

Designation: C 1703 - 08

Standard Practice for Sampling of Gaseous Uranium Hexafluoride¹

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1. Scope

- 1.1 This practice covers methods for withdrawing representative sample(s) of uranium hexafluoride (UF $_6$) during a transfer occurring in the gas phase. Such transfer in the gas phase can take place from a mother cylinder, for example in an autoclave to a receiving cylinder. It can also occur during the filling in the gas phase of a cylinder during a continuous production process, for example centrifuge enrichment facility or the distillation column in a conversion facility. Such sample(s) may be used for determining compliance with the applicable commercial specification, for example Specification C 996 or Specification C 787.
- 1.2 Since UF₆ 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 If the receiving cylinder is purged after filling and sampling, special verifications must be performed by the user to verify the representativity of the sample(s). It is then expected that the results found on volatile impurities with gas phase sampling may be conservative.
- 1.4 This practice is only applicable when the transfer occurs in the gas phase. When the transfer is performed in the liquid phase, Practice C 1052 should apply. This practice does not apply to gas sampling after the cylinder has been filled since the sample taken will not be representative of the cylinder.
- 1.5 The scope of this practice does not include provisions for preventing criticality incidents.
- 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 and health practices and determine the applicability of regulatory limitations prior to use.

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2. Referenced Documents

2.1 ASTM Standards:²

C 761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride

C 787 Specification for Uranium Hexafluoride for Enrichment

C 996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ²³⁵U

C 1052 Practice for Bulk Sampling of Liquid Uranium Hexafluoride

2.2 Other Document:

ISO/DIS 7195 Packaging of Uranium Hexafluoride (UF₆) for Transport³

3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 3.1.1 *container*—a vessel either holding or receiving by transfer, the UF₆ to be sampled; it may consist of, for example, a fixed vessel in a UF₆ handling plant or a cylinder to be used for the transport of UF₆.
- 3.1.2 sample vessel—the small vessel into which the sample of UF₆ is withdrawn for analysis in the laboratory for characterization. It can be a 1S or 2S bottle or a PCTFE (polydifluorodichloroethylene)/ PTFE (polytetrafluoroethylene) pot or tube or any other type of cylinder compatible with UF₆.

4. Summary of Practices

4.1 Two methods of withdrawing gas UF_6 for sampling are possible, namely: (1) continuous withdrawal using for example a capillary and producing only one sample, or (2) sequential withdrawals producing a composite sample. Depending on the pressure and temperature conditions during the transfer, the sampled UF_6 is either liquefied or solidified in the sample vessel.

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.

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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

- 4.2 An example of (1) is the sampling of UF₆ coming from a distillation column. In such case, the sampled gas UF₆ can be condensed in the liquid phase in the sampling vessel. The representative sample is then homogenized before analysis at the laboratory. It is assumed that the flow rate from the distillation is either constant (for example using a mass flow controller) or that the capillary will take its variation in account.
- 4.3 Examples of (2) are the sampling of UF₆ from an autoclave or from the cascades of an enrichment facility. This would apply only to a stable process. In such case, the sequential withdrawals must take into account the potential variation of flow rate which must be continuously monitored during the transfer. A composite sample is prepared and is compared to an average calculation using on line analysis.
- 4.4 For both methods of sampling, 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.

5. Significance and Use

- 5.1 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/DIS 7195). The samples are used to determine compliance with the applicable commercial specifications C 996 and C 787. The quantities involved, physical properties, chemical reactivity, and hazardous nature of UF₆ 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 UF₆ converters, enrichers, and fuel fabricators to review the effectiveness of existing procedures or as a guide to the design of equipment and procedures for future use.
- 5.2 The intention of this practice is to avoid liquid UF₆ sampling once the cylinder has been filled. For safety reasons, manipulation of large quantities of liquid UF₆ should be avoided when possible.
- 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, UF₆ is a hazardous material.

7. Principles

- 7.1 The essential purpose of the sample(s) is to be representative of the total material which has been transferred. It is the responsibility of the user to determine the way of continuous sampling or the number of samples and time distribution that are necessary to be representative, depending on the process variability. For example, in case of the presence of high level of very volatile impurities, additional samples may have to be taken at the beginning of the transfer.
- 7.1.1 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.

- 7.1.2 In case of the presence of volatile impurities close to the specification (for example within 80 % of the specification), a confirmation using liquid sampling may be necessary.
- 7.2 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 UF₆ are made from nickel, high-nickel alloys, or materials having equivalent resistance to UF₆ corrosion. The formation of an inert fluoride layer is often an important feature of UF₆ corrosion resistance, and hence, internal surfaces are generally conditioned with a suitable fluorinating agent, sometimes UF₆ itself.
- 7.3 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 analysis. Alternative procedures to prevent cross-contamination are possible and should be validated individually.

8. Procedure for Continuous Sampling During Filling of a Transport Cylinder

- 8.1 Sample Preparation:
- 8.1.1 The equipment consists of a continuous sampling vessel that has the ability to collect a desired weight/volume of UF₆ during the filling of a UF₆ transport cylinder, and a sample manifold used for obtaining the aliquot of UF₆ from the continuous sampling vessel. The sampling manifold can be a permanent (fixed) manifold, and can be the same manifold used for sampling straight from a product cylinder. The continuous sampling vessel should be fed gaseous UF₆ from a slip stream at the exit of the supplying source (for example, a distillation column) that is supplying UF₆ to a transport cylinder.
- 8.1.2 The continuous sampling vessel should be maintained at a temperature and pressure adequate for condensing and maintaining UF₆ in liquid phase, to allow for homogenization by the action of convection currents within the bulk liquid. The continuous sampling vessel should be operated so that a composite sample of UF₆ could be withdrawn during the entire filling cycle of a transport cylinder. The continuous sampling vessel should be able to be isolated from the supply so that adequate purging of the vessel and supply lines can be accomplished after the sampling cycle is complete.
- 8.1.3 The continuous sampling vessel should be opened to draw a sample from the UF₆ feed line at the beginning of filling of the transport cylinder. At the completion of filling the transport cylinder the continuous sampler should be isolated from the feed line.
- 8.1.4 The sampling manifold should be appropriately sized to contain the quantity of UF₆ required for a single sample and normally, consists of the manifold and associated pipe work or may also include an additional metering volume (pipette). The