Designation: C 761 – 04$^{\epsilon 1}$

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

This standard is issued under the fixed designation C 761; 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.

This standard has been approved for use by agencies of the Department of Defense. 4

ε[1] NOTE—Two versions of Table 1 appeared in this standard. The incorrect version was editorially deleted.

1. Scope

1.1 These test methods cover 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, as analytical methods are replaced with stand-alone analytical methods, are best addressed in subsequent publications.

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.* (For specific safeguard and safety consideration statements, see Section 6.)

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]

C 696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets

C 753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C 787 Specification for Uranium Hexafluoride for Enrichment

C 799 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions

C 996 Specification for Uranium Hexafluoride Enriched to

[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.

Current edition approved July 1, 2004. Published August 2004. Originally approved in 1973. Last previous edition approved in 2001 as C 761 – 01.

[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.

Less Than 5% ^{235}U
C 1128 Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials
C 1219 Test Methods for Arsenic in Uranium Hexafluoride
C 1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
C 1267 Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration in the Presence of Vanadium
C 1287 Test Method for Determination of Impurities in Uranium Dioxide by Inductively Coupled Plasma Mass Spectrometry
C 1295 Test Method for Gamma Energy Emission from Fission Products in Uranium Hexafluoride[2]
C 1344 Test Method for Isotopic Analysis of Uranium Hexafluoride by Single-Standard Gas Source Mass Spectrometer Method
C 1346 Practice for Dissolution of UF_6 from P-10 Tubes
C 1380 Test Method for the Determination of Uranium Content and Isotopic Composition of Isotope Dilution Mass Spectrometry
C 1413 Test Method for Isotopic Analysis of Hydrolyzed Uranium Hexafluoride and Uranyl Nitrate Solutions By Thermal Ionization Mass Spectrometry
C 1428 Test Method for Isotopic Analysis of Uranium Hexafluoride by Single-Standard Gas Source Multiple Collector Mass Spectrometer Method
C 1429 Test Method for Isotopic Analysis of Uranium Hexafluoride by Double-Standard Multi-Collector Gas Mass Spectrometer
C 1441 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
C 1474 Test Method for Analysis of Isotopic Composition of Uranium in Nuclear-Grade Fuel Material by Quadrupole Inductively Coupled Plasma-Mass Spectrometry
C 1477 Test Method for Isotopic Abundance Analysis of Uranium Hexafluoride by Multi-collector Inductively Coupled Plasma-Mass Spectrometry
C 1508 Test Method for Determination of Bromine and Chlorine in UF6 and Uranyl Nitrate by X-Ray Fluorescence
D 1193 Specification for Reagent Water
E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals
E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis
E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes

2.3 *American Chemical Society Specification:*
Reagent Chemicals [3]

2.4 *Other Specifications:*
Uranium Hexafluoride: Base Charges, Use Charges, Special Charges, Table of Enriching Services, Specifications, and Packaging[4]
USEC 651 Good Handling and Practices for UF_6

2.5 *ANSI Standards:*[5]
ANSI N 14.1 Nuclear Material- Uranium Hexafluoride-Packaging for Transport
ISO 7195 Nuclear Energy-Packaging of Uranium Hexafluoride (UF_6) for Transport

3. Significance and Use

3.1 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 Specification C 787.

4. Reagents

4.1 *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.[6] 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean reagent water conforming to Specification D 1193.[7]

5. Rejection

5.1 Rejection or acceptance criteria are described in Specifications C 787 and C 996.

6. Safety Considerations

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

6.2 *Committee C-26 Safeguards Statement*:

6.2.1 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.

[3] "Reagent Chemicals, American Chemical Society Specifications," Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, See "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

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

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

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

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

6.2.2 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 (1, 2)[6]

7. Scope

7.1 This test method is applicable to the subsampling (3) of UF_6 from bulk sample containers into smaller containers suitable for laboratory analyses. The procedure includes sample filtration that facilitates determination of both soluble and insoluble chromium compounds.

8. Summary of Test Method

8.1 The UF_6 in the bulk sample container is liquefied and homogenized by vigorous shaking. The container is inverted and connected to the top of a heated vacuum-manifold system, and the subsample container is attached to the appropriate port of the system. The system is evacuated, and the liquid UF_6 is allowed to flow by gravity into the subsample container. Table 1 summarizes the purpose of various subsamplings.

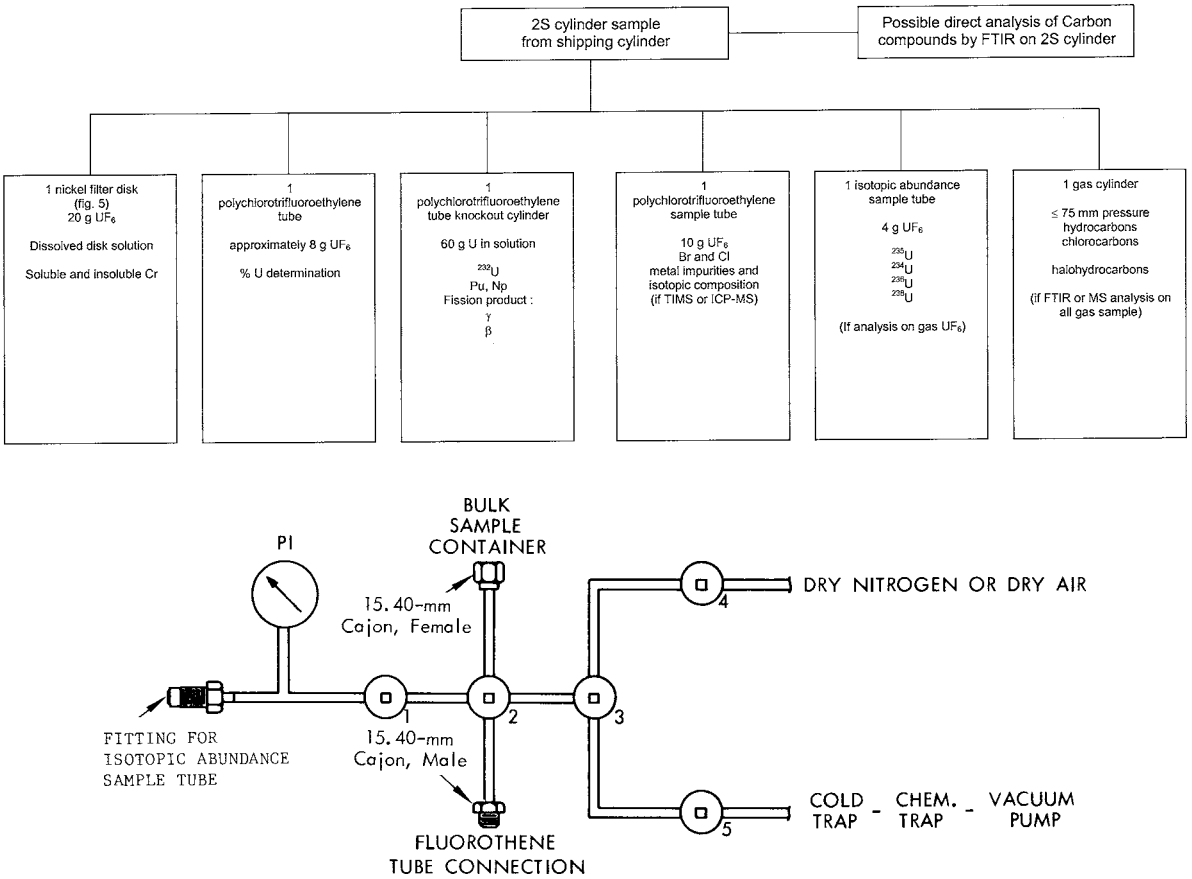
8.2 If the determination of the volatile component of an impurity is needed (for example, chromium compounds), a total gas tranfer may be necessary. In such case, the tared liquid UF_6 bulk sample container is connected to the top of the heated manifold without inversion, so that gaseous UF_6 is transferred.

9. Apparatus

9.1 *Hot Water Bath.*

9.2 *Heated Vacuum Manifold with Liquid Nitrogen Cold Trap* (Table 1).

TABLE 1 – Subsamples for UF_6 Specification Analyses



NOTE 1—All lines are ⅜-in. (9.5-mm) Monel tubing.

NOTE 2—All valves are Monel diaphragm-type valves.

NOTE 3—The valves and lines are wrapped with heating tape to maintain a system temperature of about 80°C.

NOTE 4—Valve 2 is a 3-way valve modified to make it a 4-way valve. When the valve is closed, the polychlorotrifluoroethylene tube is isolated from the system, but the lines from Valve 1 to Valve 3 and to the bulk container are open.