



Standard Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances¹

This standard is issued under the fixed designation E 267; 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 is applicable to the determination of uranium (U) and plutonium (Pu) concentrations and their isotopic abundances (Note 1) in solutions which result from the dissolution of nuclear reactor fuels either before or after irradiation. A minimum sample size of 50 μg of irradiated U will contain sufficient Pu for measurement and will minimize the effects of cross contamination by environment U.

NOTE 1—The isotopic abundance of ^{238}Pu can be determined by this test method; however, interference from ^{238}U may be encountered. This interference may be due to (1) inadequate chemical separation of uranium and plutonium, (2) uranium contamination within the mass spectrometer, and (3) uranium contamination in the filament. One indication of uranium contamination is a changing 238/239 ratio during the mass spectrometer run, in which case, a meaningful ^{238}Pu analysis cannot be obtained on that run. If inadequate separation is the problem, a second pass through the separation may remove the uranium. If contamination in the mass spectrometer or on the filaments is the problem, use of a larger sample, for example, 1 μg , on the filament may ease the problem. A recommended alternative method of determining ^{238}Pu isotopic abundance without ^{238}U interference is alpha spectroscopy using Practice D 3084. The ^{238}Pu abundance should be obtained by determining the ratio of alpha particle activity of ^{238}Pu to the sum of the activities of ^{239}Pu and ^{240}Pu . (1)² The contribution of ^{239}Pu and ^{240}Pu to the alpha activity differs from their isotopic abundances due to different specific activities.

1.2 The procedure is applicable to dissolver solutions of uranium fuels containing plutonium, aluminum, stainless steel, or zirconium. Interference from other alloying constituents has not been investigated and no provision has been made in the test method for fuels used in the ^{232}Th - ^{233}U fuel cycle.

1.3 *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:

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.

Current edition approved July 27, 1990. Published December 1990. Originally published as E 267 – 65 T. Last previous edition E 267 – 78(1985)¹.

² The boldface numbers in parentheses refer to the list of references appended to this test method.

D 1193 Specification for Reagent Water³

D 3084 Practice for Alpha-Particle Spectrometry of Water⁴

E 137 Practice for Evaluation of Mass Spectrometers for Quantitative Analysis from a Batch Inlet⁵

E 219 Test Method for Atom Percent Fission in Uranium Fuel (Radiochemical Method)⁶

E 244 Test Method for Atom Percent Fission in Uranium and Plutonium Fuel (Mass Spectrometric Method)⁶

3. Summary of Test Method

3.1 An aliquot of solution to be analyzed is spiked with known amounts of ^{233}U and ^{242}Pu (2–6). U and Pu are separated by ion exchange and analyzed mass spectrometrically.

4. Significance and Use

4.1 This test method is specified for obtaining the atom ratios and U atom percent abundances required by Test Method E 244 and the U concentration required by Test Method E 219.

NOTE 2—The isotopic abundance of ^{238}Pu normally is not required for burnup analysis of conventional light-water reactor fuel.

4.2 The separated heavy element fractions placed on mass spectrometric filaments must be very pure. The quantity required depends upon the sensitivity of the instrument detection system. If a scintillator (7) or an electron multiplier detector is used, only a few nanograms are required. If a Faraday cup is used, a few micrograms are needed. Chemical purity of the sample becomes more important as the sample size decreases, because the ion emission of the sample is repressed by impurities.

4.3 Operation at elevated temperature (for example, 50 to 60°C) (Note 3) will greatly improve the separation efficiency of ion exchange columns. Such high-temperature operation yields an iron-free U fraction and U-free Pu fraction, each of which has desirable emission characteristics.

NOTE 3—A simple glass tube column can be heated by an infrared lamp until it is warm to the touch.

4.4 Extreme care must be taken to avoid contamination of

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 11.02.

⁵ Annual Book of ASTM Standards, Vol 05.03.

⁶ Annual Book of ASTM Standards, Vol 12.02.