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Standard Test Method for Isotopic Uranium in Water by Radiochemistry[1]

This standard is issued under the fixed designation D 3972; 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-particle-emitting isotopes of uranium in water by means of chemical separations and alpha pulse-height analysis (also known as alpha-particle spectrometry). Uranium is chemically separated from a water sample by coprecipitation with ferrous hydroxide, anion exchange, and electrodeposition. The test method applies to soluble uranium as well as to any uranium that might be present in suspended matter in the water sample. This test method is applicable for uranium processing effluents as well as substitute ocean water. When suspended matter is present, an acid dissolution step is added to assure that all of the uranium dissolves. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

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 warning statements are given in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

C 859 Terminology Relating To Nuclear Materials[2]
D 1066 Practice for Sampling Steam[3]
D 1129 Terminologies Relating to Water[3]
D 1192 Guide for Equipment for Samping Water and Steam in Closed Conduits[3]
D 1193 Specification for Reagent Water[3]
D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water[3]
D 3084 Practice for Alpha-Particle Spectrometry of Water[4]
D 3370 Practices for Sampling Water[3]
D 3648 Practices for the Measurement of Radioactivity[4]
D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis[4]

3. Terminology

3.1 *Definitions*:

3.1.1 For definitions of terms used in this test method, refer to Terminologies C 859 and D 1129. For terms not included in these reference may be made to other published glossaries.[5,6]

4. Summary of Test Method

4.1 The water sample to be analyzed is acidified and ^{232}U is added to serve as an isotopic tracer before any additional operations are performed. If the sample is a seawater sample, or if it contains carbonate or bicarbonate ions, the sample must be boiled under acidic conditions to convert these ions to carbon dioxide gas which is then expelled from the solution. Carbonate ions must not be present during the precipitation step because they complex the uranium and prevent its coprecipitation. The uranium is coprecipitated from the sample with ferrous hydroxide. This precipitate is dissolved in concentrated hydrochloric acid, or is subjected to an acid dissolution with concentrated nitric and hydrofluoric acids if the hydrochloric acid fails to dissolve the precipitate.

4.2 The uranium is separated from other radionuclides by adsorption on anion-exchange resin from 8 *M* hydrochloric acid, followed by elution with 0.1 *M* hydrochloric acid. The uranium is electrodeposited onto a stainless steel disk. Isotopic uranium radioactivities are measured by alpha pulse-height analysis with a silicon surface-barrier or ion-implanted detector and a multichannel analyzer.

4.3 When ^{232}U is used as the tracer, the other isotopes of uranium listed in Table 1 can be detected in the alpha-particle spectrum of an unknown sample. From the alpha energies given in the table, it can be seen that the alpha energy of ^{232}U is more than 0.40 MeV higher than the energy of any other uranium isotope. Thus, there should be little interference from tailing of the ^{232}U into the lower energy alpha peaks. ^{233}U and ^{234}U usually cannot be resolved because their principal alpha energies differ by only 0.04 MeV. ^{235}U and ^{236}U peaks can be resolved only with difficulty. The alpha peaks from other combinations of uranium isotopes can be resolved unless the quality of the finally prepared sample is poor.

[1] This test method is under the jurisdiction of ASTM Committee D19 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.
[5] Parker, S. P., ed., *McGraw-Hill Dictionary of Chemical Terms*, McGraw-Hill Book Co., New York, NY, 1985.
[6] IUPAC, "Glossary of Terms Used in Nuclear Analytical Chemistry," *Pure and Applied Chemistry*, Vol 54, 1982, pp. 1533–1554.

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TABLE 1 Relevant Properties of Uranium Isotopes of Interest in Environmental Waters[A]

Isotope	Half Life Years	Principal Alpha Energies in MeV (Abundance)
^{232}U	68.9	5.320(68.6) 5.262(31.4)
^{233}U	1.592×10^5	4.824(83.3) 4.782(14.1)
^{234}U	2.455×10^5	4.774(72.5) 4.722(27.5)
^{235}U	7.038×10^8	4.596(5.6) 4.307 (57) 4.366 (17) 4.214(6.4)
^{236}U	2.342×10^7	4.493 (74) 4.445 (26)
^{238}U	4.468×10^9	4.198 (77) 4.151 (23)

[A] *Table of Isotopes*, Eighth Edition, Vol. 11, Richard B. Firestone, Lawrence Berkeley National Laboratory, University of California, 1996.

5. Significance and Use

5.1 This test method was developed to measure the radioactivity of uranium isotopes in environmental waters or waters released to the environment, and to determine whether the uranium-isotope concentrations are below the maximum amounts allowable by any regulatory statute.

6. Interferences

6.1 Thorium, polonium, plutonium, and americium were found not to interfere in this uranium determination.[7] The only possible alpha-emitting isotope that might interfere, based on the chemistry of this test method, is ^{231}Pa (3.28×10^4 y half-life). This isotope, however, is not likely to be present in environmental water samples. ^{231}Pa has the following alpha energies in MeV, the abundance being given in parentheses: 5.013 (25.4 %), 5.03 (23 %), 4.951 (22.5 %), 5.059 (11 %), and 4.734 (8.4 %). Thus, from Table 1, it is seen that ^{231}Pa can interfere with the determination of ^{233}U or ^{234}U. However, when the 4.951 to 5.059 MeV ^{231}Pa peaks can be resolved from the uranium peaks, a correction can be made.

6.2 When measuring very low concentrations of uranium isotopes in environmental samples, detector backgrounds and laboratory blanks must be well known. Blank determinations are made to ascertain that any contamination from reagents, glassware and other laboratory apparatus is small compared to the activity in the sample that is being analyzed. A blank determination should be made in exactly the same way as the sample determination.

7. Apparatus

7.1 *Centrifuge*, 250-mL centrifuge bottle or tube capacity.

7.2 *Ion Exchange Column*, glass or plastic, approximately 13-mm inside diameter and 150 mm long with a glass-wool plug or plastic frit and a 100- to 150-mL reservoir.

7.3 *Electrodeposition Apparatus*, consisting of a 0 to 12-V, 0 to 2-A power supply (preferably constant current) and an electrodeposition cell. The cathode is an approximately 20-mm diameter stainless steel disk polished to a mirror finish. The anode is approximately 1-mm diameter platinum wire with an approximately 8-mm diameter loop at the end of the wire parallel to the cathode disk. Cooling of the electrolyte during electrodeposition to at least 50°C is recommended. See references in Section 8 of Practice D 3084 for more details.

7.4 *Alpha Pulse-Height Analysis System*, consisting of a silicon surface-barrier or ion-implanted detector, supporting electronics, and pulse-height analyzer. A system capable of giving a resolution of 30 keV FWHM or better, when measured with a high-quality source, is recommended. 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.

8. Reagents and Materials

8.1 *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.[8] Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without reducing the precision, or increasing the bias, of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specifications D 1193, Type III, as a minimum.

8.3 *Radioactive Purity of Reagent*—Radioactive purity shall be such that the measured results of blank samples do not exceed the calculated probable error of the measurement or are within the desired precision.

8.4 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH_4OH).

8.5 *Ammonium Hydroxide Solution 0.15 M (1+99)*—Mix 1 volume of concentrated NH_4OH (sp gr 0.90) with 99 volumes of water. This solution is 0.15 *M*.

8.6 *Anion-Exchange Resin*—Strongly basic, styrene, quaternary ammonium salt, 4 % crosslinked, 100 to 200 mesh, chloride form.

8.7 *Electrolyte*—Dissolve 132 g of ammonium sulfate in water and dilute to 1 L. Slowly add concentrated NH_4OH or concentrated H_2SO_4 while stirring to adjust the pH of the solution to 3.5. The solution is 1 *M* in $(NH_4)_2SO_4$.

8.8 *Ethyl Alcohol (C_2H_5OH)*—Make slightly basic with a few drops of concentrated NH_4OH per 100 mL of alcohol. Anhydrous denatured ethanol is acceptable.

[7] Bishop, C. T., Casella, V. R., and Glosby, A. A., "Radiometric Method for the Determination of Uranium in Water: Single-Laboratory Evaluation and Interlaboratory Collaborative Study," *U.S. Environmental Protection Agency Report*, EPA *600/7-79-093*, April 1979.

[8] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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