



Designation: C 1205 – 97 (Reapproved 2002)

Standard Test Method for The Radiochemical Determination of Americium-241 in Soil by Alpha Spectrometry¹

This standard is issued under the fixed designation C 1205; 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 americium-241 in soil by means of chemical separations and alpha spectrometry. It is designed to analyze up to ten grams of soil or other sample matrices that contain up to 30 mg of combined rare earths. This method allows the determination of americium-241 concentrations from ambient levels to applicable standards. The values stated in SI units are to be regarded as standard.

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.* For specific precaution statements, see Section 9.

2. Referenced Documents

2.1 ASTM Standards:

- C 998 Standard Practice for Sampling Surface Soil for Radionuclides²
- C 999 Standard Practice for Soil Sample Preparation for the Determination of Radionuclides²
- C 1163 Standard Method for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride²
- D 1193 Specification for Reagent Water³
- D 3084 Standard Practice for Alpha Spectrometry of Water⁴
- D 3648 Standard Practices for the Measurement of Radioactivity⁴

3. Summary of Test Method

3.1 Americium-241 is determined in prepared soil samples of up to 10 g. The soil is completely dissolved by use of

pyrosulfate fusion. After an initial separation on barium sulfate and extraction with an organophosphorous compound, the americium is separated from the other trivalent actinides and the rare earths by oxidation of the americium and precipitation of the interferences. The americium is prepared for alpha spectrometry by coprecipitation with neodymium fluoride and the americium-241 determined by alpha spectrometry using americium-243 as a yield monitor.

3.2 Typical radiochemical recoveries of this method as determined by the yield monitor, are between 75 and 90 percent. Decontamination factors from other radionuclides that may interfere with the determination of americium in this energy range are 10^4 – 10^5 .

3.3 The reagent blank contains all reagents plus the americium-243 tracer. Five samples and a reagent blank can be completed and ready for alpha spectrometry in approximately 6 h. The full-width at half-maximum (FWHM) detector resolution ranges between 43 and 65 keV.

4. Significance and Use

4.1 This test method provides the speed and high decontamination factors attainable with liquid-liquid extraction of the actinides and eliminates filtration techniques that are more time consuming.

4.2 This test method provides a precise determination of americium in concentrations normally found in environmental samples.

5. Interferences

5.1 Plutonium, if inadequately separated, may interfere with the alpha spectrometric determination of americium-241. Thorium-228, identifiable by its daughter products, is a serious interference to the final determination of americium by alpha spectrometry if decontamination factors are not sufficiently high. An inadequate separation of polonium-210 may result in an inaccurate determination of the americium-243 yield monitor but this is unlikely when using the neodymium fluoride precipitation method. If high concentrations of these radionuclides are known to be present, a preliminary separation may be required.

¹ This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee D26.05 on Methods of Test.

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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.

6. Apparatus

6.1 Alpha pulse height analysis system as in Practice D 3084.

6.1.1 A system consisting of a silicon surface barrier detector capable of 50 keV or better resolution on standards electrodeposited on a flat, mirror finished disk is required. Samples prepared for alpha spectrometry using neodymium fluoride mounting by ASTM Standard Test Method C 1163 should be capable of 60 to 70 keV resolution. The resolution is defined as the width of an alpha energy peak when the counts on either side of the peak are equal to one-half of the counts at the maximum of the peak (FWHM).

6.1.2 The counting efficiency of the system (i.e., count/disintegration) should be greater than 20 % and the instrument background in the region of each energy peak used for analysis should be less than five counts in 60 000 s (1000 min).

6.2 Membrane Filter (such as cellulose nitrate or cellulose acetate), 47 mm diameter, 0.45 μ m pore size.

7. Reagents and Materials

7.1 *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. 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.

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

7.3 *Americium Tracer*—Purify the americium-243 tracer.⁵ The americium-243 tracer may be available from NIST or other recognized standards laboratories.

7.4 *Anhydrous Potassium Fluoride*.

7.5 *Anhydrous Potassium Sulfate*.

7.6 *Anhydrous Sodium Sulfate*.

7.7 *Ammonium Persulfate* (ammonium peroxydisulfate).

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

7.9 *Hydrofluoric Acid* (sp gr 1.20)—Concentrated hydrofluoric acid (HF).

7.10 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H₂SO₄).

7.11 *0.5 % Sulfuric Acid*—Mix 5 mL of concentrated sulfuric acid with water and dilute to one liter.

7.12 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃).

7.13 *25 % Potassium Metabisulfite*—Dissolve 25 g of potassium metabisulfite in water and dilute to 100 mL.

7.14 *10 % Sodium Bromate*—Dissolve 10 g of sodium bromate in water and dilute to 100 mL.

7.15 *15 % HDEHP*—Dissolve 150 mL of bis(2-ethylhexyl)phosphoric acid in 850 mL of n-heptane.

7.16 *0.5 % Barium Chloride*—Dissolve 0.5 g of barium chloride in water and dilute to 100 mL.

7.17 *5M Nitric Acid*—Mix 312 mL of concentrated nitric acid with water and dilute to one liter.

7.18 *0.5 % Silver Nitrate*—Dissolve 0.5 g of silver nitrate in water and dilute to 100 mL.

7.19 *Lanthanum Carrier (5 mg La/mL)*—Dissolve 1.17 g of lanthanum nitrate in 75 mL of 5M nitric acid and dilute to 100 mL with water.

7.20 *Phosphoric Acid (sp gr 1.83)*—Concentrated phosphoric acid (H₃PO₄).

7.21 *0.2M Ammonium Persulfate*—Dissolve 2.3 g of ammonium persulfate in water and dilute to 50 mL. Prepare daily.

7.22 *6M Ammonium Fluoride*—Dissolve 22.2 g of ammonium fluoride in water and dilute to 100 mL.

7.23 *0.10M Ammonium Persulfate–3N Ammonium Fluoride*—Mix 20 mL of 0.2M ammonium persulfate with 20 mL of 6M ammonium fluoride. Prepare daily.

7.24 *30 % Hydrogen Peroxide*.

7.25 *Perchloric Acid (sp gr 1.67)*—Concentrated perchloric acid (HClO₄).

7.26 *Neodymium Carrier (10 mg Nd/mL)*—Heat 25 mL of 12N hydrochloric acid and 1.17 g of neodymium oxide on a hot plate until the neodymium oxide is in solution. Cool the solution and dilute to 100 mL with water.

7.27 *Neodymium Carrier (0.5 mg Nd/mL)*—Dilute 5 mL of the 10 mg Nd/mL neodymium carrier solution to 100 mL with water.

8. Sampling

8.1 Collect the sample in accordance with Practice C 998.

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

9. Hazards

9.1 In addition to other precautions, adequate laboratory facilities, such as perchloric acid fume hoods and controlled ventilation, along with safe techniques must be used in this procedure. Extreme care should be exercised in using hydrofluoric acid and other hot concentrated acids, particularly hot perchloric acid. Use of safety equipment, especially safety glasses and rubber gloves, is recommended.

9.2 When diluting concentrated acids, always wear safety glasses and protective clothing, and add the acid to the water.

10. Calibration and Standardization

10.1 If a NIST-Traceable standard americium-243 solution is not available for use as a tracer, standardize a freshly prepared sample of americium-243 using the guidance in Practice D 3648. These practices may also be used to establish the counting efficiency of the alpha spectrometer which then can be used to calculate the chemical recovery.

11. Procedure

11.1 Weigh 10 g of –35 mesh soil, to 0.001 g, into a 250-mL platinum dish and add 30 g of anhydrous potassium fluoride, 10 mL of water, 10 mL of concentrated hydrofluoric acid, and 2 mL of concentrated nitric acid. Slurry the contents of the dish together and add an americium-243 tracer at the level of

⁵ For a description of the process, see Sill, C. W., *Anal. Chem.* 46, 1974, pp. 1426–1431.