



Standard Test Methods for Microquantities of Uranium in Water by Fluorometry¹

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1. Scope

1.1 These test methods cover the determination of microquantities of uranium in water in the concentration range from 0.005 to 50 mg/L.

1.2 The uranium fluorescence is quenched by many cations and some anions in the sample; it is enhanced by a few cations. If interfering ions are present, a direct fluorometric measurement is not suitable, and an extraction method shall be used to provide accurate results. The test methods and their concentration ranges are as follows:

	Concentration Range, mg/L	Sections
Test Method A—Direct Fluorometric	0.005 to 2	7 to 15
Test Method B—Extraction	0.04 to 50	16 to 24

1.3 The values stated in SI units are to be regarded as the standard.

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.* For specific hazards, see Note 1.

2. Referenced Documents

2.1 ASTM Standards:

- D 1066 Practice for Sampling Steam²
- D 1129 Terminology Relating to Water²
- D 1192 Specification for Equipment for Sampling Water and Steam²
- D 1193 Specification for Reagent Water²
- D 3370 Practices for Sampling Water²
- E 217 Test Method for Uranium by Controlled-Potential Coulometry³
- E 318 Test Method for Uranium in Aqueous Solutions by Colorimetry⁴

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test

methods refer to Terminology D 1129.

4. Significance and Use

4.1 These test methods have been referenced in the National Interim Primary Drinking Water Regulations (Title 40, Part 141; Federal Register Vol 41, No. 133, July 1976) as the approved test methods of analysis for uranium in water. However, the following limitation of these test methods should be duly noted when considering their use for determining the uranium alpha contribution to a gross alpha measurement of a drinking water sample.

4.2 Uranium occurs naturally in three isotopic forms, namely as U-238, U-235, and U-234 (U-234 being a decay product of U-238). These isotopics occur in the approximate respective mass percentages of 99.3, 0.7, and 0.0057. However, because of the different decay rates of the three isotopics, their respective alpha particle activities are 12.21, 0.55 and 13.02 Becquerels, per milligram (Bq/mg) (330, 15, and 352 picocuries per milligram) (pCi/mg) of natural uranium.

4.3 It is now known, from uranium isotopic analysis by alpha spectrometry, that the U-238/U-234 abundance ratios in ground water systems can be well out of equilibrium. Instead of the 1 to 1.07 (12.21 to 13.02) alpha activity ratio that occurs in natural uranium deposits, the isotopic alpha activity ratios in ground water systems have been found to be as much as 1 to 20. There is no single valid factor for converting measured mass units of uranium in ground water samples to uranium alpha particle activity. Therefore, a uranium mass measurement method such as this fluorometric (or colorimetric) method should not be used to determine the uranium alpha activity of water.

5. Reagents

5.1 *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.⁵ Other grades may be used, provided it is first ascertained that the reagent is of

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ Discontinued; see *1991 Annual Book of ASTM Standards*, Vol 12.01.

⁴ *Annual Book of ASTM Standards*, Vol 12.01.

⁵ *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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type III.

6. Sampling

6.1 Collect the samples in accordance with Practice D 1066, Specification D 1192, and Practices D 3370, as applicable.

6.2 To ensure continued solubility of the sample constituents, adjust the pH of the sample to approximately 2 with nitric acid (sp gr 1.42).

TEST METHOD A—DIRECT FLUOROMETRIC METHOD

7. Scope

7.1 This test method is applicable to the determination of uranium in waters containing insufficient quantities of interfering ions to either enhance or quench the fluorescence of a fused uranium-fluoride disk. The range of the test method is from 0.005 to 2.0 mg/L. Although higher concentrations of uranium can be determined by this test method, better precision and bias can be obtained with other procedures (see Test Methods E 217 and E 318).

8. Summary of Test Method

8.1 This test method is based on the measurement of the fluorescence of a fused disk of sodium fluoride, lithium fluoride, and uranium compound exposed to ultraviolet light. The intensity of the fluorescence is proportional to the uranium concentration.

8.2 An aliquot of the sample is pipeted into a platinum disk containing a sodium fluoride-lithium fluoride flux and evaporated to dryness. The mixture of sample and flux is fused with a blast burner, a muffle furnace, a tube furnace, or an induction heater. The fused disk is excited with an ultraviolet source over the wavelength range from 320 to 370 nm and the intensity of the fluorescence at 530 to 570 nm is measured by the fluorometer.

9. Interferences

9.1 There are many ions that interfere with this test method. Small quantities of cadmium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, lead, platinum, silicon, thorium, and zinc interfere by quenching the uranium fluorescence. Niobium and tantalum are reported to enhance the uranium fluorescence. In such cases use Test Method B.

10. Apparatus

10.1 *Blast Burner, Muffle Furnace, Tube Furnace, or Induction Heater*, capable of a 900°C temperature.

10.2 *Fluorometer*, having an excitation wavelength range from 320 to 370 nm and measuring the emission at a wavelength of 530 to 570 nm and capable of detecting 0.5 ng, or less, of uranium.

10.3 *Glasses*, didymium.

10.4 *Pellet Dispenser*, made by cutting a 1-mL hypodermic syringe so as to leave the full bore open.

10.5 *Pipet*—A 5-mL hypodermic syringe connected by flexible plastic tubing to a 0.5-mL Mohr pipet (graduated in 0.01-mL subdivisions) mounted on a ring stand, or equivalent.

10.6 *Platinum Disks*, the size and shape to be determined by the requirements of the fluorometer.

10.7 *Pyrometer*, with a suitable range for determining the fusion temperature of the flux.

11. Reagents and Materials

11.1 *Flux Mixture*—Mix 98 parts of sodium fluoride (NaF) and 2 parts of lithium fluoride (LiF) by weight until homogeneous. Several lots of NaF and LiF from different manufacturers should be tested to obtain material with a low blank reading and a high uranium sensitivity. Sufficient reagent to last several years should be obtained from the best lot. The powder should be sealed tightly to exclude moisture, during use and storage.

11.2 *Nitric Acid (1+1)*—Mix 1 volume of nitric acid HNO₃ (sp gr 1.42) with 1 volume of water.

11.3 *Nitric Acid (1+9)*—Mix 1 volume of HNO₃ (sp gr 1.42) with 9 volumes of water.

11.4 *Nitric Acid (1+99)*—Mix 1 volume of HNO₃ (sp gr 1.42) with 99 volumes of water.

11.5 *Potassium Pyrosulfate* (K₂S₂O₇), solid.

11.6 *Uranium Stock Solution* (1 mL = 1 mg U)—Dissolve 0.5896 g of uranous-uranium oxide (U₃O₈) in 20 mL of HNO₃ (1+1) and slowly evaporate to near dryness. Dissolve residue with 10 mL of HNO₃ (1+9) and quantitatively transfer to a 500-mL volumetric flask. Dilute to 500 mL with HNO₃ (1+99). Mix solution and transfer to a clean dry polyethylene bottle. This solution will contain 1000 mg U/L.

11.7 *Uranium Stock Solution* (1 mL = 0.05 mg U)—Pipet 25 mL of the uranium solution (1 mL = 1 mg) into a 500-mL volumetric flask. Dilute to 500 mL with HNO₃ (1+99). Mix well and transfer to a clean, dry polyethylene bottle. This solution will contain 50 mg U/L.

11.8 *Uranium Stock Solutions*—Prepare 200 mL each of standard solutions containing 10, 5, 1, 0.5, 0.1, 0.05, and 0.005 mg/L of uranium by diluting appropriate volumes of the uranium solution (1 mL = 0.05 mg U) with HNO₃ (1+99). Consecutive tenfold dilutions of the 10 and 5 mg/L U standards are suggested for improved accuracy in preparing the more dilute standards. Mix each standard well and transfer to a clean, dry polyethylene container.

12. Calibration and Standardization

12.1 *Standardization of Fusion Operations*—The fusion operation is the most critical step in the fluorometric procedure. Small variations in the duration of the fusion, temperature of the fusion, and in the method of cooling the fused disk can cause large variations in the fluorescence. Therefore, it is imperative to standardize each step of the fusion operation to obtain reproducible results.

12.1.1 Choose the method of attaining the fusion temperature from one of four acceptable methods. These methods are burner fusion (either single or multiple fusions), induction heater fusion, muffle furnace fusion, or tube furnace fusion. Although reproducible results can be obtained with each method, the methods are not necessarily interchangeable. Use the same method of attaining the fusion temperature for the