

Standard Practice for Cleaning for 1S and 2S Bottles¹

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1. Scope

1.1 This practice provides a description of the different ways to clean uranium hexafluoride (UF₆) bottles.

1.2 This practice describes two kinds of sample bottles: 1S and 2S bottles.

1.3 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.4 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 C787 Specification for Uranium Hexafluoride for Enrichment

C859 Terminology Relating to Nuclear Materials C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ²³⁵U

2.2 ANSI Standard:³

N14.1 Nuclear Materials—Uranium Hexafluoride– Packaging for Transport

3. Terminology

3.1 *Definitions*—Definitions of terms are as given in Terminology C859.

4. Significance and Use

4.1 The uranium hexfluoride (UF₆), as described in Specifications C787 and C996, has to meet different requirements:

one set of requirements being safety, health physics, and criticality and the other set being chemical, physical, and isotopic. To ensure the UF_6 is in compliance with all requirements, sampling and analysis shall be performed. Therefore, packaging may have a significant impact on the quality of UF_6 .

4.2 After sampling, the bottle will contain residues. There is contamination because of the equipment, other contamination caused by nonvolatile elements, and isotopic contamination as a result of UF_6 hydrolysis.

4.3 Cleaning shall be efficient. Special emphasis should be given to decontaminate the bottles without leaving any trace of cleaning products, make the bottles inert in UF_6 medium (passivation bottle), and minimize waste. The cleaning process should be easy, safe, and environmentally friendly.

4.4 This practice describes different protocols for cleaning bottles by gas and liquid.

5. Description of Sample Bottles

5.1 A bottle is composed of a cylinder, adaptors, and a valve (see Fig. 1).

5.2 Adaptors are brazed or welded on the valve and screwed on the cylinder.

5.3 Bottles and valves are made from nickel or nickelcopper alloy (for example, Monel).

5.4 The design pressure and temperature are indicated in ANSI N14.1.

6. Reagents

6.1 *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 may be used, provided it is first ascertained that the reagent is of sufficiently

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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.

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

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.





FIG. 1 1S and 2S Bottles

high purity to permit its use without lessening the effectiveness of the cleaning process.

6.2 Chlorine Trifluoride (ClF_3) :

6.2.1 Composition—See Table 1.

6.2.2 *Hazards*—ClF₃ is a highly reactive agent. With water, it forms hydrofluoric acid that penetrates the skin causing destruction of deep tissue layers. It is very corrosive and toxic by inhalation or contact. It is a powerful oxidizer that maintains the combustion and reacts violently with organic compounds.

6.3 Fluorine Gas (F_2) :

6.3.1 Composition—Fluorine gas used is pure.

6.3.2 *Hazards*—Fluorine gas is extremely corrosive and toxic. The free element has a characteristic pungent odor and is detectable in concentrations as low as 20 ppb, which is below the safe working level. Exposure to low concentrations causes eye and lung irritation.

6.4 Mixture of Hydrofluoric Acid, Sulfuric Acid, and Chrome Trioxide:

6.4.1 Composition—See Table 2.

6.4.2 *Hazards*—This mixture is a corrosive and an oxidant. It is toxic by inhalation, contact, and ingestion. It is a carcinogenic compound.

6.5 *Phosphoric Acid:*

6.5.1 *Composition*—Phosphoric acid is used at about 1 mol.L^{-1} .

6.5.2 Hazards-Corrosive reagent and it causes burns.

6.6 Potassium Carbonate (K_2CO_3) and Sodium Carbonate (Na_2CO_3):

TABLE 1 CIF₃ Composition

CIF3	>97 % (molar)
HF	≤0.2 % (molar)
CIF	≤1 % (molar)
Cl ₂	≤0.5 % (molar)
$CIO_2F - CIO_3F$	≤0.05 % (molar)

TABLE 2 Chrome Trioxide, Sulfuric Acid, and Hydrofluoric Acid				
Composition				

		% (in weight)
Chrome Trioxide	CrO ₃	5 to 10
Sulfuric Acid	H_2SO_4	5 to 15
Hydrofluoric Acid	HF	1 to 7

6.6.1 *Composition*—The concentration specified is about 100 g K_2CO_3/L .

6.6.2 *Hazards*—Irritation and corrosion of the skin, the eyes, and the respiratory and digestive tracts.

6.7 Hydrogen Peroxide (H_2O_2) :

6.7.1 Composition—The concentration specified is about 1 to 4 % $H_2O_2.$

6.7.2 *Hazards*—Strong oxidizer, corrosive to the eyes, and causes severe burns.

6.8 Citric Acid:

6.8.1 Composition—The concentration specified is about 150 g/L.

6.8.2 *Hazards*—Citric acid can cause severe eye irritation and possible injury.

6.9 Nitric Acid:

6.9.1 *Composition*—The concentration specified is about 0.01 mol/L.

6.9.2 *Hazards*—Nitric acid is a corrosive chemical and contact can severely irritate and burn the skin and eyes.

6.10 Acetic Acid:

6.10.1 Composition-No concentration specified.

6.10.2 *Hazards*—Causes severe eye irritation. Contact with liquid or vapor causes severe burns and possible irreversible eye damage.

7. Gaseous Cleaning

7.1 Emptying the Bottles:

7.1.1 The bottles are connected to a cleaning manifold inside a heating enclosure.

7.1.2 The equipment is tested to ensure vacuum integrity. The valves are opened.

7.1.3 The enclosure is heated to 70° C for approximately 2 h. The manifold is pumped at 10 Pa abs for approximately 1 h.

7.1.4 The bottles are filled with nitrogen to 400 kPa abs and pumped as in 7.1.3.

7.1.5 These operations are repeated twice.

7.2 *ClF*₃ *Treatment*:

7.2.1 The bottles are filled with ClF_3 at 15 kPa abs. This lasts approximately 1 h.

7.2.2 The bottles are emptied by pumping at 10 Pa abs for approximately 1 h.

7.2.3 The bottles are filled for a second time at 15 kPa abs and treated for approximately 2 h.

7.2.4 The bottles are then emptied as in 7.2.2.

7.2.5 The bottles are disconnected at room temperature and may be used for sampling.

7.3 F_2 Treatment:

7.3.1 The bottles are filled at 100 kPa abs with different concentrations of F_2 in N_2 .