



Standard Practice for Subsampling of Uranium Hexafluoride¹

This standard is issued under the fixed designation C 1689; 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 C 1052, 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.*

2. Referenced Documents

2.1 ASTM Standards:²

C 787 Specification for Uranium Hexafluoride for Enrichment

C 859 Terminology Relating to Nuclear Materials

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

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

C 1052 Practice for Bulk Sampling of Liquid Uranium Hexafluoride

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.

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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.

2.2 Other Documents:

ANSI N14.1 Uranium Hexafluoride: Packaging for Transport³

ISO/DIS 7195 Packaging of Uranium Hexafluoride (UF_6) for Transport⁴

USEC-651 The UF_6 Manual: Good Handling Practices for Uranium Hexafluoride, latest revision⁵

3. Terminology

3.1 Terms shall be defined in accordance with Terminology C 859 except for the following:

3.1.1 *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.

3.1.2 *subsample tube*—the small vessel (for example, a P10 tube⁶) 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 *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 liquefied 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

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>.

⁵ Available from United States Enrichment Corp., 6903 Rockledge Dr., Bethesda, MD 20817, <http://www.usec.com>.

⁶ 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.

tube is attached to the appropriate port of the system. The system is evacuated and the liquid UF₆ allowed to flow by gravity into the subsample tube. In the second method the sample bottle containing solid UF₆ 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) to allow for complete homogenization of the liquid UF₆ by convection. Subsequently the liquid UF₆ 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 UF₆. 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/DIS 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 characterized by reference to representative samples. The samples are used to determine compliance with the applicable commercial Specifications C 996 and C 787 by means of the

appropriate test method (for example, Test Method C 761 and references therein). The quantities involved, physical properties, chemical reactivity, and hazardous nature of UF₆ 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 UF₆. It is used by UF₆ 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 UF₆ vapor sampling are not directly representative of the chemical quality of liquid UF₆.

5.2 It is emphasized that this test guide is not meant to address conventional or nuclear criticality safety issues.

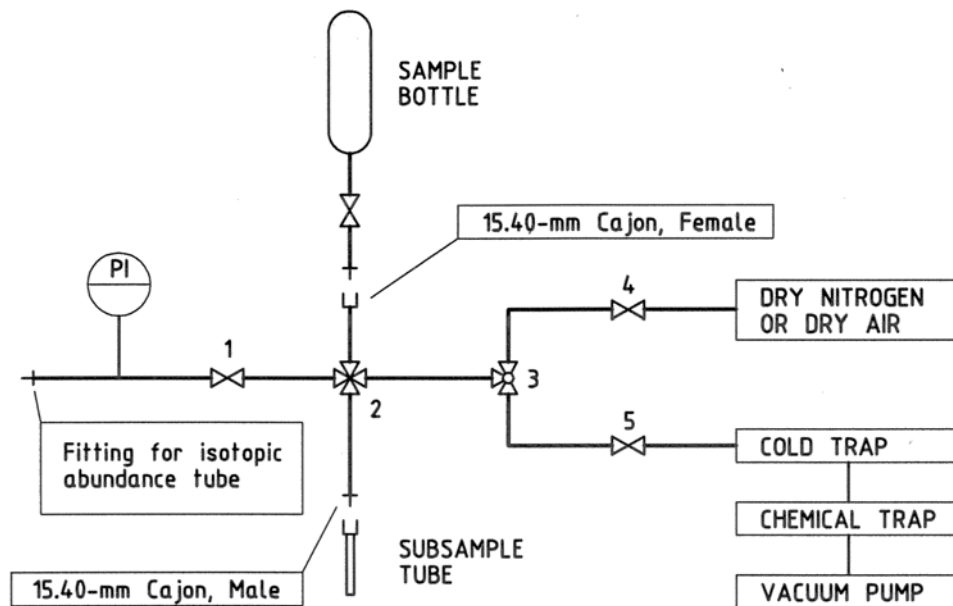
6. Apparatus

6.1 Hot Water Bath.

6.2 Subsample Rig—For Procedure 1 see Fig. 1 and Procedure 2 see Fig. 2. Materials of construction in direct contact with liquid UF₆ are made from nickel, high nickel alloys, or materials having comparable resistance to UF₆ 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 to +150°C. Materials of construction in direct contact with liquid UF₆ are made from polychlorotrifluoroethylene, PTFE/TFE (gaskets), or materials having comparable resistance to UF₆ corrosion.



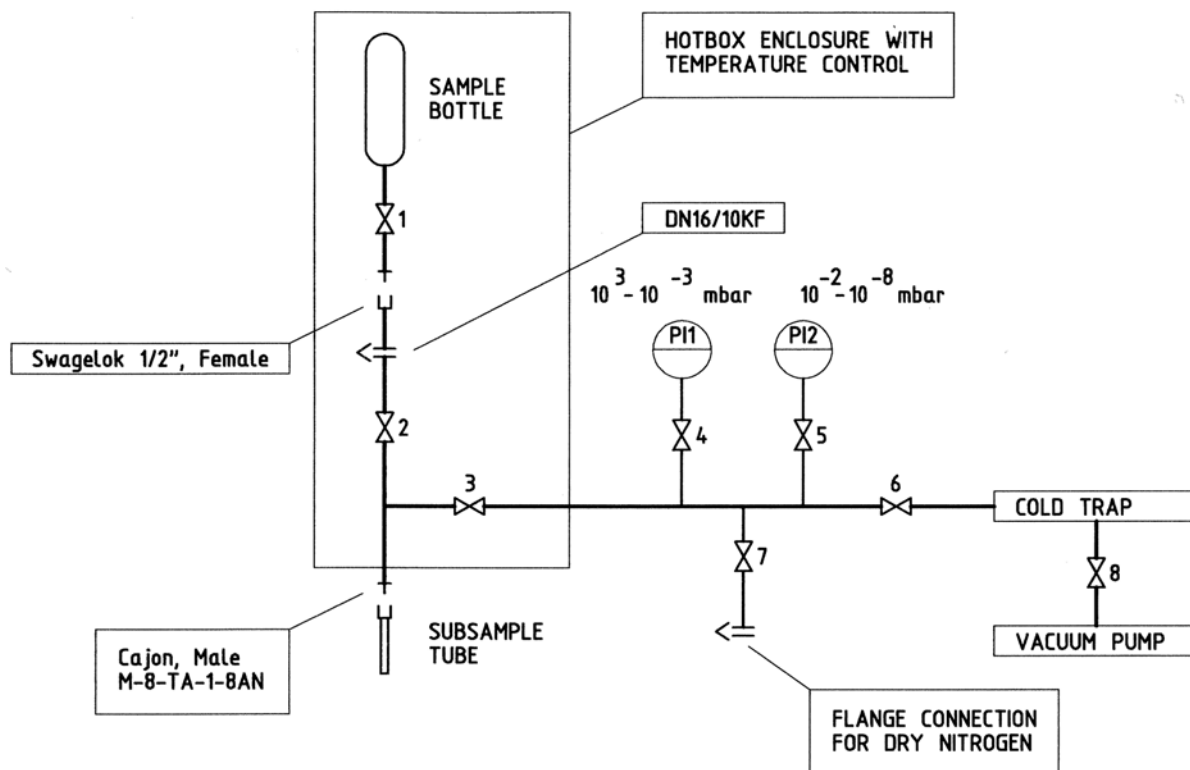
NOTE 1—All lines are 3/8 in. (9.5 mm) Monel tubing.

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.

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 UF₆ are 3/8 in. (9.5 mm) Monel tubing.
 NOTE 2—All other lines are 3/8 in. (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.

FIG. 2 Subsample Rig Used for Procedure 2

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.

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 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 (UF₆) is radioactive, toxic and highly reactive especially in the presence of reducing substances and moisture. Safe techniques must be utilized when handling UF₆. Suitable handling procedures are described in USEC-651.

7.2 Follow all safety procedures for handling UF₆ as provided by your facility.

7.3 Review Material Safety Data Sheets for UF₆ and all chemicals associated with this method prior to performance.

7.4 Perform subsampling operations in a fume hood that has been verified operable and has undergone regular inspections to ensure proper airflow.

7.5 When released to atmosphere, gaseous UF₆ reacts with moisture to produce HF gas and UO₂F₂ particulates (a white amorphous solid) and becomes readily visible as a white cloud. The corrosive nature of HF and UF₆ can result in skin burns and lung impairment. Medical evaluation is mandatory after contact with HF or UF₆. When water-soluble UO₂F₂ is inhaled or ingested in large quantities it can be toxic to the kidneys.

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 UF₆ facilitates the process of homogenization by the action of convection currents within the bulk upon heating. It

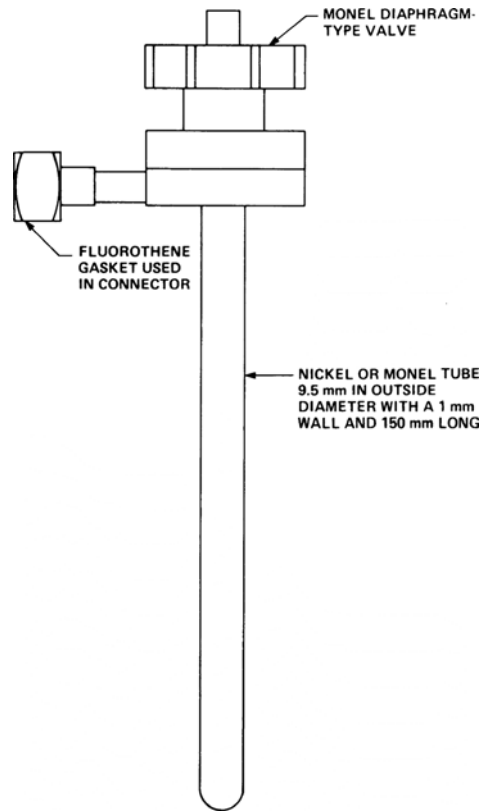


FIG. 3 Isotopic Abundance Sample Tube.

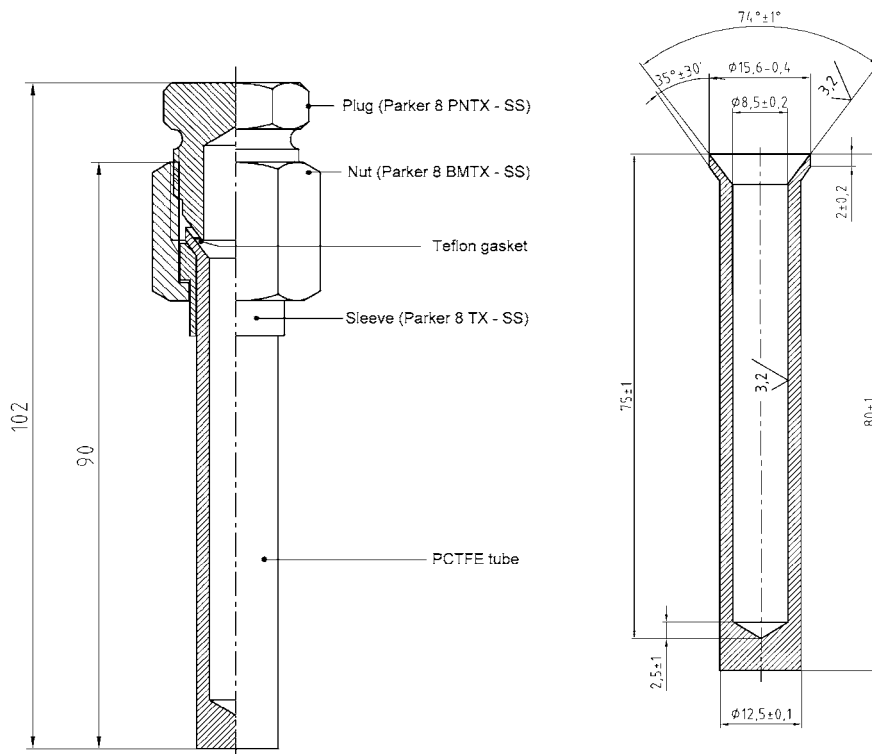


FIG. 4 Example of a Polychlorotrifluoroethylene Subsample Tube

is necessary to determine and establish for each set of subsampling equipment the physical conditions, normally a

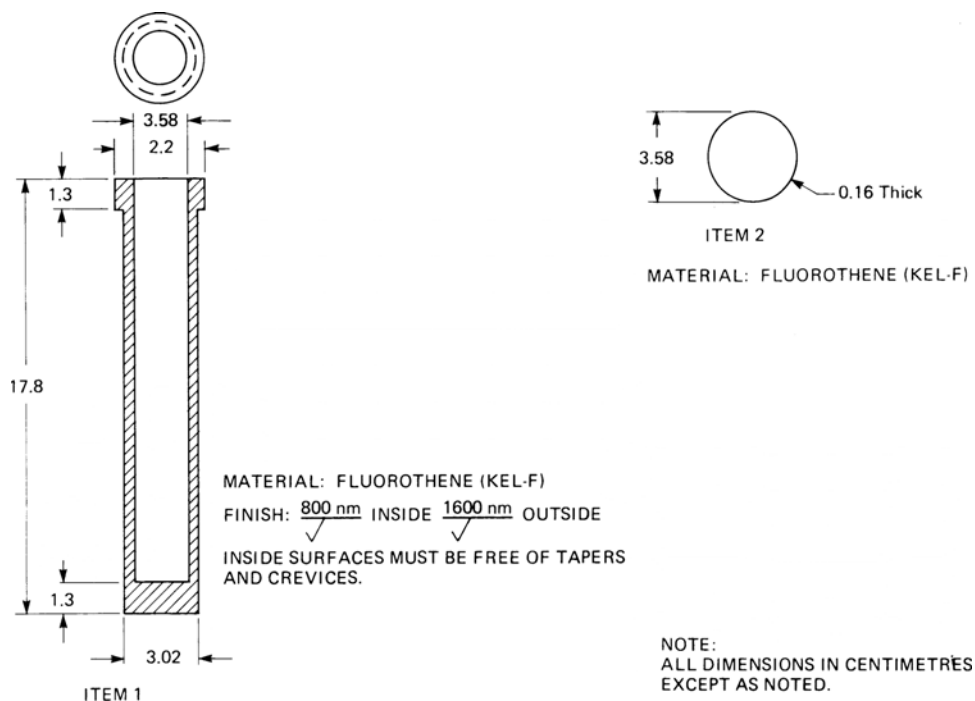


FIG. 5 Example of a Polychlorotrifluoroethylene Knock-out Tube

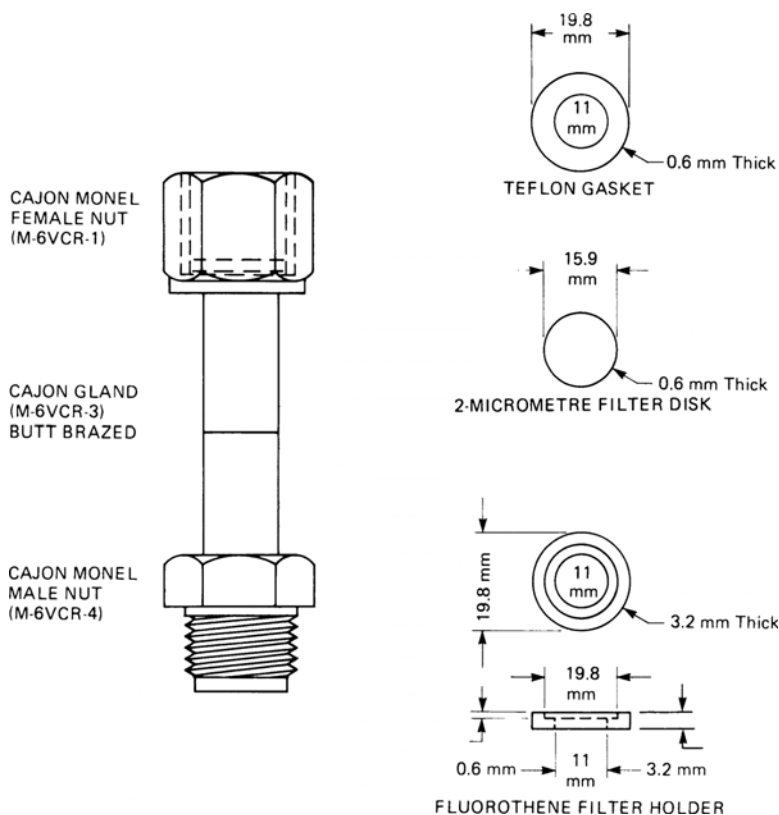


FIG. 6 Filter Disc Unit for Determination of Soluble and Insoluble Chromium

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 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.

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 UF₆ subsampling for P10 tubes or liquid UF₆ 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 UF₆ 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 requirements (natural UF₆, enriched UF₆, and particular agreements between buyer and seller) and the available equipment. Below are some typical examples of analyses that can be performed and their appropriate subsample tubes.

9.2 Uranium isotopic abundance analyses can be performed using an isotopic abundance sample tube (Fig. 3, uranium content appr. 4 g), a polychlorotrifluoroethylene tube (P10, Fig. 4, UF₆ content > 7 g) or alternatively measured directly in the gas phase using the 1S or 2S sample bottle.

9.3 For uranium purity determination a polychlorotrifluoroethylene tube (P10, Fig. 4) containing > 7 g of UF₆ can be used.

9.4 Determination of (metallic) impurities, for example B, Si, Tc, Cl, Br, etc., can be performed using a polychlorotrifluoroethylene tube (P-10, Fig. 4, UF₆ content > 7 g).

9.5 Soluble and insoluble chromium analyses can be performed using a nickel filter disc (Fig. 6) and a polychlorotrifluoroethylene tube (UF₆ content 15-20 g).

9.6 Subsampling for hydro-, chloro-, and halo-hydrocarbon analysis can be performed using a gas sample cylinder $\leq 9.99 \times 10^3$ Pa. Alternatively the analysis can be performed directly on the 1S or 2S sample bottle.

9.7 In order to achieve the typically required detection limits for the determination of impurities in reprocessed UF₆ (Pu, Np, ²³²U, γ -activity of fission products), larger amounts of UF₆ are required (typically > 50 g). For this purpose either a polychlorotrifluoroethylene knock-out cylinder (Fig. 5) or a number of polychlorotrifluoroethylene tubes (P10, Fig. 4) can be used.

9.8 Finally, it has to be emphasized that other types of subsample holders can also be used as long as the requirements in 8.3 are met.

10. Procedure 1

10.1 System Preparation:

10.1.1 Place the bulk sample container in a water bath at 90°C \pm 3°C.

10.1.2 After the UF₆ has been liquefied, remove the container from the bath, shake to homogenize the sample, and connect it at the top of the vacuum-manifold system shown in Fig. 1. The container will be inverted if liquid UF₆ must be subsampled.

10.1.3 When a liquid subsample is required for uranium analysis or chemical impurity (metal, halogen), and TMS isotopic analysis, connect a tared polychlorotrifluoroethylene sample tube at the Cajon connection at the bottom of the system. If this subsample is not required, attach a blind fitting at this point.

10.1.4 When a liquid subsample is required for gaseous isotopic analysis or all gas carbon compound determination, attach a tared isotopic abundance sample tube to the sample tube connection. If a subsample is not required, attach a cap at this point.

10.1.5 When a total gas UF₆ transfer is necessary, connect the UF₆ bulk container without inversion. Attach a polychlorotrifluoroethylene knockout cylinder at the tube connection and attach a cap at the fitting for isotopic abundance sample tube.

10.1.6 Close valve 4, then evacuate the entire system, except for the UF₆ bulk sample container.

10.2 Sample Transfer to the Polychlorotrifluoroethylene Tube:

10.2.1 Close valves 1, 2, and 3.

10.2.2 To remove solid impurities, which may be present in the bulk-container valve, open that valve and then close it quickly. Transfer this flush aliquot of liquid UF₆ to a polychlorotrifluoroethylene sample tube, as described below, and discard.

10.2.3 Open the bulk-container valve; then open valve 2 slowly, allowing liquid UF₆ to flow into the polychlorotrifluoroethylene tube. When the tube is half full of liquid UF₆, close valve 2.

10.2.4 Close the bulk-container valve.

10.2.5 Open valve 3 to remove UF₆ from the system. Open valve 1 to ensure that the system is evacuated.

10.2.6 Close valve 3.

10.2.7 Immerse the polychlorotrifluoroethylene tube in liquid nitrogen for 6 min.

10.2.8 Open valve 2 to ensure that the sample does not exert a detectable vapor pressure.

10.2.9 Close valve 5.

10.2.10 Open valves 3 and 4, and admit dry nitrogen or dry air until a pressure slightly above 1 atm ($> 1.013250 \times 10^5$ Pa) is reached.

10.2.11 Disconnect the polychlorotrifluoroethylene tube, seal with a polychlorotrifluoroethylene gasket and a Monel or RVS plug, and weigh the tube assembly.

10.2.12 Cap the manifold port and close valves 2 and 4.

10.2.13 For metals, halogens, or isotopic analysis, proceed to the hydrolysis of the UF₆.

10.2.13.1 Prepare chilled distilled water. Pour the water into a clean 500 mL polyethylene bottle. Use this water to prepare a solution of 0.1 g U/g of solution.

10.2.13.2 Cool the polychlorotrifluoroethylene tube in liquid N_2 for 15 min. Remove the metal fittings and drop the tube in the water.

10.3 *Sample Transfer to a Gaseous Isotopic Abundance Tube:*

10.3.1 Open valves 1, 3, and 5, as well as the isotopic abundance tube valve, to evacuate the tube.

10.3.2 Close valves 1, 2, and 3.

10.3.3 Immerse the lower half of the metal isotopic abundance tube, as shown in Fig. 3, in liquid nitrogen for 1 or 2 min. Immerse plastic tubing in ice water and observe desublimed UF_6 .

10.3.4 Remove the liquid nitrogen or ice water and allow frost to accumulate on the tube.

10.3.5 Open the bulk-container valve and then close.

10.3.6 Open valve 1 slowly, and observe melting of the frost as the UF_6 flows into the tube. As soon as the frost disappears, close the isotopic abundance tube valve.

10.3.7 Open valve 3 to remove the UF_6 from the manifold.

10.3.8 Close valve 5 and open valve 4 to admit dry nitrogen or dry air until a slight positive pressure is reached. Then close valve 4.

10.4 *Sample Preparation for Soluble and Insoluble Chromium in UF_6 :*

10.4.1 Attach the porous nickel filter unit with a weighed 2- μ m filter, as shown in Fig. 4, to the bottom port of the vacuum manifold, and attach a tared polychlorotrifluoroethylene tube to the bottom of the filter unit.

10.4.2 Transfer 15 to 20 g of liquid UF_6 through the filter to the polychlorotrifluoroethylene sample tube, as in 10.2.3 through 10.2.12.

10.4.3 Remove the porous nickel filter and dissolve it in 10 mL of 1.42 sp gr HNO_3 .

10.4.4 Submit the dissolved filter solution for analysis of chromium insoluble in UF_6 and the filtered UF_6 in the polychlorotrifluoroethylene sample tube for chromium soluble in UF_6 .

10.5 *Sample Transfer to Polychlorotrifluoroethylene Knockout Cylinder:*

10.5.1 Attach the polychlorotrifluoroethylene knockout cylinder, as shown in Fig. 5, to the bottom port of the manifold with a Cajon M-16 VCR-1 female nut, an M-16 VCR-4 male nut, and an M-16 VCR-3 gland.

10.5.2 Transfer approximately 50 to 200 g of liquid UF_6 to a tared polychlorotrifluoroethylene knockout cylinder, as in 10.2.3 through 10.2.12, except that in 10.2.7, the knockout cylinder is immersed in liquid nitrogen for 15 min.

10.5.3 Allow the knockout cylinder to warm to ambient temperature and then weigh to the nearest g.

10.5.4 Freeze the knockout cylinder in liquid nitrogen for 15 min, and remove the metal fittings and cover gasket. Transfer the sample to a tared, 2-L polypropylene beaker chilled in ice water, by inverting the knockout cylinder over the beaker and rapping the bottom of the knockout cylinder with a rubber mallet.

10.5.5 Immediately add chilled distilled water, to form a solution of approximately 0.1 g U/g of solution.

10.5.6 Allow the solution to reach ambient temperature while stirring periodically with a polypropylene stirring rod until all of the solid has dissolved.

10.5.7 Weigh the solution and determine the uranium concentration per g of solution.

10.5.8 Dispense aliquots of the solution for analysis according to Section 9.

10.6 *Sample Transfer for All Gas Carbon Determination:*

10.6.1 Attach a 2S cylinder to the fitting for the spectrometer isotopic abundance tube (Fig. 1).

10.6.2 Open valves 1, 3, and 5 and the gas sample cylinder valve, and evacuate the manifold and sample cylinder.

10.6.3 Close valve 3.

10.6.4 Slowly open the bulk-sample container valve to admit UF_6 until a pressure of 25 mm Hg (3.33×10^3 Pa) is reached in the sample cylinder. Close the bulk-sample container valve.

10.6.5 Open valve 3 and evacuate the manifold and sample cylinder.

10.6.6 Close valve 3.

10.6.7 Slowly open the bulk-sample container valve to admit UF_6 until a pressure of 75 mm Hg (9.99×10^3 Pa) is reached in the sample cylinder.

10.6.8 Close the gas sample cylinder valve and open valve 3 to evacuate the manifold.

10.6.9 Close valve 5, open valve 4, to bring the manifold to atmospheric pressure and remove the bulk-sample container from the manifold.

10.6.10 Plug the bulk-sample container port on the manifold, close all valves, and remove the gas sample cylinder.

10.7 *Total UF_6 Gas Transfer:*

10.7.1 Immerse the bottom of the Knockout cylinder in liquid nitrogen for at least 15 min.

10.7.2 Close valve 3 and 5 and then open the bulk container valve. The pressure will increase due to the flow the UF_6 being drawn into the manifold.

10.7.3 Wait until the pressure drops back to its original value, which will indicate a total transfer of the gaseous UF_6 in the knockout cylinder.

NOTE 3—If the pressure does not drop, it may be due to frozen UF_6 in the top of the knockout cylinder. In that case remove the liquid nitrogen from the knockout cylinder and heat the knockout cylinder until the UF_6 drops. Replace the liquid nitrogen container.

10.7.4 Close valve 2 and open valves 3 and 5 to evacuate the UF_6 in the manifold. Open valve 4 to fill manifold with dry N_2 .

10.7.5 Disconnect and weigh the UF_6 bulk container. Compare the tare weight in order to ensure that a total UF_6 transfer was performed from the mother container to the knockout cylinder. If not, reconnect the mother container and proceed from 10.7.2.

10.7.6 Proceed according to 10.5.4 to hydrolyse the UF_6 in the knockout cylinder.

11. Procedure 2

11.1 System Preparation

11.1.1 Connect the sample bottle in the Hotbox enclosure at the top of the vacuum-manifold system shown in Fig. 2. Connect a tared polychlorotrifluoroethylene tube at the bottom of the vacuum-manifold system.

11.1.2 Set the temperature of the Hotbox at $80^{\circ} \pm 3^{\circ}\text{C}$ and allow the UF_6 in the sample bottle to homogenize for at least 1.5 h.

11.1.3 Cool the coldtrap with liquid N_2 , wait at least 15 min and open the valves of the coldtrap (6 and 8). Open the valves 2, 3, 4 and 5 to evacuate the vacuum-manifold until vacuum is $< 1 \times 10^{-4}$ mbar.

11.1.4 When the UF_6 in the sample bottle is liquefied, close valves 3, 4, 5 and 8.

11.2 *Sample Transfer to the Polychlorotrifluoroethylene Tube:*

11.2.1 Slowly open valve 1, allowing liquid UF_6 to flow into the polychlorotrifluoroethylene tube. When the tube is half full with liquid UF_6 , close valve 1.

11.2.2 Immerse the polychlorotrifluoroethylene tube in liquid N_2 for 10 min, then open valve 3 and evacuate the vacuum manifold for appr. 15 min, open valve 4 and close valve 6 to ensure that the sample does not exert a detectable vapor pressure.

11.2.3 Close valve 2 and 3 and disconnect the polychlorotrifluoroethylene tube, seal with a polychlorotrifluoroethylene gasket and a plug.

11.2.4 Connect another polychlorotrifluoroethylene tube to the vacuum-manifold to evacuate the system.

12. Keywords

12.1 subsampling; uranium hexafluoride

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