



# Standard Test Method for Radiochemical Determination of Plutonium in Soil by Alpha Spectroscopy<sup>1</sup>

This standard is issued under the fixed designation C 1001; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of plutonium in soils at levels of detection dependent on count time, sample size, detector efficiency, background, and tracer yield. This test method describes one acceptable approach to the determination of plutonium in soil.

1.2 This test method is designed for 10 g of soil, previously collected and treated as described in Practices C 998 and C 999, but sample sizes up to 50 g may be analyzed by this test method. This test method may not be able to completely dissolve all forms of plutonium in the soil matrix.

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 hazard statements are given in Section 9.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

C 998 Practice for Sampling Surface Soil for Radionuclides

C 999 Practice for Soil Sample Preparation for the Determination of Radionuclides

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

C 1284 Practice for Electrodeposition of the Actinides for Alpha Spectrometry

D 1193 Specification for Reagent Water

D 3084 Practice for Alpha-Particle Spectrometry of Water

D 3648 Practice for the Measurement of Radioactivity

## 3. Summary of Test Method

3.1 Plutonium is extracted from the soil with a mixture of nitric, hydrofluoric, and hydrochloric acids in the presence of

$^{242}\text{Pu}$  or  $^{236}\text{Pu}$  isotopic tracer (See Appendix for purification and standardization of  $^{236}\text{Pu}$  tracer). Plutonium is isolated by anion exchange, then electrodeposited onto a polished metal disk for determination by alpha spectrometry. As an option, the plutonium may be prepared for alpha spectrometry measurement by using coprecipitation with neodymium fluoride. The range of chemical yield is between 40 and 90 %. The test method is based on a published procedure (1).<sup>3</sup>

## 4. Significance and Use

4.1 A soil sampling and analysis program provides a direct means of determining the concentration and distribution of radionuclides in soil. A soil analysis program has the most significance for the preoperational monitoring program to establish baseline concentrations prior to the operation of a nuclear facility. Soil analysis, although useful in special cases involving unexpected releases, is a poor technique for assessing small incremental releases and is therefore not recommended as a method for routine monitoring releases of radioactive material. Nevertheless, because soil is an integrator and a reservoir of long-lived radionuclides, and serves as an intermediary in several of the plutonium pathways of potential importance to humans, knowledge of the concentration of plutonium in soil is essential.

## 5. Apparatus

5.1 *Electrodeposition Apparatus* (2), see Practice C 1284.

5.2 *Alpha Spectrometer*, capable of 40 to 50 keV resolution on actual samples electrodeposited on a flat, mirror-finished metal planchet, and a counting efficiency greater than 17 %, and a background less than 0.010 cpm over each designated energy region. Resolution is defined as the full width half maximum (FWHM) in keV, the distance between those points on either side of the alpha peak where the count is equal to one-half the maximum count. Also see Practices D 3084 and D 3648.

NOTE 1—A regular program of measurement control operations should be conducted for the alpha spectrometry system, such as regular background checks, source check to determine system stability, control charting, and careful handling of samples during changing.

<sup>1</sup> 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 Test Methods.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

5.3 PTFE-polytetrafluoroethylene beakers, 250-mL.

## 6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available (3). Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification D 1193, Type III.

6.3 Reagent blanks should be run to determine their contribution to the sample result.

6.4 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH<sub>4</sub>OH).

6.5 *Ammonium Iodide (NH<sub>4</sub>I) (1 M)*—Dissolve 14.5 g of NH<sub>4</sub>I in 100 mL water.

6.6 *Ammonium Iodide, Hydrochloric Acid Solution (NH<sub>4</sub>I-HCl)*—Add 25 mL 1 M ammonium iodide to 500 mL concentrated hydrochloric acid. Prepare fresh prior to use.

6.7 *Analytical Grade Anion Exchange Resin Type 1, 8 % cross-linked, 100 to 200 mesh, chloride form*<sup>4</sup>—Store the resin in water in a wash bottle, transfer sufficient resin to a 1.3-cm ID ion exchange column to give a 10-cm bed of settled resin. Convert the resin to the nitrate form by passing 100 mL of HNO<sub>3</sub> (8M) through the column at maximum flow rate.

6.8 *Boric Acid (H<sub>3</sub>BO<sub>3</sub>)*.

6.9 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

6.10 *Hydrochloric Acid (3M)*—Add 250 mL of concentrated HCl (sp gr 1.19) to 500 mL of water, mix, and dilute to 1.0 L with water.

6.11 *Hydrochloric Acid (1.7M)*—Add 142 mL of concentrated HCl (sp gr 1.19) to 500 mL of water, mix, and dilute to 1.0 L with water.

6.12 *Hydrofluoric Acid (48 to 51 %)*—Concentrated hydrofluoric acid (HF).

6.13 *Iron Carrier solution (10 g Iron (III)/L)*—Dissolve 10.0 g iron metal in HCl (1.7M) and dilute to 1 L with HCl (1.7M).

6.14 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO<sub>3</sub>).

6.15 *Nitric Acid (8M)*—Add 500 mL of concentrated HNO<sub>3</sub> (sp gr 1.42) to 400 mL of water, mix, and dilute to 1.0 L with water.

6.16 *Nitric Acid (1.8M)*—Add 112 mL of concentrated HNO<sub>3</sub> (sp gr 1.42) to 500 mL of water, mix, and dilute to 1.0 L with water.

6.17 *Octyl alcohol*.

6.18 *Sodium Bisulfite (NaHSO<sub>3</sub>)*.

6.19 *Sodium Hydroxide (50%)*—Dissolve 500 g of NaOH in 500 mL water. A 50% NaOH solution is available commercially.

6.20 *Sodium Nitrite (NaNO<sub>2</sub>)*.

6.21 *Thymol Blue Indicator, Sodium Salt, 0.02 % Solution*.

6.22 *National Standard Traceable Plutonium-236 Reagent*.

6.23 *National Standard Traceable Plutonium-242 Reagent*.

## 7. Sampling

7.1 Collect the sample in accordance with Practice C 998.

7.2 Prepare the sample for analysis in accordance with Practice C 999.

7.3 Samples consisting of 10 to 50 g of soil can be readily analyzed by the procedure. In order to obtain more representative samples, as well as lowering the minimum detectable concentration, the analyses of large soil samples is desirable. In general, it is poor practice to use less than 10 g of sample, unless replicate analyses are performed, because of needed sensitivity to determine lower levels of activity.

## 8. Calibration and Standardization

8.1 The counting efficiency of the alpha spectrometer is used to determine the minimum detectable concentration (MDC), lower limit of detection (LLD), and chemical recovery. The efficiency of the alpha spectrometer is determined as the ratio of the observed count rate to the known disintegration rate times the counting efficiency of the 2π counter. The procedure is as follows:

8.1.1 Count a <sup>241</sup>Am source traceable to a national standards organization on a 2π alpha counter. The 2π counter efficiency is determined by:

$$CE_{2\pi} = C_1/(A_1)(T)(1.023) \quad (1)$$

where:

- $C_1$  = net counts of the <sup>241</sup>Am source,
- $A_1$  = certified activity of the <sup>241</sup>Am source (cps), corrected for decay,
- $T$  = duration of the count time, s, and
- 1.023 = backscatter factor correcting the counting efficiency of a source on platinum to that on stainless steel.

8.1.2 Electrodeposit <sup>239</sup>Pu on a polished metal disk, using the equipment and procedure listed in this method, and counting on the 2π counter. This gives the known disintegration rate,  $C_{2\pi}$ .

8.1.3 The counting efficiency of the alpha spectrometer is determined as follows:

$$CE = (C_s)(CE_{2\pi})/(C_{2\pi}) \quad (2)$$

where:

- $C_s$  = net count rate of the electroplated source over the entire energy region on the alpha spectrometer (cps); the observed count rate,
- $CE_{2\pi}$  = counting efficiency of the 2π counter, and
- $C_{2\pi}$  = net counting rate of the same source on the 2π counter (cps).

8.2 The quantity of the tracer to be used should be in the expected range (but not less than 0.17 Bq) of the isotopic activity being determined so that the statistical uncertainty in

<sup>4</sup> Resin obtainable from BioRad Laboratories (Hercules, CA) and from Eichrom Technologies, Inc. (Darien, IL).