



Designation: **C1415 – 01a (Reapproved 2007) C1415 – 14**

Standard Test Method for ^{238}Pu Isotopic Abundance By Alpha Spectrometry¹

This standard is issued under the fixed designation C1415; 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 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 ~~less reliable than those from alpha spectrometry because of 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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](#)

[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](#)

[D1193 Specification for Reagent Water](#)

3. Terminology

3.1 For definitions of pertinent terms not listed here, see Terminology [C859](#).

4. Summary of Test Method

4.1 This determination method involves the measurement of the alpha-activity ratio of ^{238}Pu and $^{239}\text{Pu} + ^{240}\text{Pu}$. The isotopic analysis of plutonium for the ^{238}Pu isotope requires the prior separation of ~~interferences, potentially interfering species.~~ After dissolution of the sample (see Practice [C1168](#)), the plutonium is separated from interferences by an anion-exchange purification technique. 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). 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 $^{239}\text{Pu} + ^{240}\text{Pu}$ 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 $^{239}\text{Pu} + ^{240}\text{Pu}$. 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 ~~determined~~ determination of ^{238}Pu isotopic abundance is required for plutonium samples.

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 Feb. 15, 2007; June 1, 2014. Published April 2007; July 2014. Originally approved in 1999. Last previous edition approved in 2004; 2007 as [C1415 – 01a](#); [C1415 – 01a \(2007\)](#). DOI: [10.1520/C1415-01AR07-10.1520/C1415-14](#).

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 ~~²⁴¹Am~~²⁴¹Am-241 is always present as a result of ²⁴¹Pu decay and is a direct interference that must be removed prior to the determination of ²³⁸Pu. The very small amount of ²³⁰Th and ²³²Th which could be present causes insignificant interference with the determination of ²³⁸Pu at the level of uncertainty of this test method. ~~Other nuclides that would interfere, such as~~ Other nuclides that could interfere, such as ²³⁸U, ²⁴³Am, ²⁴⁵Cm, and ²⁴⁹Bk, are removed by the anion-exchange separation. 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 sensitivity ~~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 ~~should typically may~~ consist of the following ~~components: individual components but~~ more typically as an integrated system that is readily interfaced to a computer:

7.2.1 *Silicon surface barrier detector*, with an active area of at least 100 mm², a depletion depth of greater than 100 μm, and a resolution of 30 keV or less full width at half maximum (FWHM) (for ²⁴¹Am 5.486 MeV alpha).

7.2.2 *Evacuatable, 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~~ 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 10⁶ counts full scale per channel.

6.2.8 ~~A computer printer~~ for output printing of the data from the multichannel analyzer (optional).

7.3 *Heat lamp*.

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

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

7.6 *Bottles or vials*.

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 (Specification **D1193**).

8.3 *Nitric acid (HNO₃)*. 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*,⁴ 100-200 mesh, containing quaternary ammonium ~~function~~ functional groups.

9. Calibration of the Alpha Spectrometer

9.1 ~~Initial Alignment~~ ~~=~~ 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 ²¹⁰Po, or the 5.49 MeV particle of ²⁴¹Am, for energy calibration. Establish the system gain at some convenient ~~value, such as 5 keV per channel.~~ value that will cover the needed energy range.

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.

⁴ Disposable, prefilled columns packed with AG 1-X 8, available from Bio-Rad, Richmond, CA, have been found to be acceptable.