Designation: C 1539 – 02

Standard Test Method for Determination of Technetium-99 in Uranium Hexafluoride by Liquid Scintillation Counting[1]

This standard is issued under the fixed designation C 1539; 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 test method is a quantitative method used to determine technetium-99 (^{99}Tc) in uranium hexafluoride (UF_6) by liquid scintillation counting.

1.2 *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 787 Specification for Uranium Hexafluoride for Enrichment[2]

C 996 Specification for Uranium Hexafluoride Enriched to Less that 5 % ^{235}U[2]

C 1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry[2]

2.2 *Other Document:*

USEC-651 Uranium Hexafluoride: A Manual of Good Handling Practices[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *quench standard curve*—a relationship between sample quench and detection efficiency. A quench curve for an isotope in a given cocktail and vial combination is developed by counting a series of standards containing the same activity of that isotope, but each with different quench. Sample quench is typically quantified by a variety of parameters.

4. Summary of Test Method

4.1 A measured portion of hydrolyzed uranium hexafluoride (UF_6) containing approximately 0.8 to 1.2 g of uranium or a volume of sample less than or equal to 30 mL is transferred to a centrifuge tube. The uranium is precipitated using ammonium hydroxide. After centrifuging, the decanted supernatant is acidified with sulfuric acid and extracted with tributyl phosphate. An aliquot of the extract is transferred to a scintillation vial, where stannous chloride in hydrochloric acid and liquid scintillation cocktail are added. The ^{99}Tc beta activity is then determined by liquid scintillation counting.

5. Significance and Use

5.1 Uranium hexafluoride is a basic material used to prepare nuclear reactor fuel. To be suitable for this purpose, the material must meet the criteria for technetium composition. This test method is designed to determine whether the material meets the requirements described in Specifications C 787 and C 996.

5.2 Using the specified instrumentation and parameters, this method has a lower detection limit of 0.0004μgTc/gU.

NOTE 1—Different instrumentation or parameters may provide varying detection limits, as calculated in 11.4.

6. Apparatus

6.1 *Liquid Scintillation Counter*[4], with alpha/beta discrimination and enhanced low level discrimination over the entire energy range of 0 to 2000 keV.

6.2 *Centrifuge*.

6.3 *Analytical Balance*, 1 mg sensitivity.

6.4 *Separatory Funnel*, 125 mL volume.

[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] *Annual Book of ASTM Standards*, Vol 12.01.

[3] Available from U.S. Enrichment Corporation, 6903 Rockledge Drive, Bethesda, MD 20817.

[4] Packard Tri-Carb Model 1905 AB/LA has been found to be acceptable.

6.5 *Liquid Scintillation Vials*, 20 mL.
6.6 *Centrifuge Tubes with Caps*, 50 mL.
6.7 *Laboratory Wipes*, lint free disposable.

7. Reagents and Materials

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 of Analytical Reagents of the American Chemical Society where specifications are available.[5]

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean laboratory accepted deionized water.

7.3 *Ammonium Hydroxide (NH_4OH)*, concentrated (14.5*M*).

7.4 *Hydrochloric Acid (HCl)*, concentrated (12*M*).

7.5 *Hydrochloric Acid (HCl)(1M)*. Add 82 mL of concentrated (12*M*) HCl to 900 mL of water, dilute to a final volume of 1000 mL, and mix.

7.6 *Liquid Scintillation Cocktail*[6].

7.7 *Potassium Permanganate ($KMnO_4$)*, 1 % W/V in water. Dissolve 1 g of $KMnO_4$ in 100 mL of water, and mix.

7.8 *Stannous Chloride ($SnCl_2$)*, 20 % (W/V) $SnCl_2$ in concentrated hydrochloric acid. Dissolve 20 g of $SnCl_2$ in 100 mL of concentrated hydrochloric acid, and mix.

7.9 *Sulfuric Acid (H_2SO_4)*, concentrated 18*M*.

7.10 *Sulfuric Acid (H_2SO_4)*. 9*M*. Add 500 mL concentrated H_2SO_4 (18*M*) to 400 mL water, dilute to a final volume of 1000 mL, and mix.

7.11 *Sulfuric Acid (H_2SO_4)*. 3*M*. Add 168 mL of concentrated H_2SO_4 (18*M*) to 800 mL of water, dilute to a final volume of 1000 mL, and mix.

7.12 *Sulfuric Acid (H_2SO_4)*. 1*M*. Add 56 mL of concentrated H_2SO_4 (18*M*) to 900 mL of water, dilute to a final volume of 1000 mL, and mix.

7.13 *Technetium Standard(s) in a Basic Aqueous Solution*.

7.14 *Tributyl Phosphate (TBP $C_{12}H_{27}O_4P$)*, saturated solution. Equilibrate 500 mL TBP with 500 mL 3*M* H_2SO_4. Shake for approximately 2 min. Allow to separate and discard aqueous layer.

8. Hazards

8.1 Since UF_6 is radioactive, toxic, and highly reactive, especially when reducing substances and moisture are present (see USEC-651), appropriate facilities and practices must be provided.

9. Procedure

9.1 Transfer an aliquot up to 30 mL of one of the following solutions, as applicable, to a 50 mL centrifuge tube:

9.1.1 *Hydrolyzed UF_6 Sample*—Unknown UF_6 sample hydrolyzed in water.

9.1.2 *Standard*—Laboratory control sample with a known ^{99}Tc concentration.

9.1.3 *Spike Solution*—UF_6 sample spiked with a known concentration of ^{99}Tc (approximately ten times the sample activity).

9.2 Add 2 drops of potassium permanganate solution (1 % W/V) and swirl to mix.

9.3 Dilute with water to approximately 35 mL and swirl to mix.

9.4 Add 5 mL concentrated ammonium hydroxide to precipitate uranium.

9.5 Dilute with deionized water to 50 mL.

9.6 Cap and shake vigorously to break up large particles of ammonium diuranate.

9.7 Centrifuge for approximately 10 min at approximately 1500 rpm.

9.8 Add 25 mL 9*M* H_2SO_4 to a clean 125-mL separatory funnel.

9.9 Decant the supernatant containing the technetium into the 125-mL separatory funnel.

NOTE 2—The precipitated uranium remains in the centrifuge tube.

9.10 Add 5 mL of TBP solution to the separatory funnel.

9.11 Stopper or cap the funnel and shake for approximately 60 s.

9.12 Allow phases to separate a minimum of 5 min.

9.13 Drain off aqueous (lower) phase into a waste beaker.

9.14 Add 20 mL of 3*M* H_2SO_4.

9.15 Stopper or cap the funnel and shake for approximately 30 to 45 s.

9.16 Allow phases to separate for a minimum of 5 min.

9.17 Drain off aqueous (lower) phase into a waste beaker.

9.18 Pipette up to 4 mL of the extract from the funnel into a 20 mL scintillation vial.

9.19 Pipette 0.2 mL stannous chloride solution into the vial.

9.20 Pipette 12 mL liquid scintillation cocktail into the vial.

NOTE 3—This test method has proven acceptable for 12 mL of liquid scintillation cocktail, but up to 16 mL can be added depending on the user's instrumentation.

9.21 Cap the vial and shake vigorously for approximately 5 to 10 s.

9.22 Wipe the outside of the vial with a damp laboratory wipe to remove static electricity, if necessary.

9.23 Place the vial in the liquid scintillation counter.

9.24 Allow vial to stand for approximately 15 min prior to counting.

10. Counting

10.1 Program the liquid scintillation counter according to the manufacturer's guidelines.[7]

10.2 Place reagent blank in position one and allow instrument to subtract background counts to obtain net counts per minute (cpm), as needed.

10.3 Count vial for three consecutive 10 min counts, and average (avg).

[5] 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*, Merk Ltd., Poole, Dorset, U.K. and the *United States Pharmacopeia and National Formulary* , U.S. Pharmacopoeil Convention, Inc. (USPC), Rockville, MD, or equivalent.

[6] Insta-Gel® has been found to be acceptable.

[7] Packard Tri-Carb Model1905 AB/LA uses a 0.8 to 293 keV counting window.