



Standard Test Method for Gamma Energy Emission from Fission and Decay 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 (UF_6) and uranyl nitrate solution. This test method may also be used to measure the concentration of some uranium decay products. It is intended to provide a method for demonstrating compliance with UF_6 specifications C787 and C996, uranyl nitrate specification C788, and uranium ore concentrate specification C967.

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 fission product nuclides to be measured are ^{106}Ru / ^{106}Rh , ^{103}Ru , ^{137}Cs , ^{144}Ce , ^{144}Pr , ^{141}Ce , ^{95}Zr , ^{95}Nb , and ^{125}Sb . Among the uranium decay product nuclides that may be measured is ^{231}Pa . Other gamma energy-emitting fission and uranium decay nuclides present in the spectrum at detectable levels should be identified and quantified as required by the data quality objectives.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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.*

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

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2. Referenced Documents

2.1 ASTM Standards:²

- C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride
- C787 Specification for Uranium Hexafluoride for Enrichment
- C788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals
- C859 Terminology Relating to Nuclear Materials
- C967 Specification for Uranium Ore Concentrate
- C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U
- C1022 Test Methods for Chemical and Atomic Absorption Analysis of Uranium-Ore Concentrate
- D3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water
- E181 Test Methods for Detector Calibration and Analysis of Radionuclides

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminology C859.

4. Summary of Test Method

4.1 A solution of the uranium sample is counted on a high-resolution gamma-ray spectrometry system. The resulting spectrum is analyzed to determine the identity and activity of the gamma-ray-emitting radioactive fission and decay products. The number of counts recorded from one or more of the peaks identified with each fission nuclide is converted to disintegrations of that nuclide per second (Bq). The gamma-ray energy for a fission nuclide is calculated by multiplying the number of disintegrations per second of the nuclide by the

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

mean gamma-ray energy emission rate of the nuclide. The calculated gamma-ray energy emission rates for all observed fission 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. Decay product nuclides such as ^{231}Pa will be separately quantified and reported based on specific needs.

5. Significance and Use

5.1 Specific gamma-ray emitting radionuclides in UF_6 are identified and quantified using a high-resolution gamma-ray energy analysis system, which includes a high-resolution germanium detector. This test method shall be used to meet the health and safety specifications of C787, C788, and C996 regarding applicable fission products in reprocessed uranium solutions. This test method may also be used to provide information to parties such as conversion facilities on the level of uranium decay products in such materials. Pa-231 is a specific uranium decay product that may be present in uranium ore concentrate and is amenable to analysis by gamma spectrometry.

6. Apparatus

6.1 *High-Resolution Gamma-Ray Spectrometry System*, as specified in Practice D3649. The energy response range of the spectrometry system may need to be tailored to address all the needed fission and uranium decay product nuclides that need to be analyzed for.

6.2 *Sample Container with Fitted Cap*—A leak-proof plastic container capable of holding the required sample volume. The dimensions must be consistent between containers used for samples and standard to keep the counting geometry constant. The greatest detection efficiency will be achieved with a low-height sample container with a diameter slightly smaller than the detector being used.

6.3 *Sample Holder*, shall be used to position the sample container such that the detector view of the sample is reproducible. To reduce 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.

7. Calibration and Standardization of Detector

7.1 Prepare a mixed radionuclide calibration standard stock solution covering the energy range of approximately 50 to 2000 keV.

7.1.1 Commercial calibration standards are available which are traceable to NIST or other national standards laboratories.

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

7.3 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. Test Methods E181 and Practice D3649 provides information on calibration of detector energy, efficiency, resolution, and other parameters.

7.4 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 duration) must be identical to those that will be used for analysis of the actual sample.

7.5 Determine the net counts under each peak of every nuclide in the mixed radionuclide standard, then divide by the count duration (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.

7.6 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).

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

TABLE 1 Gamma-Ray-Emitting Fission and Decay Products Found in UF_6

| Nuclide | Half-Life | Decay Constant (λ_i) | Measurement Peaks, MeV | Abundance Gamma/Disintegration (G_i) | Mean Gamma Energy Disintegration, MeV Bq (E_i) |
|-----------------------------------|-----------|--------------------------------|------------------------|--|--|
| $^{103}\text{Ru}/^{103}\text{Rh}$ | 39.35d | 0.01761/d | 0.4971 0.6103 | 0.889 0.056 | 0.484 |
| $^{106}\text{Ru}/^{106}\text{Rh}$ | 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 | 0.0718 |
| $^{144}\text{Ce}/^{144}\text{Pr}$ | 284.5d | 0.002436/d | 0.1335 | 0.1110 | 0.0518 |
| $^{137}\text{Cs}/^{137}\text{Ba}$ | 30.17y | 0.02297/y | 0.6616 | 0.851 | 0.5655 |
| ^{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 |
| ^{231}Pa | 32760y | 2.1158E-05/y | 0.002736 | 0.103 | n/a |