



Designation: C1022 – 17 (Reapproved 2022)

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

This standard is issued under the fixed designation C1022; 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 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
Phosphorus by Spectrophotometry	52 to 60
Determination of Silicon	61
Determination of Thorium	62
Calcium, Iron, Magnesium, Molybdenum, Titanium, and Vanadium by Atomic Absorption Spectrophotometry	63 to 72
Potassium and Sodium by Atomic Absorption Spectrophotometry	73 to 82
Boron by Spectrophotometry	83 to 92

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. A specific precautionary statement is given in Section 7.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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 Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma-Atomic Emission Spectroscopy (Withdrawn 2014)³
- C1219 Test Methods for Arsenic in Uranium Hexafluoride (Withdrawn 2015)³
- 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
- C1843 Test Method for Determining Moisture Content in Uranium-Ore Concentrate
- D1193 Specification for Reagent Water
- E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

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.

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.

³ The last approved version of this historical standard is referenced on www.astm.org.

4.2 Because of the variability of matrices of uranium-ore concentrate and the limited availability of suitable reference or calibration 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.⁴ 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 type I water in 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 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. Familiarization and compliance with the Safety Data Sheet is essential.

7.3 Chloroform is a dangerous chemical causing acute toxicity with repeated inhalation, dermal, or oral exposure. Many health hazards are associated with exposure to chloroform including its potential to cause cancer. Familiarization and compliance with the Safety Data Sheet is essential.

⁴ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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. Sampling

8.1 Collect samples in accordance with Specification C967.

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 Practice C1347. The laboratory must validate the performance of Practice C1347 using characterized UOC samples. If Practice 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 °C 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 °C ± 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*, ashless of medium porosity, and a medium flow rate with a particle retention of 8 µm has been found to be suitable.