



Standard Test Method for Radiochemical Determination of Plutonium in Soil by Alpha Spectroscopy¹

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 (ϵ) 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:²

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}Pu or ^{236}Pu isotopic tracer (See Appendix for purification and standardization of ^{236}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).³

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.

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

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 Test Methods.

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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.

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 (NH₄OH).

6.5 *Ammonium Iodide (NH₄I) (1 M)*—Dissolve 14.5 g of NH₄I in 100 mL water.

6.6 *Ammonium Iodide, Hydrochloric Acid Solution (NH₄I-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*⁴—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 HNO₃ (8M) through the column at maximum flow rate.

6.8 *Boric Acid (H₃BO₃)*.

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 (HNO₃).

6.15 *Nitric Acid (8M)*—Add 500 mL of concentrated HNO₃ (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 HNO₃ (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 (NaHSO₃).*

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 (NaNO₂).*

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 ²⁴¹Am 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 ²⁴¹Am source,

A_1 = certified activity of the ²⁴¹Am 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 ²³⁹Pu 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

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

the yield determination will not be larger than that of the nuclide being determined.

9. Precautions

9.1 Adequate laboratory facilities, such as fume hoods and controlled ventilation, along with safe techniques, must be used in this procedure. Extreme care should be exercised in using hydrofluoric and other hot, concentrated acids. Use of proper gloves is recommended. Refer to the laboratory's chemical hygiene plan and other applicable guidance for handling chemical and radioactive materials and for the management of radioactive, mixed, and hazardous waste.

9.2 Hydrofluoric acid is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid is similar to other acids in that the initial extent of a burn depends on the concentration, the temperature, and the duration of contact with the acid. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left unattended. Due to the serious consequences of hydrofluoric acid burns, prevention of exposure or injury of personnel is the primary goal. Utilization of appropriate laboratory controls (hoods) and wearing adequate personal protective equipment to protect from skin and eye contact is essential.

10. Procedure

10.1 Weigh a 10 ± 0.01 g soil aliquot into a 250 mL PTFE beaker.

10.2 Wet sample with distilled water and add a known quantity of ^{236}Pu or ^{242}Pu tracer.

10.3 Add concentrated HNO_3 (sp gr 1.42) a few drops at a time as fast as the frothing and vigor of the reaction will permit until the entire sample is covered.

10.4 Add 60 mL of the concentrated HNO_3 (sp gr 1.42) and 30 mL of concentrated HF (48 to 51 %) and digest on a hotplate with frequent stirring (TFE fluorocarbon stirring rod) for about 1 h (Note 2 and Note 3).

NOTE 2—For organic soils, first add the nitric acid only in small portions while stirring. If the solution threatens to overflow as a result of froth generation, add a few drops of octyl alcohol and stir. Digest on a hotplate until the evolution of reddish-brown fumes is reduced to a barely visible level. Cool to room temperature before carefully adding the concentrated hydrofluoric acid (48 to 51 %) and digesting for an hour.

NOTE 3—For larger soil aliquots, larger amounts of the acids (in the same proportions) should be used. For example, for a 50 g sample, use 200 mL concentrated HNO_3 and 100 mL HF, etc., with appropriately sized containers.

10.5 Remove from the hotplate and cool somewhat before adding 30 mL concentrated HNO_3 (sp gr 1.42) and 30 mL concentrated HF (48 to 51 %). Digest on the hotplate with intermittent stirring for an additional 1 h.

10.6 Remove from the hotplate and cool. Carefully add 20 mL concentrated HCl (sp gr 1.19) and stir. Heat on hotplate for 45 min with occasional stirring.

10.7 Add about 5 g of powdered boric acid and digest for an additional 15 min with occasional stirring.

10.8 Add approximately 200 mg of sodium bisulfite and continue heating until the solution has evaporated to a liquid volume of approximately 20 mL.

10.9 Add 50 mL of water and digest on a hotplate while stirring for 10 min to dissolve soluble salts.

10.10 Cool and transfer approximately equal parts of the total sample into centrifuge bottles with a minimum of water from a wash bottle. If equipment for large volume centrifugation is not available, the two precipitations in 10.11-10.21 may be performed in a beaker, allowing the precipitate to settle, decanting the supernate, and then completing the separation by centrifugation on a smaller scale.

10.11 Add 1.0 mL of iron carrier solution (10 mg Fe (III)/mL) to each centrifuge bottle and mix (Note 4).

NOTE 4—It may not be necessary to add the iron carrier if a sufficient amount of iron is present in the soil.

10.12 Add NaOH (50 %) with mixing to each bottle to a pH of about 9 (using pH paper) to precipitate ferric hydroxide. Add 5 to 10 mL excess NaOH and mix for 1 min.

10.13 Centrifuge for approximately 5 min, decant, and discard the supernate.

10.14 Dissolve each precipitate with about 30 mL HNO_3 (8M) (60 mL total) saturated with boric acid. (Approximately 7 g of boric acid/30 mL HNO_3 (8M).) Digest in a hot water bath for 10 min.

10.15 Cool and centrifuge for approximately 5 min. Decant the supernate into the original 250 mL PTFE beaker and save.

10.16 Wash each residue with approximately 10 to 20 mL (20 to 40 mL total) of HNO_3 (8M) saturated with boric acid. Centrifuge for 5 min and combine the supernates with that in 10.15. Discard the undigested soil.

10.17 Heat the supernate on a hotplate and evaporate to approximately 5 mL.

10.18 Add approximately 30 mL water and heat to dissolve the salts. Cool and transfer into a centrifuge tube.

10.19 Add concentrated ammonium hydroxide dropwise with mixing to a pH of approximately 9 (using pH paper) to precipitate ferric hydroxide.

10.20 Centrifuge and discard the supernate.

10.21 Dissolve the precipitate with a volume of concentrated nitric acid approximately equal to the volume of the precipitate and transfer using nitric acid (8M) into a 250-mL beaker. Add nitric acid (8M) to a total volume of approximately 75 mL. If the volume of the hydroxide precipitate is considerably greater than should be expected from the 10 mg of Fe(III) added, the final volume should be brought up to approximately 100 mL with nitric acid (8M) or, alternatively, the dissolved hydroxides should be evaporated to salts before the addition of the nitric acid (8M) solution. The final molarity of the solution is not extremely critical, but should be in the range of 7 to 9.

10.22 Add approximately 200 mg of sodium nitrite crystals and stir with a stirring rod. Bring to a quick gentle boil on a hotplate and cool. Avoid prolonged heating.

10.23 Prepare an anion-exchange column as described in 6.7.

10.24 Pass the sample (at maximum flow rate) through the anion-exchange resin column.

10.25 When the solution just drains to the top of the resin bed, wash the column with six column volumes of HNO₃ (8M). A column volume is approximately 9.5 mL.

10.26 Wash the resin column with six column volumes of concentrated HCl.

10.27 Elute the plutonium with four column volumes of freshly prepared NH₄I-HCl solution, and collect in a 150-mL beaker.

10.28 Evaporate the solution to approximately 5 mL on a hotplate. Rinse down the sides of the beaker dropwise with 1 to 2 mL of concentrated HNO₃. Add six drops of concentrated HCl and evaporate to near dryness.

10.29 Add 50 mL of HNO₃(8M) and repeat steps 10.22-10.27, using a fresh anion-exchange resin column.

10.30 Continue heating the final plutonium elution just to dryness on the hotplate. Rinse down the sides of the beaker with a few mL of concentrated HCl, and evaporate to approximately 0.5 mL. See Note 5. Assemble the electrodeposition cell as described in Practice C 1284.

NOTE 5—Plutonium can also be prepared for alpha spectrometry by coprecipitation with neodymium fluoride, see Test Method C 1163. If samples are determined by coprecipitation, then the counting standards must be prepared in a similar manner.

10.31 Add 1.5 to 2 mL of HCl (3M) solution into the beaker, rinse as above, and add the rinses to the plating cell.

10.32 Add 3 drops of thymol blue indicator solution and then add concentrated NH₄OH dropwise until the color changes to yellow.

10.33 Add HCl (1.7M) dropwise to a salmon pink endpoint.

10.34 Electroplate at 1.5 amps for 20 min. See Note 6.

NOTE 6—**Caution:** The electrodeposition should be performed in a fume hood due to the chlorine gas evolved during the electrodeposition.

10.35 At the end of 20 min, quickly add 2 to 3 mL of concentrated NH₄OH and leave the current on for another 20 s.

10.36 Turn off the current, rinse out the solution into a beaker with water, and dismantle the cell. Rinse the disk with water and dry it in a clean planchet on a hotplate at medium heat for 5 min, or flame with a burner.

10.37 Count the sample in an alpha spectrometer long enough to obtain the desired sensitivity (see Table 1).

TABLE 1 Decay Characteristics (4)

Isotope	Half-life	Maximum Alpha Energy, MeV (Abundance)
²³⁶ Pu	2.851 years	5.768 (68.1 %), 5.721 (31.7 %)
²³⁸ Pu	87.74 years	5.499 (71.6 %), 5.456 (28.3 %)
²³⁹ Pu	2.411 × 10 ⁴ years	5.155 (73.2 %), 5.143 (15.1 %), 5.105 (10.6 %)
²⁴² Pu	3.763 × 10 ⁵ years	4.901 (78 %), 4.856 (22.4 %)

11. Calculation of Plutonium Concentration

11.1 The activity of ²³⁹Pu or ²³⁸Pu in the aliquot of soil taken for analysis (*A_s* in Bq/g) is calculated as follows:

$$A_s = \frac{(C_s/T_s - C_b/T_b)}{C_{si}/T_s - C_{bi}/T_b} (S/V) - A_r \quad (3)$$

where:

A_s = sample activity for isotope expressed in Bq/g,
S = activity (Bq) of internal standard isotope (²³⁶Pu) added to the sample,

C_s = sample gross counts of isotope (²³⁹Pu or ²³⁸Pu),
C_{si} = sample gross counts for internal standard (²³⁶Pu),
C_b = detector background gross counts for isotope (²³⁹Pu or ²³⁸Pu),

C_{bi} = detector background gross count for internal standard isotope (²³⁶Pu),

T_s = sample count time, s,

T_b = detector background count time, s,

V = sample unit volume or weight in g, and

A_r = activity of laboratory reagent blank for isotope (²³⁹Pu or ²³⁸Pu), Bq/g, as follows:

$$A_r = \frac{C_r/T_r - C_b/T_b}{(C_{si}/T_s - C_{bi}/T_b)} (S/V) \quad (4)$$

where:

C_r = reagent blank gross counts of isotope (²³⁹Pu or ²³⁸Pu), and

T_r = reagent blank count time, s.

11.1.1 Because spectrometry cannot distinguish between plutonium-239 and plutonium-240, when plutonium-239 is mentioned, the alpha activity due to the plutonium-240 is also included.

11.2 Most of the available plutonium-236 contains significant impurities of the isotopes being sought. Thus a correction must be made to account for contaminant contributions to the analyte regions. The correction must take into account a predetermined ratio of tracer to contaminant isotopes activity to ensure that is adequately reflects the changing contribution based on varying recovery of the tracer in the sample. This interference must also be reflected in the statistics reported in association with the result.

12. Precision and Bias

12.1 Reference samples and standards were sent to 15 laboratories. Analytical data were returned by six laboratories (2). It is not known if these six laboratories are typical of the laboratories that will use this test method. The responses of two of the laboratories were judged as outliers and excluded from the analyses. Thus, the following estimates are based on twelve measurements per sample, three at each of four laboratories:

Summary of Plutonium Results

Known Value (Bq/g)	No. of Laboratories	\bar{X} (Bq/g)	Absolute Percent Bias	Percent Relative Standard Deviation	
				Within Laboratory	Between Laboratories
0.00188	4	0.0023	26	6.7	17.0
0.0023	4	0.0026	13	8.1	15.2
0.0150	4	0.0170	12	7.7	13.4
0.0172	4	0.0203	18	7.7	17.2
0.150	4	0.172	15	3.6	16.9

13. Keywords

13.1 alpha spectrometry; plutonium; radiochemistry; soil

APPENDIXES

(Nonmandatory Information)

X1. PURIFICATION OF THE PLUTONIUM-236 TRACER (5)

X1.1 In order to accurately calibrate the plutonium-236 tracer by 2π counting and alpha spectrometry, it will be necessary to ensure the absence of plutonium-236 daughters (uranium-232, thorium-228, radium-224, etc.) in the tracer. The following purification must be performed just prior to the initial calibration and annually thereafter if additional calibrations are desired.

X1.1.1 Add approximately 4.2×10^2 Bq of plutonium-236 to a 250-mL beaker containing 25 mL of nitric acid (sp gr 1.42).

X1.1.2 Evaporate the solution on a hotplate to a volume of approximately 10 mL.

X1.1.3 Remove from the hotplate and add 25 mL of HNO_3 (8M) and 10 mL of distilled water to adjust the HNO_3 concentration to 8 M.

X1.1.4 Add ~ 200 mg of sodium nitrite crystals and stir with a glass stirring rod. Bring the solution to a quick gentle boil on a hotplate and cool. Avoid prolonged heating.

X1.1.5 Pass through an anion-exchange resin column (nitrate form) prepared as described in 6.7 at maximum flow rate.

X1.1.6 When the solution just drains to the top of the resin bed, add two column volumes of HNO_3 (8M) to the column reservoir and wash the resin column at the maximum flow rate. Discard the effluents from the sample and washes.

X1.1.7 Repeat Step 6 twice until the resin column has been washed with a total of six column volumes of HNO_3 (8M).

X1.1.8 Wash the resin column at maximum flow rate with six column volumes of HCl (sp gr 1.19) using the same technique. Discard the hydrochloric acid washes.

X1.1.9 Elute the plutonium with four column volumes of freshly prepared ammonium iodide-hydrochloric acid solution and collect in a 150-mL beaker.

X1.1.10 Evaporate the solution to approximately 5 mL on a hotplate. Rinse down the sides of the beaker dropwise with 1 to 2 mL of HNO_3 (sp gr 1.42). Add six drops of HCl (sp gr 1.19) and evaporate just to dryness on the hotplate.

X1.1.11 Add 5 mL each of HCl (sp gr 1.19) and HNO_3 (sp gr 1.42) to the beaker and evaporate to about 2 mL.

X1.1.12 Add 15 mL of HNO_3 (sp gr 1.42) and boil down to about 5 mL to ensure complete dissolution of the plutonium and complete oxidation of chlorides as indicated by the absence of color or fumes of chlorine oxide or nitrogen oxide, or both.

X1.1.13 Cool, add 25 mL of distilled water and filter the solution through a DM-450 membrane filter in a filtering chimney. Wash the flask and filter with enough distilled water to give a final volume of 50 mL.

X1.1.14 Dilute aliquots of the 8.3 Bq/mL stock solution with HNO_3 (8M) to give concentrations desired for use. Store all tracers in tightly capped TFE-fluorocarbon FEP bottles.

X2. STANDARDIZATION OF THE PLUTONIUM-236 TRACER (5)

X2.1 Transfer a 1-mL aliquot of the purified plutonium-236 stock tracer (~ 8.3 Bq/mL in nitric acid (1 + 1)) onto a polished metal planchet with a calibrated silicone-treated pipet and slowly evaporate to near dryness under an infrared lamp to minimize any loss. Keep the activity in the center of the planchet in an area limited to approximately 2 cm ($\frac{3}{4}$ in.) in diameter by alternately adding the tracer a few drops at a time and evaporating. The partially filled silicone-treated pipet can be placed on its side between additions with no loss of solution. To ensure quantitative transfer of the tracer, carefully blow out the last few drops with a rubber bulb.

X2.2 When the last of the tracer has been transferred to the planchet and evaporated nearly to dryness, add 2 or 3 drops of HNO_3 (sp gr 1.42) to help keep the activity spread as uniformly as possible and evaporate to complete dryness.

X2.3 Heat the dry planchet over a blast burner just to the first dull red glow. Then quickly lower the temperature by placing the planchet on a cold steel surface to minimize oxidation of the plate.

X2.4 Count in the 2π alpha counter immediately after

cooling to avoid any possibility of absorption of water vapor from the air. Collect at least 5×10^4 counts for the standard to ensure adequate statistical precision.

X2.5 Prepare and count a duplicate source by repeating X2.1-X2.4.

X2.6 The 2π counting rate of the plutonium-236 sources must be corrected by determining the fraction of the total alpha activity due to plutonium-236.

X2.6.1 Transfer 2 mL of the purified ~ 8.3 Bq/mL plutonium-236 tracer to a 150-mL beaker and add 2 mL each of HNO_3 (sp gr 1.42) and HCl (sp gr 1.19).

X2.6.2 Evaporate carefully on a hotplate just to dryness. Rinse down the sides of the beaker with a few millilitres of HCl (sp gr 1.19) and evaporate to approximately 0.5 mL.

X2.6.3 Treat and electrodeposit as described in 10.31-10.36.

X2.6.4 Count the electroplated source on an alpha spectrometer for 15 000 s over an energy range of 3 to 8 MeV. Determine the fraction of the total number of counts in the alpha spectrum that is due to plutonium-236 in the source. This

fraction is the correction factor to be applied to the counting rate of the plutonium-236 evaporated source in the 2π proportional counter.

NOTE X2.1—Prolonged and repeated counting of high level plutonium-236 sources on the alpha spectrometer should be avoided to minimize daughter recoil contamination of the alpha detector. Alternatively, such contamination virtually can be eliminated by leaving a small amount of air in the counting chamber and applying a small negative potential to the source plate.

X2.7 Calculate the activity concentration of the plutonium-236 tracer (Bq plutonium-236/mL) by multiplying the observed 2π counting rates of the evaporated sources by the correction factor, and dividing by the 2π counter efficiency and the volume of tracer used to prepare the evaporated sources.

X3. LIMIT OF DETECTION (6)

X3.1 The Lower Limit of Detection (LLD), in Bq, is calculated as follows for each isotope of interest:

$$LLD = \frac{2.71 + 4.66(C_b/T_b)^{1/2}}{CE} \quad (X3.1)$$

where:

- C_b = counter and reagent background, in cps,
- CE = counter efficiency,
- LLD = lower limit of detection, and
- T_b = detector background count time for isotope, s.

X3.2 To determine the Minimum Detectable Concentration (MDC), divide the LLD value by the volume and yield as follows:

$$MDC = \frac{LLD}{VY} \quad (X3.2)$$

where:

- MDC = minimum detectable concentration,
- V = sample size, g, and
- Y = tracer yield.

X3.2.1 Typical conditions for method are as follows:

CE (%)	=	25	
CB (cps)	=	0.00002	
Y (%)	=	80	
V (g)	=	10	
LLD	=	2 400 s (Bq)	0.0017
LLD	=	60 000 s (Bq)	0.00034
LLD	=	300 000 s (Bq)	0.00015
MDC	=	2 400 s (Bq/g)	0.0002
MDC	=	60 000 (Bq/g)	0.00004
MDC	=	300 000 (Bq/g)	0.00002

REFERENCES

- (1) "Measurements of Radionuclides in the Environment; Sampling and Analysis of Plutonium in Soil," Atomic Energy Commission Regulatory Guide 4.5, May 1974.
- (2) "Acid Dissolution Method for the Analysis of Plutonium in Soil," Environmental Protection Agency 600/7-79-081, p. 30.
- (3) "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., New York, NY, and the "United States Pharmacopeia."
- (4) Browne, E., and Firestone, R. B., (Shirley, V. S., Ed.), *Table of Radioactive Isotopes*, John Wiley and Sons, Inc., 1986.
- (5) "Acid Dissolution Method for the Analysis of Plutonium in Soil," Environmental Protection Agency 600/7-79-081, pp. 37–41.
- (6) "Upgrading Environmental Radiation Data," Environmental Protection Agency, 520/1-80-012, pp. 6–24.

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