

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 $115 \pm 65 \mu\text{g/g}$.

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.

Washington, D.C. 20234
May 11, 1982

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

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 1 are *not certified*, but given for information only.

Table 1

<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 ²³⁸Pu, 87.74; ²³⁹Pu, 24119; ²⁴⁰Pu, 6560; ²⁴¹Pu, 14.35; and ²⁴²Pu, 387.000.

Table 2

Plutonium Assay, Percent

<u>1980</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. Grill, 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).