



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 (3 pCi/L) in water by means of chemical separations and alpha pulse-height analysis (alpha-particle spectrometry). The isotopes, plutonium-239, 240, and plutonium-238, are 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 *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:

- C 859 Terminology Relating to Nuclear Materials²
- D 1129 Terminology Relating to Water³
- D 1193 Specification for Reagent Water³
- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water³
- D 3084 Practice for Alpha Spectrometry of Water⁴
- D 3370 Practices for Sampling Water³
- D 3648 Practices for the Measurement of Radioactivity⁴

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D 1129, and C 859.

4. Summary of Test Method

4.1 The water sample is acidified and plutonium-242 is added as a tracer before any chemical separations are per-

formed. 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 HNO₃ 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 and hydrofluoric 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. From the recovery of the plutonium-242 tracer, the absolute activities of plutonium-238 and plutonium-239, 240 can be calculated.

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 at concentrations 100 times or greater than plutonium-238 has been found to interfere with the determination of plutonium-238. Some thorium-228 comes through the chemical separation procedure and is electrodeposited with the plutonium. If the disk is poorly plated and if the resolution of the sample as determined by the alpha spectrometer is not better than 60 keV, the plutonium-238 and the thorium-228 may appear as one peak; the principal alpha energy of plutonium-238 is 5.50 MeV while that of thorium-228 is 5.42 MeV.

7. Apparatus

7.1 *Alpha Pulse—Height Analysis System*, consisting of a silicon surface barrier, or ion-implanted detector, supporting electronics, and pulse-height analyzer capable of giving a resolution of 50 keV WHM or better with a sample electrodeposited on a flat, mirror-finished stainless steel disk. The counting efficiency of the system should be greater than 15 % and the background in the energy region of each peak should be less than ten counts in 60 000 s.

7.2 *Electrodeposition Apparatus*, consisting of a 0 to 12 V, (0 to 2 A power supply (preferably constant current) and a

¹ This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

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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