



Designation: C1883 – 19

Standard Practice for Sampling of Gaseous Enriched Uranium Hexafluoride¹

This standard is issued under the fixed designation C1883; 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 covers the methods for withdrawing samples of enriched uranium hexafluoride (UF_6) during and after a transfer occurring in the gas phase in order to determine compliance with Specification C996. It is applicable to the filling of a transport UF_6 container (30B cylinder or 1 m³ vertical container) at a centrifuge enrichment facility that has been fed with UF_6 that complies with Specification C787.

1.2 Since UF_6 sampling is taken during the filling process, this practice does not address any special additional arrangements that may be agreed upon between the buyer and the seller when the sampled bulk material is being added to residues already present in a container (“heels recycle”). Such arrangements will be based on QA procedures such as traceability of cylinder origin (to prevent, for example, contamination with irradiated material).

1.3 This practice is only applicable when the transfer occurs in the gas phase. When the transfer is performed in the liquid phase, Practice C1052 should apply.

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

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses 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.*

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. 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

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

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

C1842 Test Method for The Analysis of Boron and Silicon in Uranium Hexafluoride via Fourier-Transform Infrared (FTIR) Spectroscopy

C1838 Practice for Cleaning for 1S and 2S Bottles

2.2 ISO Standards:³

ISO 7195:2005 Nuclear energy—Packaging of uranium hexafluoride (UF_6) for transport

3. Terminology

3.1 Terms shall be defined in accordance with Terminology C859, except for the following:

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *container, n*—a vessel either holding or receiving by transfer, the UF_6 to be sampled; it may consist of, for example, a fixed vessel in a UF_6 handling plant or a cylinder to be used for the transport of UF_6 .

3.2.2 *sample vessel, n*—the small vessel into which the sample of UF_6 is withdrawn for analysis in the laboratory for characterization. It can be a 1S or 2S bottle or a PCTFE

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.

³ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandinnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

(polychlorotrifluoroethylene)/PTFE (polytetrafluoroethylene) pot or tube or any other type of cylinder compatible with UF₆.

4. Summary of Practice

4.1 The method is based on three different steps for UF₆ characterization, namely: (1) continuous on-line analysis by mass spectrometry (MS) for isotopic measurement, (2) sequential withdrawals during filling to produce a representative composite sample, (3) gas sampling of container after the filling is complete, to determine volatile impurities.

4.2 The conformity of the container is determined by sampling (1) for isotopic composition; by sampling (3) for HF to determine UF₆ concentration, for very volatile impurities compared to UF₆, such as BF₃, SiF₄, and low molecular weight hydrocarbon, chlorocarbon, and partially substituted halohydrocarbon compounds; and by sampling (2) for other requirements: Pu, Np, fission products, ⁹⁹Tc, if they are necessary. The sample can also be used for counter-analyses or supplied to the customer. Additionally, the sample may be used to determine UF₆ concentration. Figs. 1 and 2 summarize the principle of the three sampling steps.

4.3 Determining concentration of ²³⁵U (²³⁴U, ²³⁶U) in a batch of product extracted is carried out according to the results of hourly mass-spectrometry isotope analysis. Weighted average value calculated over the entire control period is assigned to a batch of transport UF₆ containers processed simultaneously.

4.4 Content of radioactive admixtures (²³²U, ²³⁷Np, Pu isotopes, ⁹⁹Tc, gamma-emitting uranium fission products) shall be determined according to analysis results related to a sample collected from the process flow intermittently one time per hour during the entire period UF₆ batch is extracted. Refer to Test Methods C761 for test methods.

4.5 Quality control of UF₆ by UF₆ concentration is carried out using an indirect method based on performed IR-spectrometry analysis for HF content in gas phase of container once it is filled.

4.6 Compliance of UF₆ quality with Specification C996 by content of boron, silicon, and hydrocarbon, chlorocarbons, and partially substituted halohydrocarbons is confirmed according to the results of IR-spectrometry analysis of samples collected from gas phase of container (reservoir) once it is filled.

4.7 The intention of this practice is to avoid liquid UF₆ sampling once the cylinder has been filled. For safety reasons, manipulation or large quantities of liquid UF₆ should be avoided when possible. This practice is applicable only if the process of gas phase filling is sufficiently stable over time, ²³⁵U concentration fluctuations in the process flow are characterized by the relative standard deviation equal to 0.2 % rel. This practice does not address cylinders filled during transitional regime of the cascade.

4.8 In case of the presence of volatile impurities close to the specification (for example, 80 %) or in case of HF concentration close 0.5 % or less, a confirmation using liquid sampling may be necessary.

4.9 It is recommended to validate the gas sampling using a comparison on several cylinders with liquid sampling after filling. Statistically significant sampling basis and requirement should be established. Adequacy shall be demonstrated by quality assurance procedures.

4.10 The presence of residues may have significant implications for the quality of the UF₆. For safety and quality reasons, cylinders and bottles shall be clean, dry, and empty before filling.

4.11 Cross-contamination may occur between subsequent samples taken using the same equipment, and appropriate precautions must be taken to prevent this. It is therefore recommended that, before taking definitive samples, the equipment is flushed through with an aliquot of the material to be sampled. This is normally accomplished by taking an initial volume which is then rejected and not used for definitive

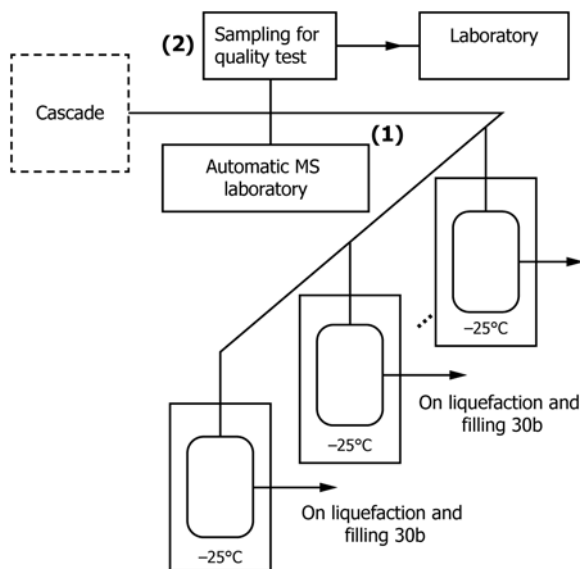


FIG. 1 Schematic Arrangement for Online Filling (3) Measurement (1)

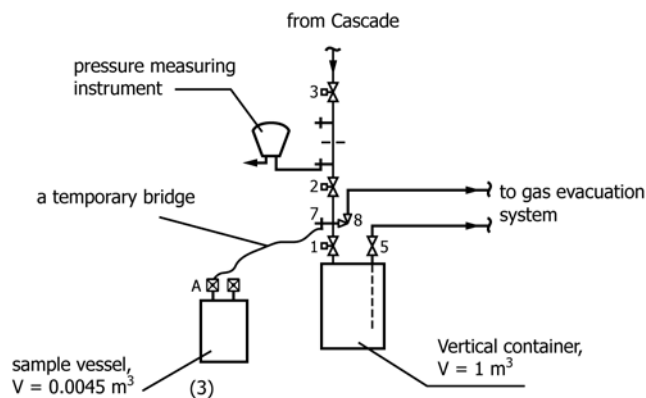


FIG. 2 Schematic Arrangement for Gas Sampling After and Composite Sample (2)

analysis. Alternative procedures to prevent cross-contamination are possible and should be validated individually.

5. Significance and Use

5.1 The standard method for taking representative UF_6 sample from a cylinder is collecting from UF_6 liquid phase. Homogeneity of material is obtained by the basic indicator, that is, content of uranium isotope. Representativeness of a sample by other indicators such as content of highly volatile admixtures (boron fluoride, silicon fluoride, organic impurities), admixtures generating non-volatile fluorides may not be satisfactory owing to differences between physical properties of admixture and UF_6 . Nevertheless, such sampling is widely applied during the product quality control due to the fact that liquid phase process is applied when filling transport containers both for feedstock UF_6 and enriched one. Refer to Practice C1052 for UF_6 liquid phase sampling practice.

5.2 Uranium hexafluoride is normally produced and handled in large (typically 1 to 14-ton) quantities and must, therefore, be characterized by reference to representative samples (see ISO 7195:2005). The samples are used to determine compliance with the applicable commercial Specification C996. The quantities involved, physical properties, chemical reactivity, and hazardous nature of UF_6 are such that for representative sampling, specially designed equipment must be used and operated in accordance with the most carefully controlled and stringent procedures. This practice can be used by UF_6 enrichers to review the effectiveness of existing procedures or as a guide to the design of equipment and procedures for future use.

5.3 It is emphasized that this practice is not meant to address conventional or nuclear criticality safety issues.

6. Hazards

6.1 Because of its chemical, radiochemical, and toxic properties, UF_6 is a hazardous material.

6.2 Uranium hexafluoride is very reactive and corrosive. It reacts readily with water, atmospheric moisture, certain metals, and many organic materials. For reasons of safety and to avoid contamination, precautions must be taken to avoid contact with such materials. The sampling equipment is therefore fabricated

to appropriate high standards of vacuum and high temperature integrity, and components in direct contact with UF_6 are made from nickel, high-nickel alloys, or materials having equivalent resistance to UF_6 corrosion. The formation of an inert fluoride layer is often an important feature of UF_6 corrosion resistance, and hence, internal surfaces are generally conditioned with a suitable fluorinating agent, sometimes UF_6 itself.

6.3 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. Procedure for Continuous On-line Isotopic Analysis by MS

7.1 Determining concentration of ^{235}U (^{234}U , ^{236}U) in a batch of product extracted is carried out according to the results of mass-spectrometry isotope analysis of gas flow admitted for condensation. Concentration of uranium isotopes in UF_6 shall be monitored with frequency of single measurement per hour (hourly). To ensure measurement conducted is reliable and precise, monitoring is carried out simultaneously using two magnet sector four-manifold “gas” mass-spectrometers operating in automatic mode. Precision measurement is conducted using referent samples of uranium isotope composition in the form of UF_6 . Refer to Fig. 3 for connection diagram of mass-spectrometers and process flow. During the measurement period UF_6 flow via measuring vessels shall be blocked.

7.2 Weighted average values calculated over the entire control period are assigned to a batch of containers (reservoirs) processed simultaneously. Calculation of uranium isotope mass fractions C_i , %, in a product batch extracted at a constant value of flow to the cylinders is performed based on calculated arithmetical average using Eq 1:

$$C = \frac{\sum C_i}{N}$$