



# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 960

#### Uranium Metal

Uranium Assay . . . . . 99.975 ± 0.017 Weight Percent

This metal standard of normal isotopic composition is issued as a primary assay standard for uranium determinations. The value of the atomic weight of this material is 238.0289 as determined at NBS by thermal ionization mass spectrometry.

The uranium assay is based on the constant-current coulometric reduction of uranyl ion with electrogenerated titanous ion in 7M sulfuric acid. The value of the assay has been corrected for 42 ppm of iron and 4 ppm of vanadium which are the titratable impurities present in the metal. The certified value, 99.975 weight percent, represents the mean of 21 determinations. The precision of the method, expressed in terms of the standard deviation of a single determination is 0.008 percent. The estimated value of the uncertainty of the mean assay is 0.006 percent. This figure includes the estimates of all known sources of error inherent to this determination: the random error component, 0.004 percent (the 95 percent confidence interval for the mean based on 20 degrees of freedom), and an additional 0.002 percent error term as an allowance for all known possible sources of systematic error. An overall mass balance of 99.9970 percent is obtained when the estimate of total impurities present in the material (223 ppm) is taken into account.

The uncertainty ascribed to the certified assay value is the 95 percent confidence interval for a single determination.

The metal as received will contain a significant amount of surface oxide. In assaying the material, the oxide was removed from the uranium samples just prior to weighing. The metal surface was cleaned by the procedure outlined on the back of this certificate.

This material was prepared by the United States Atomic Energy Commission. Impurities were analyzed by the AEC Paducah Laboratory, Paducah, Kentucky. Assay of the material was performed by G. Marinenko and E. S. Etz, the iron content was determined polarographically by E. J. Maienthal, and the atomic weight was determined by isotopic ratio measurements performed by E. L. Garner, all of the NBS Analytical Chemistry Division.

The overall direction and coordination of the technical measurements leading to the certification were performed under the chairmanship of W. R. Shields.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234  
May 12, 1972

J. Paul Cali, Chief  
Office of Standard Reference Materials

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### Removal of Surface Impurities

The following cleaning procedure was used on all uranium assay samples after being cut to the approximate size for analysis. For accurate analytical work, this procedure or an equivalent one should be followed.

Dip the uranium sample in 1:1  $\text{HNO}_3$  for a period of up to 10 minutes to remove all visible surface oxide. Rinse in distilled water. Etch in 1:3  $\text{HCl}$  for 5 minutes. Rinse thoroughly in distilled water. Remove excess surface water before placing the sample in a vacuum desiccator for attainment of constant weight. Removal of surface moisture is accelerated and re-oxidation of the metal surface is retarded by drying under vacuum for a suitable length of time (evacuation for 1/2 hour is sufficient).