



Standard Test Method for Plutonium in Water¹

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1. Scope

1.1 This test method covers the determination of alpha-particle-emitting isotopes of plutonium concentrations over 0.01 Bq/L (0.3 pCi/L) in water by means of chemical separations and alpha pulse-height analysis (alpha-particle spectrometry). Due to overlapping alpha-particle energies, this method cannot distinguish ^{239}Pu from ^{240}Pu . Plutonium is chemically separated from a 1-L water sample by coprecipitation with ferric hydroxide, anion exchange and electrodeposition. The test method applies to soluble plutonium and to suspended particulate matter containing plutonium. In the latter situation, an acid dissolution step is required to assure that all of the plutonium dissolves.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.* Specific hazards are given in Section 9.

2. Referenced Documents

2.1 ASTM Standards:²

- C859 Terminology Relating to Nuclear Materials
- C1163 Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride
- C1284 Practice for Electrodeposition of the Actinides for Alpha Spectrometry
- D1129 Terminology Relating to Water
- D1193 Specification for Reagent Water
- D2777 Practice for Determination of Precision and Bias of

Applicable Test Methods of Committee D19 on Water
D3084 Practice for Alpha-Particle Spectrometry of Water
D3370 Practices for Sampling Water from Closed Conduits
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D1129 and Terminology C859.

4. Summary of Test Method

4.1 The water sample is acidified and a plutonium isotopic tracer, for example ^{236}Pu or ^{242}Pu , is added as a tracer before any chemical separations are performed. Iron is added to the water as iron (III), and the plutonium is coprecipitated with the iron as ferric hydroxide. After decantation and centrifugation, the ferric hydroxide precipitate containing the coprecipitated plutonium is dissolved, and the solution is adjusted to 8 M in HNO_3 for anion exchange separation. When the sample fails to dissolve because of the presence of insoluble residue, the residue is treated by a rigorous acid dissolution using concentrated nitric, hydrofluoric, and hydrochloric acids.

4.2 After an anion exchange separation, the plutonium is electrodeposited onto a stainless steel disk for counting by alpha pulse-height analysis using a silicon surface barrier or ion-implanted detector. Table 1 shows the alpha energies of the isotopes of interest in this test method. The absolute activities of ^{238}Pu and $^{239/240}\text{Pu}$ are calculated independent of discrete detector efficiency and chemical yield corrections by directly comparing the number of counts in each peak relative to counts observed from a known activity of ^{236}Pu or ^{242}Pu tracer (see Eq 1).

5. Significance and Use

5.1 This test method was developed to measure plutonium in environmental waters or waters released to the environment and to determine whether or not the plutonium concentration exceeds the maximum amount allowable by regulatory statutes.

6. Interferences

6.1 Thorium-228, when present in the original water sample at concentrations 100 times or greater than ^{238}Pu has been

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

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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.