Designation: ~~C1689 – 08a (Reapproved 2014)~~ C1689 – 21

Standard Practice for Subsampling of Uranium Hexafluoride[1]

This standard is issued under the fixed designation C1689; 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 subsampling uranium hexafluoride (UF_6), using heat liquefaction techniques, from bulk containers, obtained in conformance with Practices C1052, C1703, and C1883, into smaller sample containers, which are required for laboratory analyses.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 It is assumed that the liquid UF_6 being sampled comprises a single quality and quantity of material. This practice does not address any special additional arrangement that might be required for taking proportional or composite samples.

1.4 The number of samples to be taken, their nominal sample weight, and their disposition shall be agreed upon between the parties.

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.*

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:*[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 % ^{235}U
~~C761~~C1052 ~~Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of~~

[1] This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.
Current edition approved ~~Jan. 1, 2014~~Feb. 1, 2021. Published ~~February 2014~~March 2021. Originally approved in 2008. Last previous edition approved in ~~2008~~2014 as ~~C1689 – 08a.~~C1689 – 08a (2014). DOI: ~~10.1520/C1689-08aR14.~~10.1520/C1689-21.

[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.

Practice for Bulk Sampling of Liquid Uranium Hexafluoride
C1052C1703 Practice for Bulk Sampling of LiquidGaseous Uranium Hexafluoride for Enrichment
C1883 Practice for Sampling of Gaseous Enriched Uranium Hexafluoride
2.2 *Other Documents:*
ANSI N14.1 Uranium Hexafluoride: Packaging for Transport[3]
ISO/DISISO 7195 Packaging of Uranium Hexafluoride (UF_6) for Transport[4]
USEC-651 The UF_6 Manual: Good Handling Practices for Uranium Hexafluoride, latest revision[5]

3. Terminology

3.1 *Definitions:*

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

3.1.2 *sample bottle*—the vessel (typically a 1S or 2S bottle) into which the sample of UF_6 is withdrawn from the container for transfer to the laboratory, analysis or dispatch to the customer.

3.1.3 *subsample tube*—the small vessel (for example, a P10 tubetube)) into which a subsample of UF_6 is withdrawn from the sample bottle for analysis of UF_6 quality or dispatch to the customer.

3.1.3.1 *Discussion—*
Polychlorotrifluoroethylene P10 tubes are widely accepted by the industry for subsample collection and subsequent UF_6 quality analyses or dispatch to the customer. Other types of subsample tubes, for example P-20, P-80, or P100 , can be used for internal subsample collection and processing. Dispatch of these subsample tubes may be agreed upon by buyer and seller and subject to (local) transport regulations.

3.1.4 *subsample rig*—the equipment to perform the transfer of liquid UF_6 from the sample bottle into the subsample tube, typically a vacuum manifold equipped with heating and a liquid nitrogen trap.

4. Summary of Practice

4.1 Two methods of withdrawing a subsample of UF_6 are described which differ based on safety requirements namely: *(1)* homogenizing of liquefied UF_6 by agitation before liquid transfer, and *(2)* homogenizing of liquefied UF_6 by convection before liquid transfer. The first method involves homogenization of liquifiedliquefied UF_6 in a sample bottle by vigorous shaking. Subsequently the sample bottle is inverted and connected to the top of a heated vacuum-manifold system, and the subsample tube is attached to the appropriate port of the system. The system is evacuated and the liquid UF_6 allowed to flow by gravity into the subsample tube. In the second method the sample bottle containing solid UF_6 is connected to the top of a manifold system, and a subsample tube is attached to the appropriate port of the system. The whole system is enclosed in secondary containment that can be heated (hot-box). After evacuation the complete system is heated for specific period (typically > 1.5 hr) >1.5 h) to allow for complete homogenization of the liquid UF_6 by convection. Subsequently the liquid UF_6 is allowed to flow by gravity either directly or via graduated volume into the subsample tube.

4.2 For both methods of sampling, the presence of residues may have significant implications for the quality of UF_6. For safety and quality reasons, sample bottles and subsample tubes shall be clean, dry, and empty before filling.

4.3 Various types of sample bottles and tubes are in use and are described in detail in the applicable national and international standards, for example, ANSI N14.1 and ISO/DISISO 7195. For a given type of sample bottle, the detailed configuration, for example valve orientation, terminal fittings and the like, may vary. Hence the type and configuration of bottles used for the withdrawal of samples shall be agreed upon between the parties.

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

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

[4] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

[5] Available from United States Enrichment Corp., 6903 Rockledge Dr., Bethesda, MD 20817, http://www.usec.com.Centrus Energy Corporation, 6901 Rockledge Drive, Bethesda, MD 20817.

characterized by reference to representative samples. The samples are used to determine compliance with the applicable commercial Specifications C996 and C787 by means of the appropriate test method (for example, Test Method C761 and references therein). The quantities involved, physical properties, chemical reactivity, and hazardous nature of UF_6 are such that for representative sampling, specially designated equipment must be used and operated in accordance with the most carefully controlled and stringent procedures. This practice indicates appropriate principles, equipment and procedures currently in use for subsampling of liquid UF_6. It is used by UF_6 converters, enrichers and fuel fabricators to review the effectiveness of existing procedures or to design equipment and procedures for future use. Other subsampling procedures such as UF_6 vapor sampling are not directly representative of the chemical quality of liquid UF_6.

5.2 It is emphasized that this test guide is not meant to address conventional or nuclear criticality safety ~~issues.~~issues, nor does it address the conditioning of subsample tubes to make them suitable for transport.

6. Apparatus

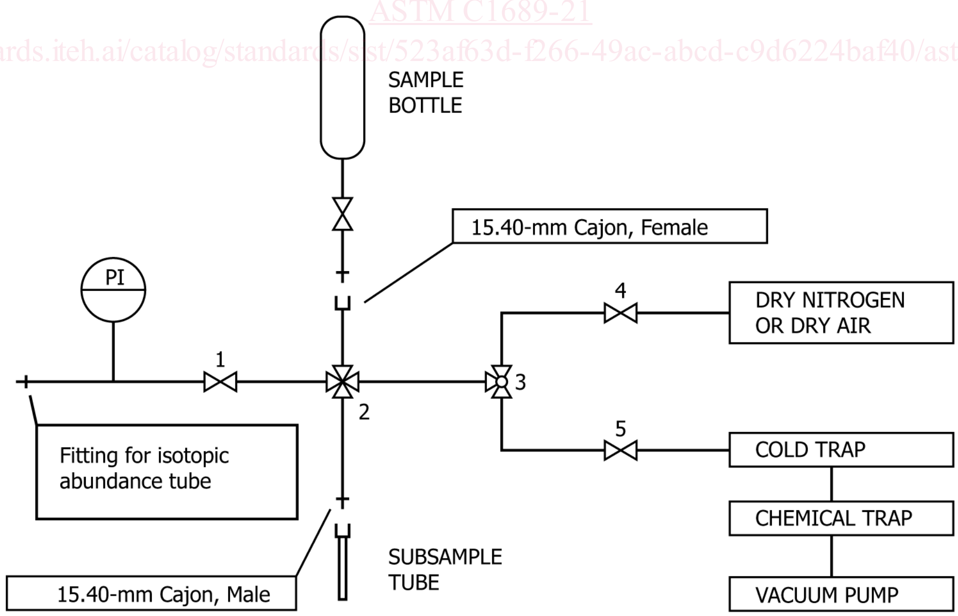
6.1 *Hot Water Bath.*

6.2 *Subsample Rig*—For ~~Procedure~~Procedures 1 ~~see Fig. 1~~and ~~Procedure~~ 2 see ~~Fig. 2~~Figs. 1 and 2~~.~~, respectively. Materials of construction in direct contact with liquid UF_6 are made from nickel, high nickel alloys, or materials having comparable resistance to UF_6 corrosion.

6.3 *Gaseous Isotopic Abundance Sample Tube* (Fig. 3).

6.4 *Polychlorotrifluoroethylene Subsample Tube and Closing Disc* (Fig. 4~~)—~~)—The tube must be of uniform density, free from cracks or occlusions and able to withstand temperatures from ~~−195°C~~−195 °C to ~~+150°C.~~ +150 °C. Materials of construction in direct contact with liquid UF_6 are made from polychlorotrifluoroethylene, PTFE/TFE (gaskets), or materials having comparable resistance to UF_6 corrosion.

6.5 *Flare Nut and Plug*—Flare nuts and plugs for subsample tube closure, storage and transport can be constructed from Monel, nickel, high nickel alloys or 316 SS.



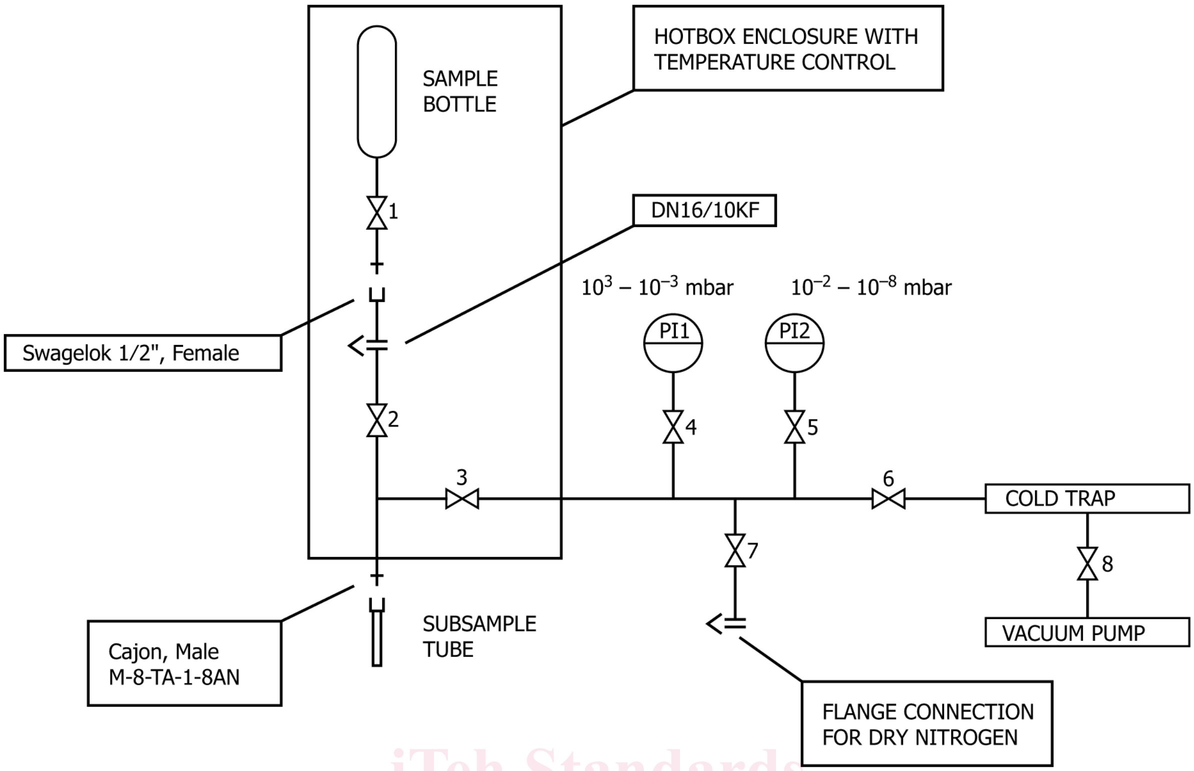
NOTE 1—All lines are ⅜ in. (9.5 mm) Monel tubing.

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.~~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.

FIG. 1 Subsample Rig Used for Procedure 1



NOTE 1—All lines in direct contact with liquid UF_6 are ~~⅜ in. (9.5 mm)~~ 9.5 mm Monel tubing.

NOTE 2—All other lines are ~~⅜ in. (9.5 mm)~~ 9.5 mm 316 SS tubing.

NOTE 3—Valves 1–3 are Monel below sealed valves that can be operated from outside the hotbox.

NOTE 4—Valves 4–8 are 316 SS below sealed valves.

NOTE 5—Flange connections are equipped with ~~helicoflex~~ (high pressure) ~~gaskets.~~ gaskets resistant to liquid UF_6, viton and helicoflex have been found to be acceptable for this application

FIG. 2 Subsample Rig Used for Procedure 2

6.6 *Polychlorotrifluoroethylene Knockout Cylinder* (Fig. 5),closed with a Cajon M-16 VCR-1 female nut and an M-16 VCR-4 male nut or equivalent.

NOTE 1—Brand names mentioned in this practice are intended to be typical, not limiting. Another brand with comparable characteristics could perform equally well.

6.7 *Nickel Filter Disc,* porous, 2µm, free of chromium (Fig. 6).

NOTE 2—The ~~filterdisc~~ filter disc should weigh approximately 1 g. It should be made of nickel powder produced from carbonyl nickel and formed by the no pressure sintering method in graphite or ceramic molds.

6.8 *Gas Sample Cylinder.*

6.9 *Heat Sources*—Heat gun (or an equivalent) and heat lamps.

6.10 *Dewar Flask,* for liquid nitrogen, stainless steel.

7. Hazards

7.1 Uranium hexafluoride (UF_6) is radioactive, toxic, and highly reactive especially ~~in the presence of~~ with reducing substances and moisture. ~~Safe techniques must~~Appropriate laboratory facilities, materials of construction, and techniques shall be utilized when handling UF_6~~. Suitable handling procedures are described in USEC-651.~~ (see for example USEC-651).

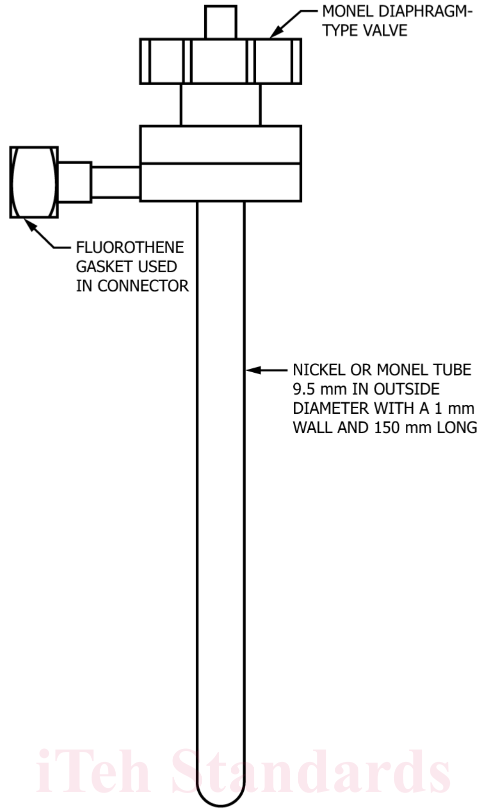


FIG. 3 Isotopic Abundance Sample ~~Tube.~~Tube

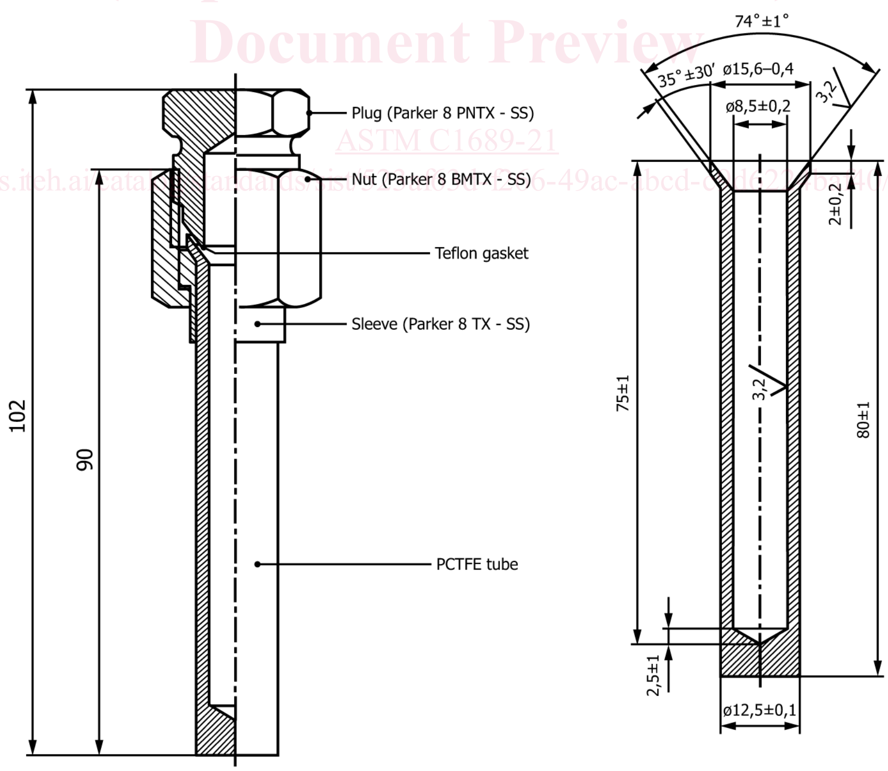


FIG. 4 Example of ~~a~~ Polychlorotrifluoroethylene Subsample Tube

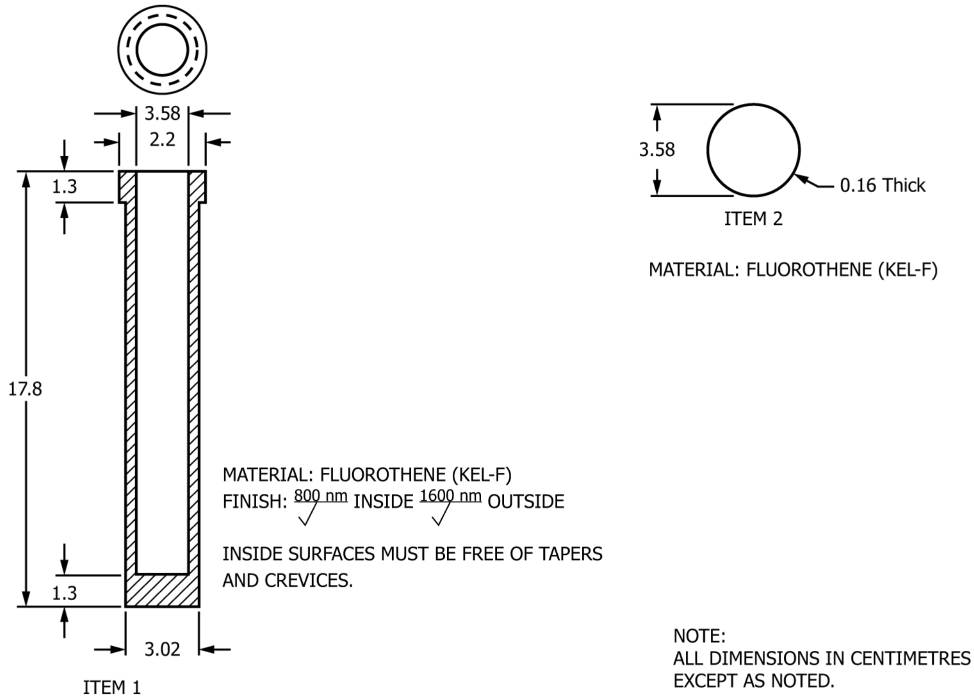


FIG. 5 Example of ~~a~~ Polychlorotrifluoroethylene Knock-out Tube

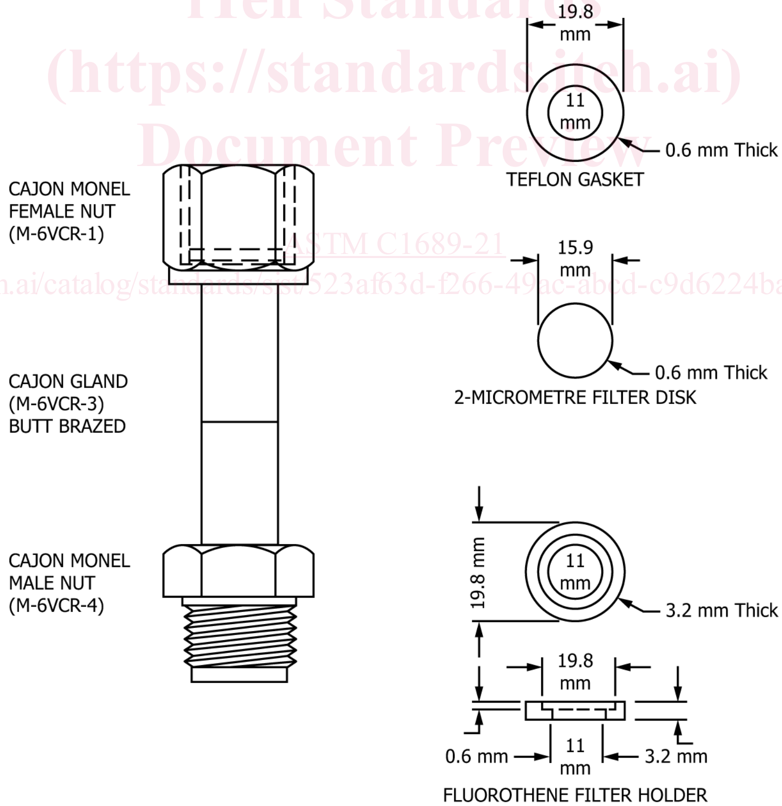


FIG. 6 Filter Disc Unit for Determination of Soluble and Insoluble Chromium

7.2 Follow all safety procedures for handling uranium and UF_6 as provided by your facility.the facility. Review the Safety Data Sheet (SDS) for UF6 prior to performing the procedure.

7.3 Review Material Safety Data Sheets for UF_6 and all chemicals associated with this method prior to performance.

7.3 Perform subsampling operations in a fume hood that has been verified operable and has undergone regular inspections to ensure proper airflow.laboratory hood. Hoods should be regularly inspected for proper air flow

7.4 When released to atmosphere, gaseous UF_6 reacts with moisture to produce HF gas and toxic UO_2F_2 particulates (a white amorphous solid) and becomes readily visible as a white cloud. The corrosive nature of HF and UFparticulates. Use sufficient ventilation $_6$ can result in skin burns and lung impairment. Medical evaluation is mandatory after contact with HF or UFor respiratory$_6$. When water-soluble UO protection to avoid$_2$F$_2$ is inhaled or ingested in large quantities it can be toxic to the kidneys.breathing fumes. Use appropriate personal protective equipment such as gloves, eye, and face protection.

7.5 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

7.5.1 Use gloves designed for use with cryogenic substances, and wear goggles or a face shield when handling bulk quantities of liquid nitrogen.

8. Principles

8.1 The essential purpose of the sample is to be representative of the bulk material for the purpose of determining compliance with the applicable material specification. To ensure that the sample is representative for this purpose, certain principles, as described below, must be observed.

8.2 Special attention must be given to ensuring that the bulk material, from which the sample is withdrawn, is homogeneous. In practice, the low viscosity, and hence easy mobility of liquid UF_6 facilitates the process of homogenization by the action of convection currents within the bulk upon heating. It is necessary to determine and establish for each set of subsampling equipment the physical conditions, normally a combination of the minimum time and temperature for which liquefied uranium hexafluoride is held, which guaranty homogeneity of the bulk UF_6.

8.3 Uranium hexafluoride is very reactive and corrosive. It reacts readily with water, atmospheric moisture and many organic materials. For reasons of safety and to avoid contamination, precautions must be taken to avoid contact with such materials. The subsampling equipment and subsample tube are therefore fabricated to appropriate high standards of vacuum integrity, and components in direct contact with liquid UF_6 are made from nickel, high nickel alloys, or materials having comparable 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.

8.4 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.

8.5 If sample bottles are taken for an analytical need such as liquid UF_6 subsampling for P10 tubes or liquid UF_6 transfer for FTIR quantification, it is recommended, in order to minimize the gas phase contribution to the sample bottle, to fill the bottle with more than 10 % of its total volume.

9. Subsampling Schemes for UF_6 Specification Analyses

9.1 The number and type of subsamples taken from a sample bottle (typically 1S or 2S bottle) depends both on the sampling