



Standard Test Methods for Chemical and Atomic Absorption Analysis of Uranium-Ore Concentrate¹

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^{ε1} NOTE—Sections 1.4 and 7.2 were editorially corrected in August 2010.

1. Scope

1.1 These test methods cover procedures for the chemical and atomic absorption analysis of uranium-ore concentrates to determine compliance with the requirements prescribed in Specification C967.

1.2 The analytical procedures appear in the following order:

	Sections
Uranium by Ferrous Sulfate Reduction—Potassium Dichromate Titrimetry	9
Nitric Acid-Insoluble Uranium	10 to 18
Extractable Organic Material	19 to 26
Determination of Arsenic	27
Carbonate by CO ₂ Gravimetry	28 to 34
Fluoride by Ion-Selective Electrode	35 to 42
Halides by Volhard Titration	43 to 50
Moisture by Loss of Weight at 110°C	51 to 57
Phosphorus by Spectrophotometry	58 to 66
Determination of Silicon	67
Determination of Thorium	68
Calcium, Iron, Magnesium, Molybdenum, Titanium, and Vanadium by Atomic Absorption Spectrophotometry	69 to 78
Potassium and Sodium by Atomic Absorption Spectrophotometry	79 to 88
Boron by Spectrophotometry	89 to 98

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.* A specific precautionary statement is given in Section 7.

¹ 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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2. Referenced Documents

2.1 ASTM Standards:²

- C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride
- C859 Terminology Relating to Nuclear Materials
- C967 Specification for Uranium Ore Concentrate
- C1110 Practice for Sample Preparation for X-Ray Emission Spectrometric Analysis of Uranium in Ores Using the Glass Fusion or Pressed Powder Method
- C1219 Test Methods for Arsenic in Uranium Hexafluoride
- 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
- C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis
- D1193 Specification for Reagent Water
- E60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology C859.

4. Significance and Use

4.1 The test methods in this standard are designed to show whether a given material meets the specifications prescribed in Specification C967.

4.2 Because of the variability of matrices of uranium-ore concentrate and the lack of suitable reference or calibration

² 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.

materials, the precision and bias of these test methods should be established by each individual laboratory that will use them. The precision and bias statements given for each test method are those reported by various laboratories and can be used as a guideline.

4.3 Instrumental test methods such as X-ray fluorescence and emission spectroscopy can be used for the determination of some impurities where such equipment is available.

5. Interferences

5.1 Interferences are identified in the individual test methods.

5.2 Ore concentrates are of a very variable nature; therefore, all interferences are very difficult to predict. The individual user should verify the applicability of each procedure for specific ore concentrates.

6. Reagents

6.1 *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.³ 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.

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

7. Precautions

7.1 Proper precautions should be taken to prevent inhalation or ingestion of uranium during sample preparation and any subsequent sample analysis.

7.2 Hydrofluoric acid is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid is similar to other acids in that the initial extent of a burn depends on the concentration, the temperature, and the duration of contact with the acid. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left untreated. Due to serious consequences of hydrofluoric acid burns, prevention of exposure or injury of personnel is the primary goal. Utilization of appropriate laboratory controls (hoods) and wearing adequate personal protective equipment to protect from skin and eye contact is essential.

8. Sampling

8.1 Collect samples in accordance with Specification **C967**.

³ *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.

8.2 Special requirements for subsampling are given in the individual test methods.

URANIUM BY FERROUS SULFATE REDUCTION—POTASSIUM DICHROMATE TITRIMETRY

9. Scope

9.1 This test method covers the determination of uranium in uranium-ore concentrates. This test method was discontinued in January 2002 and replaced with Test Method **C1267**.

9.2 The uranium content of the sample may also be determined using Test Method **C1254**. The user's laboratory must establish and document method performance.

NOTE 1—Dissolution of UOC samples may be achieved using the techniques or combination of techniques described in **C1347**. The laboratory must validate the performance of **C1347** using characterized UOC samples. If **C1347** methods are not suitable for UOC sample dissolution, the user may establish and document applicable dissolution methods.

NITRIC ACID-INSOLUBLE URANIUM

10. Scope

10.1 This test method covers the determination of that quantity of uranium in uranium-ore concentrate that is not soluble in nitric acid.

11. Summary of Test Method

11.1 A sample of ore concentrate is digested in 10 *M* nitric acid at 95 to 100°C for 1 h. The slurry is filtered and the residue washed with 1 *M* nitric acid until the filtrate gives a negative test for uranium. The washed residue is then dried and ignited at 1000 ± 25°C for 1 h. The uranium content is determined on the ignited residue by spectrophotometry.

12. Interference

12.1 At the specification limit for nitric acid insoluble uranium usually established for uranium-ore concentrates, interference effects are insignificant.

13. Apparatus

13.1 *Digestion Flask*, 500-mL, with side entry tube and attached reservoir.

13.2 *Stirring Apparatus*, with sleeve-type stirrer.

13.3 *Heating Mantle*, 250-W, controlled by a variable transformer.

13.4 *Büchner Funnel*.

13.5 *Porcelain Crucibles*, 40-mL.

13.6 *Muffle Furnace*.

13.7 *Filter Paper*,⁴ of medium porosity.

13.8 *Spectrophotometer*, with 1-cm cells that are in accordance with Practice **E60**.

14. Reagents

14.1 *Nitric Acid* (10 *M*)—Dilute 62.5 mL of HNO₃ (sp gr 1.42) to 100 mL with distilled water.

⁴ Whatman brand No. 40 or its equivalent has been found suitable.

14.2 *Nitric Acid* (1 *M*)—Dilute 62.5 mL of HNO₃ (sp gr 1.42) to 1 L with distilled water.

14.3 *Sodium Hydroxide* (100 g/L)—Dissolve 10 g of NaOH in 100 mL of water.

14.4 *Hydrogen Peroxide* (H₂O₂, 30 %).

14.5 *Hydrochloric Acid* (HCl, sp gr 1.19).

14.6 *Hydrofluoric Acid* (HF, 48 %).

14.7 *Sulfuric Acid* (9 *M*)—Add 500 mL H₂SO₄ (sp gr 1.84) to 500 mL of iced water with constant stirring. Cool and dilute to 1 L with water.

15. Procedure

15.1 Weigh a 50.0 ± 0.1-g sample directly into the digestion flask.

15.2 Place the flask in the heating mantle and adjust the support ring so that the joints of the flask and sleeve stirrer are engaged, and the stirrer blades turn freely but just clear the bottom of the flask.

15.3 Transfer 95 mL of 10 *M* nitric acid to a 250-mL beaker and heat between 95 to 100°C.

15.4 Slowly transfer the heated nitric acid solution to the digestion flask through the entry side tube with the stirrer turning.

NOTE 2—The stirrer is started before the acid is added to prevent material from sticking to the flask.

15.5 Align a thermometer in such a manner that the mercury chamber of the thermometer is immersed in the stirring slurry, but adequately clears the turning stirrer blades.

15.6 Quickly bring the sample to 97°C and digest between 95 to 100°C for 1 h while stirring. (Measure the 1-h digestion time after the temperature of the slurry has reached 97°C.)

15.7 Turn off the variable transformer, but allow the stirrer to continue turning.

15.8 Remove the thermometer and carefully rinse with water all slurry that adheres to it.

15.9 Wipe the immersed portion of the thermometer with one fourth of a circle of filter paper and transfer the paper to a prepared Büchner funnel fitted with a filter paper.

15.10 Add 10 mL of paper pulp to the slurry and continue stirring for about 5 min.

15.11 Turn off the stirrer, then lower the flask and mantle.

15.12 Carefully wash the slurry that adheres to the stirrer shaft and blades into the flask with water.

15.13 Wipe the shaft and blades with one fourth of a circle of filter paper and transfer the filter paper to the Büchner funnel.

15.14 Filter the slurry through the Büchner funnel and wash contents of the flask into the funnel.

15.15 Wash the residue with 1 *M* nitric acid until a 10-mL portion of the filtrate shows no detectable yellow color when made basic with sodium hydroxide and after a few drops of H₂O₂ (30 %) have been added as a color developer.

15.16 Wash the residue several times with water after a negative test is obtained.

15.17 Draw air through the filter until the residue and filter pad are dry.

15.18 Scrape the residue and paper into a preignited (1000°C) tared 40-mL crucible, place on a hot plate and slowly char off the organic material.

15.19 Ignite the residue for 1 h at 1000°C in a muffle furnace.

15.20 Cool the crucible in a desiccator and weigh.

15.21 Calculate the percentage of solids in accordance with 17.1.

NOTE 3—If the percentage of solids (insoluble residue) is greater than 0.1 %, grind and mix the residue and determine the total milligrams of uranium in the residue by the photometric procedure in 16.1-16.10.

16. Photometric Procedure for Uranium

16.1 Transfer the ground, blended residue from 15.20 to a 100-mL beaker.

16.2 Add 10 mL of water and 10 mL of HCl (sp gr 1.19), cover, and boil for 10 min.

16.3 Add 5 mL of HNO₃ (sp gr 1.42) and boil until fuming of NO₂ ceases. Remove cover glass.

16.4 Add 5 mL of 9 *M* H₂SO₄ and 2 mL of HF (48 %), then heat to dryness on the hotplate. Bake to fume off remaining H₂SO₄ and cool.

16.5 Wash down sides of beaker with water and add 5 mL of HNO₃.

16.6 Cover with a watchglass and digest for approximately 10 min near the boiling point.

16.7 Quantitatively transfer the solution to a 250-mL volumetric flask. Add 25 mL of NaOH solution and a few drops of H₂O₂. Make up to mark with water and mix.

NOTE 4—The solution must be basic for yellow sodium peruranate color to develop.

16.8 Measure the absorbance of the solution in a spectrophotometer at 425 nm in a 1-cm cell using a blank as reference. The blank is prepared by diluting 25 mL of NaOH, plus a few drops of H₂O₂, to 250 mL with water.

16.9 Prepare a calibration curve covering the range from 0 to 50 mg of uranium from aliquots of a standard uranium solution. Proceed as in 16.5-16.8. Plot the milligrams of uranium against absorbance readings.

16.10 Determine the total milligrams of uranium in the sample solution from the calibration curve.

NOTE 5—If the sample solution falls outside the calibration range, dilute a portion with the reference-blank solution and read again.

17. Calculation

17.1 Calculate the percentage of insoluble residue, *R*, present as follows:

$$R = \frac{R_w \times 100}{S_w} \quad (1)$$

where:

R_w = weight of residue (see 15.20), g, and

S_w = weight of samples, g.

17.2 If the insoluble residue exceeds 0.1 %, calculate the percentage of nitric acid-insoluble uranium, *U_N*, and present as follows:

$$U_N = \frac{U}{S_w \times 10} \quad (2)$$