



Designation: C 1295 – 98

Standard Test Method for Gamma Energy Emission from Fission Products in Uranium Hexafluoride and Uranyl Nitrate Solution¹

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1. Scope

1.1 This test method covers the measurement of gamma energy emitted from fission products in uranium hexafluoride (UF₆) and uranyl nitrate solution. It is intended to provide a method for demonstrating compliance with UF₆ Specifications C 787 and C 996 and replace the ion chamber measurement technique.

1.2 The lower limit of detection is 5000 MeV Bq/kg (MeV/kg per second) of uranium and is the square root of the sum of the squares of the individual reporting limits of the nuclides to be measured. The limit of detection was determined on a pure, aged natural uranium (ANU) solution. The value is dependent upon detector efficiency and background.

1.3 The nuclides to be measured are ¹⁰⁶Ru/¹⁰⁶Rh, ¹⁰³Ru, ¹⁴⁴Ce, ¹⁴⁴Pr, ¹⁴¹Ce, ⁹⁵Zr, ⁹⁵Nb, and ¹²⁵Sb. Other gamma energy-emitting fission nuclides that are identified should be measured and reported.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride²

C 787 Specification for Uranium Hexafluoride for Enrichment²

C 996 Specification for Uranium Hexafluoride Enriched to Less than 5 % ²³⁵U²

3. Summary of Test Method

3.1 A solution of the uranium sample is counted on a high-resolution gamma-ray spectroscopy system. The resulting

spectrum is analyzed to determine the identity and activity of the gamma-ray-emitting radioactive fission products. The number of counts recorded from one or more of the peaks identified with each nuclide is converted to disintegrations of that nuclide per second (Bq). The gamma-ray energy for a nuclide is calculated by multiplying the number of disintegrations per second of the nuclide by the mean gamma-ray energy emission rate of the nuclide. The calculated gamma-ray energy emission rates for all observed nuclides are summed, then divided by the mass of the uranium in the sample to calculate the overall rate of gamma energy production in units of million electron volts per second per kilogram of uranium.

4. Significance and Use

4.1 The fission products in UF₆ are identified and quantified using a high-resolution gamma-ray energy analysis system, which includes a high-resolution germanium detector.

5. Apparatus

5.1 High-Resolution Germanium Detector.

5.1.1 A relative efficiency of 10 % or higher (ratio of area under the 1.33-MeV photopeak of Cobalt-60 to that obtained with a 76- by 76-mm (3- by 3-in.) NaI(Tl) detector, at a source-to-detector distance of 25 cm).

5.1.2 Full width at half maximum (FWHM) resolution of 2.3 keV or less at 1.33 MeV.

5.2 *Gamma-Ray Analysis System*, containing the following components:

5.2.1 High voltage bias power supply.

5.2.2 Spectroscopy amplifier with peak shaping time constants of 1, 2 and 4 μs, baseline restoration, pulse pileup rejection, and live time correction.

5.2.3 Multichannel analyzer with a minimum of 4096 data channels and circuits compatible with the pulse pileup rejection and live time correction circuits of the spectroscopy amplifier.

5.2.4 Computer system and input/output (I/O) devices for storage of calibration data, control of analyzer functions, calculation of results, and report generation.

5.3 *Heavy Metal Shield*, for the detector and sample.

¹ This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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² *Annual Book of ASTM Standards*, Vol 12.01.

5.4 *Sample Container Fitted with Cap*, internal diameter 56 mm, outside diameter 59 mm, and height 65 mm. The dimensions must be consistent between sample containers to keep the counting geometry constant.

5.5 *Sample Holder*, shall accommodate the sample container and position it so that the detector view of the sample container is reproducible. To minimize the effects of coincident summing, the sample holder shall provide a minimum separation of 5 mm between the sample container and the detector end cap.

6. Calibration and Standardization of Detector

6.1 Prepare a mixed radionuclide calibration standard stock solution³ covering the energy range of 5 to 2000 keV.

6.2 Prepare a solution of ANU at 6.74 gU/100 g. The uranium and its daughter products' relationship must not have been altered for at least eight months.

6.3 Transfer a known, suitable activity of the mixed nuclide calibration standard stock solution (40 to 50 kBq) to a container identical to that used for the sample measurement. Add ANU solution to the mixed nuclide standard so that the final volume and uranium concentration match those expected in the sample measurement. This test method is used to calibrate the energy scale of the detector and determine the counter efficiency.

6.4 The detector energy scale and efficiency are calibrated by placing the container with the mixed nuclide calibration standard in a sample holder that provides a reproducible geometry relative to the detector. Collect a spectrum over a period up to 1 h that includes all the gamma photopeaks in the energy range up to 2000 keV. All counting conditions (except count time) must be identical to those that will be used for analysis of the actual sample.

6.5 Determine the net counts under each peak of every nuclide in the mixed radionuclide standard, then divide by the count time (live time) to determine the rate in counts per second for each radionuclide. If a background count on the detector shows any net peak area for the peaks of interest, these must be subtracted from the standard counts per second.

6.6 Divide the observed count rate determined for each gamma peak by the calculated emission rate of the gamma ray that produced the peak in the mixed calibration standard (gammas per second).

6.6.1 Calculation of the gamma emission rate for each peak from the mixed calibration standard must account for the following:

6.6.1.1 Activity of the nuclide that produces the peak in its original standard (disintegrations/second/unit volume). This is taken from the standard certificate of measurement supplied with the standard.

6.6.1.2 Volume of each isotopic standard taken for the mixed standard and the final volume of the mixed standard.

6.6.1.3 Volume of the mixed standard taken for counting.

³ The mixed radionuclide calibration standard QCY48 from Amersham International, White Lion Road, Amersham, Buckinghamshire, HP7 9LL, England, has been found to be suitable.

6.6.1.4 Decay of the activity of the standard between its date of characterization and the date of counting according to the equation:

$$A = A_0 e^{-\lambda t} \quad (1)$$

where:

A = activity on the date of counting disintegration/s;

A_0 = activity on the date of standard characterization in disintegration/s;

λ = decay constant in units of time^{-1} , values from Table 1; and

t = time between calibration reference date and the date of counting. Time units must be the same as the decay constant.

6.6.1.5 The abundance of gamma rays of the energy of interest emitted by each disintegration (see Table 1).

6.7 Plot a detector efficiency curve of counts/gamma versus gamma energy. Some multichannel analyzers and data reduction programs may be able to store individual values from this curve or the equation of the curve for later use.

6.8 This efficiency calibration will remain valid provided none of the sample or instrument parameters are changed (for example, volume of sample, container geometry, distance from detector, and detector) and instrument response to the control standard remains within the statistical limits established.

7. Measurement of Control Standard Solution

7.1 Measure the control standard solution prepared in 6.3 with the geometry as used during detector efficiency calibration. Ten measurements of the control standard solution are made. The calculated data for the fission products is used to establish precision and bias of the test method.

7.1.1 Many multichannel analyzers have automatic routines for determining the net counts under single peaks and double peaks that are not resolved. If the available analyzer does not have such capabilities, refer to Reilly⁴ for single-peak analysis methods and 7.2.1 and 7.2.2 for double-peak problems that are likely to be encountered.

⁴ Reilly, T. D., and Parker, J. L., *A Guide to Gamma Ray Assay for Nuclear Materials Accountability*, LA-5794M, Los Alamos National Laboratory, 1975.

TABLE 1 Gamma-Ray-Emitting Fission Products Found in UF₆

Nuclide	Half-Life	Decay Constant	Measurement Peaks, MeV	Abundance Gamma/Disintegration	Mean Gamma Energy Disintegration, MeV
¹⁰³ Ru/ ¹⁰³ Rh	39.35d	0.01761/d	0.4971 0.6103	0.889 0.056	0.497
¹⁰⁶ Ru/ ¹⁰⁶ Rh	366.5d	0.001891/d	0.5119 0.6222	0.207 0.0981	0.209
¹⁴¹ Ce	32.55d	0.02129/d	0.1454	0.484	0.0765
¹⁴⁴ Ce/ ¹⁴⁴ Pr	284.5d	0.002436/d	0.1335	0.1110	0.0479
¹³⁴ Cs	2.062y	0.3362/y	0.7958	0.851	1.554
¹³⁷ Cs/ ¹³⁷ Ba	30.17y	0.02297/y	0.6616	0.899	0.597
⁹⁵ Nb	34.97d	0.01982/d	0.7658	1.000	0.766
⁹⁵ Zr	63.98d	0.01083/d	0.7242 0.7567	0.444 0.549	0.737
¹²⁵ Sb	2.71y	0.256/y	0.4279 0.6008	0.294 0.178	0.433