



Standard Guide for Determination of Plutonium and Neptunium in Uranium Hexafluoride by Alpha Spectrometry¹

This standard is issued under the fixed designation C 1561; 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 method covers the determination of plutonium and neptunium isotopes in uranium hexafluoride by alpha spectroscopy. The method could also be applicable to any matrix that may be converted to a nitric acid system.

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 requirements prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 787 Specification for Uranium Hexafluoride for Enrichment²

C 996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U ²

C 1163 Test Method for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride²

C 1475 Guide for the Determination of Neptunium-237 in Soil²

D 1193 Specification for Reagent Water³

D 3084 Practice for Alpha-Particle Spectrometry of Water⁴

D 3648 Practices for the Measurement of Radioactivity⁴

3. Terminology

3.1 *reagent blank*—DI water processed the same as the samples; used in the determination of the minimum detectable activity.

3.2 *region-of-interest (ROI)*—the channels, or region, in the alpha spectra in which the counts due to a specific radioisotope appear on a functioning calibrated alpha spectrometry system.

4. Summary of Test Method

4.1 An aliquot of hydrolyzed uranium hexafluoride equivalent to approximately 0.5 g of uranium is converted to an oxalic acid-nitric acid system and the uranium is selectively removed via solid phase extraction. Plutonium and neptunium are further purified by additional solid phase extractions. The plutonium and neptunium are then co-precipitated with neodymium as the fluorides and counted by alpha spectrometry.

4.2 Tracer recoveries using this method are typically between 75 and 90 %. The resolution of the tracer is typically less than 40 keV full-width at half-maximum.

4.3 The minimum detectable activity will vary with tracer recovery, sample size, instrument background, and counting efficiency.

5. Significance and Use

5.1 The method is applicable to the analysis of materials to demonstrate compliance with the specifications set forth in Specifications C 787 and C 996.

6. Interferences

6.1 Incomplete removal of U-234 from the neptunium fraction could result in a false positive for the Np-237 analysis. The method has been shown to adequately remove uranium at enrichments up to 5 %. If the method is used for the analysis of materials at greater than 5 % enrichment, a blank consisting of uranium at the same enrichment as the samples should be analyzed to show adequate removal of the U-234.

6.2 A Pu tracer is used to monitor the chemical recovery of the Np. Spiked analyses should be performed to confirm the appropriateness of this correction; fractionation of Np and Pu during the separation could lead to incorrect test results.

7. Instrumentation

7.1 *Alpha Spectrometry System*—See Practices D 3084 and D 3648 for a description of the apparatus.

8. Apparatus

8.1 *Ion Exchange Columns*, able to hold a 10 mL resin bed and 15 mL solution washes.

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 10, 2003. Published March 2003.

² *Annual Book of ASTM Standards*, Vol 12.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.02.