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# Standard Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions<sup>1</sup>

This standard is issued under the fixed designation C799; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover procedures for the chemical, mass spectrometric, spectrochemical, nuclear, and radiochemical analysis of nuclear-grade uranyl nitrate solution to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

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1.3 *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 precautionary statements are given in Section 5.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- C696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets
- C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride
- C788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals
- C1219 Test Methods for Arsenic in Uranium Hexafluoride
- C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
- C1254 Test Method for Determination of Uranium in Mineral Acids by X-Ray Fluorescence
- C1267 Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration in the Presence of Vanadium
- C1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry
- C1295 Test Method for Gamma Energy Emission from Fission Products in Uranium Hexafluoride and Uranyl Nitrate Solution
- C1296 Test Method for Determination of Sulfur in Uranium Oxides and Uranyl Nitrate Solutions by X-Ray Fluorescence (XRF)<sup>3</sup>
- C1380 Test Method for the Determination of Uranium Content and Isotopic Composition by Isotope Dilution Mass Spectrometry
- C1413 Test Method for Isotopic Analysis of Hydrolyzed Uranium Hexafluoride and Uranyl Nitrate Solutions by Thermal Ionization Mass Spectrometry
- D1193 Specification for Reagent Water
- E12 Terminology Relating to Density and Specific Gravity

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.05 on Methods of Test.

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<sup>2</sup> 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.

<sup>3</sup> Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

of Solids, Liquids, and Gases

**E60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry**

2.2 *American Chemical Society Specification:*

Reagent Chemicals<sup>4</sup>

2.3 *Other Documents:*

**ISO 7097** Determination of Uranium in Uranium Product Solutions and Solids with Cerium IV Oxidation Titrimetric Method<sup>5</sup>

### 3. Significance and Use

3.1 Uranyl nitrate solution is used as a feed material for conversion to the hexafluoride as well as for direct conversion to the oxide. In order to be suitable for this purpose, the material must meet certain criteria for uranium content, isotopic composition, acidity, radioactivity, and impurity content. These methods are designed to show whether a given material meets the specifications for these items described in Specification **C788**.

3.1.1 An assay is performed to determine whether the material has the specified uranium content.

3.1.2 Determination of the isotopic content of the uranium is made to establish whether the effective fissile content is in accordance with the purchaser's specifications.

3.1.3 Acidity, organic content, and alpha, beta, and gamma activity are measured to establish that they do not exceed their maximum limits.

3.1.4 Impurity content is determined to ensure that the maximum concentration limit of certain impurity elements is not exceeded. Impurity concentrations are also required for calculation of the equivalent boron content (EBC), and the total equivalent boron content (TEBC).

### 4. Reagents

4.1 *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>4</sup> 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D1193**.

### 5. Safety Precautions

5.1 Use of this standard does not relieve the user of the obligation to be aware of and to conform to all health and safety requirements.

<sup>4</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 Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>5</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

5.2 The user should also be cognizant of and adhere to all federal, state, and local regulations for processing, shipping, or in any way using uranyl nitrate solutions.

### 6. Sampling

6.1 Criteria for sampling this material are given in Specification **C788**.

## DETERMINATION OF URANIUM

### 7. Scope

7.1 Uranium can be determined using iron (II) reduction and dichromate titration. Test Method **C1267** can be used.

7.2 Uranium can also be determined using cerium (IV) oxidation titrimetry. **ISO 7097** Test Method can be used.

7.3 Uranium can also be determined by X-Ray Fluorescence using Test Method **C1254**.

7.4 Previous sections have been deleted.

## URANIUM BY IGNITION GRAVIMETRY

### 8. Scope

8.1 This test method covers the determination of uranium in nuclear-grade uranyl nitrate solution. Appropriate size sample aliquots are chosen to obtain 5 to 10 g of  $U_3O_8$ .

### 9. Summary of Test Method

9.1 The uranyl nitrate solution is evaporated to dryness, ignited to  $U_3O_8$ , and weighed. Corrections are made for any impurities present (**1, 2**).

### 10. Interferences

10.1 The weight of  $U_3O_8$  is corrected for the nonvolatile impurities present as determined by spectrographic analysis.

10.2 Volatile anions that are difficult to decompose require an extended ignition period.

### 11. Apparatus

11.1 *Heat Lamp*, infrared.

11.2 *Hot Plate*.

11.3 *Muffle Furnace*.

### 12. Procedure

12.1 Transfer a weighed portion of uranyl nitrate solution containing 5 to 10 g of uranium into a preweighed platinum dish and add 2 drops of HF (48 %).

12.2 Position the dish under the heat lamp and evaporate the solution to dryness.

12.3 Place the dish on a hot plate with a surface temperature of about 300°C and heat until most of the nitrate has decomposed.

12.4 Transfer the dish to a muffle furnace and ignite for 2 h at 900°C.

12.5 Remove the dish to a desiccator and allow to cool to room temperature.

12.6 Weigh the dish; then repeat **12.4-12.6** until a constant weight is obtained.

### 13. Calculation

13.1 Calculate the uranium content as follows:

$$\text{Uranium, g/g} = ((B - C)/A) D \quad (1)$$

where:

*A* = sample, g,

*B* =  $U_3O_8$  obtained, g,

*C* = impurity-element oxides, g, and

*D* = gravimetric factor, grams of uranium/grams of  $U_3O_8$  (varies according to uranium enrichment).

### 14. Precision

14.1 The limit of error at the 95 % confidence level for a single determination is  $\pm 0.03$  %.

## SPECIFIC GRAVITY BY PYCNOMETRY

### 15. Scope

15.1 This test method covers the determination of the specific gravity of a solution of uranyl nitrate to  $\pm 0.0004$ .

### 16. Summary of Test Method

16.1 A known volume of the solution adjusted at a controlled temperature is weighed and compared to the weight of water measured in the same container (Terminology E12).

### 17. Apparatus

17.1 *Volumetric Flasks*, 50-mL, Class A.

17.2 *Water Bath*, temperature controlled to  $\pm 0.1^\circ\text{C}$  at a temperature slightly above normal room temperature, and provided with clips for holding volumetric flasks.

### 18. Procedure

18.1 Weigh the clean, dry volumetric flask and its stopper to the nearest 0.1 mg.

18.2 Fill the volumetric flask with the uranyl nitrate solution to a point close to the volume mark, using a thinstemmed funnel and a glass dropper.

18.3 Place the stoppered volumetric flask in the water bath for 30 min.

18.4 Use a finely drawn glass dropper to adjust the liquid volume to the mark.

18.5 Leave the flask in the water bath an additional 10 min to make sure that the bath temperature has been reached.

18.6 Dry and weigh the flask to the nearest 0.1 mg.

18.7 Repeat 18.2-18.6 using boiled and cooled distilled water instead of the uranyl nitrate solution.

### 19. Calculation

19.1 Very accurate determinations of specific gravity require that vacuo corrections be made, but if a median correction figure in terms of grams per grams of sample is applied to the solution weights in all cases the resulting error will not exceed 0.05 %.

$$\text{Sp gr} = \frac{B - A + 0.0007(B - A)}{C - A + 0.0010(C - A)} \quad (2)$$

where:

*B* = sample plus flask in air, g,

*A* = flask in air, g,

*C* = water plus flask in air, g,

0.0007 g/g = correction factor applicable for densities of 1.3 to 1.5, and

0.0010 g/g = correction factor for water.

### 20. Precision

20.1 The limit of error at the 95 % level for a single determination is  $\pm 0.03$  %.

## FREE ACID BY OXALATE COMPLEXATION

### 21. Scope

21.1 This test method covers the determination of the free acid content of uranyl nitrate solutions that may contain a ratio of up to 5 moles of acid to 1 mole of uranium.

### 22. Summary of Test Method

22.1 To a diluted solution of uranyl nitrate, solid, pulverized potassium oxalate is added until a pH of about 4.7 is reached. The solution is then titrated with standard NaOH solution by the delta pH method to obtain the inflection point (3).

### 23. Apparatus

23.1 *pH Meter*, with glass and calomel electrodes.

23.2 *Buret*, Class A, 50-mL.

### 24. Reagents

24.1 *Nitric Acid (2.0 N)*—Dilute 130 mL of  $HNO_3$  (sp gr 1.42) to 1 L with water. Standardize with sodium hydroxide solution (see 24.3).

24.2 *Potassium Oxalate ( $K_2C_2O_4 \cdot H_2O$ )*, crystals.

24.3 *Sodium Hydroxide Solution (0.3 N)*—Dissolve 12.0 g of NaOH in 1 L of water. Standardize with acid potassium phthalate.<sup>6</sup>

### 25. Procedure

25.1 Transfer a 5-mL sample aliquot into a 250-mL beaker.

25.2 Add 100 mL of distilled water or such volume that the uranium concentration will be between 7 and 50 g/L.

25.3 Add a spike of sufficient 2.0 *N* standard  $HNO_3$  to make the sample definitely acid if the sample is neutral or acid deficient.

25.4 Add pulverized  $K_2C_2O_4 \cdot H_2O$  slowly and with constant stirring until a pH of 4.7 to 4.9 is reached.

25.5 Immerse the titration beaker in an ice bath. (Titrations made at room temperature are possible but are less sharp.)

25.6 Titrate with 0.3 *N* NaOH using 0.20-mL increments and determine the inflection point by the delta pH or “analytical” method.

NOTE 1—This test method of locating the end point depends on the fact that the second derivative  $\Delta^2\text{pH}/\Delta\text{vol}^2$  is zero at the point where the slope  $\Delta\text{pH}/\Delta\text{vol}$  is a maximum.

### 26. Calculation

26.1 Calculate the free acid normality, *N*, as follows:

$$N = (A \times N_B - C \times N_A)/5 \quad (3)$$

<sup>6</sup> NBS SRM 84h.

where:

- A = NaOH solution used in the titration, mL
- $N_B$  = normality of the NaOH solution,
- C = HNO<sub>3</sub> solution used in the spike, mL, and
- $N_A$  = normality of HNO<sub>3</sub> solution.

NOTE 2—Negative values of free acid indicate an acid deficiency.

### 27. Precision

27.1 The limit of error at the 95 % confidence level for a single determination is  $\pm 3$  %.

## DETERMINATION OF THORIUM

### 28. Scope

28.1 The determination of thorium by the arsenazo (III) (photometric) method has been discontinued, (see C799-93).

28.2 As an alternative, thorium can be determined using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). See Test Method C1287.

28.3 Previous sections have been deleted.

## DETERMINATION OF CHROMIUM

### 29. Scope

29.1 The determination of chromium by the diphenyl carbazide method has been discontinued, (see C799-93).

29.2 As an alternative, chromium can be determined using inductively coupled plasma atomic emission spectrometry (ICP-AES). Test Method C761 can be used providing a transformation to U<sub>3</sub>O<sub>8</sub> so described hereafter in sections 117–120 A direct conversion to the ammonium fluoride plus nitric acid solution can also be used, (see C761).

29.3 As an alternative, chromium can be determined using atomic absorption spectroscopy. Test Method C761 can be used.

29.4 As an alternative, chromium can be determined using ICP-MS. Test Method C1287 can be used.

29.5 Previous sections have been deleted.

## DETERMINATION OF MOLYBDENUM

### 30. Scope

30.1 The determination of molybdenum by the thiocyanate (photometric) method has been discontinued, (See C799-93).

30.2 As an alternative, molybdenum can be determined using ICP-MS. Test Method C1287 can be used.

30.3 As an alternative, molybdenum can be determined using ICP-AES. Test Method C761, sections 251 to 271 can be used providing a transformation to U<sub>3</sub>O<sub>8</sub> as described hereafter in sections 71-74. A direct conversion to the ammonium fluoride plus nitric acid solution can also be used, (see C761, section 251).

30.4 Previous sections have been deleted.

## HALOGENS SEPARATION BY STEAM DISTILLATION

### 31. Scope

31.1 This test method covers the separation of the halogens by means of a steam distillation.

### 32. Summary of Test Method

32.1 A sample aliquot is mixed with a solution containing ferrous ammonium sulfate, sulfamic acid, phosphoric acid, and sulfuric acid. The halogens are then steam distilled at a temperature of 140°C.

### 33. Apparatus

33.1 *Steam Distillation Apparatus* (see Fig. 1).

33.1.1 *Distilling Flask*, 200-mL with thermometer well.

33.1.2 *Condenser*.

33.1.3 *Heating Mantle*.

33.1.4 *Steam Boiler*, 500-mL flask.

### 34. Reagents

34.1 *Absorber Solution (4 M Potassium Hydroxide)*—Dissolve 22.4 g KOH pellets in water and dilute to 100 mL.

34.2 *Acid Mixture*—Mix 0.2 M ferrous ammonium sulfate-0.5 M sulfamic acid (see 34.3), phosphoric acid (85 %), and sulfuric acid (sp gr 1.84) in the ratio of 1 + 2 + 5.

34.3 *Ferrous Ammonium Sulfate Solution (0.2 M)-Sulfamic Acid (0.5 M) Solution*—Dissolve 78.4 g Fe (NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>·6 H<sub>2</sub>O and 48.6 g NH<sub>2</sub>SO<sub>3</sub>H in H<sub>2</sub>SO<sub>4</sub>(5 + 95) and dilute to 1 L with H<sub>2</sub>SO<sub>4</sub>(5 + 95).

34.4 *Phenolphthalein Solution (10 g/L)*—Dissolve 1 g of phenolphthalein in 50 mL of ethanol and add 50 mL of water.

### 35. Procedure

35.1 Place a weighed portion of about 15 mL containing approximately 5 g of uranium in the distillation flask.

35.2 Add 25 mL of the acid mixture to the distillation flask.

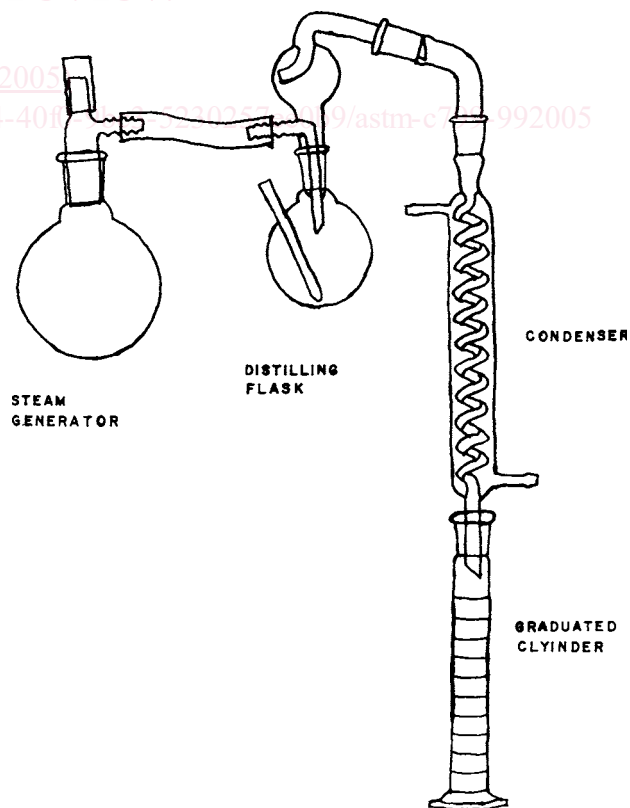


FIG. 1 Halogen Distillation Apparatus



35.3 Transfer 5 mL of the KOH solution to a 100-mL graduated cylinder and position it under the condenser tip.

35.4 Heat the distillation flask until the thermometer in the well reaches 140°C.

35.5 Pass steam through from the boiler, and maintain at a temperature of 140°C until a volume of 90 mL is collected.

35.6 Add 2 drops of phenolphthalein solution and adjust the pH of the distillate with KOH or HNO<sub>3</sub>, to the phenolphthalein end point. Make the volume to 100 mL.

35.7 Repeat the distillation, omitting the uranium sample, to use as the matrix for the fluoride standard curve.

35.8 Reserve the distillate for the fluoride and combined halide determinations.

### FLUORIDE BY SPECIFIC ION ELECTRODE

#### 36. Scope

36.1 This test method covers the determination of as low as 2 µg F/g U in distillate containing all the halogens.

#### 37. Summary of Test Method

37.1 An aliquot of the distillate representing 1 g of uranium is measured by specific ion electrode and compared to a standard curve prepared by spiking equivalent-size aliquots taken from a blank distillation (4,5).

#### 38. Apparatus

38.1 *pH Meter*, expanded scale.

38.2 *Ion-Selective Electrode*, fluoride.

38.3 *Reference Electrode*, single-junction.

#### 39. Reagents

39.1 *Buffer Solution (0.001 N)*—Dissolve 0.1 g of potassium acetate (KC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>) in water. Add 0.050 mL of acetic acid (sp gr 1.05) and dilute to 1 L.

39.2 *Fluoride Standard Solution A (1 mL = 1 mg F)*—Dissolve 0.220 g of dried sodium fluoride (NaF) in 25 mL of water and dilute to 100 mL.

39.3 *Fluoride Standard Solution B (1 mL = 5 µg F)*—Dilute 5 mL of the fluoride standard Solution A (see 39.2) to 1 L with water.

#### 40. Procedure

40.1 Pipet a 20-mL aliquot of the sample distillate (representing about 1 g of uranium) into a 25-mL flask and make to volume with the buffer solution.

40.2 Prepare a standard curve by pipetting 20-mL aliquots from the blank distillate into 25-mL flasks and adding F<sup>-</sup> standard solution to make 0, 5, 10, and 20 µg F<sup>-</sup>/25 mL.

40.3 Measure all of the solutions with the fluoride ion-selective electrode.

#### 41. Calculation

41.1 Calculate the F<sup>-</sup> content as follows:

$$F^-, \mu\text{g/g} = A/B \quad (4)$$

where:

A = F<sup>-</sup> found in the sample distillate aliquot, µg, and

B = uranium represented by the sample distillate aliquot, g.

#### 42. Precision

42.1 The limit of error at the 95 % confidence level for a single determination is ±25 %.

### HALOGEN DISTILLATE ANALYSIS: CHLORIDE, BROMIDE, AND IODIDE BY AMPEROMETRIC MICROTITRIMETRY

#### 43. Scope

43.1 The determination of chloride, bromide and iodide by microtitrimetric method has been discontinued, (see C799-93).

43.2 Previous sections have been deleted.

### DETERMINATION OF CHLORIDE AND BROMIDE

#### 44. Scope

44.1 Determination of bromide by the fluorescein (photo-metric) method has been discontinued, (see C799-93).

44.2 As an alternative, bromide and chloride can be determined by X-Ray Fluorescence. Halogens are precipitated by silver nitrate and filtrated. The precipitate is washed and counted by X-Ray Fluorescence.

44.3 Previous sections have been deleted.

### DETERMINATION OF SULFUR BY X-RAY FLUORESCENCE

#### 45. Scope

45.1 Sulfur can be determined using X-Ray Fluorescence. See Test Method C1296.

### SULFATE SULFUR BY (PHOTOMETRIC) TURBIDIMETRY

#### 46. Scope

46.1 This test method covers the determination of the sulfur concentration, which exists as sulfate in uranyl nitrate solutions, in the range from 100 to 1000 µg S/g of uranium.

#### 47. Summary of Test Method

47.1 The uranium in the sample is removed by extraction with tributyl phosphate (TBP). The sulfate is then precipitated as barium sulfate (BaSO<sub>4</sub>) in the presence of excess salt and acid and is held in suspension in a glycerin matrix. Sulfate is determined turbidimetrically using a spectrophotometer (6, 7).

#### 48. Interferences

48.1 Any anions that form insoluble precipitates with barium, such as phosphate, oxalate, and chromate, will interfere.

48.2 Many variables, although not classed as interferants, effect the precision of this test method. Careful control of the following parameters must be maintained to achieve the stated precision: particle size of the barium chloride (BaCl<sub>2</sub>), particle size of the BaSO<sub>4</sub> formed, total ionic concentration of the final solution, degree of mixing of sample and reagents (number of times the flask is inverted), concentration of hydrogen ion in the final solution, and the length of time of standing of the supernatant before the absorbance is measured.

## 49. Apparatus

49.1 *Spectrophotometer*—See Practice E60.

## 50. Reagents

50.1 *Barium Chloride* ( $BaCl_2$ ), crystals. Sift the salt and use only the portion that passes through a 28-mesh screen and is retained on a 35-mesh screen.

50.2 *Sodium Chloride-Glycerin Solution* (16 g/L)—Dissolve 40 g of NaCl in 60 mL of HCl (sp gr 1.19). Add 833 mL of glycerin and dilute to 2.5 L with water.

50.3 *Sulfate Standard Solution* (1 mL = 1000  $\mu\text{g SO}_4^-$ )—Dissolve 1.1813 g of  $K_2SO_4$ , dried at 110°C for 1 h, and dilute to 1 L with water.

50.4 *Tributyl Phosphate Solution* (3 + 7)—Dilute 300 mL of TBP with 700 mL of kerosine and equilibrate with 8 M  $HNO_3$ .

## 51. Procedure

51.1 Transfer a weighed aliquot of sample that contains approximately 1 g of uranium to a 60-mL separatory funnel. Adjust the nitric acid concentration to 4 to 5 M and the volume to 5 mL.

51.2 Add 10 mL of TBP solution (see 50.4) and equilibrate the solutions.

51.3 Allow the layers to separate and transfer the aqueous layer to 50-mL volumetric flask containing 30 mL of distilled water. Use a minimum volume of 1 N  $HNO_3$  wash solution to ensure quantitative transfer of the aqueous layer to the 50-mL flask.

51.4 Pipet 10 mL of NaCl-glycerin solution into the 50-mL flask and dilute to volume with water.

51.5 Add 0.50 g of  $BaCl_2$  (see 50.1), stopper the flask, and invert the solution 20 times to dissolve the  $BaCl_2$ .

NOTE 3—The conditions of mixing and the time of standing prior to measuring the absorbance *must* be the same for sample and standards.

51.6 Allow the solution to stand  $60 \pm 5$  min; then measure the absorbance at 450 nm in 5-cm cells with a blank containing all of the reagents except sample as the reference.

51.7 Prepare a calibration curve by transferring 0.200, 0.500, 1.000, 1.500, and 2.000-mL aliquots of the standard sulfate solution into 60-mL separatory funnels that contain 5 mL of 4 to 5 M nitric acid and process in accordance with 51.2-51.6.

## 52. Calculation

52.1 Calculate the sulfur content in micrograms per gram of uranium as follows:

$$\text{Sulfur, } \mu\text{g/g} = (A \times B)/C \quad (5)$$

where:

A =  $SO_4$  = found in the sample solution,  $\mu\text{g}$ ,

B = 0.334, the gravimetric factor converting  $SO_4$  = to S, and

C = uranium in the sample solution aliquot, g.

## 53. Precision

53.1 The limit of error at the 95 % confidence level for a single determination is  $\pm 3$  %.

## PHOSPHORUS BY THE MOLYBDENUM BLUE (PHOTOMETRIC) METHOD

### 54. Scope

54.1 This test method covers determination of phosphorus in nuclear-grade uranyl nitrate solutions. Appropriate dilution may be made to facilitate obtaining samples containing 0 to 60  $\mu\text{g P}$ .

### 55. Summary of Test Method

55.1 Phosphorus is determined by the formation of heteropoly molybdophosphoric acid and its subsequent reduction to molybdenum blue. Sodium molybdate is used to complex the P in an acid solution containing the sample. The yellow complex is then extracted into isobutanol. After the excess molybdate is washed out with water, the organic phase is contacted with an acid solution of stannous chloride to reduce the complex. The resulting molybdenum blue is read at 725 nm using a spectrophotometer (8).

### 56. Interferences

56.1 The molybdenum blue reaction is not specific for phosphorus; however, adjustment of the acidity to above 0.9 N avoids the formation of molybdosilicic acid.

56.2 Fluoride and chloride must be fumed off before the heteropoly acid is formed.

### 57. Apparatus

57.1 *Spectrophotometer*—See Practice E60.

### 58. Reagents

58.1 *Isobutanol*.

58.2 *Phosphorus Standard Solution A* (1 mL = 100  $\mu\text{g P}$ )—Dissolve 0.4393 g of  $KH_2PO_4$ , which has been dried at 110°C for 1 h, in 1 L of water.

58.3 *Phosphorus Standard Solution B* (1 mL = 10  $\mu\text{g P}$ )—Dilute 10.0 mL of Solution A to 100 mL with water.

58.4 *Sodium Molybdate Solution* (10 g/L)—Dissolve 25 g of  $Na_2MoO_4 \cdot 2 H_2O$  in 250 mL of water. Filter if turbid and store in a polyethylene bottle.

58.5 *Stannous Chloride Solution* (2 N in HCl)—Dissolve 2.38 g of  $SnCl_2 \cdot 2 H_2O$  in 170 mL of HCl (sp gr 1.19), and dilute to 1 L with water. Store in a polyethylene bottle containing 1 to 2 g of tin metal pellets or granules.

### 59. Procedure

59.1 Transfer a weighed portion of sample solution containing 0.1 g of uranium or an appropriate dilution to a 150-mL beaker.

59.2 Add 3 mL of  $HClO_4$  (72 %) or  $H_2SO_4$  (sp gr 1.84) to the beaker and heat to strong fumes.

59.3 Add 40 mL of water and 5 mL of  $Na_2MoO_4 \cdot 2 H_2O$  solution and let stand 5 min.

59.4 Transfer to a 125-mL separatory funnel.

59.5 Add 40 mL of isobutanol and extract for 1 min.

59.6 Discard the aqueous layer.

59.7 Wash the organic layer with two 25-mL portions of water and discard the aqueous layers.

59.8 Add 25 mL of  $SnCl_2$  solution and shake for 15 s.