Designation: C1415 – 18

Standard Test Method for ^{238}Pu Isotopic Abundance By Alpha Spectrometry[1]

1. Scope

1.1 This test method covers the use of alpha spectrometry for determining the ^{238}Pu isotopic abundance in plutonium samples. It is particularly useful for samples in which the ^{238}Pu content is less than 1 % of the total plutonium content. For such samples, mass spectrometric results are vulnerable to bias because of potential interference from any ^{238}U isobar remaining after ion exchange.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets
C859 Terminology Relating to Nuclear Materials
C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis
C1411 Practice for The Ion Exchange Separation of Uranium and Plutonium Prior to Isotopic Analysis
C1625 Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances by Thermal Ionization Mass Spectrometry
C1672 Test Method for Determination of Uranium or Plutonium Isotopic Composition or Concentration by the Total Evaporation Method Using a Thermal Ionization Mass Spectrometer
C1816 Practice for The Ion Exchange Separation of Small Volume Samples Containing Uranium, Americium, and Plutonium Prior to Isotopic Abundance and Content Analysis
D1193 Specification for Reagent Water

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 This determination method involves the measurement of the alpha-activity ratio of ^{238}Pu and ^{239}Pu + ^{240}Pu. The isotopic analysis of plutonium for the ^{238}Pu isotope requires the prior separation of potentially interfering species. After dissolution of the sample (see Practice C1168), the plutonium is separated from interferences by purification techniques such as given in this test method or in Practices C1411 or C1816.

4.2 In this test method, nitric acid (HNO_3) is used to adsorb the plutonium fraction on the resin. Interfering ions, principally uranium and americium, are not adsorbed. The plutonium is then eluted with dilute HNO_3 (0.1 *M*).

4.3 Because an alpha-activity ratio is used, quantitative recovery of the plutonium is not required. The alpha spectrum in the 5 to 6-MeV region is obtained. The total counts in the ^{238}Pu and the ^{239}Pu + ^{240}Pu peaks are obtained and corrected for background. The ^{238}Pu abundance is calculated from the ratio of the alpha activity due to ^{238}Pu and that due to ^{239}Pu + ^{240}Pu. The abundance of ^{239}Pu and ^{240}Pu is determined by mass spectrometry (see Test Methods C697, C1625, or C1672) on a separate portion of the purified sample.

5. Significance and Use

5.1 This test method is used when the determination of ^{238}Pu isotopic abundance is required for plutonium samples.

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[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] 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.

6. Interferences

6.1 Am-241 is always present as a result of ^{241}Pu decay and is a direct interference that must be removed prior to the determination of ^{238}Pu. The very small amount of ^{230}Th and ^{232}Th which could be present causes insignificant interference with the determination of ^{238}Pu at the level of uncertainty of this test method. Other nuclides that could interfere, such as ^{238}U, ^{243}Am, ^{245}Cm, and ^{249}Bk, are removed by purification prior to analysis. Any residual uranium, while it does not directly interfere with the alpha-pulse height determination, can raise the salt content of the sample. A high salt content can decrease the resolution of the alpha spectra, and, consequently, decrease the accuracy of the test method.

7. Apparatus

7.1 *Counting disks of polished platinum, tantalum, or stainless steel,* sized to fit the detection chamber. A disk 25 mm in diameter and 0.5 mm thick has been found to be acceptable.

7.2 *Alpha spectrometer.* This instrument may consist of the following individual components but more typically as an integrated system that is readily interfaced to a computer:

7.2.1 *Silicon based alpha detector,* with an active area of at least 100 mm^2, a depletion depth of 100 µm or more, and a resolution of 50 keV or less full width at half maximum (FWHM) (for ^{241}Am 5.486 MeV alpha).

NOTE 1—A FWHM of 30 keV or less is desirable, but a FWHM up to 50 keV can be tolerated.

7.2.2 *Evacuable, light-tight chamber* in which the detector and the counting plate on its support can be mounted.

7.2.3 *Preamplifier (charge-sensitive field-effect transistor)* with noise less than 4.6 keV when used with above detector (100 pF capacitance).

7.2.4 *Detector bias supply,* 0 to 150 V, continuously variable, well-regulated and stable, with noise and ripple less than 0.0002 %.

7.2.5 *Main spectroscopy amplifier,* low noise, with variable shaping constants and baseline restoration.

7.2.6 *Biased amplifier and pulse stretcher,* with continuously adjustable post-gain and automatic pile-up rejection.

7.2.7 *Multichannel pulse-height analyzer.* A multichannel analyzer is most versatile and convenient, since it can be used for the acquisition of data from one to four detectors, simultaneously. Even if only one detector is used, such an analyzer has the advantages that background may be stored in another subgroup and subtracted electronically from the spectrum of interest, and that several spectra can be stored and compared. An analyzer that permits the analyst to set windows around the peaks of interest and perform electronic integration is especially convenient. The analyzer should accept pulses 0 to 10 V and 3 to 6 µs in width and should have a capacity of at least 10^6 counts full scale per channel.

7.3 *Heat lamp.*

7.4 *Bunsen burner,* or similar heat source device.

7.5 *Sample beaker,* 30-mL, borosilicate glass.

7.6 *Bottles or vials.*

7.7 *Hot plate.*

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals are used in all tests and conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[3]

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled or deionized water in conformance with Specification D1193, Type I.

8.3 *Nitric acid (HNO_3).* Concentrated (sp gr 1.42).

8.4 *Nitric acid, 4 M.* Add 250 mL of nitric acid (sp gr 1.42) to <750 mL of water and dilute to 1 L.

8.5 *Nitric acid, 0.1 M.* Add 6.2 mL of nitric acid (sp gr 1.42) to <950 mL of water and dilute to 1 L.

8.6 *Anion-exchange resin and column,* 74-149 µm (100-200 mesh) dry size, containing quaternary ammonium functional groups.

9. Calibration of the Alpha Spectrometer

9.1 *Initial Calibration*—Set the amplifier so that channel zero is about 4.75 MeV. Use a standard alpha source such as the 5.30 MeV particle of ^{210}Po, or the 5.49 MeV particle of ^{241}Am, for energy calibration. Establish the system gain at some convenient value that will cover the needed energy range.

9.2 *Resolution*—Count the standard source and determine the energy span at half the peak height. A full width half maximum of 30 keV or less is desirable, but a FWHM up to 50 keV can be tolerated.

9.3 *Background*—Obtain a background spectrum with a clean counting disk in the chamber.

9.4 *Frequency of Calibration*—The energy calibration and energy resolution should be checked periodically, to maintain the same operating conditions and to check whether the spectrometer has deteriorated. Daily or similarly frequent background counting is advisable, to ensure that neither the chamber nor the detector has been overly contaminated and that an appropriate background correction is accomplished.

10. Procedure

NOTE 2—Procedure steps 10.2 through 10.7 were developed based on pre-packed columns containing 2 mL of Bio-Rad AG 1-X8 anion exchange resin. Other resins with similar characteristics (for example, those with quaternary ammonium functional groups and a high degree of crosslinkage) can be used. Adjustments in acid concentration, or volume, or both, as described in these steps may be required.

NOTE 3—Purification as described in Practices C1411 or C1816 or using extraction chromatography resins may be used as an alternative to steps 10.1 through 10.7 for separation of plutonium from uranium and other impurities prior to alpha spectrometry.

10.1 Transfer an aliquot of approximately 1 mg Pu from a sample obtained from Practice C1168 or equivalent procedure into a 30-mL beaker and take to dryness on a hot plate.

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd. Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.