



## Standard Test Methods for Microquantities of Uranium in Water by Fluorometry<sup>1</sup>

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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 Steam<sup>2</sup>
- D 1129 Terminology Relating to Water<sup>2</sup>
- D 1192 Specification for Equipment for Sampling Water and Steam<sup>2</sup>
- D 1193 Specification for Reagent Water<sup>2</sup>
- D 3370 Practices for Sampling Water<sup>2</sup>
- E 217 Test Method for Uranium by Controlled-Potential Coulometry<sup>3</sup>
- E 318 Test Method for Uranium in Aqueous Solutions by Colorimetry<sup>4</sup>

### 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.<sup>5</sup> Other grades may be used, provided it is first ascertained that the reagent is of

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> Discontinued; see 1991 *Annual Book of ASTM Standards*, Vol 12.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 12.01.

<sup>5</sup> *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 HNO<sub>3</sub> (sp gr 1.42) with 1 volume of water.

11.3 *Nitric Acid (1+9)*—Mix 1 volume of HNO<sub>3</sub> (sp gr 1.42) with 9 volumes of water.

11.4 *Nitric Acid (1+99)*—Mix 1 volume of HNO<sub>3</sub> (sp gr 1.42) with 99 volumes of water.

11.5 *Potassium Pyrosulfate* (K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>), solid.

11.6 *Uranium Stock Solution* (1 mL = 1 mg U)—Dissolve 0.5896 g of uranous-uranium oxide (U<sub>3</sub>O<sub>8</sub>) in 20 mL of HNO<sub>3</sub> (1+1) and slowly evaporate to near dryness. Dissolve residue with 10 mL of HNO<sub>3</sub> (1+9) and quantitatively transfer to a 500-mL volumetric flask. Dilute to 500 mL with HNO<sub>3</sub> (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 HNO<sub>3</sub> (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 HNO<sub>3</sub> (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

samples, blanks, and standards. If a burner is used for the fusion operation, use a reducing flame since an oxidizing flame increases the reaction rate between the flux and the platinum disk. The dissolved platinum will subsequently quench the fluorescence of the fused disk. Avoid excessively high temperature fusions or prolonged fusions, since it increases the amount of dissolved platinum in the fused disk. Very short fusions, however, can result in poor distribution of the uranium.

**NOTE 1—Caution:** Perform all fusion operations in a hood to avoid breathing the NaF-LiF fumes. Wear didymium glasses during burner fusions for eye protection and for ease in observing the molten flux.

12.1.2 The quantity of flux, which affects several variables in the fusion operation, cannot be specified in this procedure due to variations in the sizes of fluorometer dishes. Therefore use the following criteria to standardize the fusion operation:

12.1.2.1 Determine the melting point of the flux with a pyrometer.

12.1.2.2 Standardize the fusion temperature at 50°C above the melting point of the flux.

12.1.2.3 Determine the quantity of flux that will completely wet the depression of the platinum dish during the fusion.

12.1.2.4 Determine the amount of time required to completely melt the quantity of flux established in 12.1.2.3 at the temperature established in 12.1.2.2.

12.1.2.5 Standardize the fusion time by multiplying the value obtained in 12.1.2.4 by 1.5 and rounding to the nearest 1 min.

12.1.2.6 Provide for an annealing or a slow cooling step following the fusion to give higher sensitivity and better reproducibility of the physical properties of the fused disk.

12.2 *Calibration of Fluorometer*—The instructions for the operation and adjustment of the fluorometer will be provided by the manufacturers. Although a daily factor is used to convert the fluorometer readings to mg U/L for each method, perform a preliminary calibration to confirm a linear relationship between the fluorometer readings and the uranium concentrations.

12.2.1 Add an NaF-LiF flux pellet, using the quantity of flux established in 12.1.2.3, to 20 platinum dishes. These platinum dishes will be used for two blanks and nine duplicate uranium standards. Duplicate 0.1 and 0.2-mL aliquots of the 0.005, 0.05, 0.50, and 5.0 mg U/L standards and duplicate 0.1 aliquots of the 50 mg U/L solution shall be used to calibrate the fluorometer. These aliquots will simulate samples containing 0.005, 0.010, 0.050, 0.10, 0.50, 1.0, 5.0, 10.0, and 50.0 mg U/L, respectively. After evaporation of the blanks and standards under an infrared lamp, fuse the blank and standard pellets in accordance with the standardized conditions established in 12.1.2. Cool the fused disks to room temperature, and either measure the fluorescence of the blanks and the standards with the fluorometer or zero the fluorometer with the blanks and measure the fluorescence of the standards. Follow the manufacturer's recommendations to either zero the fluorometer with the blank or record the reading of the blank. If the blanks have not been used to zero the fluorometer, subtract the average value of the blanks from the average readings of each standard. Plot the uranium concentration in milligrams per litre versus

the product of the fluorometer readings and the scale factor for each standard. A straight line should be obtained.

12.2.2 It is necessary to recalibrate the fluorometer whenever any change is made in the fluorometer, reagents, or fusion conditions.

### 13. Procedure

13.1 Using the quantity of the NaF-LiF flux established in 12.1.2.3, add a flux pellet to each of the eight platinum dishes with the pellet dispenser. These platinum dishes will be used for two blanks and six uranium standards. Add a pellet of the flux to each platinum dish required for the samples using two platinum dishes per sample.

13.2 Pipet duplicate 0.1-mL aliquots of the 0.01, 0.10, and 1.0 mg/L uranium standards into six of the platinum dishes.

13.3 Pipet duplicate 0.1-mL aliquots of the samples into platinum dishes.

13.4 Evaporate samples and standards to dryness under an infrared lamp.

13.5 Using the fusion operation established in 12.1.2, fuse the samples, blanks, and uranium standards.

13.6 After the fused disks have cooled to room temperature, measure the fluorescence of the samples, blanks, and uranium standards with the fluorometer.

13.7 *Cleaning the Platinum Dishes*—Remove the disk from the platinum dish and wash the dish in hot water. Fuse each dish with potassium pyrosulfate ( $K_2S_2O_7$ ), cool, and dissolve the residue in hot water. Store the dishes in dilute  $HNO_3$  (1+9) until needed. Rinse in water prior to use.

### 14. Calculation

14.1 Calculate the uranium concentration in milligrams per litre as follows:

$$\text{Uranium, mg/L} = F \times R \times S \quad (1)$$

where:

$R$  = fluorometer reading of the sample,

$S$  = fluorometer scale, and

$F = \{[0.01/(R_1 \times S_1)] + [0.10/(R_2 \times S_2)] + [1.0/(R_3 \times S_3)]\}/3$

where:

0.01, 0.10, and 1.0 = mg/L of the three uranium standards,

$R_1, R_2,$  and  $R_3$  = fluorometer reading of the three standards, and

$S_1, S_2,$  and  $S_3$  = fluorometer scale used for the three uranium standards.

14.2 Calculate the total propagated uncertainty (1s) for the uranium concentration as follows:

$$S_c(\text{mg/L}) = C[(S_R/R)^2 + (S_F/F)^2 + (S_V/V)^2]^{1/2} \quad (2)$$

where:

$C$  = uranium concentration in mg/L,

$S_R$  = one standard deviation of the fluorometer reading of the sample,

$S_F$  = one standard deviation in the calibration factor,

$S_V$  = one standard deviation in the sample volume, and

$V$  = sample volume in mL and the other items are as previously defined.

$S_R$  and  $S_F$  are functions of sample concentration and the

variability in sample preparation and instrument fluctuations. Estimates of the standard deviations would have to be obtained using replicate standardizations and determinations at different concentrations and under a variety of operator conditions.

14.3 Estimate the minimum detectable concentration (*MDC*) from replicate measurements of a reagent blank as follows:

$$MDC \text{ (mg/L)} = 4.65 * S_b \quad (3)$$

where:

$S_b$  = one standard deviation of replicate reagent blank analyses.

NOTE 2—If the fluorometer is not zeroed with the blank, subtract the reading of the blank from the reading of the samples and the uranium standards.

## 15. Precision and Bias <sup>6</sup>

15.1 An interlaboratory study was conducted which involved seven operators from four laboratories, each operator analyzing six samples in triplicate.

15.2 Statistical results of the interlaboratory study are given in Table 1.

## TEST METHOD B—EXTRACTION

### 16. Scope

16.1 This test method is applicable to the determination of uranium in waters known to contain sufficient quantities of impurities to interfere with a direct fluorometric determination. The range of the test method is from 0.05 to 50 mg/L. Although this range can be extended, better precision and bias can be obtained at 50 mg/L and above with other procedures (see Test Methods E 217 and E 318).

### 17. Summary of Test Method

17.1 The uranium in the sample is separated by an extraction with hexone (methyl isobutyl ketone) using an acid-deficient aluminum nitrate salting solution containing tetrapropylammonium nitrate (TPAN). The extracted uranium is fused with a sodium fluoride-lithium fluoride pellet. The uranium content is determined by measuring the ultraviolet activated fluorescence of the fused disk with a fluorometer. The fluorometer is calibrated with standard uranium solutions that have been extracted by the same procedure.

### 18. Interferences

18.1 Excessive quantities of sulfate ion or hydrogen ion interfere with this test method by inhibiting the extraction of

the uranium. These interferences are eliminated by evaporating the acidified solution to near dryness and dissolving the residue in nitric acid (38+62). With a specified sample aliquot, the tolerance limits are 5 *M* for sulfate and 16 *N* for acid. Alkaline solutions shall be acidified with concentrated nitric acid before proceeding with the analysis.

### 19. Apparatus

19.1 For a description of the furnace, pellet dispenser, platinum dishes, pyrometer, and accessories required for the preparation of fused disks, see Section 10.

19.2 *Fluorometer*—See 10.2.

19.3 *Mechanical Test Tube Shaker*.

19.4 *Pipet*—See 10.5.

### 20. Reagents

20.1 *Acetone*.

20.2 *Aluminum Nitrate* [Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O], crystalline.

20.3 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide NH<sub>4</sub>OH. Keep tightly closed. Discard if cloudy.

20.4 *Flux Mixture*—See 11.1.

20.5 *Hexone (Methyl Isobutyl Ketone)*.

20.6 *Hydroxylamine Hydrochloride Solution* (139 g/L)—Dissolve 13.9 g of hydroxylamine hydrochloride (NH<sub>2</sub>OH·HCl) in water and dilute to 100 mL.

20.7 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO<sub>3</sub>).

20.8 *Nitric Acid* (38+62)—Mix 380 mL of HNO<sub>3</sub> (sp gr 1.42) with 620 mL of water.

20.9 *Potassium Permanganate Solution* (31.6 g/L)—Dissolve 7.9 g of potassium permanganate (KMnO<sub>4</sub>) in water and dilute to 250 mL.

20.10 *Tetrapropylammonium Hydroxide (TPAH)*.

20.11 *TPAH Salting Solution*—Dissolve 1050 g of aluminum nitrate [Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O] in 700 mL of water with the aid of heat. Add 135 mL of NH<sub>4</sub>OH (sp gr 0.90) and 10 mL of a 10 % solution of tetrapropylammonium hydroxide and dilute to about 950 mL with water. Stir until dissolved, then extract with 250 mL of hexone in a separatory funnel and discard extract. Filter the aqueous phase through a large fine porosity, sintered-glass Büchner funnel, add 10 mL of 10 % tetrapropylammonium hydroxide, and dilute to 1 L with water.

20.12 *Uranium Standards*—See 11.7 and 11.8.

### 21. Calibration and Standardization

21.1 *Standardization of Fusion Operation*—Follow the procedure given in 12.1.

21.2 *Calibration of Fluorometer*—Proceed as directed in 12.2 except that the 0.005-mL/L uranium standard may be omitted.

### 22. Procedure

22.1 If the sulfate concentration exceeds 5 *M* or if the acid concentration exceeds 16 *N*, evaporate an 0.5-mL aliquot of the sample to near dryness and redissolve in 0.15 mL of HNO<sub>3</sub> (38+62).

22.2 Pipet 0.5 mL of the sample, or transfer the redissolved aliquot from 22.1, into a 13 by 100-mm test tube. For neutral

<sup>6</sup> Supporting data are available from ASTM, Request RR: D19-1005.

**TABLE 1 Test Method A—Results of Interlaboratory Study**

Added, mg U/L	Found, mg U/L	Bias, %
0.0063	0.0074	+17.5
0.013	0.019	+46.1
0.026	0.031	+19.2
0.125	0.120	-4.0
0.523	0.544	+4.0
2.09	2.27	+8.6

or alkaline samples, add 0.2 mL of HNO<sub>3</sub> (sp gr 1.42). With each group of samples, include two blanks and duplicate 0.5-mL aliquots of the 0.10, 1.0, and 10.0 mg/L uranium standards.

22.3 While swirling, add 0.2 N KMnO<sub>4</sub> solution, 1 drop at a time, until the pink color persists.

22.4 While swirling, add 1 drop of NH<sub>2</sub>OH·HCl solution. If the pink color persists, add another drop. If the pink color still persists, too much KMnO<sub>4</sub> solution was added in 22.3. Start again with a fresh aliquot.

22.5 Add 4.0 mL of the TPAH salting solution.

22.6 Pipet 2.0 mL of hexone into the test tube, stopper, mix well for 3 min with a mechanical mixer or 1 min if hand mixed. Allow to stand until the phases separate. If complete separation does not occur, centrifuge for 2 min.

22.7 Place a pellet of the fusion flux into each of two platinum dishes.

22.8 Pipet 0.2 mL of the organic phase onto each of the two pellets. Each time, rinse the pipet with acetone and add the rinsings to the pellet.

22.9 Continue as directed in 13.4-13.7.

### 23. Calculation

23.1 See Section 14. The 0.10-g, 1.0 and 10.0 mg U/L standards are used to calculate the factor *F*.

### 24. Precision and Bias

24.1 An interlaboratory study was conducted which involved seven operators from four laboratories, each operator analyzing four samples in triplicate.

24.2 Statistical results of the interlaboratory study are given in Table 2.

### 25. Quality Control Applicable to Both Test Methods

25.1 Whenever possible, the project leader, as part of the external quality control program, should submit quality control samples to the analyst along with routine samples in such a way that the analyst does not know which of the samples are the quality control samples. These external quality control samples which usually include duplicate and blank samples, should test sample collection and preparation as well as sample analysis whenever this is possible. In addition, analysts are expected to run internal quality control samples that will indicate to them whether the analytical procedures are in control. Both the external and internal quality control samples should be prepared in such a way as to duplicate the chemical matrix of the routine samples, insofar as this is practical. The quality control samples that are routinely used consist of five basic types: blank samples, replicate samples, reference materials, control samples and "spiked" samples.

### 26. Keywords

26.1 fluorometry; uranium; water

TABLE 2 Test Method B—Results of Interlaboratory Study

Added, mg U/L	Found, mg U/L	Bias, %
0.063	0.071	+12.7
0.523	0.611	+16.8
6.27	7.27	+15.9
52.3	49.3	-5.7

## APPENDIXES

### (Nonmandatory Information)

#### X1. SUMMARY OF ROUND-ROBIN TESTING OF DIRECT FLUOROMETRIC URANIUM TEST METHOD (TEST METHOD A)

X1.1 The 0.0016 and 0.00024 values for the formulas for calculating the *S<sub>o</sub>* and *S<sub>i</sub>* were determined by trial and error. The other constants were determined with a computer programmed

for the least squares fit for a curve of the general type,  $y = ax^B$ .

N	Added, mg U/L	Found, mg U/L	Predicted <sup>A</sup>		Predicted <sup>B</sup>		Bias, %
			<i>S<sub>o</sub></i> , mg U/L	<i>S<sub>o</sub></i> , mg U/L	<i>S<sub>i</sub></i> , mg U/L	<i>S<sub>i</sub></i> , mg U/L	
24	0.0063	0.0074	0.0019	0.0019	0.0025	0.0025	+17.5
26	0.013	0.019	0.002	0.002	0.003	0.003	+46.1
21	0.026	0.031	0.009	0.003	0.012	0.003	+19.2
18	0.125	0.120	0.009	0.010	0.012	0.010	-4.0
21	0.523	0.544	0.053	0.055	0.072	0.081	+4.0
21	2.09	2.27	0.35	0.32	0.72	0.70	+8.6

<sup>A</sup>Formula  $S_o - 0.0016 = 0.1144x^{1.2468}$  or  $\log(S_o - 0.0016) = \log 0.1144 + 1.2468 \log x$   
*x* = uranium concentration

<sup>B</sup>Formula  $S_i - 0.0024 = 0.2001x^{1.5293}$  or  $\log(S_i - 0.0024) = \log 0.2001 + 1.5293 \log x$

X1.2 A plot of the standard deviations versus uranium concentrations on log-log paper indicated that the *S<sub>o</sub>* and *S<sub>i</sub>* for the 0.026 mg U/L standard were biased high. These two points

were disregarded in the determination of the formulas for *S<sub>o</sub>* and *S<sub>i</sub>*.

**X2. SUMMARY OF ROUND-ROBIN TESTING OF DIRECT FLUOROMETRIC URANIUM TEST METHOD  
(TEST METHOD B)**

X2.1 The contents were determined with a computer programmed for the least squares fit for a curve of the general type,  $y = ax$ .<sup>B</sup>

N	Added, mg U/L	Found, mg U/L	Predicted <sup>A</sup>		Predicted <sup>B</sup>		Bias, %
			S <sub>o</sub> mg U/L	S <sub>e</sub> mg U/L	S <sub>f</sub> mg U/L	S <sub>r</sub> mg U/L	
28	0.063	0.071	0.014	0.014	0.020	0.019	+12.7
29	0.523	0.611	0.093	0.107	0.126	0.148	+16.8
29	6.27	7.27	1.50	1.09	1.91	1.58	+15.9
28	52.3	49.3	5.51	6.59	9.02	9.80	-5.7

<sup>A</sup>Formula  $S_o = 0.1691x^{0.9397}$  or  
 $\log(S_o) = \log 0.1691 + 0.9397 \log x$   
 $x =$  uranium concentration

<sup>B</sup>Formula  $S_r = 0.2369x^{0.9551}$  or  
 $\log(S_r) = \log 0.2369 + 0.9551 \log x$

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