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Designation: C1415 - 01a (Reapproved 2007)

StandardTest Method for ²³⁸Pu 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 ²³⁸Pu isotopic abundance in plutonium samples. It is particularly useful for samples in which the ²³⁸Pu 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 interference from any ²³⁸U isobar remaining after ion exchange.

1.2 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
- C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis
- D1193 Specification for Reagent Water

13. Summary of Test Method g/standards/sist/542690e0-b6a

3.1 This determination method involves the measurement of the alpha-activity ratio of ²³⁸Pu and ²³⁹ Pu + ²⁴⁰Pu. The isotopic analysis of plutonium for the ²³⁸Pu isotope requires the prior separation of interferences. After dissolution of the sample (see Practice C1168), the plutonium is separated from interferences by an anion-exchange purification technique. Nitric acid (HNO₃) 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 HNO₃ (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 ²³⁸Pu and the ²³⁹Pu + ²⁴⁰Pu peaks are obtained and corrected for background. The ²³⁸Pu abundance is calculated from the ratio of the alpha activity due to ²³⁸Pu and that due to ²³⁹Pu + ²⁴⁰Pu. The abundance of ²³⁹Pu and ²⁴⁰Pu is determined by mass spectrometry (see Test Methods C697) on a separate portion of the purified sample.

4. Significance and Use

4.1 This test method is used when the determined of ²³⁸Pu isotopic abundance is required for plutonium samples.

5. Interferences

5.1 ²⁴¹Am 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 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 of the test method.

6. Apparatus

6.1 *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.

6.2 *Alpha spectrometer*. This instrument should typically consist of the following components:

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

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

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

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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.

Current edition approved Feb. 15, 2007. Published April 2007. Originally approved in 1999. Last previous edition approved in 2001 as C1415 – 01a. DOI: 10.1520/C1415-01AR07.

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.