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Standard Practice for Dissolution of UF₆ from P-10 Tubes^{1,2}

This standard is issued under the fixed designation C 1346; 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 UF₆ from a P-10 tube to provide solutions for analysis.

1.2

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

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

C 787 Specification for Uranium Hexafluoride for Enrichment² Specification for Uranium Hexafluoride for Enrichment

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

D 1193 Specification for Reagent Water

3. Summary of Practice

3.1 UF₆ 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 UF₆, a fluorothene PCTFE gasket to cover the tube's opening, and a Monel nut and plug (Monel or SS) to seal the gasket to the tube.

3.2 The UF₆ tube is weighed, cooled in liquid nitrogen, and quickly opened and immersed in ice-cold 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 U₃O₈, or diluted to an appropriate concentration for dispensing into aliquants/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, metallic impurities, hydrocarbon, composition and partially substituted halohydrocarbon content metallic impurities in Specification C 787 and C 996. This practice results in the complete dissolution of the sample for uranium and impurities analysis, and determination of isotopic distribution by thermal ionization mass spectrometry as described in, for example, Test Methods C 761. Highly volatile impurities should be determined directly on UF₆.

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 9.2.16 is used. The oven should be adjustable to 80°C at a pressure of -29 in. of Hg. 9.2.14

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. 10, 2002. Published April 2002. Originally published as C1346-96. Last previous edition C1346-96

Current edition approved June 1, 2008. Published July 2008. Originally approved in 1996. Last previous edition approved in 2002 as C 1346 – 02

² Annual Book of ASTM Standards, Vol 12.01.

² Polychlorotrifluoroethylene P-10 tubes are widely accepted by the industry for subsample collection and subsequent UF₆ 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 hydrolysis of UF₆ and avoid excessive heat evolution.

³ 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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.

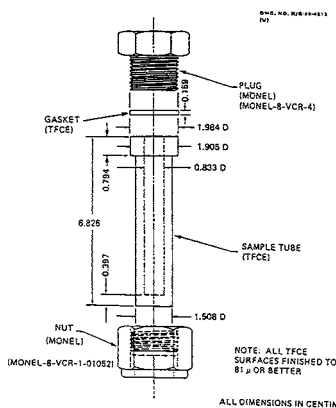


FIG. 1—This figure is from Example 10 of the P-10 Sample Tube reference in Footnote 4.

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, $1\frac{5}{16}$ in.

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

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

5.11 Desiccator, optional, 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 fluorothene 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 UF₆ tube (a maximum of approximately 13.0 g UF₆) limits the number and size of the aliquants 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 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean laboratory-accepted demineralized or deionized water.

7.3 Liquid nitrogen.

7.4 Water, deionized distilled, cooled to about 4°C, approximately 100 mL per sample.

7.5 Ethanol or other suitable, volatile organic solvent.

7.2 Liquid nitrogen.

⁴Hedge, 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.

⁴Reagent 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 Annual 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.