Designation: E692 – 08

StandardTest Method for Determining the Content of Cesium-137 in Irradiated Nuclear Fuels by High-Resolution Gamma-Ray Spectral Analysis[1]

This standard is issued under the fixed designation E692; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the number of atoms of ^{137}Cs in aqueous solutions of irradiated uranium and plutonium nuclear fuel. When combined with a method for determining the initial number of fissile atoms in the fuel, the results of this analysis allows atom percent fission (burn-up) to be calculated (**1**).[2] The determination of atom percent fission, uranium and plutonium concentrations, and isotopic abundances are covered in Test Methods E267 and E321.

1.2 ^{137}Cs is not suitable as a fission monitor for samples that may have lost cesium during reactor operation. For example, a large temperature gradient enhances ^{137}Cs migration from the fuel region to cooler regions such as the radial fuel-clad gap, or, to a lesser extent, towards the axial fuel end.

1.3 A nonuniform ^{137}Cs distribution should alert the analyst to the potential loss of the fission product nuclide. The ^{137}Cs distribution may be ascertained by an axial gamma-ray scan of the fuel element to be assayed. In a mixed-oxide fuel, comparison of the ^{137}Cs distribution with the distribution of non-migrating fission-product nuclides such as ^{95}Zr or ^{144}Ce would indicate the relative degree of ^{137}Cs migration.

1.4 The values stated in SI units are to be regarded as standard. No other unites 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E170 Terminology Relating to Radiation Measurements and Dosimetry

E181 Test Methods for Detector Calibration and Analysis of Radionuclides

E219 Test Method for Atom Percent Fission in Uranium Fuel (Radiochemical Method) (Discontinued 2001) (Withdrawn 2001)[4]

E267 Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances

E321 Test Method for Atom Percent Fission in Uranium and Plutonium Fuel (Neodymium-148 Method)

3. Summary of Test Method

3.1 ^{137}Cs is assayed by measuring the 662[5] keV gamma-ray emission rate from the isomeric transition of its metastable 2.6[6] min ^{137m}Ba daughter, using a high-resolution germanium detector and multichannel pulse-height analyzer. Refer to Test Methods E181.

3.2 The number of atoms of ^{137}Cs in a sample is computed from the measured net gamma-ray count rate relative to the measured net gamma-ray count rate from a standard ^{137}Cs solution.

4. Significance and Use

4.1 This test method uses a high-resolution gamma-ray spectrometer as a basis for measuring the gamma-ray emission

[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] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[3] 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.

[4] The last approved version of this historical standard is referenced on www.astm.org.

[5] The energy of the gamma ray is more precisely given in Ref (**2**) as 661.637 keV. For simplicity, all citations of this energy in this standard will be given as 662 keV.

[6] The half-life of this state is more precisely given in Ref (**3**) as 2.552 min. For simplicity, all citations of this half-life listed in this standard will be given as 2.6 min.

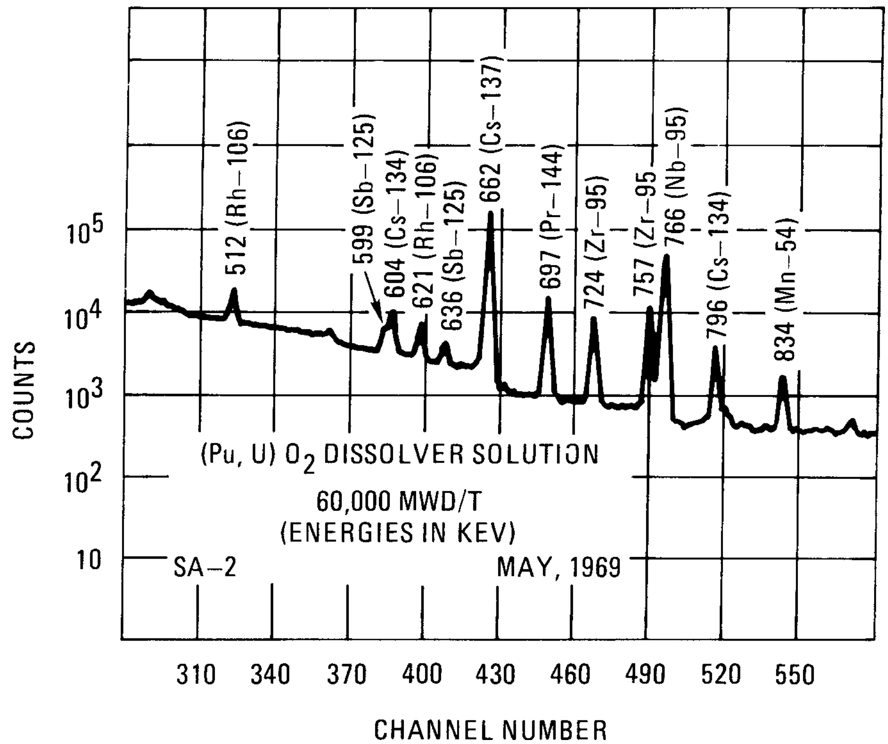

FIG. 1 Gamma-Ray Spectrum of a (Pu,U)O_2 Fuel, Irradiated to 6 Atom % Fission, and Decayed for Seven Months

rate of ^{137}Cs-^{137m}Ba in a dilute nitric acid solution containing 10 mg/L of cesium carrier. No chemical separation of the cesium from the dissolved-fuel solution is required. The principal steps consist of diluting a weighed aliquot of the dissolved-fuel solution with a known mass of 1 *M* nitric acid (HNO_3) and measuring the 662 keV gamma-ray count rate from the sample, then measuring the 662 keV gamma-ray count rate from a standard source that has the same physical form and counting geometry as the sample.

4.2 The amount of fuel sample required for the analysis is small. For a sample containing 0.1 g of fuel irradiated to one atom percent fission, a net count rate of approximately 10^5 counts per second will be observed for a counting geometry that yields a full-energy peak efficiency fraction of 1×10^{-3}. The advantage of this small amount of sample is that the concentration of fuel material can be kept at levels well below 1 g/L, which results in negligible self-absorption in the sample aliquot and a small radiation hazard to the analyst.

5. Precautions

5.1 Interferences from other gamma-ray emitting fission products are lessened by the use of a germanium detector with a minimum resolution of 3 keV full-width at half-maximum (FWHM) at 1332 keV, and by allowing four months or more for the sample to decay prior to measurement (**4**). Under these conditions, the gamma rays nearest to the 662 keV gamma ray of ^{137m}Ba will be the 637 keV gamma ray of ^{125}Sb and the 697 keV gamma ray of ^{144}Pr.

5.2 A slight complication of this test method is that the 662 keV gamma ray is superimposed on the Compton edge (defined in Terminology E170) from the 766 keV gamma ray of ^{95}Nb and from the 796 keV gamma ray of ^{134}Cs, as shown in Fig. 1.

5.3 This test method requires accurately weighing an aliquot of the sample of fuel material containing sufficient ^{137}Cs-^{137m}Ba activity into a sample vial. In order to achieve the precision of which this test method is capable, the analyst should exercise great care when preparing the sample. To reduce the uncertainty associated with the sample quantity, aliquots should be prepared by weighing to an accuracy of better than 0.2 %. Weighing also reduces the calculation task, because in the case of burn-up analysis, the quantity of heavy-element atoms in the sample will have been determined on a mass-aliquot basis. The aliquot of sample solution should contain a weight of fuel sample of not less than 0.1 g weighed to an accuracy of 0.01 mg, and is to be diluted to the same total mass as the working standard. Uncertainties caused by slight variations in the counting-geometry among samples are negligible provided that the masses of the individual diluted samples are within ±0.01 g of each other.

5.4 The preparation of the ^{137}Cs reference standard should receive particular attention. Preferably, the number of ^{137}Cs atoms per gram of standard should have been determined by isotope-dilution mass spectrometry. An aliquot of not less than 0.1 g of the standard solution, weighed to ±0.1 mg, is diluted to total mass of 10.00 g with 1 *M* HNO_3 in a sample vial, which is then flame-sealed. A series of working standards with different concentrations should be prepared so that a working standard may be selected that will have approximately the same number of ^{137}Cs atoms as the sample to be measured.