INTERNATIONAL

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Standard Test Methods for Arsenic in Uranium Hexafluoride[1]

This standard is issued under the fixed designation C1219; 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 are applicable to the determination of total arsenic in uranium hexafluoride (UF_6) by atomic absorption spectrometry. Two test methods are given: Test Method A—Arsine Generation-Atomic Absorption (Sections 5-10), and Test Method B—Graphite Furnace Atomic Absorption (Appendix X1).

1.2 The test methods are equivalent. The limit of detection for each test method is 0.1 µg As/g U when using a sample containing 0.5 to 1.0 g U. Test Method B does not have the complete collection details for precision and bias data thus the method appears as an appendix.

1.3 Test Method A covers the measurement of arsenic in uranyl fluoride (UO_2F_2) solutions by converting arsenic to arsine and measuring the arsine vapor by flame atomic absorption spectrometry.

1.4 Test Method B utilizes a solvent extraction to remove the uranium from the UO_2F_2 solution prior to measurement of the arsenic by graphite furnace atomic absorption spectrometry.

1.5 Both insoluble and soluble arsenic are measured when UF_6 is prepared according to Test Method C761.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride

C787 Specification for Uranium Hexafluoride for Enrichment

D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 *Arsine Generation-Atomic Absorption Spectrometry Method*—The sample of UF_6 is hydrolyzed and the UO_2F_2 solution is fumed with sulfuric acid in the presence of boric acid to complex the fluoride. Potassium iodide is used to reduce arsenic(V) to arsenic(III). Sodium borohydride is used to generate arsine vapor in a hydride generator with subsequent measurement by flame atomic absorption spectrometry.

3.2 *Graphite Furnace Atomic Absorption Spectrometry Method*—The sample of UF_6 is hydrolyzed, and the uranium in the UO_2F_2 solution is removed by extraction with tri(2-ethylhexyl)phosphate/heptane. The aqueous phase containing the arsenic is analyzed by graphite furnace atomic absorption.

4. Significance and Use

4.1 Arsenic compounds are suspected to cause corrosion in some materials used in UF_6 handling equipment. Arsenic originates as a contaminant in fluorspar (CaF_2) used to produce anhydrous hydrogen fluoride which is used subsequently in the production of UF_6.

[1] This 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] 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.

4.2 These test methods are used to measure the arsenic content in UO_2F_2 solutions prepared from the hydrolysis of UF_6 for determination of conformance to Specification C787.

TEST METHOD A—ARSINE GENERATION-ATOMIC ABSORPTION SPECTROMETRY

5. Interferences

5.1 The presence of hydrofluoric acid in the sample suppresses arsine generation when using sodium borohydride. Boric acid is added to complex the fluoride present at a molar excess of 250 %.[3]

5.2 Arsenic(V) must be reduced to arsenic(III) otherwise arsine will not be generated using sodium borohydride and hydrochloric acid.

5.3 The reduction of arsenic(V) by potassium iodide is time dependent at room temperature requiring strict adherence to the procedure.

5.4 Do not use platinum labware.

6. Apparatus

6.1 *Atomic Absorption Spectrometer,* equipped with an air-acetylene burner, arsenic hollow cathode lamp and hydride generator, gas/liquid separator, and hydride absorption cell.

6.2 *Hot Plate,* capable of reaching a surface temperature of 500°C.

7. Reagents and Materials

7.1 *Reagents:*

7.1.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents 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.

7.1.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean reagent water Type II conforming to Specification D1193.

7.1.3 *Acetylene* (C_2H_2), 99.6 % minimum purity.

7.1.4 *Air,* compressed breathing air or equivalent.

7.1.5 *Arsenic Standard Stock Solution* (1000 mg As/L)—Dissolve 1.320 g of arsenic trioxide (As_2O_3) in 100 mL of hydrochloric acid (1 + 2) and dilute to 1 L. Commercially available stock solutions traceable to NIST primary standards may be used.

TABLE 1 Atomic Absorption Operating Parameters

Element	arsenic
Wavelength, nm	193.7
Lamp current, mA	10
Slit width, nm	0.5
Gas	C_2H_2/air
Acetylene, psig	9
Air, psig	40
Argon, psig	50
Fuel flow, L/min	1.5
Oxidant flow, L/min	4.0

7.1.6 *Arsenic Standard Solution* (0.10 mg As/L)—Pipet 10 mL of 1000 mg/L arsenic stock solution into a 1-L volumetric flask containing 500 mL of water. Add 20 mL of concentrated hydrochloric acid, dilute to volume with water and mix. This (10 mg/L) solution should be kept no longer than one month. Pipet 2 mL of the 10 mg/L arsenic solution into a 200-mL volumetric flask containing 100 mL of water. Add 4 mL of concentrated hydrochloric acid and dilute to volume with water.

NOTE 1—The 0.10-mg As/L solution must not be kept longer than one day.

7.1.7 *Boric Acid* (H_3BO_3).

7.1.8 *Hydrochloric Acid* (sp gr 1.18)—Concentrated hydrochloric acid (HCl).

7.1.9 *Hydrochloric Acid* (1 + 1)—Add one volume of concentrated hydrochloric acid to one volume of water.

7.1.10 *Hydrochloric Acid* (1 + 2)—Add one volume of concentrated hydrochloric acid to two volumes of water.

7.1.11 *Nitrogen* (N_2), 99.9 % minimum purity.

7.1.12 *Potassium Iodide Solution* (50 % w/v)—Dissolve 50 g of potassium iodide in water and dilute to 100 mL in a volumetric flask. Store in a brown bottle.

NOTE 2—The colorless solution is stable for two days. A yellow tinge indicates the solution has deteriorated.

7.1.13 *Sodium Borohydride Solution* (6.0 g/L)—Dissolve 3.0 g of sodium borohydride ($NaBH_4$) and 2.5 g of sodium hydroxide (NaOH) in water and dilute to 500 mL in a volumetric flask. This solution should be prepared weekly.

7.1.14 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

8. Calibration and Standardization

8.1 *Instrument Parameters*—A set of suggested atomic absorption operating parameters is listed in Tables 1 and 2. The parameters may vary with the type of instrument used and the manufacturer's instructions.

8.2 *Preparation of Calibration Solutions:*

8.2.1 Aliquot 0, 2, 5, 10, 20, and 30 mL of the 0.10 mg As/L solution into 100-mL volumetric flasks. Add 2 mL concentrated H_2SO_4 and 10 mL concentrated HCl to each flask.

8.2.2 Add 2 mL of 50 % potassium iodide solution and dilute to volume with water 75 min before running the calibration solutions.

8.3 *Calibration:*

[3] Petrik, K., and Krivan, V., "Radiotracer Investigation of the Interference of Hydrofluoric Acid in the Determination of Arsenic and Antimony by Hydride Generation Atomic Absorption Spectroscopy," *Analytical Chemistry, Vol 59*, No. 20 (1987), pp. 2426–2427.

[4] *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.