Designation: C1346 – 08 (Reapproved 2014)

Standard Practice for Dissolution of UF_6 from P-10 Tubes[1,2]

This standard is issued under the fixed designation C1346; 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 dissolution of UF_6 from a P-10 tube to provide solutions for analysis.

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 *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.* For specific safeguard and safety precaution statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride

C787 Specification for Uranium Hexafluoride for Enrichment

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

D1193 Specification for Reagent Water

3. Summary of Practice

3.1 UF_6 samples intended for analysis are packaged in P-10 tubes to prevent sublimation and reaction with moisture in the air. The P-10 tube assembly (Fig. 1) consists of a Polychlorotrifluoroethylene (PCTFE) tube containing the UF_6, a PCTFE gasket to cover the tube's opening, and a nut and plug (Monel or SS) to seal the gasket to the tube.

3.2 The UF_6 tube is weighed, cooled in liquid nitrogen, and quickly opened and immersed in water for dissolution. The pieces of the tube's assembly are removed from the resulting solution, rinsed, dried, reassembled, and weighed. The solution is dried for gravimetric conversion to U_3O_8, or diluted to an appropriate concentration for dispensing into aliquots for subsequent analysis.

4. Significance and Use

4.1 Uranium hexafluoride is a basic material used to prepare nuclear reactor fuel. To be suitable for this purpose the material must meet criteria for uranium content, isotopic composition and metallic impurities in Specification C787 and C996. This practice results in the complete dissolution of the sample for uranium and impurities analysis, and determination of isotopic distribution by mass spectrometry as described in, for example, Test Methods C761.

5. Apparatus

5.1 *Steam bath,* in a hood, if optional step 9.2.13 is used.

5.2 *Vacuum oven,* if option 2 of 9.2.14 is used. The oven should be adjustable to 80°C at an absolute pressure of 3×10^3 Pa.

5.3 *Dewar flask,* wide-mouth.

5.4 *Vise,* small lab-bench model or similar type of holder.

5.5 *Wrench,* 15⁄16 in.

5.6 *Plastic clamping forceps,* 12 to 13 cm long, with a claw-like bent tip, to securely hold the cylindrical PCTFE tube.

NOTE 1—These forceps are not commercially available. Bend the ends of a straight-tip forceps by heating over a moderate flame, shaping, and maintaining the shape until cool.

5.7 *TFE-fluorocarbon-coated spatula,* 0.5- to 1-cm wide at its flat end, optional.

5.8 *Platinum or PCTFE rod,* optional.

5.9 *Platinum dishes or plastic beakers with compatible HF resistance (typically PolyEthylene; PE),* large enough to contain a completely submerged P-10 tube.

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[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] Polychlorotrifluoroethylene P-10 tubes are widely accepted by the industry for subsample collection and subsequent UF_6 quality analyses or dispatch to the customer. The procedure for subsample collection and dissolution can also be used for other types of subsample tubes, for example, P-20, P-80 or P-100 , in that case the amount of water has to be adjusted to ensure complete hydrolisation of UF_6 and avoid excessive heat evolution.

[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.

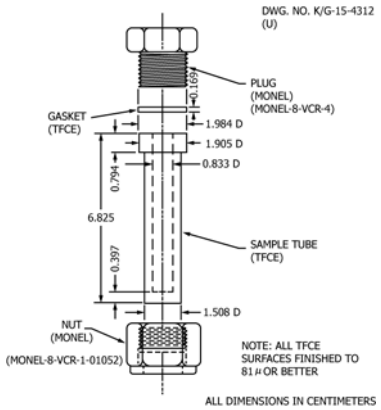

FIG. 1 Example of a P-10 Sample Tube

5.10 *Copper wires,* optional. The wires should be flexible and looped at one end to loosely fit around the PCTFE tube without allowing the flare nut to pass through.

5.11 *Desiccator,* optional.

5.12 *Balance,* ≥100-g capacity, readable to at least 0.1 mg, preferably 0.01 mg.

NOTE 2—Use of a balance with lower sensitivity will negatively impact on sampling error.

6. Interferences

6.1 The weight of the PCTFE tube is affected by atmospheric humidity. Keep the P-10 tube assembly in a desiccator between weighings until constant weight is attained.

6.2 The capacity of the UF_6 tube (a maximum of approximately 13.0 g UF_6) limits the number and size of the aliquots that can be obtained from each tube. See analytical procedures for their requirements.

7. Reagents

7.1 *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.[4] Other grades of reagents 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.

7.2 Liquid nitrogen.

7.3 Deionized distilled water in accordance with Specification D1193, approximately 50–100 cm^3 per sample.

7.4 Ethanol or other suitable, volatile organic solvent.

[4] *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.

8. Hazards

8.1 Since UF_6 materials are radioactive, toxic, and highly reactive, especially with reducing substances and moisture, adequate laboratory facilities and fume hoods along with safe techniques must be used in handling samples containing these materials. A detailed discussion of all necessary precautions is beyond the scope of this practice. However, personnel who handle radioactive materials should be familiar with the safe handling practices of the facility.

8.2 Follow all safety procedures for handling uranium and UF_6 provided by the facility. Review the Material Safety Data Sheet (MSDS) for UF_6 prior to performing the procedure.

8.3 Perform dissolutions in a laboratory hood. Hoods should be regularly inspected for proper air flow.

8.4 Gaseous UF_6, when released to the atmosphere, reacts with moisture to form HF gas and UO_2F_2 particulate (a white amorphous solid that settles on all surfaces). Release of UF_6 to the atmosphere is readily visible as a white cloud. The corrosive nature of HF and UF_6 can cause skin burns and lung impairment. Medical evaluation is mandatory for all situations where there may have been inhalation or contact with HF or UF_6. Water soluble UO_2F_2, when inhaled or ingested in large quantities, is toxic to the kidneys.

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

9. Procedure

9.1 *Preparation:*

9.1.1 Wipe the outside of the tube with a lintless tissue moistened with a suitable, volatile organic solvent (for example, ethanol) and allow to air-dry. Allow the tube to stand overnight to equilibrate with room air, or place the P-10 tube in a dessicator for at least one hour.

NOTE 3—P-10 tubes can occasionally exhibit some discoloration due to trace amounts of impurities. These tubes can be used for further analyses provided that these subsequent analyses confirm compliance with the impurity limits as stated in Specification C787 and C996. Discoloration could necessitate further investigation into the causes.

9.1.2 Using a 4- or 5- decimal place balance, weigh the sample tube to constant weight. Identify this initial mass weight as W_g.

9.1.3 To reduce any loss of liquid nitrogen during the dissolution procedure, the Dewar flask and the P-10 tube may be cooled in a refrigerator prior to use (optional).

9.2 *Dissolution:*

9.2.1 Wearing cryogenic gloves and a face shield or goggles, fill the Dewar with liquid nitrogen, optionally covered with a lid such as aluminum foil during transport, and place it in the hood.

9.2.2 *Option 1*—Slip the P-10 tube into a loop of copper wire. Holding on to the end of the wire, lower the tube into the liquid nitrogen without submerging the fittings. Secure the wire by bending it over the top edge of the Dewar flask. Cover the Dewar flask with aluminum foil or other suitable covering.

9.2.3 *Option 2*—Submerge the entire P-10 tube into the liquid nitrogen. The Dewar flask may be covered with aluminum foil or other suitable covering.

9.2.4 Leave the tube suspended in liquid nitrogen for at least ten minutes. Immediately before removing the tube, pour approximately 50–100 cm^3 distilled deionized water into a platinum dish or PE beaker.

NOTE 4—The volume of distilled deionized water must be sufficient to cover the opening in the P-10 tube.

NOTE 5—For steps 9.2.5 through 9.2.9, try to minimize elapsed time while maximizing care in handling.

9.2.5 Wearing cryogenic gloves remove the P-10 tube from the liquid nitrogen. Quickly position the tube vertically in the vise, with the fittings on top.

9.2.6 Use a wrench to loosen the plug. Remove the plug and place it in a stainless steel beaker or plastic dish or on a plastic cover.

9.2.7 Gently push (the flat end of a TFE-fluorocarbon spatula, may be used) the PCTFE tube upward through the nut until just enough of the tube emerges to securely grasp the PCTFE tube. Hold the gasket gently but firmly in place with a gloved index finger.

9.2.8 Pull the tube through its nut, and lay it on its side in a platinum dish or PE beaker containing the distilled, deionized water. Either a platinum or PCTFE rod and bent-tip forceps, or the rod alone, or the forceps alone may be used, as necessary, to dislodge the gasket and facilitate the flow of water into the tube.

9.2.9 Remove the nut from the vise and place it in the stainless steel beaker or plastic dish or on the plastic cover with the plug.

9.2.10 With the tips of the bent-tips forceps partially opened, push the gasket up on the wall of the platinum dish or PE beaker. As the gasket emerges above the solution, grasp it securely with the forceps.

9.2.11 Carefully rinse the gasket and forceps with distilled deionized water into the solution and place the gasket in the stainless steel beaker or plastic dish or on the plastic cover with the fittings.

9.2.12 Place the platinum dish in the hood for at least 2–4 h to ensure that dissolution is complete. (Dissolution is complete when yellow solution completely fills the tube.) A plastic cover may be placed on the platinum dish or PE beaker at this time.

NOTE 6—In order to reduce the volume of the of the solution, the platinum dish (with P-10 tube) can be placed in a heating apparatus for approx. 1 h after the dissolution appears to be complete. Remove the platinum dish from the heating apparatus and allow to cool to ambient temperature before proceeding with 9.2.13.

9.2.13 After dissolution appears to be complete, carefully remove the empty tube from the solution using either the bent-tip clamping forceps or PCTFE rod, as appropriate, and rinse the tube with distilled deionized water into the solution. Do not splash. Place the tube in the stainless steel beaker or plastic dish or on the plastic cover with the fittings and gasket.

9.2.14 *Option 1*—Allow the emptied tube to air-dry overnight. Place the parts in a desiccator for at least one hour to remove adsorbed water, then reassemble.

9.2.15 *Option 2*—Place the P-10 tube parts in a vacuum oven at 80°C and at an absolute pressure of 3×10^3 Pa for 2 h. Remove the P-10 tube parts from the vacuum oven and allow the tube to come to ambient temperature (2 h minimum), then reassemble.

9.2.16 Weigh the tube to constant weight using the same balance as in 9.1.2. Record all weights. Identify the final weight as W_t.

9.2.17 The solution from 9.2.13 may either be dried for gravimetric conversion to U_3O_8, or transferred to an appropriate container for dilution and subsampling for chemical or isotopic analysis.

10. Calculations

10.1 *Buoyancy Corrections:*

10.1.1 Weight of UF_6 dissolved (W_c), corrected for air buoyancy and cover gas, in grams.[5,6]

$$W_c = (-0.0058) + (1.00047)\,(W_g - W_t) \quad (1)$$

where:

W_g = weight of P-10 tube containing UF_6, in grams, and
W_t = weight of empty P-10 tube, in grams.

NOTE 7—This buoyancy correction applies to the sample tube in Fig. 1. The constants in the equation may differ for different sample tubes.

11. Keywords

11.1 dissolution; P-10 tube; uranium hexafluoride; uranium hexafluoride dissolution

[5] Hedge, W. D., "Empirical Cover Gas Correction, Sample Freezing Time, and Air Buoyancy Adjustment for the Analysis of Uranium in Uranium Hexafluoride," *Report K-2051*, Oak Ridge Gaseous Diffusion Plant, Martin Marietta Energy Systems, Inc., Oak Ridge, TN, July 31, 1985.

[6] Hedge, W. D., "Composite Net UF_6 Weight Data," Martin Marietta Energy Systems, Inc., Oak Ridge Gaseous Diffusion Plant, ANALIS correspondence to R. E. Simmons, Paducah Gaseous Diffusion Plant; H. H. Sullivan, Oak Ridge Gaseous Diffusion Plant; and O. A. Vita, Goodyear Atomic Corporation, May 28, 1986.