Designation: C1880 – 19

Standard Practice for Sampling Gaseous Uranium Hexafluoride using Alumina Pellets[1]

This standard is issued under the fixed designation C1880; 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 This practice is applicable to sampling gaseous uranium hexafluoride (UF_6) from processing facilities, isotope enrichment cascades or storage cylinders, using the sorbent properties of aluminum oxide (Al_2O_3).

1.2 It is based on the 'ABACC-Cristallini Method' (**1, 2**)[2] and is intended to be used for the determination of uranium (U) isotopic composition required for nuclear material safeguards as well as other applications.

1.3 The application of this practice assures the resulting sample vessel contains no UF_6 and hydrogen fluoride (HF); therefore, it may be handled and categorized for transport under less stringent constraints.

1.4 The scope of this practice does not include provisions for preventing criticality.

1.5 *Units*—The values stated in SI units are to be regarded as the standard. When non-SI units are provided, they are for information only.

1.6 *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.*

1.7 *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 *ASTM Standards:*[3]

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

C787 Specification for Uranium Hexafluoride for Enrichment

C859 Terminology Relating to Nuclear Materials

C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U

C1052 Practice for Bulk Sampling of Liquid Uranium Hexafluoride

C1346 Practice for Dissolution of UF_6 from P-10 Tubes

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

C1672 Test Method for Determination of Uranium or Plutonium Isotopic Composition or Concentration by the Total Evaporation Method Using a Thermal Ionization Mass Spectrometer

C1703 Practice for Sampling of Gaseous Uranium Hexafluoride for Enrichment

C1832 Test Method for Determination of Uranium Isotopic Composition by the Modified Total Evaporation (MTE) Method Using a Thermal Ionization Mass Spectrometer

C1871 Test Method for Determination of Uranium Isotopic Composition by the Double Spike Method Using a Thermal Ionization Mass Spectrometer

D1193 Specification for Reagent Water

[1] This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.02 on Fuel and Fertile Material Specifications.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[3] 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.

2.2 *Other Documents:*

USEC-651 Revision 10 The UF_6 Manual – Good Handling Practices for Uranium Hexafluoride[4]

IAEA-TECDOC-771 Manual on Safe Production, Transport, Handling and Storage of Uranium Hexafluoride[5]

3. Terminology

3.1 Terminology C859 contains terms, definitions, descriptions of terms, nomenclature, and explanations of acronyms and symbols specifically associated with standards under the jurisdiction of Committee C26 on Nuclear Fuel Cycle.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *sample vessel*—the small vessel, for example a P-10 tube, into which the UF_6 sample is transferred and stored for characterization in the analytical laboratory. It can be made of polychlorotrifluorethylene (PCTFE), polytetrafluoroethylene (PTFE) or any other material chemically resistant to UF_6. The dimensions of the P-10 tube and its components, gasket, nut and plug, can be found in Practice C1346.

3.2.2 *sampling manifold*—a set-up used to connect the sample vessel to the UF_6 processing lines, isotope enrichment cascades or storage cylinders, allowing vacuum pumping or pressurization by the introduction of an inert gas.

4. Summary of Practice

4.1 A tared P-10 tube filled with a weighed quantity of aluminum oxide (Al_2O_3) pellets, hereafter referred to as alumina, is attached to a sampling manifold, and exposed to gaseous UF_6 for a timed period, typically 10 to 30 min, depending on the gas pressure in the facility and the desired amount of U amount to be deposited in the P-10 tube. UF_6 readily reacts in contact with alumina pellets generating predominantly uranyl fluoride (UO_2F_2).

4.2 When the sampling period is over, the P-10 tube and the manifold are evacuated to remove remaining gaseous compounds. Next, the valve that connects the manifold to the processing line is closed and the P-10 tube and the manifold are pressurized to atmospheric pressure with dry nitrogen (N_2) to prevent moisture build-up within them.

4.3 These evacuation and pressurization operations must be repeated twice to assure no UF_6 or HF is present in the vessel, but only solid UO_2F_2 on the alumina support. This is of utmost importance to achieve a less stringent sample vessel categorization for transport.

4.4 The P-10 tube can now be safely removed from the manifold and sealed. The amount of U in the P-10 tube can be determined by re-weighing the P-10 tube.

4.5 Using a P-10 tube loaded with 1 g of alumina pellets, and sampling from UF_6 processing lines, enrichment cascades or storage cylinders under typical conditions will easily allow the adsorption of 100 to 300 mg of U, based on the gas pressure, temperature and exposure time. The maximum adsorption capacity is approximately 600 mg of U per gram of alumina.

4.6 At the analytical laboratory, the alumina pellets are transferred from the P-10 tube to an Erlenmeyer flask and leached initially with distilled water and finally with nitric acid (HNO_3). The alumina fines produced must be carefully removed from the solution so that it can be prepared for the determination of the U isotopic composition using, for example, Test Methods C1672, C1832, C1871, C1474, or C1477.

5. Significance and Use

5.1 Facility operators and safeguards inspectors routinely take UF_6 samples from processing lines, isotopic enrichment cascades or storage cylinders to determine its U isotopic composition, most important the $n(^{235}U)/n(^{238}U)$ isotope ratio, needed to calculate the amount of the fissile ^{235}U in the sample. The current version of the "International Target Values for Measurement Uncertainties in Safeguarding Nuclear Materials" (**3**) contains recommended guidelines for these measurements.

5.2 The conventional sampling practice collects UF_6 samples in the range of 1-10 g and requires the use of liquid nitrogen to condense them in sample vessels, metallic bottles or P-10 tubes. These samples must then be transported to external analytical laboratories for verification of the declared data, especially the isotope ratios. Transport includes, among other things, public roads and intercontinental air shipment. Due to the hazards of UF_6, air transport is becoming increasingly difficult, with many transport operators and regulators refusing to carry the material.

5.3 This sampling practice was developed to meet the following requirements:

5.3.1 Fit for Purpose: to enable the verification of the declarations of amounts of nuclear materials.

5.3.2 Simplicity: to ensure a simple and fast execution.

5.3.3 Flexibility: to be applied in a wide range of facilities.

5.3.4 Robustness: to ensure sufficient material is sampled even when operational parameters slightly change.

5.3.5 Reliability: to provide measurement results in agreement with those obtained using the conventional sampling practice.

5.3.6 Confidentiality: to respect the facility's operational procedure and confidentiality of data.

5.3.7 Safety: to reduce the risks associated with the sampling, handling and transport of radioactive and hazardous materials.

5.4 This sampling practice offers significant advantages over the conventional sampling practice because it allows handling non-reactive, non-volatile, solid UO_2F_2 instead of highly reactive and volatile UF_6.

5.5 A smaller UO_2F_2 sample can be transported with lower radioactivity level and reduced radiological problems in case of accident. Additionally, there is no risk of airborne uranium particle and HF release.

[4] Available from Centrus Energy Corp., 6901 Rockledge Drive, Bethesda, MD 20817, http://centrusenergy.com.

[5] Available from International Atomic Energy Agency (IAEA), Vienna International Center, PO Box 100, A-1400 Vienna, Austria, http://www.iaea.org.

5.6 The U isotope ratios measured in UF_6 sampled by the conventional and this sampling practice provide measurement results which are in good agreement within the stated uncertainties (**4, 5**).

5.7 It is strongly recommended to discard used P-10 tubes to avoid the possibility of isotopic cross contamination, mainly because this practice is associated with the processing of very small amounts of U.

5.8 In case recycled P-10 tubes are used, a very efficient and reliable cleaning procedure must be employed to assure a complete removal of U from the P-10 tube inner surfaces.

5.9 This practice provides guidance to obtain samples for determining the U composition for material nuclear safeguards as well as other applications. Such samples should not be used for determining compliance with Specifications C787 and C996. For these cases, the recommendations of Practices C1052 or C1703 must be followed.

5.10 The test methods describing procedures for subsampling, mass spectrometric, spectrochemical, nuclear, and radiochemical analysis of uranium hexafluoride are presented in Test Methods C761. Most of them are routinely used to determine the compliance with Specifications C787 and C996.

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 conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[6] Other grades of reagents may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

6.2 *Alumina Pellets Specification*[7]—Aluminum oxide type gamma has been succesfully demonstrated. These pellets are cylinders of typically 3 mm of diameter and 5-6 mm of length, with density of 0.39 g/cm^3 and a total pore volume of 1.14 cm^3/g. The specific surface area is around 250 m^2/g measured by the BET Method. The alumina pellets must have low levels of impurities, especially U, to prevent systematic bias in the measured isotopic composition. It is strongly recommended to dry the alumina pellets to 120°C before use. Then, keep it in a desiccator until it reaches room temperature. Following this recommendation, the absorbed water content should be at about 0.07 % by weight. Pellets containing more than 7-8 % water will be so reactive that the pores will become blocked during hydrolysis and U loading will be confined to the pellet exterior surfaces. In case pellets absorb too much water, it will be difficult to reduce the pressure in the manifold and in the P-10 tube to at least 10 Pa, and moisture may be introduced into the processing line.

6.3 *Purity of Water*—Unless otherwise indicated, references to water should be understood to mean laboratory accepted demineralized or deionized water as described by Type I of Specification D1193.

6.4 *HNO_3,* concentrated acid, specific gravity 1.42, 15.8M.

6.5 *HNO_3,* 0.3M: add approximately 18 mL of concentrated HNO_3 to 500 mL of water and dilute to 1 L.

6.6 *HNO_3,* 4-8 M: add approximately 250-500 mL of concentrated HNO_3 to 400 mL of water and dilute to 1 L.

6.7 *Dry N_2 Gas,* purity grade 99.5 %, water content lower than 10 ppm.

7. Hazards

7.1 UF_6 is radioactive, corrosive, toxic and highly reactive. At ambient conditions it is a nearly white crystalline solid with high vapor pressure. Its reaction with H_2O is exothermic and generates corrosive HF and toxic UO_2F_2. It attacks most metals, some plastics, rubber and coatings. It is also incompatible with aromatic hydrocarbons and hydroxy compounds.

7.2 UO_2F_2 is radioactive, corrosive and toxic. It is a yellow solid very soluble in H_2O. When heated to decomposition, above 300°C, it emits toxic fluoride fumes.

7.3 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.

7.4 When released to atmosphere, gaseous UF_6 reacts with moisture to produce HF gas and UO_2F_2 particulates. This latter becomes readily visible as a white cloud. The corrosive nature of UF_6, HF and UO_2F_2 can result in skin burns and lung impairment. Medical evaluation is mandatory after contact with these compounds. When water-soluble UO_2F_2 is inhaled or ingested in large quantities it can be toxic to the kidneys.

7.5 Adequate laboratory facilities and safe techniques must be used when handling samples containing these materials. Personnel handling radioactive materials must adhere to the recommend practices in place in the respective nuclear facility.

7.6 Before performing this practice, review the Safety Data Sheet (SDS) for U, UF_6, UO_2F_2, HF and HNO_3.

7.7 Perform all chemical operations in a laboratory fume hood that is verified operable and has undergone inspections to ensure proper airflow before each use.

8. Sampling Procedure

8.1 Sampling is performed directly or using a manifold connected to the sampling point, bypassing the main stream.

[6] *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. Pharmacopeia Convention, Inc. (USPC), Rockville, MD.

[7] Alumina pellets from Alfa Aesar brand, Johnson Matthey Company, catalog number 43832, have been found to perform satisfactorily. This does not preclude the use of materials from other manufacturers providing an equivalent performance.