Designation: C761 – 18

Standard Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride[1]

This standard is issued under the fixed designation C761; 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 or give reference to procedures for subsampling and for chemical, mass spectrometric, spectrochemical, nuclear, and radiochemical analysis of uranium hexafluoride (UF_6). Most of these test methods are in routine use to determine conformance to UF_6 specifications in the Enrichment and Conversion Facilities.

1.2 The analytical procedures in this document appear in the following order:

NOTE 1—Subcommittee C26.05 will confer with C26.02 concerning the renumbered section in Test Methods C761 to determine how concerns with renumbering these sections are best addressed in subsequent publications as analytical methods are replaced with stand-alone analytical methods.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* (For specific safeguard and safety consideration statements, see 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.*

2. Referenced Documents

2.1 The following documents of the issue in effect on date of material procurement form a part of this specification to the extent referenced herein:

2.2 *ASTM Standards:*[2]

C787 Specification for Uranium Hexafluoride for Enrichment

C799 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions

C859 Terminology Relating to Nuclear Materials

[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] 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.

C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U
C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials
C1219 Test Methods for Arsenic in Uranium Hexafluoride (Withdrawn 2015)[3]
C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
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 and Decay Products in Uranium Hexafluoride and Uranyl Nitrate Solution
C1344 Test Method for Isotopic Analysis of Uranium Hexafluoride by Single-Standard Gas Source Mass Spectrometer Method
C1346 Practice for Dissolution of UF_6 from P-10 Tubes
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
C1428 Test Method for Isotopic Analysis of Uranium Hexafluoride by Single–Standard Gas Source Multiple Collector Mass Spectrometer Method
C1429 Test Method for Isotopic Analysis of Uranium Hexafluoride by Double-Standard Multi-Collector Gas Mass Spectrometer
C1441 Test Method for The Analysis of Refrigerant 114, Plus Other Carbon-Containing and Fluorine-Containing Compounds in Uranium Hexafluoride via Fourier-Transform Infrared (FTIR) Spectroscopy
C1474 Test Method for Analysis of Isotopic Composition of Uranium in Nuclear-Grade Fuel Material by Quadrupole Inductively Coupled Plasma-Mass Spectrometry
C1477 Test Method for Isotopic Abundance Analysis of Uranium Hexafluoride and Uranyl Nitrate Solutions by Multi-Collector, Inductively Coupled Plasma-Mass Spectrometry
C1508 Test Method for Determination of Bromine and Chlorine in UF_6 and Uranyl Nitrate by X-Ray Fluorescence (XRF) Spectroscopy
C1539 Test Method for Determination of Technetium-99 in Uranium Hexafluoride by Liquid Scintillation Counting
C1561 Guide for Determination of Plutonium and Neptunium in Uranium Hexafluoride and U-Rich Matrix by Alpha Spectrometry
C1636 Guide for the Determination of Uranium-232 in Uranium Hexafluoride
C1689 Practice for Subsampling of Uranium Hexafluoride
C1742 Test Method for Isotopic Analysis of Uranium Hexafluoride by Double Standard Single-Collector Gas Mass Spectrometer Method
D1193 Specification for Reagent Water
D3084 Practice for Alpha-Particle Spectrometry of Water
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

2.3 *American Chemical Society Specification:*
Reagent Chemicals [4]

2.4 *Other Specifications:*
Uranium Hexafluoride: Base Charges, Use Charges, Special Charges, Table of Enriching Services, Specifications, and Packaging[5]
USEC 651 Good Handling and Practices for UF_6

2.5 *ANSI Standards:*[6]
ANSI N 14.1 Nuclear Material-Uranium Hexafluoride-Packaging for Transport

2.6 *ISO Standards:*
ISO 7195 Nuclear Energy-Packaging of Uranium Hexafluoride (UF_6) for Transport

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms relating to the nuclear fuel cycle, refer to Terminology C859.

4. Significance and Use

4.1 Uranium hexafluoride is a basic material used to prepare nuclear reactor fuel. To be suitable for this purpose the material must meet criteria for uranium content, isotopic composition, metallic impurities, hydrocarbon and halohydrocarbon content. These test methods are designed to determine whether the material meets the requirements described in Specifications C787 and C996.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all procedures. Unless otherwise indicated, 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 that it is first established that the reagent to be used is of 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 mean reagent water conforming to Specification D1193.[7]

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

[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.

[5] United States Department of Energy, Oak Ridge, TN 37830.

[6] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

[7] Type 1 and 2 water have been found to be suitable.

6. Rejection

6.1 Rejection or acceptance criteria are described in Specifications C787 and C996.

7. Safety Considerations

7.1 Since UF_6 is radioactive, toxic, and highly reactive, especially with reducing substances and moisture (see Uranium Hexafluoride: Handling Procedures and Container Criteria, sections 2.4 through 2.6), appropriate facilities and practices for sampling and analysis must be provided.

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 *Committee C26 Safeguards Statement:*

7.3.1 The material (uranium hexafluoride) to which these test methods apply, is subject to nuclear safeguards regulations governing its possession and use. The following analytical procedures in these test methods have been designated as technically acceptable for generating safeguards accountability measurement data: Gravimetric Determination of Uranium; Titrimetric Determination of Uranium; All Isotopic Analyses.

7.3.2 When used in conjunction with appropriate certified Reference Materials (CRMs), these procedures can demonstrate traceability to the national measurement base. However, adherence to these procedures does not automatically guarantee regulatory acceptance of the resulting safeguards measurements. It remains the sole responsibility of the user of these test methods to assure that its application to safeguards has the approval of the proper regulatory authorities.

SUBSAMPLING OF URANIUM HEXAFLUORIDE

8. Scope

8.1 This test method has been discontinued (see C761 – 04$^{\varepsilon 1}$). The subsampling of UF_6 from bulk sample containers into smaller containers suitable for laboratory analyses has been published as a separate Practice C1689.

GRAVIMETRIC DETERMINATION OF URANIUM

9. Scope

9.1 Practice C1346 is applicable to the hydrolysis of uranium hexafluoride in polychlorotrifluoroethylene (P10) tubes. The following test method is then applicable to the direct gravimetric determination of uranium.

10. Summary of Test Method

10.1 A sample of uranium hexafluoride is weighed, cooled in liquid nitrogen, and hydrolyzed with water. The uranyl fluoride solution produced is evaporated to dryness and converted to uranic oxide by pyrohydrolysis. The uranium content is determined from the weight of the uranium oxide after correcting for stoichiometry based on isotopic content, ignition conditions, and nonvolatile impurities. Ref. **(1-4)**.[8]

11. Interferences

11.1 Nonvolatile impurities affect the accuracy of the method and must be measured by spectrographic analysis with corrections applied.

12. Apparatus

12.1 *Polytrifluorochloroethylene (PTFCE) Sample Tube, TFCE Gasket, Flare Nut, and Plug,* see Fig. 1.

12.2 *Platinum Boat and Cover*—The cover should be platinum gauze (52 mesh) and shaped to cover the boat (Fig. 2).

12.3 *Muffle Furnace,* must be capable of operating continuously at 875°C and maintain this temperature within ±25°C. The furnace shall be equipped with a steam supply that is passed through a tube furnace to preheat the steam to 875°C.

12.4 *Tube Furnace,* must be capable of operating continuously at 875°C and maintain this temperature within 25°C.

12.5 *Infrared Heat Lamps,* 250 watts.

12.6 *Analytical Balance.*

12.7 *Vacuum Oven.*

12.8 *Dewar Flask,* stainless steel.

12.9 *Spatula,* platinum.

12.10 *PTFCE Rod,* 120 mm long and 1.6 mm in diameter.

12.11 *Forceps,* platinum tipped.

12.12 *Jig,* suitable for holding the TFCE sample tube so that it can be opened with a wrench.

12.13 *Box Wrench,* to fit sample tube plug.

12.14 *Beaker,* stainless steel, 125 mL capacity.

13. Reagents

13.1 *Liquid Nitrogen.*

13.2 *Nitric Acid (sp gr 1.42)*—concentrated nitric acid (HNO_3).

13.3 *Nitric Acid* (4*M*)—Mix 500 mL of concentrated HNO_3 with 1500 mL of distilled water.

13.4 *Detergent.*

14. Sampling

14.1 A UF_6 sample is taken as described in Practice C1689.

15. Procedure

15.1 Inspect the PTFCE sample tube for leaks.

NOTE 2—An indication of a leak is a yellow-green residue on the flare nut and cap or a yellow discoloration in the tube. Discard the sample if a leak is indicated.

15.2 Allow the sample tube to stand overnight in the laboratory.

[8] The boldface numbers in parentheses refer to a list of references at the end of these test methods.