



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

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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 interference from any ^{238}U 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:

C 697 Test Method for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets²

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis²

D 1193 Specification for Reagent Water³

3. Summary of Test Method

3.1 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. After dissolution of the sample (see Practice C 1168), 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 is determined by mass spectrometry (see Test Methods C 697) on a separate portion of the purified sample.

4. Significance and Use

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

5. Interferences

^{241}Am 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 would interfere, such as ^{238}U , ^{243}Am , ^{245}Cm , and ^{249}Bk , 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 μm , and a resolution of 30 keV or less full width at half maximum (FWHM) (for ^{241}Am 5.486 MeV alpha).

6.2.2 *Evacuatable, 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).

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

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

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

6.2.7 *Multichannel pulse-height analyzer*. A multichannel

¹ This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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² Annual Book of ASTM Standards, Vol 12.01.

³ Annual Book of ASTM Standards, Vol 11.01.