



Standard Test Method for Determination of Bromine and Chlorine in UF₆ and Uranyl Nitrate by X-Ray Fluorescence (XRF) Spectroscopy¹

This standard is issued under the fixed designation C 1508; 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 method covers the determination of bromine (Br) and chlorine (Cl) in uranium hexafluoride (UF₆) and uranyl nitrate solution. The method as written covers the determination of bromine in UF₆ over the concentration range of 0.2 to 8 µg/g, uranium basis. The chlorine in UF₆ can be determined over the range of 4 to 160 µg/g, uranium basis. Higher concentrations may be covered by appropriate dilutions. The detection limit for Br is 0.2 µg/g uranium basis and for Cl is 4 µg/g uranium basis.

1.2 *This standard may involve hazardous materials, operations and equipment. 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:

- C 761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear and Radiochemical Analysis of Uranium Hexafluoride²
- C 787 Specification for Uranium Hexafluoride for Enrichment²
- C 788 Specification for Uranyl Nitrate²
- C 1118 Guide for Selecting Components for Wavelength Dispersive X-Ray Fluorescence (XRF) Systems²
- D 1193 Specification for Reagent Water³

3. Summary of Test Method

3.1 A sample of hydrolyzed UF₆ (uranyl fluoride) or uranyl nitrate solution is treated with sodium nitrite to reduce oxidized forms of bromine and chlorine (bromates and chlorates) to their respective halide ions. Addition of silver nitrate precipitates the silver halides. Spike recoveries can be improved by the addition of potassium iodide causing coprecipitation of the halides. The halides are collected on filter paper and are

analyzed by X-ray fluorescence using two different crystal/detector systems.

4. Significance and Use

4.1 The method is designed to show whether or not the tested materials meet the specifications as given in C 787 and C 788.

5. Interferences

5.1 Plastic equipment must be used throughout the method for uranyl fluoride as the hydrofluoric acid in the uranyl fluoride leaches chloride from glassware causing a high bias.

5.2 Low recoveries may occur as the precipitate can be difficult to transfer quantitatively to the filter paper. A surfactant can be added (optional step) to minimize the adhesion of the precipitate to the walls of the beakers and the funnel.

6. Apparatus

- 6.1 *X-Ray Spectrometer*, see Guide C 1118 for the selection of the X-ray Spectrometer.
- 6.2 *Plastic Vacuum Filtration Apparatus*, for 47 mm diameter filter paper.
- 6.3 *Filter Paper*, 0.45 micron, 47 mm diameter.⁴
- 6.4 *Beakers*, polypropylene, 250 mL.
- 6.5 *Stirring Rods*, plastic or Teflon.
- 6.6 *X-ray Sample Support, Rings*. Inner diameter approximately 40 mm.

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.⁵ Other grades 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 reagent water conforming

¹ This test method 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 August 10, 2001. Published September 2001.

² *Annual Book of ASTM Standards*, Vol 12.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ The filter must be Cl and Br free. Millipore membrane filter type HABP (www.millipore.com) has been successfully used. An alternate is 15A from S.C.B. BP6, RN86, 07130 Soyons France. Any other equivalent is acceptable.

⁵ Reagent Chemicals, American Society Specifications, American Chemical Society, Washington, DC.