



Designation: C1000 – 05

Standard Test Method for Radiochemical Determination of Uranium Isotopes in Soil by Alpha Spectrometry¹

This standard is issued under the fixed designation C1000; 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 alpha-emitting uranium isotopes in soil. This test method describes one acceptable approach to the determination of uranium isotopes in soil.²

1.2 The test method is designed to analyze 10 g of soil; however, the sample size may be varied to 50 g depending on the activity level. This test method may not be able to completely dissolve all forms of uranium in the soil matrix. Studies have indicated that the use of hydrofluoric acid to dissolve soil has resulted in lower values than results using total dissolution by fusion.

1.3 The lower limit of detection is dependent on count time, sample size, detector efficiency, background, and tracer yield. The chemical recovery averaged 78 % in a single laboratory evaluation, and 66 % in an interlaboratory collaborative study.

1.4 *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.* A specific precautionary statement is given in Section 10.

2. Referenced Documents

2.1 *ASTM Standards:*³

C998 Practice for Sampling Surface Soil for Radionuclides

C999 Practice for Soil Sample Preparation for the Determination of Radionuclides

C1163 Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride

C1284 Practice for Electrodeposition of the Actinides for Alpha Spectrometry

D1193 Specification for Reagent Water

D3084 Practice for Alpha-Particle Spectrometry of Water

D3648 Practices for the Measurement of Radioactivity

3. Summary of Test Method

3.1 A soil sample with uranium-232 tracer added is heated to destroy organic matter and dissolved with a mixture of hydrofluoric acid and nitric acid. The uranium is coprecipitated with ferric hydroxide and the precipitate is dissolved with hydrochloric acid. Iron is removed by extraction with isopropyl ether, and plutonium, radium, and thorium are separated from uranium by anion exchange. Uranium is electrodeposited on a stainless steel disk and determined by alpha spectrometry. As an option, the uranium may be prepared for alpha spectrometric measurement by using coprecipitation with neodymium fluoride.

4. Significance and Use

4.1 This test method is used to analyze soil for alpha-emitting uranium isotopes. It can be used to establish baseline uranium levels and to monitor depositions from nuclear facilities.

5. Interferences

5.1 Protactinium-231 may not be completely separated by the procedure and could interfere with the determination of uranium-233 or uranium-234 because it has the following alpha energies in MeV: 5.06, 5.03, 5.01, 4.95 and 4.73 (see **Appendix X1**). If neptunium is present in the sample in the plus four oxidation state, it will co-elute with the uranium.

5.2 Since uranium-232 is added as a tracer, it can not be determined in soil. Uranium-232 is rarely present in soil samples. If present in significant quantities relative to the

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² Casella, V. A., Bishop, C. T., and Glosby, A. A., "Radiometric Method for the Determination of Uranium in Soil and Air," U.S. Environmental Protection Agency, EPA-600/7-80-019, Las Vegas, NV, February 1980; and in Practices **D3084** and **D3648**.

³ 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.

activity of uranium-232 tracer added, uranium-232 will lead to an overestimation of the chemical yield and a low bias in uranium results.

6. Apparatus

6.1 *Alpha Pulse Height Analysis System*:²

6.1.1 A system consisting of a charged particle detector capable of 50 keV or less resolution on samples electrodeposited on a flat mirror-finished stainless steel disk is required.

6.1.2 The resolution is defined as the width of an alpha peak when the counts on either side of the peak are equal to one-half of the counts at the maximum of the peak (full width at half maximum height (FWHM)).

6.1.3 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 0.010 cpm.

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

6.2 *Beakers and Covers* (TFE-fluorocarbon), 250 mL.

6.3 *Porcelain Crucible*, 60 mL.

6.4 *Centrifuge and Bottles*, 250-mL capacity.

6.5 *Ion Exchange Columns*, 1.3 cm inside diameter by 15 cm long with 100 mL reservoir.

6.6 *Automatic Pipettes*, 0.1 to 1.0 mL with disposable tips.

6.7 *Furnace*, able to maintain 600°C.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ 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, references to water shall be understood to mean reagent water as defined in Specification **D1193**, Type I.

7.3 Reagent purity shall be such that the measured radioactivity of blank samples does not exceed the calculated probable error of measurement.

7.4 *Ammonium Hydroxide* (0.15 M)—Mix 10 mL of concentrated ammonium hydroxide with water and dilute to 1 L.

7.5 *Ammonium Hydroxide* (*sp gr* 0.90)—Concentrated ammonium hydroxide (NH₄OH). Keep tightly capped to minimize the uptake of carbon dioxide.

7.6 *Ammonium Sulfate Solution* (1 M)—Dissolve 132 g of (NH₄)₂SO₄ in water and dilute to 1 L.

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.7 *Anion Exchange Resin*⁵—Type 1 anion exchange resin, 8% cross-linked, 100-200 mesh, chloride form. Prepare a resin slurry by soaking the resin in 8M HCl and transfer the slurry to an ion exchange column so that the resin column is approximately 10 cm high.

7.8 *Ferric Chloride Solution* (0.18 M in 0.5 M HCl)—Dissolve 48 g of FeCl₃ · 6H₂O in 0.5 M HCl and dilute to 1 L.

7.9 *Hydriodic Acid* (48 %)—Concentrated hydriodic acid (HI).

7.10 *Hydrochloric Acid* (0.5 M)—Mix 42 mL of concentrated HCl with water and dilute to 1 L.

7.11 *Hydrochloric Acid* (1 M)—Mix 83 mL of concentrated HCl with water and dilute to 1 L.

7.12 *Hydrochloric Acid* (6 M)—Mix 500 mL of concentrated HCl with water and dilute to 1 L.

7.13 *Hydrochloric Acid* (8 M)—Mix 667 mL of concentrated HCl with water and dilute to 1 L.

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

7.15 *Hydrochloric Acid-Hydriodic Acid Solution* (HCl-HI)—Mix 1 mL of concentrated HI with 50 mL of 6 M HCl. Prepare immediately before use.

7.16 *Hydrofluoric Acid* (48 %)—Concentrated hydrofluoric acid (HF).

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

7.18 *Uranium-232, Standard Solution*.⁶

7.19 *Boric Acid*—Solid.

7.20 *Isopropyl Ether*.

8. Sampling

8.1 Collect the sample in accordance with Practice **C998**.

8.2 Prepare the sample for analysis in accordance with Practice **C999**.

9. Calibration and Standardization

9.1 If a standard uranium-232 solution is not available for use as a tracer, standardize a freshly prepared sample of uranium-232; for guidance refer to Practices **D3648**. This standard may also be used to establish the counting efficiency of the alpha spectrometer which then can be used to calculate the chemical recovery and lower limit of detection (LLD) of the test method.

10. Precautions

10.1 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.

⁵ Ag1-X8 from BioRad Laboratories (Hercules, CA) and A8-B500-M-Cl from Eichrom Technologies, Inc. (Darien, IL) have been found to be satisfactory.

⁶ Uranium-232 is available from the National Institute of Standards and Technology, Gaithersburg, MD 20899, as a Standard Reference Material.