Designation: ~~C1508 – 01 (Reapproved 2011)~~ C1508 – 18

Standard Test Method for Determination of Bromine and Chlorine in UF_6 and Uranyl Nitrate by X-Ray Fluorescence (XRF) Spectroscopy[1]

This standard is issued under the fixed designation C1508; 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 (UF_6) and uranyl nitrate solution. The method as written covers the determination of bromine in UF_6 over the concentration range of 0.2 to 8 µg/g, uranium basis. The chlorine in UF_6 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 The values stated in SI units are to be regarded as the standard.

1.3 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

C787 Specification for Uranium Hexafluoride for Enrichment

C788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals

~~C1118~~C859 ~~Guide for Selecting Components for Wavelength-Dispersive X-Ray Fluorescence (XRF) Systems~~Terminology Relating to Nuclear Materials ~~(Withdrawn 2011)~~

D1193 Specification for Reagent Water

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms relating to the nuclear fuel cycle, refer to Terminology C859.

4. Summary of Test Method

4.1 A sample of hydrolyzed UF_6 (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.

5. Significance and Use

5.1 The method is designed to show whether or not the tested materials meet the specifications as given in Specifications C787 and C788.

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[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.

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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.

6. Interferences

6.1 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.

6.2 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.

7. Apparatus

7.1 *X-Ray Spectrometer,* ~~see Guide~~ appropriate ~~C1118~~for the ~~selection of the X-ray Spectrometer.~~intended use.

7.2 *Plastic Vacuum Filtration Apparatus,* for 47 mm diameter filter paper.

7.3 *Filter Paper,* 0.45 micron, 47 mm diameter.[3]

7.4 *Beakers,* polypropylene, 250 mL.

7.5 *Stirring Rods,* plastic or Teflon.

7.6 *X-ray Sample Support, Rings.* Inner diameter approximately 40 mm.

8. Reagents

8.1 *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 high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type I.

8.3 *Ammonium Hydroxide,* NH_4OH, concentrated, specific gravity 0.90.

8.4 *Ammonium Hydroxide Solution,* 1 + 3 (3.7 M). Dilute 1 part NH_4OH with 3 parts water.

8.5 *Surfactant,* bromine and chlorine free.[5] (Optional).

8.6 *Surfactant Solution,* 1 + 999. Add 1 mL of surfactant to approximately 1 litre of water. (Optional).

8.7 *Nitric Acid,* HNO_3, concentrated, specific gravity 1.42.

8.8 *Nitric Acid Solution,* 1 + 999 (0.016 M). Add 1 mL of concentrated HNO_3 to approximately 200 mL of water. Add 1 mL of the surfactant (~~7.6~~8.6). (Optional). Dilute to 1 litre.

8.9 *Sodium Nitrite,* $NaNO_2$.

8.10 *Sodium Nitrite Solution,* 2 g/L. Dissolve 1 g of sodium nitrite in water and dilute to 500 mL. Prepare fresh immediately before use.

8.11 *Silver Nitrate,* $AgNO_3$.

8.12 *Silver Nitrate Solution,* 2 g/L. Dissolve 2 g of silver nitrate in water and dilute to 1 litre. Keep away from light in an opaque bottle. The solution should be made fresh weekly. Silver is an RCRA listed hazardous waste. Make up only as much of this solution as required to minimize excess waste.

8.13 *Potassium Bromide,* KBr.

8.14 *Bromide Solution,* 500 mg Br/L. Dissolve 0.1861 g of KBr (dried at 110°C for 1 hour) in water and dilute to 250 mL in a volumetric flask.

8.15 *Sodium Chloride,* NaCl.

8.16 *Sodium Chloride Solution,* 1000 mg Cl/L. Dissolve 1.648 g NaCl (dried at 110° C for 1 hour) in water and dilute to 1 litre in a volumetric flask.

8.17 *Spike Solution,* 5 mg Br/L, 100 mg Cl/L. Transfer 10 mL of 500 mg ~~Br /L~~ Br/L solution into a 1 litre volumetric flask by pipette. Transfer 100 mL of 1000 mg ~~Cl /L~~ Cl/L into the flask by pipette. Dilute to volume.

~~[3] The last approved version of this historical standard is referenced on www.astm.org.~~

[3] 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.

[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.

[5] Triton X-100, Rohm and Haas, has been successfully used.