



Designation: C1913 – 21

Standard Practice for Sampling Gaseous Uranium Hexafluoride Using Zeolite in Single-Use Destructive Assay Sampler¹

This standard is issued under the fixed designation C1913; 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 zeolite in a single-use destructive assay (SUDA) sampler.

1.2 This practice is based on the SUDA method developed at Pacific Northwest National Laboratory (**1**)² for collection of samples of UF_6 for determination of uranium isotopic content for nuclear material safeguards and other applications.

1.3 The UF_6 collected is converted to uranyl fluoride (UO_2F_2), allowing samples to be handled and categorized for transport under less stringent conditions than are required for UF_6 .

1.4 This practice can be used to collect samples for safeguards measurements. Safeguards samples collected with this practice have been shown to provide suitable isotopic measurements (**1**).

1.5 This practice has not been demonstrated for suitability for compliance with Specifications **C787** and **C996**. Practices **C1052** or **C1703** can be used to collect samples for compliance with these specifications.

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

1.7 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.8 *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 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.

Current edition approved June 1, 2021. Published July 2021. DOI: 10.1520/C1913-21.

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

1.9 *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

C787 Specification for Uranium Hexafluoride for Enrichment

C859 Terminology Relating to Nuclear Materials

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

C1052 Practice for Bulk Sampling of Liquid Uranium Hexafluoride

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 Modified Total Evaporation (MTE) Method Using Thermal Ionization Mass Spectrometer

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

C1880 Practice for Sampling Gaseous Uranium Hexafluoride using Alumina Pellets

D1193 Specification for Reagent Water

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.

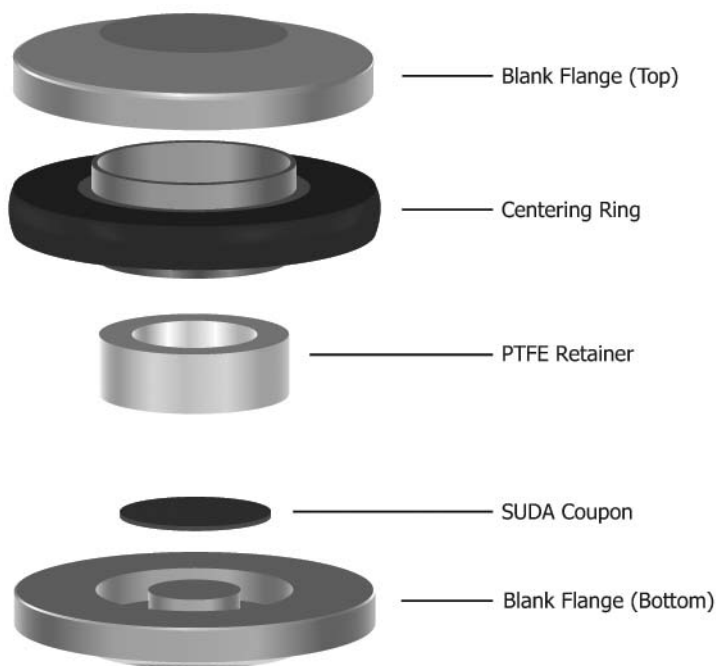


FIG. 1 Exploded View of SUDA Sampler

D1418 Practice for Rubber and Rubber Latices—
Nomenclature

2.2 ISO Standards:⁴

ISO 2861:2020 Vacuum technology — Dimensions of
clamped-type quick-release couplings

3. Terminology

3.1 For definitions of terms used in this practice but not defined herein, refer to Terminology C859.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *sampling coupon, n*—silicon wafer coated with an engineered zeolite sorbent thin film (see 7.5).

3.2.2 *sampling manifold, n*—piping with apertures used to connect the SUDA sampler to the UF₆ processing lines, isotope enrichment cascades or storage cylinders, and tap valves to allow introduction of UF₆ to the sampling coupon.

3.2.3 *SUDA sampler, n*—apparatus that is connected to the sampling manifold for sample collection (see Fig. 1 and Section 6).

4. Summary of Practice

4.1 A SUDA sampler (see Figs. 1 and 2) is attached to a sampling manifold and exposed to gaseous UF₆ for a specified time period. At a gas pressure of 5.3 kPa to 10.6 kPa (40 Torr to 80 Torr), this is typically 2 to 5 min (1, 2). UF₆ is hydrolyzed by the water content in the sorbent film to form chemically-stable uranyl fluoride, which typically forms as a hydrate, UO₂F₂·2H₂O, in the presence of water.

4.2 At the end of the sampling period, the tap valve is closed. A total uranium mass of 0.1 mg to 3 mg is collected per SUDA sampler using the sampler described in this practice (3). A larger SUDA sampler is available that can collect up to 100 mg of total uranium mass; this sampler is not described in this practice.

4.3 The SUDA sampler is removed from the sampling manifold, reassembled, and may be stored indefinitely until it can be transported for laboratory analysis.

4.4 Samples of depleted uranium or natural uranium may be transported as unregulated radioactive materials. Samples of low enriched uranium (LEU) may be packaged and transported in excepted quantities (for example, as UN 2910 excepted packages (4)), which are significantly less burdensome to transport than gaseous UF₆ samples (UN 3507).

4.5 At the analytical laboratory, the UO₂F₂ is extracted from the sampling coupon with 2 % nitric acid or water. The uranium isotopic composition is then determined by mass spectrometric techniques such as thermal ionization mass spectrometry (TIMS) using Test Methods C1672, C1832, or C1871, multi-collector inductively coupled plasma mass spectrometry (MC-ICP-MS) using Test Method C1477, or quadrupole inductively coupled plasma mass spectrometry (Q-ICP-MS) using Test Method C1474.

5. Significance and Use

5.1 Facility operators and safeguards inspectors routinely collect UF₆ samples from processing lines, isotopic enrichment cascades or storage cylinders to determine uranium isotopic composition. The isotope ratio $n(^{235}\text{U})/n(^{238}\text{U})$ is particularly important since it is used to calculate the amount of fissile ²³⁵U in the sample.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandinnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <https://www.iso.org>.



FIG. 2 Fully Assembled SUDA Sampler Including Hinge Clamp

5.2 Conventional sampling practices (such as Practices C1052 and C1703) collect samples of UF_6 , usually in quantities greater than one gram. Due to the chemical hazards of UF_6 (and in some cases the high collection mass), an increasing number of air transport operators are unwilling to transport such samples. In contrast, SUDA samples are expected to be transported as excepted quantities (for example, under UN 2910 (3)), as the conversion to a less hazardous, more stable chemical species avoids the chemical hazards of UF_6 similar to Practice C1880. Additionally, the decreased shipping requirement and small collection mass of SUDA samplers (less than Practice C1880) allow for multiple SUDA samples to be transported in the same shipment.

5.3 For safeguards applications, isotopic measurements that fall within the 2010 International Target Value (ITV) ranges (5) have been demonstrated (1).

5.4 This practice provides the following qualities:

5.4.1 Fitness for purpose in verifying nuclear material declarations.

5.4.2 A safe, simple and fast procedure for the sample collector that minimizes sample handling and potential for cross-contamination.

5.4.3 Flexibility for use in a wide variety of facilities.

5.4.4 Robustness to adapt to minor changes in facility operating parameters.

5.4.5 Confidentiality for the operating facility from which the sample is collected.

5.4.6 Safety in sample handling and transport since the sample is a less hazardous, more stable form (specifically, UO_2F_2 is more stable and less volatile than UF_6 gas).

5.4.7 Ease of sample preparation in the laboratory with reduced processing hazards during recovery of the uranium content (1).

5.5 Samples collected using this practice are suitable for determination of uranium isotopic composition, as described in 4.5, for safeguards applications. Care must be taken to ensure cleanliness of the sampling tap to be used for SUDA samples, as any UF_6 holdup in the sampling tap from previous sample collection could affect sample collection and isotopic measurements (see Section 9 for further details regarding this issue). Other applications of this practice are possible but require validation prior to use.

6. Apparatus

6.1 *SUDA Sampler* (see Fig. 1 for exploded view and Fig. 2 for fully assembled view), including the following:

6.1.1 *Sampler Body*, consisting of two blank flanges of size KF-16 as described in ISO 2861:2020.

6.1.2 *Hinge Clamp*, suitable for the sampler body.

6.1.3 *Centering Ring*, a commercially available KF-16 centering ring made of a stainless steel or aluminum ring and fluorinated rubber (class FKM as described in Practice D1418).

6.1.4 *Insert, Polytetrafluoroethylene (PTFE)*, to hold the sampling coupon in place within the sampler body.

6.1.5 *Sampling Coupon*, prepared as described in 7.5.

7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 of Specification D1193.

7.2 *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.⁵ 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.

7.3 *Nitric Acid (HNO_3)*, concentrated, spectroscopic grade, sp gr 1.42, 15.8 M.

7.4 *Nitric Acid (HNO_3)*, 2 % (v/v)—Carefully and slowly add 20 mL of concentrated HNO_3 to 500 mL water in a 1 L volumetric flask, then dilute to volume with water.

7.5 *Sampling Coupon*, consisting of the following:

7.5.1 *Substrate*, consisting of a commercially available silicon wafer (or similar, providing an equivalent performance).

7.5.2 *Engineered Zeolite Thin Film*, hydrated aluminosilicate crystals are synthesized as described in Ref (6) and impregnated onto the substrate followed by calcination at 450 °C for 48 h.

NOTE 1—Other materials that provide an equivalent performance may also be acceptable.

⁵ *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.

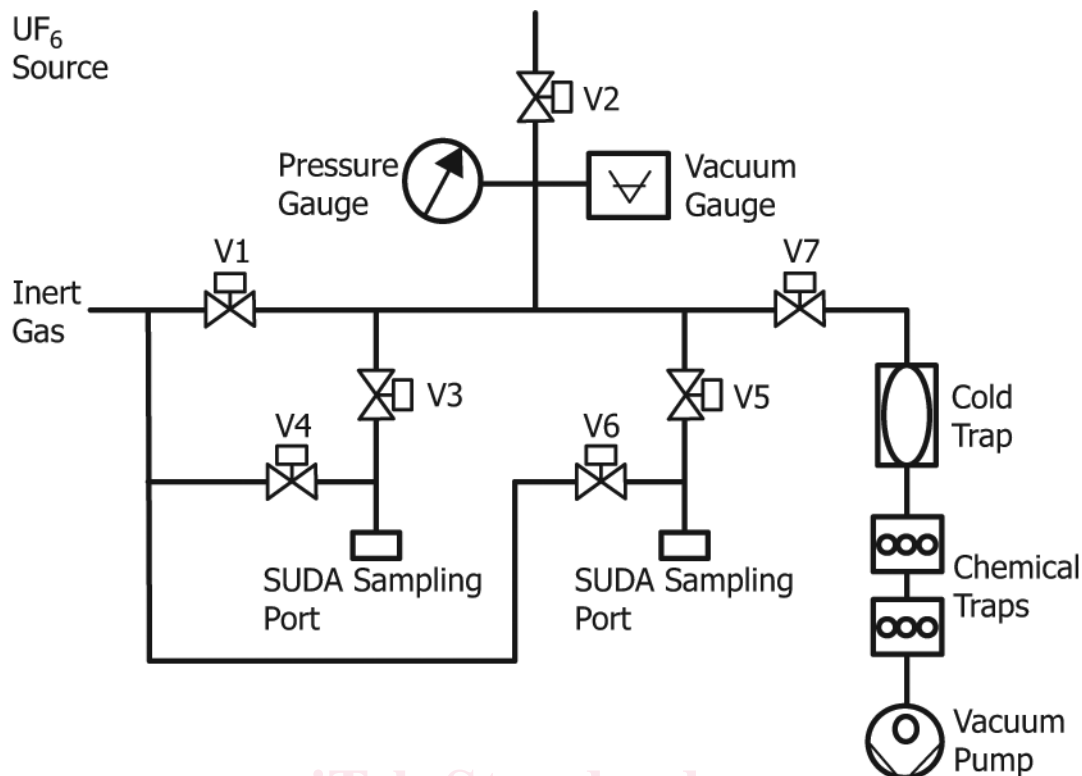


FIG. 3 Example Schematic Arrangement for SUDA Sampling of UF₆

NOTE 2—Impregnating the film by spraying is recommended to ensure uniform coverage.

NOTE 3—The zeolite thin film mass and water content can be adjusted to collect more or less uranium based on the film thickness and area on the substrate.

8. Hazards

8.1 UF₆ is radioactive, corrosive, toxic and highly reactive. At ambient conditions it is a nearly white crystalline solid with high vapor pressure. Its reaction with H₂O is exothermic and generates corrosive HF and toxic UO₂F₂. It attacks most metals, some plastics, rubber and coatings. It is also incompatible with aromatic hydrocarbons and hydroxy compounds.

8.2 UO₂F₂ is radioactive, corrosive and toxic. It is a yellow solid very soluble in H₂O. When heated to decomposition, above 300 °C, it emits toxic fluoride fumes.

8.3 Hydrofluoric acid (HF) is a highly corrosive and toxic 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.

9. Sampling Procedure

9.1 Sampling is performed using a sampling manifold connected to the sampling point. Fig. 3 provides an example arrangement for sampling gaseous UF₆ from a processing line,

enrichment cascade or storage cylinder, using SUDA samplers (one or more) attached to a sampling manifold.

NOTE 4—In Fig. 3, an inert gas such as nitrogen or argon can be used.

9.2 Prepare and assemble a SUDA sampler, including sampling coupon, and secure it with a hinge clamp.

9.3 Transport the sampler to the manifold or sampling point. Remove the hinge clamp and top blank flange immediately before connecting to the manifold or sampling point.

9.4 Connect the SUDA sampler to the manifold or sampling point. If using a sampling manifold, connect the manifold to the vacuum line with a cold trap, chemical trap and dry nitrogen supply. Connect the manifold to the processing line, enrichment cascade or storage cylinder.

9.5 Ensure all valves are closed and open valves (V3), (V5), and (V7) to evacuate the SUDA sampler and the manifold. It is recommended that a pressure lower than 10 Pa be established. If this pressure is not achievable due to equipment limitation, then it is recommended to heat the sampling manifold (60 °C). When a stable pressure is achieved, close the valve (V7) to isolate the SUDA sampler(s) and manifold from the pumping line. Ensure the pressure is stable, confirming that the set-up has a good seal.

9.6 Ensure that valve (V2) is closed. Close valve (V7) and open valve (V1) to introduce an inert dry gas (nitrogen or argon) to pressurize the manifold and SUDA sampler up to atmospheric pressure.