in 1960. Therefore, the plutonium in SRM 946 and 947 certainly originated after 1960. There is no doubt that a large portion of Am would have been removed in the ion exchange process. There is reasonably strong analytical evidence in the literature that the separation of Am from the product Pu was at least near-quantitative and that only traces of Am remained after the 1970 ion exchange purifications.

For example:

1. "Age determination of plutonium material in nuclear forensics by thermal ionization mass spectrometery"; Wallenius, M; Mayer, K; Fresnius J Anal Chem (2000) 366:234-238.

   The authors determined the ages of SRMs 946 and 947 in late 1997 to mid-1998 using 4 different chronometers (^{238}Pu/^{234}U; ^{239}Pu/^{235}U; ^{240}Pu/^{236}U and finally ^{241}Pu/^{241}Am) using both gamma and mass spectrometric measurements. To generalize, all age measurements via gamma and TIMS agreed to within about 6 months for each material. For SRM 946 the average determined age was about 27.5 years, and for SRM 947 the average determined age was about 27.0 years. This would place the date of last purification at right around 1970, which agrees very well with the documentary evidence. Furthermore, in this paper the authors note that

> “The ^{241}Pu/^{241}Am ratio gave mostly slightly higher ages compared to the Pu/U parent-daughter ratios. A possible explanation can be that the initial Am separation was not completely successful and traces of it were left in the NBS material after reprocessing. Gamma spectrometry measurements supported this theory, because also a small amount of ^{243}Am was found, which is not generated from decay of Pu isotopes”.

2. “The ‘age’ of plutonium particles”; Wallenius, M et al; Radiochim. Acta 89, 55-58 (2001).

3. Letter to U. I. Narayanan (NBL) from L. Preston (NBL) of November 5, 1991. “H.W. Kirby and W.E. Sheehan, Mound Lab, assayed CRM 137 (SRM 947) for americium ingrowth using alpha spec over a period of 5 ½ years. They calculated an effective purification date of Sept 26, 1970 for this material. In the report of this work, they cite C. Leaf of NIST who indicated a purification date of September 30, 1970”.

So while there was likely some amount of ^{241}Am remaining in the SRM’s after ion exchange purification in 1970, it clearly was not a significant amount and would likely be properly characterized as a ‘trace’ amount.

Appendix 1

Source of SRM 946 and 947 Material

The following is excerpted from a February 1996 DOE Report entitled *“Plutonium Recovery from Spent Fuel Reprocessing by Nuclear Fuel Services at West Valley, New York from 1966 to 1972”.*

It is available courtesy of the Office of Scientific and Technical Information (OSTI) website, is publically available, and may be found at:

https://www.osti.gov/opennet/document/purecov/nfsrepo.html

Reprocessing by Nuclear Fuel Services of Yankee and Dresden Fuels

This report provides a detailed accounting of the separated plutonium received by the U.S. Atomic Energy Commission (AEC), a predecessor to the Department of Energy (DOE), from Nuclear Fuel Services (NFS), which operated a commercial spent fuel reprocessing facility located near West Valley, New York, 35 miles south of Buffalo. The NFS facility was the first and only private plant in the U.S. to reprocess spent nuclear fuel. NFS began receiving spent nuclear fuel at West Valley in 1965, and operated the facility from 1966 to 1972 to chemically separate and recover plutonium and uranium from the fuel.

In total, the plant recovered 1926 kilograms (kg) of plutonium and shipped almost 80% of the material (1530 kg) to the AEC. The remaining plutonium, 396 kg, either was retained by the utility companies, sold to industry by the utilities, or purchased by NFS and later re-sold to industry for use in plutonium recycle operations.

Of the 1530 kg of separated plutonium received by the AEC from the NFS West Valley facility, 635 kg originated from fuel or reactors that were AEC-owned and 895 kg came from commercial power- reactor fuel. The AEC purchased the 895 kg of commercial power-reactor plutonium from the utility companies under a program named the Plutonium Credit Activity which was established by the U.S. Congress in the Atomic Energy Act of 1954.

- Of the 635 kg of AEC-origin plutonium, most came from N-Reactor, a plutonium production reactor at the Hanford site near Richland, Washington. Specifically, 534 kg of plutonium came from N-Reactor, 95 kg from the Nuclear Fuels Services facility in Erwin, Tennessee, and 6 kg from the Bonus Reactor, an AEC-owned demonstration reactor in Puerto Rico.

- Of the 895 kg of separated plutonium purchased by the AEC from the utility companies, 436 kg was from Yankee Atomic Electric Company (Yankee Rowe), 285 kg from Commonwealth Edison Company (Dresden-1), 63 kg from Consumers Power Company (Big Rock Point), 7 kg from Northern States Power Company (Pathfinder), and 104 kg from Consolidated Edison Company (Indian Point-1).

All of the AEC-owned and -purchased plutonium was shipped as plutonium nitrate solution from NFS to the Hanford site. These shipments were made by commercial truck in accordance with applicable transportation regulations. Most of the plutonium received by the AEC from NFS was used in breeder reactor and zero-power reactor programs. To meet the isotopic and physical requirements for these programs, the NFS plutonium was blended with other plutonium and converted to either metal or oxide.

The purpose of this report is to provide a detailed account of the separated plutonium received by the AEC from NFS, a commercial spent fuel reprocessing facility located near West Valley, New York, 35 miles south of Buffalo. This document is part of a larger effort to respond to the Secretary of Energy's June 27, 1994, announced goal to declassify and release detailed plutonium information (See the DOE report, Plutonium: the First 50 Years, February 1996). This report is the first comprehensive look at NFS West Valley reprocessing operations and is the result of an exhaustive search of open literature, historical memoranda, and nuclear material accountability records.

It is available courtesy of the Office of Scientific and Technical Information (OSTI) website, is publically available, and may be found at:

https://www.osti.gov/opennet/servlets/purl/219368/219368.pdf

The NFS West Valley facility was the first and only private plant in the U.S. to reprocess spent nuclear fuel. The NFS facility was a PUREX (Plutonium Uranium Extraction) process plant with a design capacity of 300 tons of fuel per year. The PUREX process included storing spent fuel assemblies; chopping the assembly rods; dissolving the uranium, plutonium, and radioactive products in acid; separating and storing the radioactive wastes, and separating uranium nitrate from plutonium nitrate. Two other commercial reprocessing facilities were built in the United States, but never operated.

- The General Electric's Midwest Fuel Recovery Plant (also 300 tons per year) at Morris, Illinois, adjacent to the site of the Commonwealth Edison Company Dresden reactors, was completed at a cost of $64 million but was declared inoperable in 1974.
- In 1970, Allied General Nuclear Services (AGNS) began construction of a 1500 tons per year reprocessing plant at Barnwell, South Carolina, adjacent to the DOE Savannah River site. The Barnwell facility was due to begin operation in 1974, but following delays in construction and licensing, it still had not been completed or licensed when in 1977 President Carter decided to defer indefinitely all reprocessing of commercial irradiated fuel.

In the spring of 1963, the AEC issued the necessary permits to NFS, a subsidiary of the W.R. Grace Company (NFS was acquired by the Getty Oil Company in 1969), to begin construction of a fuel reprocessing facility. NFS was granted a license on May 27, 1965 to receive and store fuel at its reprocessing facility in West Valley. The first shipment of fuel, from the Yankee Rowe reactor in Massachusetts, was placed in the fuel storage pool at West Valley on June 5, 1965. Government and commercially-generated fuel continued to be received at NFS until 1973. There were a total of 756 truck and rail shipments.

The AEC encouraged NFS to focus on commercial fuel reprocessing; however, the AEC guaranteed a minimum quantity of government fuel to NFS in the absence of sufficient commercial supplies. Sufficient commercial supplies were not available because there were not many operating commercial reactors during the NFS reprocessing period of 1966 to 1972. As a result, approximately 60% of the facility's supply of fuel and 33% of the plutonium came from AEC reactors. Specifically, a majority of this came from N-Reactor.

In 1972, NFS (now owned by the Getty Oil Company) halted all reprocessing operations in order to increase reprocessing capacity, and to alter the facility to meet new regulatory requirements. However, subsequent difficulties were encountered in retrofitting the facility to meet these requirements and, after four years of fruitless negotiations with federal and state regulatory authorities, NFS announced its intention to cease reprocessing operations and transfer the management and long-term storage of approximately 600,000 gallons of high-level radioactive liquids and sludges at the West Valley Site to the site's landlord, the New York State Energy Research and Development Authority. This transfer was in accordance with contractual obligations.

There were a total of 27 processing campaigns performed at West Valley, however, only the first 26 campaigns reprocessed intact reactor fuel. The last campaign involved processing liquid residues received from Nuclear Fuels Services facility in Erwin, Tennessee, generated during the fabrication of fuel for the SEFOR reactor. The material reprocessed by NFS was both government and commercially-generated. The quantities of "Plutonium Received" below were based on shipper's data, i.e., theoretical calculations of plutonium produced in the reactors.

The AEC-owned plutonium came from the following:

- The Hanford N- Reactor, a production reactor formerly called the New Production Reactor, is located near Richland, Washington, about 150 miles southeast of Seattle. This reactor is owned and was operated by the AEC. N-Reactor was designed as a dual-purpose reactor for the production of plutonium and the production of by-product steam for electricity generation. It was a graphite-moderated, pressurized light water- cooled reactor. N-Reactor operated from 1963 to 1987.

- The Bonus Reactor, a demonstration boiling water reactor, was located at Ricon, about 75 miles west of San Juan, Puerto Rico. This reactor featured high-temperature, superheated steam and was owned by the AEC but operated by the Puerto Rico Water Resources Authority. The Bonus reactor began operation in 1964 and was permanently shut down in 1968.

- The remainder of the material came from the Nuclear Fuels Services facility in Erwin, Tennessee, and was in the form of liquid residues generated during the fabrication of reactor fuel for the Southwest Experimental Fast Oxide Reactor (SEFOR), an experimental reactor, located near Strickler, Arkansas. SEFOR was built by the Southwest Atomic Energy Associates for testing liquid metal fast breeder reactor fuel. SEFOR began operations in 1969 and was permanently shut down in 1972.

The remaining fuel came from seven commercial nuclear power reactors that were owned and operated by commercial utility companies.

- The Big Rock Point Nuclear Power Plant. This boiling water reactor, owned and operated by Consumers Power Company, is located on Lake Michigan near Charlevoix, Michigan, about 200 miles northwest of Detroit. Big Rock Point has operated since 1963.

- CVTR, Carolinas-Virginia Tube Reactor. This pressurized heavy water tube reactor, owned and operated by Carolinas-Virginia Nuclear Power Associates was located in Parr, South Carolina, about 25 miles northwest of Columbia. This reactor began operation in 1964 and was permanently shut down in 1967.

- Dresden Nuclear Power Station, Unit #1. This boiling water reactor, owned and operated by Commonwealth Edison Company, was located near Morris, Illinois, about 50 miles southwest of Chicago. Dresden-1 commenced operation in 1960 and was permanently shut down in 1978.

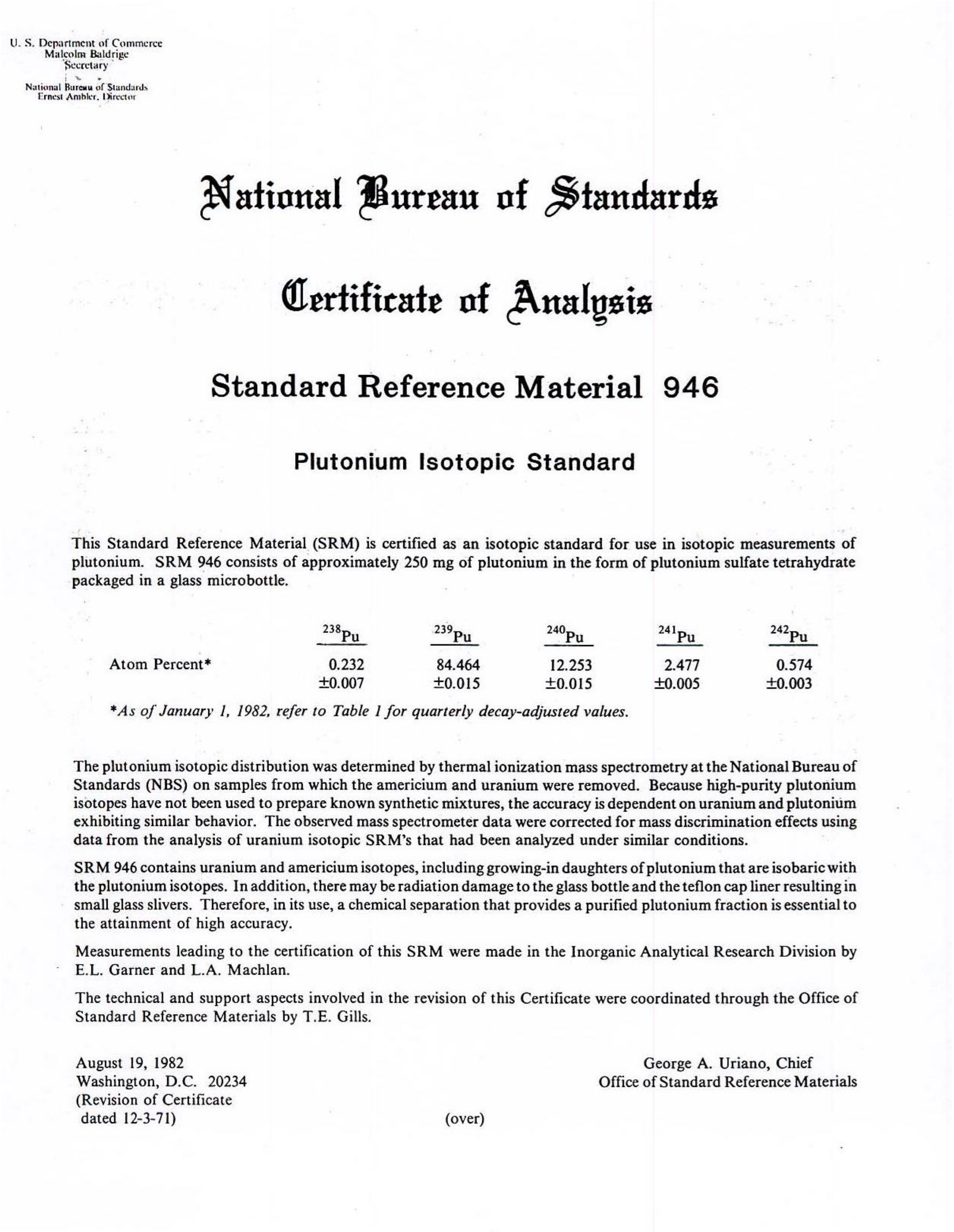
- Humboldt Bay Nuclear Plant. This boiling water reactor, owned and operated by the Pacific Gas and Electric Company, was located on Humboldt Bay near Eureka, California, about 200 miles north of San Francisco. This plant commenced operation in 1963 and was permanently shut down in 1976.

- Indian Point Nuclear Power Station, Unit #1. This pressurized water reactor, owned and operated by the Consolidated Edison Company, was located on the Hudson River at Buchanan, New York, about 35 miles north of New York City. Indian Point began operation in 1962 and was permanently shut down in 1974.

- Pathfinder Nuclear Power Plant. This experimental, boiling water reactor, owned and operated by the Northern States Power Company, was located on the Big Sioux River, near Sioux Falls, South Dakota. Pathfinder began operations in 1964 and was permanently shut down in 1967.

- Yankee Atomic Electric Power Station. This pressurized water reactor, owned and operated by the Yankee Atomic Electric Company, was located near Rowe, Massachusetts, about 45 miles east of Albany, New York. This reactor began operation in 1960 and was permanently shut down in 1992.

CERTIFICATES



U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 946

Plutonium Isotopic Standard

This Standard Reference Material (SRM) is certified as an isotopic standard for use in isotopic measurements of plutonium. SRM 946 consists of approximately 250 mg of plutonium in the form of plutonium sulfate tetrahydrate packaged in a glass microbottle.

	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
Atom Percent*	0.232 ±0.007	84.464 ±0.015	12.253 ±0.015	2.477 ±0.005	0.574 ±0.003

**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 the 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.

SRM 946 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 the 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 and L.A. Machlan.

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)

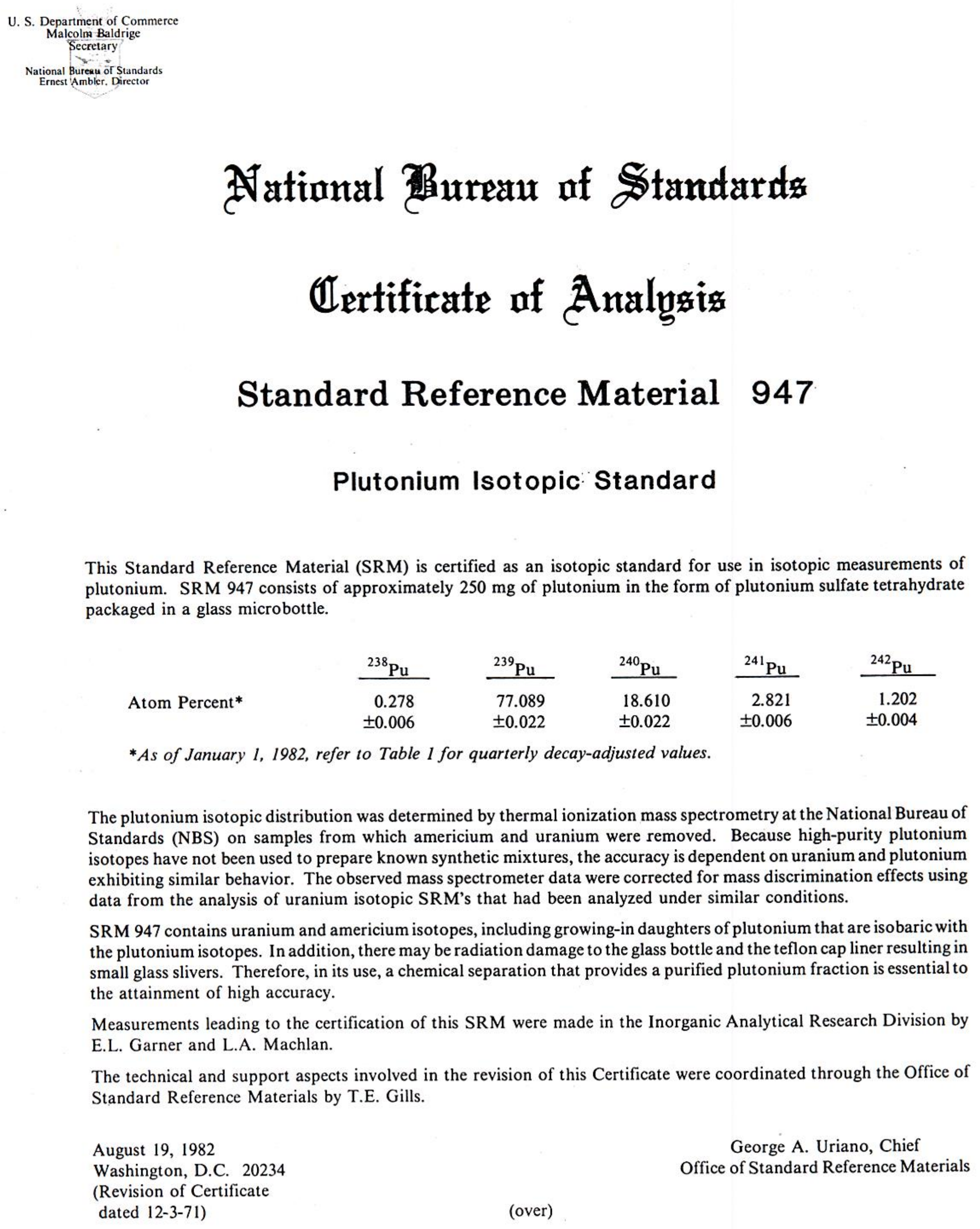
George A. Uriano, Chief
Office of Standard Reference Materials

(over)

The decay-adjusted values for the plutonium isotopic composition, in atom percent, are tabulated below in Table 1. 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 1
Decay-Adjusted Plutonium Isotopic Composition
Atom Percent

Date	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
January 1, 1982	0.232	84.464	12.253	2.477	0.574
April 1, 1982	.231	84.490	12.257	2.448	.574
July 1, 1982	.231	84.515	12.260	2.419	.575
October 1, 1982	.230	84.540	12.264	2.391	.575
January 1, 1983	.230	84.565	12.267	2.363	.575
April 1, 1983	.230	84.590	12.270	2.335	.575
July 1, 1983	.229	84.614	12.274	2.308	.575
October 1, 1983	.229	84.638	12.277	2.281	.575
January 1, 1984	.229	84.662	12.280	2.253	.576
April 1, 1984	.228	84.685	12.283	2.227	.576
July 1, 1984	.228	84.709	12.286	2.201	.576
October 1, 1984	.227	84.732	12.290	2.175	.576
January 1, 1985	.227	84.755	12.293	2.149	.576
April 1, 1985	.227	84.777	12.296	2.125	.576
July 1, 1985	.226	84.799	12.299	2.100	.577
October 1, 1985	.226	84.821	12.302	2.075	.577
January 1, 1986	.225	84.843	12.305	2.050	.577
April 1, 1986	.225	84.864	12.307	2.026	.577
July 1, 1986	.225	84.885	12.310	2.003	.577
October 1, 1986	.224	84.906	12.313	1.979	.577
95% Confidence Limit:	±0.007	±0.015	±0.015	±0.005	±0.003



U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 947

Plutonium Isotopic Standard

This Standard Reference Material (SRM) is certified as an isotopic standard for use in isotopic measurements of plutonium. SRM 947 consists of approximately 250 mg of plutonium in the form of plutonium sulfate tetrahydrate packaged in a glass microbottle.

	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
Atom Percent*	0.278 ±0.006	77.089 ±0.022	18.610 ±0.022	2.821 ±0.006	1.202 ±0.004

**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.

SRM 947 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 the 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 and L.A. Machlan.

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)

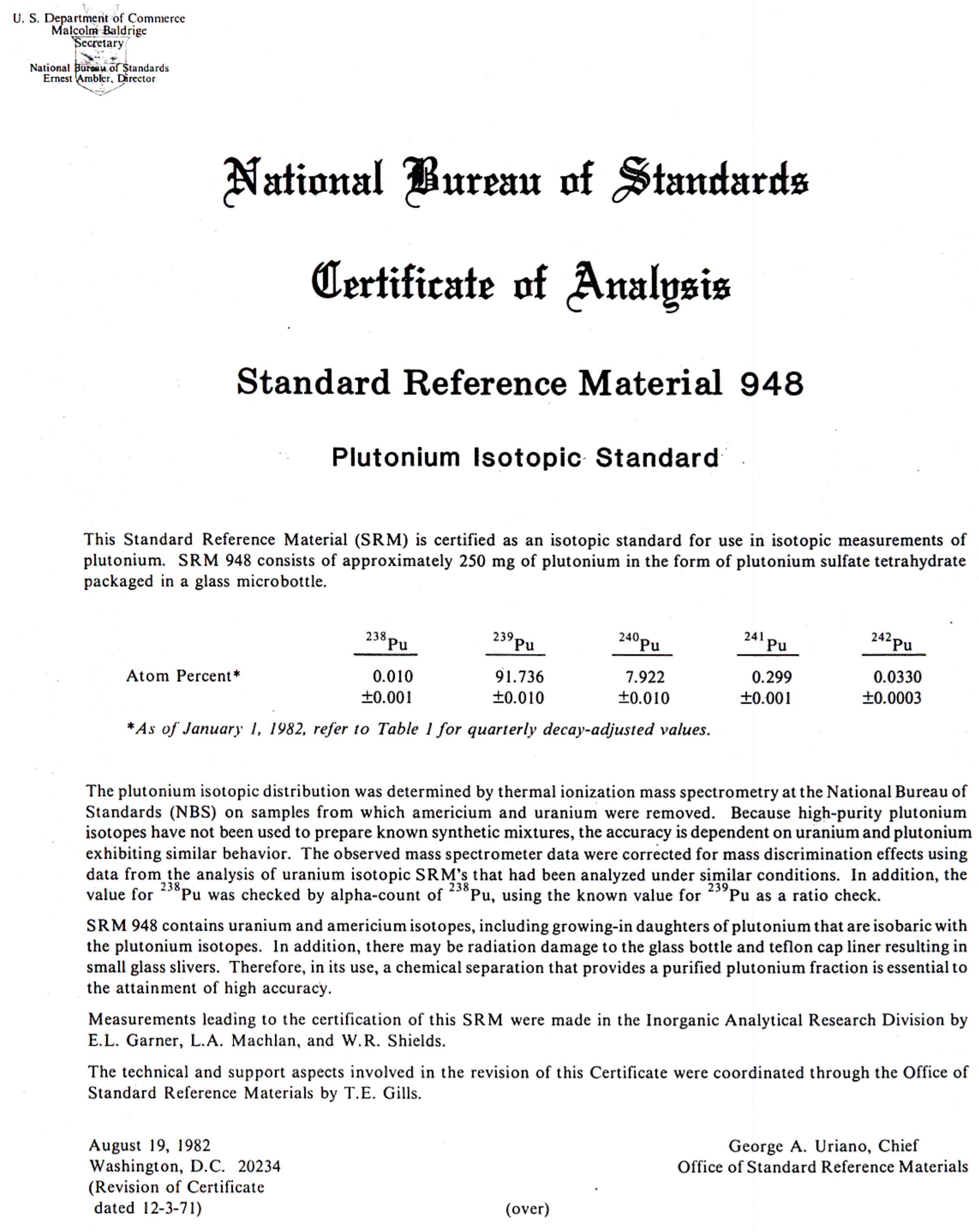
George A. Uriano, Chief
Office of Standard Reference Materials

(over)

The decay-adjusted values for the plutonium isotopic composition, in atom percent, are tabulated below in Table 1. 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 1
Decay-Adjusted Plutonium Isotopic Composition
Atom Percent

Date	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
January 1, 1982	0.278	77.089	18.610	2.821	1.202
April 1, 1982	.278	77.115	18.616	2.789	1.202
July 1, 1982	.277	77.142	18.622	2.756	1.203
October 1, 1982	.277	77.168	18.628	2.724	1.203
January 1, 1983	.276	77.194	18.634	2.692	1.204
April 1, 1983	.276	77.219	18.640	2.661	1.204
July 1, 1983	.275	77.245	18.645	2.630	1.205
October 1, 1983	.275	77.270	18.651	2.599	1.205
January 1, 1984	.275	77.295	18.657	2.568	1.205
April 1, 1984	.274	77.319	18.662	2.538	1.206
July 1, 1984	.274	77.344	18.668	2.509	1.206
October 1, 1984	.273	77.368	18.673	2.479	1.206
January 1, 1985	.273	77.392	18.679	2.450	1.207
April 1, 1985	.272	77.415	18.684	2.422	1.207
July 1, 1985	.272	77.438	18.689	2.393	1.208
October 1, 1985	.271	77.461	18.694	2.365	1.208
January 1, 1986	.271	77.484	18.700	2.337	1.208
April 1, 1986	.270	77.506	18.704	2.310	1.209
July 1, 1986	.270	77.528	18.709	2.283	1.209
October 1, 1986	.270	77.550	18.714	2.256	1.209
95% Confidence Limit:	±0.006	±0.022	±0.022	±0.006	±0.004



U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

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.

	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
Atom Percent*	0.010 ±0.001	91.736 ±0.010	7.922 ±0.010	0.299 ±0.001	0.0330 ±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)

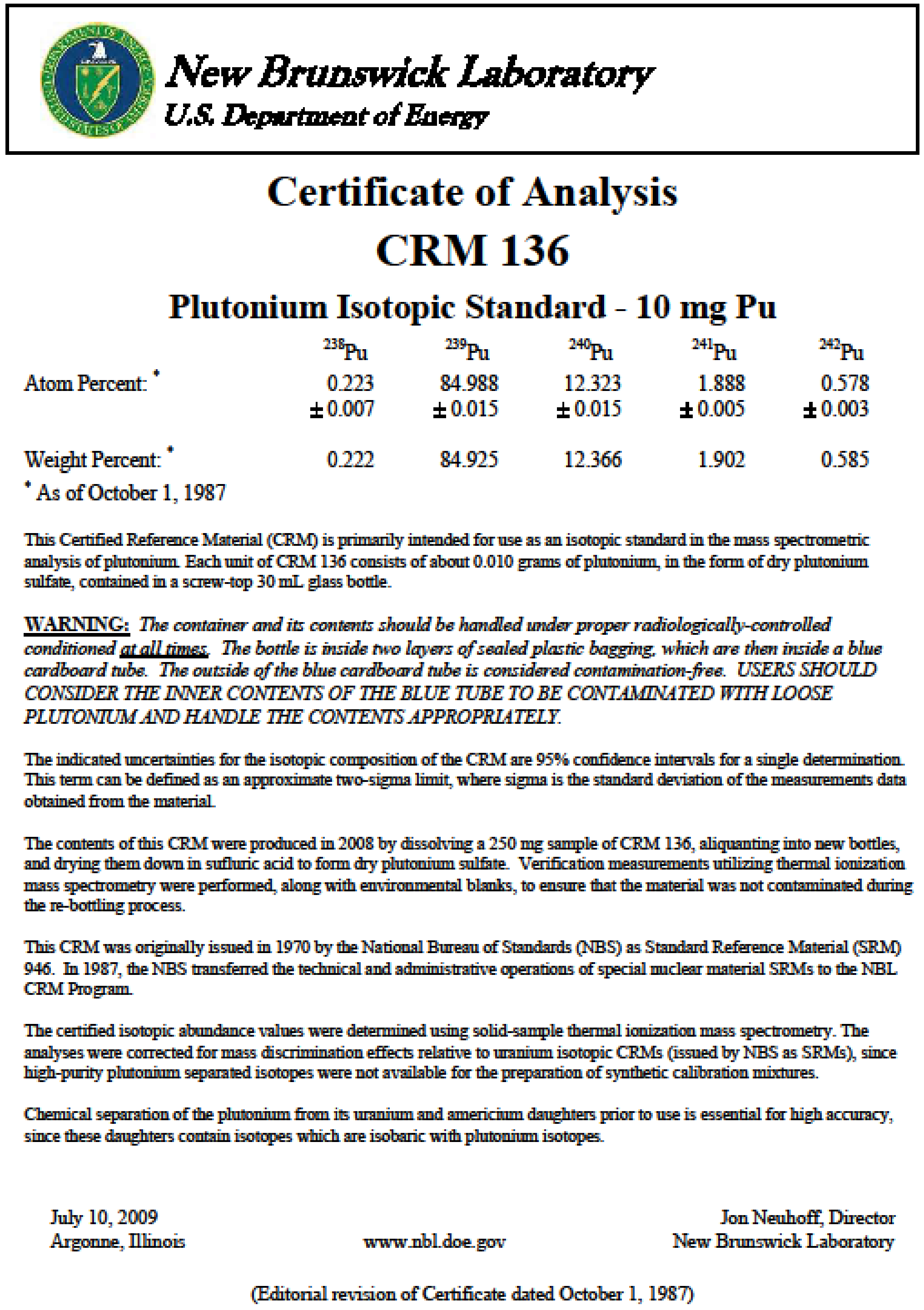
George A. Uriano, Chief
Office of Standard Reference Materials

(over)

The decay-adjusted values for the plutonium isotopic composition, in atom percent, are tabulated below in Table 1. 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 1
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



New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis

CRM 136

Plutonium Isotopic Standard - 10 mg Pu

	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
Atom Percent: *	0.223 ± 0.007	84.988 ± 0.015	12.323 ± 0.015	1.888 ± 0.005	0.578 ± 0.003
Weight Percent: *	0.222	84.925	12.366	1.902	0.585

* As of October 1, 1987

This Certified Reference Material (CRM) is primarily intended for use as an isotopic standard in the mass spectrometric analysis of plutonium. Each unit of CRM 136 consists of about 0.010 grams of plutonium, in the form of dry plutonium sulfate, contained in a screw-top 30 mL glass bottle.

WARNING: *The container and its contents should be handled under proper radiologically-controlled conditioned at all times. The bottle is inside two layers of sealed plastic bagging, which are then inside a blue cardboard tube. The outside of the blue cardboard tube is considered contamination-free. USERS SHOULD CONSIDER THE INNER CONTENTS OF THE BLUE TUBE TO BE CONTAMINATED WITH LOOSE PLUTONIUM AND HANDLE THE CONTENTS APPROPRIATELY.*

The indicated uncertainties for the isotopic composition of the CRM are 95% confidence intervals for a single determination. This term can be defined as an approximate two-sigma limit, where sigma is the standard deviation of the measurements data obtained from the material.

The contents of this CRM were produced in 2008 by dissolving a 250 mg sample of CRM 136, aliquanting into new bottles, and drying them down in sufluric acid to form dry plutonium sulfate. Verification measurements utilizing thermal ionization mass spectrometry were performed, along with environmental blanks, to ensure that the material was not contaminated during the re-bottling process.

This CRM was originally issued in 1970 by the National Bureau of Standards (NBS) as Standard Reference Material (SRM) 946. In 1987, the NBS transferred the technical and administrative operations of special nuclear material SRMs to the NBL CRM Program.

The certified isotopic abundance values were determined using solid-sample thermal ionization mass spectrometry. The analyses were corrected for mass discrimination effects relative to uranium isotopic CRMs (issued by NBS as SRMs), since high-purity plutonium separated isotopes were not available for the preparation of synthetic calibration mixtures.

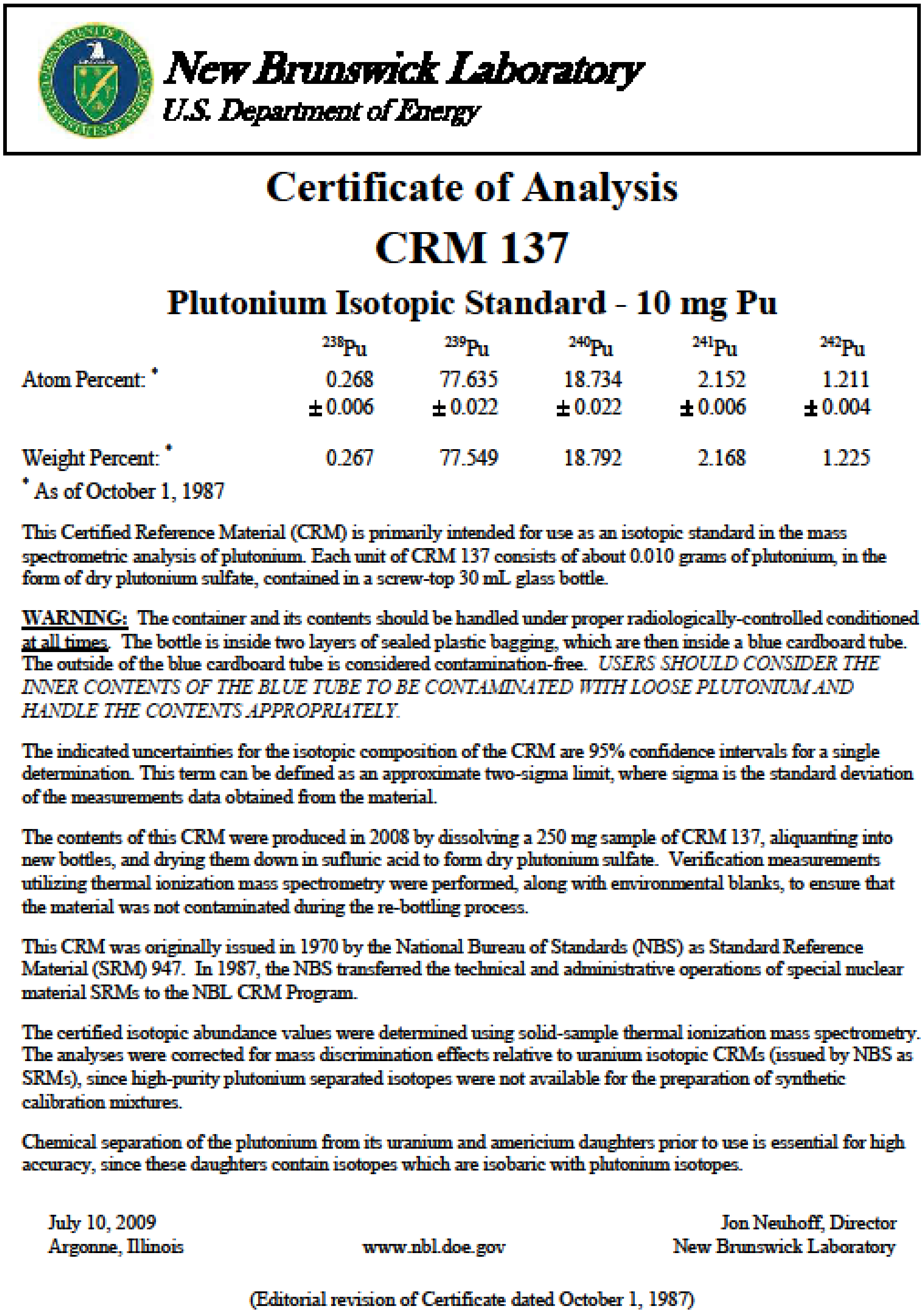
Chemical separation of the plutonium from its uranium and americium daughters prior to use is essential for high accuracy, since these daughters contain isotopes which are isobaric with plutonium isotopes.

July 10, 2009
Argonne, Illinois

www.nbl.doe.gov

Jon Neuhoff, Director
New Brunswick Laboratory

(Editorial revision of Certificate dated October 1, 1987)



New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis

CRM 137

Plutonium Isotopic Standard - 10 mg Pu

	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
Atom Percent: *	0.268 ± 0.006	77.635 ± 0.022	18.734 ± 0.022	2.152 ± 0.006	1.211 ± 0.004
Weight Percent: *	0.267	77.549	18.792	2.168	1.225

* As of October 1, 1987

This Certified Reference Material (CRM) is primarily intended for use as an isotopic standard in the mass spectrometric analysis of plutonium. Each unit of CRM 137 consists of about 0.010 grams of plutonium, in the form of dry plutonium sulfate, contained in a screw-top 30 mL glass bottle.

WARNING: The container and its contents should be handled under proper radiologically-controlled conditioned at all times. The bottle is inside two layers of sealed plastic bagging, which are then inside a blue cardboard tube. The outside of the blue cardboard tube is considered contamination-free. *USERS SHOULD CONSIDER THE INNER CONTENTS OF THE BLUE TUBE TO BE CONTAMINATED WITH LOOSE PLUTONIUM AND HANDLE THE CONTENTS APPROPRIATELY.*

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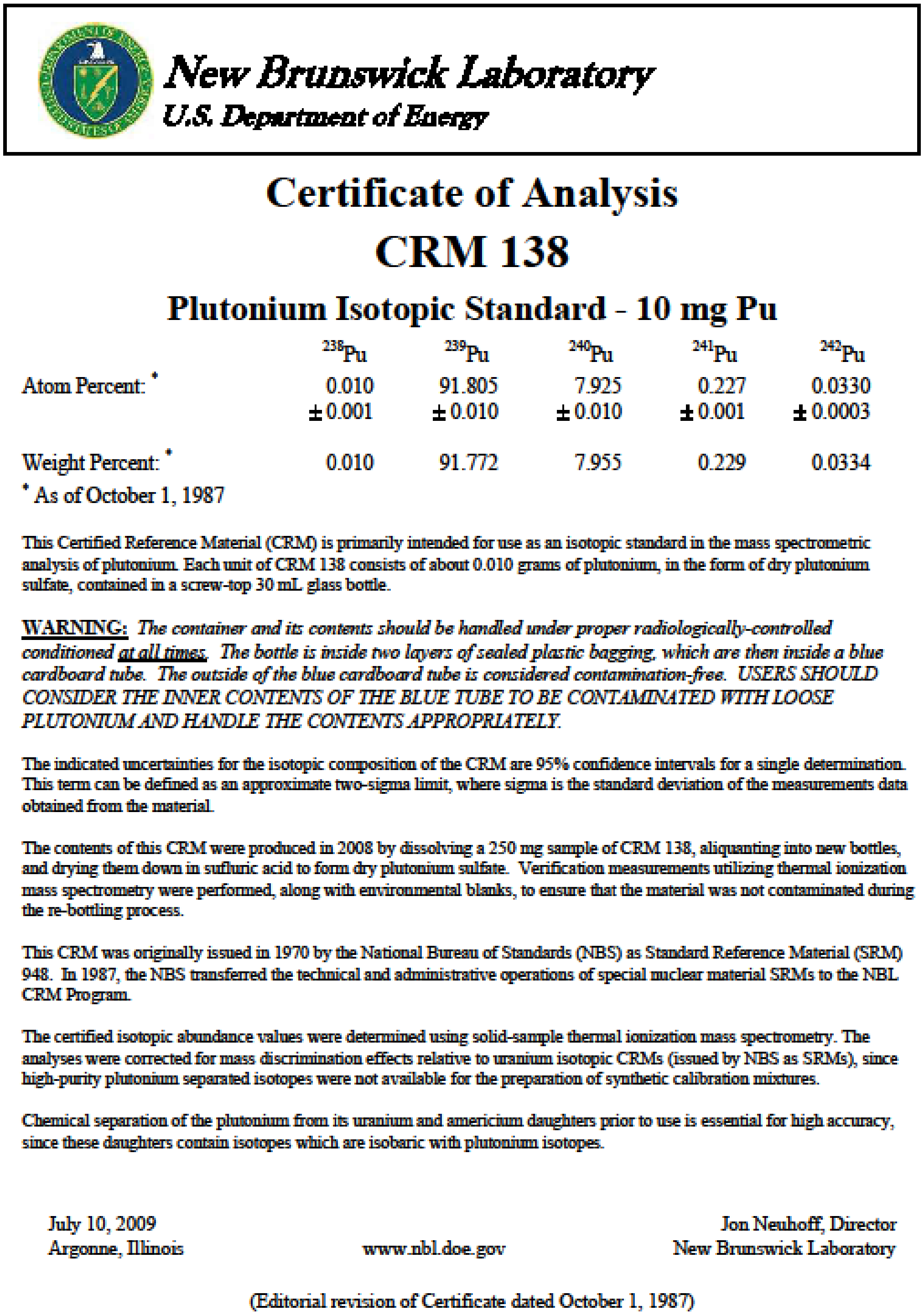

New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis
CRM 138
Plutonium Isotopic Standard - 10 mg Pu

	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
Atom Percent: *	0.010 ± 0.001	91.805 ± 0.010	7.925 ± 0.010	0.227 ± 0.001	0.0330 ± 0.0003
Weight Percent: *	0.010	91.772	7.955	0.229	0.0334

* As of October 1, 1987

This Certified Reference Material (CRM) is primarily intended for use as an isotopic standard in the mass spectrometric analysis of plutonium. Each unit of CRM 138 consists of about 0.010 grams of plutonium, in the form of dry plutonium sulfate, contained in a screw-top 30 mL glass bottle.

<u>WARNING:</u> *The container and its contents should be handled under proper radiologically-controlled conditioned at all times. The bottle is inside two layers of sealed plastic bagging, which are then inside a blue cardboard tube. The outside of the blue cardboard tube is considered contamination-free. USERS SHOULD CONSIDER THE INNER CONTENTS OF THE BLUE TUBE TO BE CONTAMINATED WITH LOOSE PLUTONIUM AND HANDLE THE CONTENTS APPROPRIATELY.*

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This CRM was originally issued in 1970 by the National Bureau of Standards (NBS) as Standard Reference Material (SRM) 948. In 1987, the NBS transferred the technical and administrative operations of special nuclear material SRMs to the NBL CRM Program.

The certified isotopic abundance values were determined using solid-sample thermal ionization mass spectrometry. The analyses were corrected for mass discrimination effects relative to uranium isotopic CRMs (issued by NBS as SRMs), since high-purity plutonium separated isotopes were not available for the preparation of synthetic calibration mixtures.

Chemical separation of the plutonium from its uranium and americium daughters prior to use is essential for high accuracy, since these daughters contain isotopes which are isobaric with plutonium isotopes.

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