Designation: C1838 − 16

Standard Practice for Cleaning for 1S and 2S Bottles[1]

This standard is issued under the fixed designation C1838; 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 provides a description of the different ways to clean uranium hexafluoride (UF_6) 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 % ^{235}U

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 (UF_6), 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 nickel-copper 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

[1] This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.02 on Fuel and Fertile Material Specifications.

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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*—ClF_3 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 ClF_3 Composition

ClF_3	>97 % (molar)
HF	≤0.2 % (molar)
ClF	≤1 % (molar)
Cl_2	≤0.5 % (molar)
ClO_2F – ClO_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.

7.3.2 The first treatment is performed at 10 % of F_2.

7.3.3 The last treatment is performed at 100 % of F_2.

7.3.4 Between each treatment, the bottles are emptied by pumping at 10 Pa abs.

8. Liquid Cleaning

8.1 *Operations before Washing:*

8.1.1 *External Cleaning*—Use a solvent for degreasing and cleaning.

8.1.2 *Bottle Dismantling:*

8.1.2.1 The bottles are drained.

8.1.2.2 The bottles are placed in a vise and the valve loosened to finger tightness.

8.1.2.3 The bottles are frozen for approximately 1 min in liquid nitrogen.

8.1.2.4 The valves are removed from the bottles.

8.2 *Operations after Washing:*

8.2.1 *Flushing and Drying:*

8.2.1.1 The liquid is decanted from the bottles and the valves in a waste container.

8.2.1.2 The bottles and the valves are rinsed with demineralized water. The liquid is decanted into a waste container.

8.2.1.3 The bottles and the valves are dried, inside and outside, using inert gas and a heating enclosure.

8.2.1.4 The internal surfaces of the dried bottles are inspected. If needed, repeat cleaning.

8.2.2 *Bottle Assembling:*

8.2.2.1 A lubricant suited for use with UF_6 or polytetrafluoroethylene tape is placed on the bottle thread to obtain a seal with the valves.

8.2.2.2 The bottles are assembled.

8.2.3 *Control of the Tightness:*

8.2.3.1 This control includes:

(1) An internal and external inspection,

(2) A hydrostatic test,

(3) A leak test of valves and caps, and

(4) A test of the thickness of walls if the corrosion is significant.

8.2.3.2 All the objectives of the control are described in ANSI N14.1.

8.3 *Washing Process:*

8.3.1 *Washing Sequences with Sodium Carbonate and Mixture of Hydrofluoric Acid, Sulfuric Acid, and Chrome Trioxide Cleaning:*

8.3.1.1 The bottles are soaked in a basic (Na_2CO_3) bath.

8.3.1.2 The bottles are rinsed with water.

8.3.1.3 The bottles are soaked in the mixture for 1 to 3 min, depending on state.

8.3.1.4 The bottles are rinsed with demineralized water.

8.3.1.5 The bottles are soaked in ethyl alcohol.

8.3.2 *Washing Sequences with Phosphoric Acid and Potassium Carbonate Cleaning:*

8.3.2.1 The bottles are rinsed with water and tripolyphosphate under pressure (150 kPa, 80°C).

8.3.2.2 The bottles are rinsed with demineralized water.

8.3.2.3 The bottles are soaked in a basic (K_2CO_3) bath.

8.3.2.4 The bottles are rinsed with water.

8.3.2.5 The bottles are soaked in an acid bath (phosphoric acid smear).

8.3.2.6 The bottles are rinsed with water.

8.3.3 *Washing Sequences with Potassium Carbonate and Hydrogen Peroxide Cleaning:*

8.3.3.1 The bottles are evacuated.

8.3.3.2 The solution is introduced in the bottle. There is no valve dismantling.

8.3.3.3 The bottles are inverted and shaken for about 2 to 3 min.

8.3.3.4 The solution is emptied using vacuum.

8.3.3.5 This method is repeated four to five times until the wash solution is clear.

8.3.3.6 The bottles are rinsed with deionized water four times in the same fashion.

8.3.4 *Washing Sequences with Citric Acid (Nitric Acid) Cleaning:*

8.3.4.1 The bottles are placed upside down in a citric acid container and are gently tapped with a wooden mallet to dislodge any loose material.

8.3.4.2 The vacuum is removed from the valves and then the valves are opened.

8.3.4.3 All of the bottles components are placed in a tray of water to await decontamination.

8.3.4.4 The bottles are filled with citric acid.

8.3.4.5 A rubber bung is placed in the bottle neck. The bottles are shaken well for 10 s, then allowed to stand for 30 min.

8.3.4.6 The valves are removed from the tray and water is run through to confirm that there is no blockage.

8.3.4.7 The valves are connected to form a vertical chain.

8.3.4.8 A pump outlet pipe is connected to the top of the chain. A pump inlet pipe is immersed into a stainless steel beaker containing a citric acid.

8.3.4.9 The liquid circulates for 1 h.

8.3.4.10 If the citric acid turns yellow, then replace with fresh solution.

8.3.4.11 Nitric acid could be used in place of citric acid, but the concentration and the treatment period shall be shorter to reduce the corrosion effect.

8.3.5 *Washing Sequences with Acetic Acid Solution and Sodium Carbonate with Hydrogen Peroxide Cleaning Method:*

8.3.5.1 The bottles are connected to the system.

8.3.5.2 The bottles are drained.

8.3.5.3 The bottles are rinsed with a decontamination solution (sodium carbonate with hydrogen peroxide) for 30 min and drained.

8.3.5.4 The bottles are rinsed with hot water for approximately 2 min and drained.

8.3.5.5 The bottles are rinsed with acetic acid solution for approximately 2 min and drained.

8.3.5.6 The bottles are rinsed with hot water for three cycles of approximately 2 min and drained.

9. Choice of Treatment

9.1 *Performance:*

9.1.1 Carbonates are decontaminants known in the nuclear field.

9.1.2 Acids are powerful reagents for decontamination.

9.1.3 Hydrogen peroxide is used as a supplement to other reagents to aid with the oxidation of uranium IV.

9.1.4 ClF_3 is very reactive and allows, at the same time, to eliminate impurities and passivate the metal of the bottle.

9.2 *Process Comparison (see Table 3):*

9.2.1 Gaseous treatments are easier to operate because they do not require dismantling of the bottle.

9.2.2 Liquid treatments require more time.

9.3 *Hazards:*

9.3.1 ClF_3 is a chemical that is extremely dangerous to use.

9.3.2 Acids and hydrogen peroxide are corrosive and toxic but their use is mastered well in the industry.

9.3.3 Carbonates present little risks.

9.4 *Effluents:*

9.4.1 For ClF_3 treatment, little product is used. Effluents are washed in a scrubber.

9.4.2 Liquid treatments generate more effluents. These reagents are commonly used in the industry. The management of effluents remains accessible.

9.4.3 The mixture of hydrofluoric acid, sulfuric acid, and chrome trioxide is very corrosive and hazardous to the environment. This reagent requires a specific additional treatment.

10. Keywords

10.1 1S 2S bottle cleaning; UF_6

TABLE 3 Comparison Process

Reagent	Decontamination Efficiency	Corrosion Effect	Process	Hazards	Waste
ClF_3	Only for U	Passivation effect	Not dismantling of bottles	Extremely dangerous	Need a gas treatment
F_2	No effect	Passivation effect	Not dismantling of bottles	Extremely dangerous	Need a gas treatment
HNO_3	Moderate on U, Th, Ni, Fe	Important risks	Need time (washing, water rincing, drying)	Toxic but well known	Need a waste treatment but well known
HF, H_2SO_4, CrO_3	Moderate on U, Th, Ni, Fe	Important risks	Need time (washing, water rincing, drying)	Toxic	Need a waste treatment
H_3PO_4 & Citric and Acetic Acid	Moderate on U, Th, Ni, Fe	Low risks	Need time (washing, water rincing, drying)	Low toxicity	Need a waste treatment but well known
K_2CO_3 or Na_2CO_3 + H_2O_2	High efficiency for U and Th and leave a deposit of metallic impurities	No corrosion	Need time (washing, water rincing, drying)	Low toxicity	Need a waste treatment but well known