S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards Certificate

Standard Reference Material 996

Plutonium-244 Spike Assay and Isotopic Standard

(In cooperation with the Department of Energy, New Brunswick Laboratory, Argonne, Illinois, and The University of California Los Alamos National Laboratory, Los Alamos, New Mexico)

This Standard Reference Material (SRM) is certified as an assay and isotopic standard for use as a spike for plutonium determination by isotope dilution mass spectrometry (IDMS). This SRM consists of approximately 1 mg of enriched ^{244}Pu as evaporated plutonium nitrate in a 125 mL Teflon bottle. Each bottle (SRM unit) contains a unique quantity of plutonium and is assigned a serial number for identification and reference.

Plutonium - 244 content: ± μmole

Serial No:

The plutonium content in SRM 996 is certified based on analyses and results by the National Bureau of Standards (NBS), the New Brunswick Laboratory (NBL), and the Los Alamos National Laboratory. Methods utilized were IDMS (^{239}Pu spike) and coulometry. The New Brunswick Laboratory used a controlled-potential coulometric method with quality assurance verification to NBS SRM 949e [1]. The Los Alamos National Laboratory used a similar controlled-potential coulometric method with chemical calibration relative to SRM 949e [2]. The NBS method was that of isotope-dilution mass spectrometry using SRM 949e as a spike. All three assays are traceable to the NBS SRM 949e, Plutonium Metal. The indicated uncertainty for the plutonium assay expressed at the 95 percent confidence interval includes combined errors of imprecision of measurements and weighing errors of individual units.

Plutonium isotopic distribution was determined by thermal ionization mass spectrometry at NBS. The NBS measurements were corrected for mass discrimination effects relative to NBS plutonium isotopic SRM's 947 and 948 which, in turn, are corrected relative to NBS uranium isotopic SRM's.

Plutonium Isotope:	238	239	240	241	242	244
Atom Percent:	0.005	0.034	0.677	0.092	1.325	97.867
95% Confidence Limit:	±.001	±.001	±.004	±.002	±.004	± .008

Measurements at NBS leading to the certification of this SRM were made in the Inorganic Analytical Research Division by J.D. Fassett, H.M. Kingston, and L.A. Machlan.

The overall direction and coordination of the technical measurements leading to certification were under the chairmanship of E. Garner, Chief of the Inorganic Analytical Research Division.

The statistical assessment of the data used for the certification of this SRM was performed by J. Mandel, Statistical Consultant to the NBS National Measurement Laboratories.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Washington, D.C. 20234
June 10, 1981

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

SRM 996 had a radioactivity of about 106 μCi per unit on May 17, 1980, which is dominated by relatively short-lived ^{241}Pu. This SRM is believed to be stable in the Teflon bottle for periods of 20 years or more. Since this stability has not been rigorously assessed, periodic reanalysis will be performed and if significant changes are observed, the purchasers of this SRM will be notified.

SRM 996 contains uranium and americium isotopes, including growing-in daughters of plutonium that are isobaric with the plutonium isotopes. Therefore, in its use, a chemical separation that provides a purified plutonium fraction is essential to the attainment of high accuracy.

The decay-adjusted values for plutonium isotopes 238 through 242 relative to ^{244}Pu at 1-year intervals for a 10-year elapsed time are tabulated below. In this time, the decay of ^{244}Pu (with a half-life of ~ 8 x 10^7 years) is not significant. 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.

Decay-Adjusted Plutonium Isotopic Ratios

	Ratios				
Date	^{238}Pu/^{244}Pu	^{239}Pu/^{244}Pu	^{240}Pu/^{244}Pu	^{241}Pu/^{244}Pu	^{242}Pu/^{244}Pu
5/17/80	0.00005	0.00035	0.00692	0.00094	0.01354
5/17/81	0.00005	0.00035	0.00692	0.00090	0.01354
5/17/82	0.00005	0.00035	0.00692	0.00085	0.01354
5/17/83	0.00005	0.00035	0.00691	0.00081	0.01354
5/17/84	0.00005	0.00035	0.00691	0.00077	0.01354
5/17/85	0.00005	0.00035	0.00691	0.00074	0.01354
5/17/86	0.00005	0.00035	0.00691	0.00070	0.01354
5/17/87	0.00005	0.00035	0.00691	0.00067	0.01354
5/17/88	0.00005	0.00035	0.00691	0.00064	0.01354
5/17/89	0.00005	0.00035	0.00691	0.00061	0.01354
5/17/90	0.00005	0.00035	0.00691	0.00058	0.01354

RECOMMENDED PROCEDURE FOR USING SRM 996

The package is designed to prepare a solution having a known concentration of Pu on a weight basis. Once prepared, it is suggested that all the solution be distributed as subportions for later use as individual spikes.

Wipe the bottle with a damp, lintless cloth or chamois to dissipate static charge and weigh it. Unscrew the cap, disengage the Teflon film, which may have adhering Pu, from the bottle threads, and carefully push the film into the bottle. Add a desired quantity, usually 50 to 100 g, of a 8M HNO_3 - 0.01M HF mixture and heat in a distilled-water bath at 90 ± 5 °C for 1 to 2 hours. Replace and tighten the cap, then shake the bottle vigorously for at least 2 minutes. Let cool to ambient temperature, preferably overnight. Loosen the cap to equalize air pressure, retighten the cap, wipe the bottle with a damp cloth or chamois, and weigh. Shake vigorously for at least 2 minutes and distribute all the solution as weighed portions into suitable containers for use as spikes. Calculate the Pu concentration by

$$^{244}\text{Pu } \mu\text{moles/g} = \frac{\text{(Certified content of } ^{244}\text{Pu } \mu\text{moles)}}{\text{(wt. of bottle and solution, (grams)) - (tare of bottle, (grams) - 0.0015)}}$$

in which 0.0015 of solution is the nominal grams of evaporated plutonium nitrate residue.

If a more dilute solution is desired, dissolve the residue as above, transfer the solution quantitatively to a larger tared container, weigh, mix vigorously, and distribute all the solution as weighed portions.

REFERENCES

1. M.K. Holland, J.R. Weiss, and C.E. Pietri, "Controlled-Potential Coulometric Determination of Plutonium," Anal. Chem. 50, 236-240 (1978).
2. D.D. Jackson, R.M. Hollen, F.R. Roensch, and J.E. Rein, "Controlled-Potential Coulometric Determination of Plutonium with a Hydrochloric Acid-Sulfamic Acid Electrolyte and Phosphate Complexing," Anal. Chim. Acta 117, 205-15 (1980).