. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 969

Uranium Isotopic Standard Reference Material for Gamma Spectrometry Measurements

(In Cooperation with the Commission of the European Communities, Central Bureau for Nuclear Measurements, Geel, Belgium, and the U.S. Department of Energy, New Brunswick Laboratory, Argonne, Illinois.)

This Standard Reference Material (SRM) is intended for use in the calibration and evaluation of gamma-ray counting procedures for the nondestructive determination of the $^{235}U/U$ isotope abundance in uranium bulk materials. SRM 969 consists of a set of five different U_3O_8 powders, with nominal ^{235}U abundances of 0.31, 0.71, 1.94, 2.95, and 4.46 mass percent, encased in aluminum cans that have been manufactured to rigid specifications (See attached specifications) and thoroughly tested. In addition an empty can is provided for use when measuring uranium materials of unknown ^{235}U abundances. SRM 969 was prepared as a set to permit measurement of materials containing uranium by using the theoretically expected linear relationship between ^{235}U abundance and the counting rate of the 185.7 keV gamma-ray of ^{235}U. Each SRM subunit is made up of 200 g of U_3O_8 powder. Since SRM 969 consists of 5 different containers, and each is unique in dimensions, the attached data sheets should be used with the specified container number. Individual data sheets are provided for each set along with the certificate.

The certified $^{235}U/U$ isotope abundances are shown in Table 1. The isotope abundances for $^{234}U/U$, $^{236}U/U$, and $^{238}U/U$ are given in Table 2.

Table 1

Certified $^{235}U/U$ Abundances in SRM 969

Material ID:	031	071	194	295	446
Atom Percent:	0.3206	0.7209	1.9664	2.9857	4.5168
	±0.0002	±0.0005	±0.0014	±0.0021	±0.0032
Mass Percent	0.3166	0.7119	1.9420	2.9492	4.4623
	±0.0002	±0.0005	±0.0014	±0.0021	±0.0032

Statement of Uncertainty

The overall uncertainty of the $^{235}U/U$ abundance of each individual reference sample was estimated by combining the different uncertainty components from the mass spectrometry measurements and the $^{235}U/U$ homogeneity. The resulting values were conservatively enlarged to 0.07% to include other possible measurement errors.

Isotope Certification: The uranium isotopic abundances were determined by thermal ionization mass spectrometry (THIMS) at the National Bureau of Standards (NBS) and by uranium hexafluoride mass spectrometry (UF_6 MS) and THIMS at the Central Bureau of Nuclear Measurements (CBNM). These measurements were corrected for mass discrimination effects relative to NBS uranium isotopic SRM's or synthetic isotope mixtures.

Additional measurements supporting the certification were made by the U.S. Department of Energy, New Brunswick Laboratory (NBL), using THIMS and by NBS using gamma spectrometry (see summary of the final results in Table 3).

Measurements by Gamma Spectrometry: The isotope abundance measurements and verification by gamma spectrometry were performed at NBS and CBNM utilizing the 185.7 keV gamma-ray of ^{235}U. All measurements were made using a high-resolution germanium detector. The heterogeneity of the $^{235}U/U$ in each SRM subunit is $\leq$0.05% relative.

June 27, 1985
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Table 2. $^{234}U/U$, $^{236}U/U$ and $^{238}U/U$, Abundances in SRM 969

Material		Uranium Isotopes		
		$^{234}U/U$	$^{236}U/U$	$^{238}U/U$
031	atom	0.0020	0.0147	99.6627
	2s	±0.0002	±0.0003	± 0.0004
	mass	0.0020	0.0146	99.6668
071	atom	0.0053	<0.00002	99.2738
	2s	±0.0002		± 0.0004
	mass	0.0052	<0.00002	99.2828
194	atom	0.0174	0.0003	98.0159
	2s	±0.0002	±0.0001	± 0.0018
	mass	0.0171	0.0003	98.0406
295	atom	0.0284	0.0033	96.9826
	2s	±0.0004	±0.0002	± 0.0029
	mass	0.0279	0.0033	97.0196
446	atom	0.0365	0.0069	95.4398
	2s	±0.0003	±0.0002	± 0.0032
	mass	0.0359	0.0068	95.4950

Measurements leading to the development and certification of this SRM were made at NBS in the Inorganic Analytical Research Division by B.S. Carpenter, J.W. Gramlich, R.R. Greenberg, and L.A. Machlan; at CBNM by E. Bouwmeester, R. Damen, P. De Biévre, W. De Bolle, H.L. Eschbach, R. Eyckens, M. Gallet, W. Lycke, H. Meyer, G. Müschenborn, W. Nagel, F. Quik, and J. Van Audenhove; at U.S. Department of Energy New Brunswick Laboratory (NBL), by V.E. Connolly and A.C. Zook in the Safeguards Assessment and Reference Materials Branch.

The sampling plan used for the certification measurements was developed by W. Liggett, NBS Center for Applied Mathematics. The statistical assessment of the data used for the certification of this SRM was performed by R. Werz, CBNM.

The overall scope and coordination of the technical measurements leading to certification were performed by B.S. Carpenter, NBS, and P. De Biévre, CBNM.

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.

Supplemental Information

Description of SRM 969

The uranium oxides used for this SRM are from the same lots of material used and jointly certified by CBNM and NBS to produce this SRM and the Certified Nuclear Reference Material (EC NRM 171) for the Commission of European Communities.

For unique identification and for checking the integrity of the subunit, the plugs used for sealing the cans are equipped with ultrasonic seals, each having a unique ultrasonic spectrum.

Parameters that are of special interest for abundance measurements by gamma spectrometry are summarized below:

Chemical Purity of the U_3O_8

Material:	U_3O_8 powder
Total impurities:	<0.05 mass% of U_3O_8 content
Moisture content:	<0.3 mass% of U_3O_8 content

Uranium Minor Isotopes (that could interfere with gamma spectrometry measurements)

(Note: Data is presented as a ratio of the minor isotope gamma-ray line intensity to ^{235}U gamma-ray line intensity)

Material ID	$^{232}U/^{235}U$	$^{233}U/^{235}U$	$(^{237}U + ^{237}Np)/^{235}U$	Chemical Separation Date
031	$8x10^{-10}$	$<5x10^{-5}$	$<3x10^{-6}$	1977
071	$<0.3x10^{-10}$	$<5x10^{-5}$	$<3x10^{-6}$	1977
194	$0.3x10^{-10}$	$<5x10^{-5}$	$<3.10^{-6}$	1977
295	$0.1x10^{-10}$	$<5x10^{-5}$	$<3x10^{-6}$	1977
446	$1x10^{-10}$	$<5x10^{-5}$	$<3x10^{-6}$	1979

U_3O_8 Filling Information

Material ID	Mass (g)	Filling Height (mm)	Diameter (mm)	U_3O_8 Density (g/cm^2)
031	200.1 ±0.2	20.8 ± 0.5	70.00	5.2 ± 0.3
071	200.1 ±0.2	20.8 ± 0.5	70.00	5.2 ± 0.3
194	200.1 ±0.2	20.8 ± 0.5	70.00	5.2 ± 0.3
295	200.1 ±0.2	20.8 ± 0.5	70.00	5.2 ± 0.3
446	200.1 ±0.2	15.8 ± 0.5	70.00	5.2 ± 0.3

Container Material Characteristics and Specifications

Container Material:	Aluminum type 6061 (ASTM-GS T6) (All containers manufactured from the same base material)		
Constituent Elements: (in Base Material, Wt.%)	Mg 0.8 - 1.2 Si 0.4 - 0.8 Cu 0.15 - 0.4	Zn $\leq$0.25 Ti $\leq$0.15 Fe $\leq$0.7	U $\leq$0.00025 Mn $\leq$0.15
	Cr 0.04 - 0.35	Total other Elements <0.15	

Container dimensions: See attached specifications

Use of SRM 969

Ideally, physical materials used for the evaluation of nondestructive measurements should be representative of the unknown samples with respect to all parameters that influence the measurement. One of the most crucial factors in gamma spectrometry is the strong attenuation of the gamma ray in the sample material itself and in the sample container. This attenuation is generally influenced by parameters such as sample size, shape, material density, and matrix composition. In addition, characteristics such as container material, wall thicknesses and container size can also influence attenuation.

SRM 969 is ideally suited for use with U_3O_8 materials contained in aluminum cans with 2mm bottom wall thickness. To be useful in the calibration of assay systems using other types of uranium samples, correction factors are needed. These factors are intended to normalize the gamma-ray response with respect to differences in both the matrix composition and the container. Typical correction factors are given in a special user's manual (Report KfK 3752(1984)) that has been prepared to facilitate the correct use of SRM 969 and EC NRM 171. Note: The attenuation correction factors given are based on theoretical values for photon cross sections. They represent a possible source of systematic error for those cases where the required corrections are large. Therfore, the correction given and the range of application should be experimentally validated to eliminate possible systematic errors from the gamma spectrometry abundance measurements.

Because the enrichment meter principle is based on the assumption that a sample is "quasi-infinitely thick" for the 185.7 keV gamma ray, the application of the method is inherently restricted to relatively large samples. About 200 g of unknown material is required when standard containers, with 7cm diameter, are used. For many applications it may be desirable to have reference samples that both physically and chemically differ from SRM 969. These reference samples can be calibrated against SRM 969.

Notice and Warnings

Container: The aluminum cans for SRM 969 have numbers engraved in the cylindrical wall. The numbering system on each can reflects the nominal isotope abundance of ^{235}U in mass percent and a sequence number. The cans within a set should have the same sequence number.

SRM 969 should be handled with great care to avoid any damage or deformation to the bottoms of the cans, since the bottoms serve as a window for the emitted gamma radiation. A special transport and storage case is supplied with this SRM.

Identification: A unique tamperproof system, making use of an ultrasonic "fingerprint" identification device, has been placed into the plug of each can.

Documentation

An NBS Special Publication, 260-96, and a European Commission Publication COM 4153, have been issued describing the preparation and characterization of this SRM, and should be used in conjunction with SRM 969/EC NRM 171. An additional User's Guide has been prepared and published as report KfK 3752.

Storage

The SRM subunits should be stored above −10 °C and below 40 °C.

Table 3. Summary of ^{235}U/U Isotope Abundance Measurements, in atom percent with 2s uncertainties, by Method and Laboratory

Material ID Laboratory/Method	031	071	194	295	446
CBNM-UF6	0.32049 ±0.00016	0.72096 ±0.00017	1.96575 ±0.00037	2.98432 ±0.00048	4.51668 ±0.00075
CBNM-THIMS	0.32061 ±0.00047	0.72119 ±0.00098	1.9675 ±0.0017	2.9875 ±0.0039	4.5201 ±0.0052
NBS-THIMS	0.32076 ±0.00052	0.72069 ±0.00068	1.9664 ±0.0017	2.9869 ±0.0022	4.5138 ±0.0033
NBL-THIMS	0.3203 ±0.0007	0.7207 ±0.0007	1.9657 ±0.0015	2.9843 ±0.0039	4.5162 ±0.0039
NBS-Gamma	0.32053 ±0.00026	0.72081 ±0.00058	1.9660 ±0.0016	2.9830 ±0.0024	4.5158 ±0.0036