Designation: C1429 – 99 (Reapproved 2014)

Standard Test Method for Isotopic Analysis of Uranium Hexafluoride by Double-Standard Multi-Collector Gas Mass Spectrometer[1]

1. Scope

1.1 This test method covers a quantitative test method applicable to determining the mass percent of uranium isotopes in uranium hexafluoride (UF_6) samples. This method as described is for concentrations of ^{235}U between 0.1 and 10 mass %, and ^{234}U and ^{236}U between 0.0001 and 0.1 mass %.

1.2 This test method is for laboratory analysis by a gas mass spectrometer with a multi-collector.

1.3 This standard complements Test Methods C761, the double-standard method for gas mass spectrometers using a single collector, by providing a method for spectrometers using a multi-collector.

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]

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

C787 Specification for Uranium Hexafluoride for Enrichment

C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U

C1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *A standard, n*—the low-value standard of a standard pair that brackets the sample.

3.1.2 *B standard, n*—the high-value standard of a standard pair that brackets the sample.

3.1.3 *determination, n*—a single isotopic value, calculated from a sequence of ratios; the most basic isotopic value calculated.

3.1.4 *Lagrange's interpolation formula, n*—a mathematical equation designed to estimate values between two or more known values.

3.1.5 *run, n*—a completed, six-entry symmetrical sequence consisting of A standard, sample, B standard, B standard, sample, and A standard from which a determination can be calculated for one or more isotopes.

3.1.6 *standard spread, n*—the difference between the high and low standards; sometimes called standard range.

3.1.7 *test result, n*—a reported value; the mean of two or more determinations.

4. Summary of Test Method

4.1 Uranium hexafluoride gas is introduced into an ionization source. The resulting ions are accelerated down the flight tube into the magnetic field. The magnetic field separates the ions into ion beams in accordance with the *m/e* ratio. Four collectors are stationed so the $^{234}UF_5^+$, $^{235}UF_5^+$, $^{236}UF_5^+$, and $^{238}UF_5^+$ ion beams strike individual collectors.

4.2 Two standards are chosen whose values bracket the desired isotope of the sample. The sample and two standards are introduced in a six-entry, symmetrical sequence. Then, measurements are taken that give the mole ratio of the desired isotope to ^{238}U.

4.3 Through Lagrange's interpolation formula, these measurements are used to calculate the mass percent of the desired isotope. If standards are available that bracket all isotopes, then the ^{234}U, ^{235}U, and ^{236}U mass percents are calculated from the same six-entry run.

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[1] This test method is under the jurisdiction of ASTM CommitteeC26 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.

4.4 The results of two six-entry, symmetrical-sequence runs are averaged to find test results for each isotope. The ^{238}U mass percent is obtained by subtraction.

5. Significance and Use

5.1 Uranium hexafluoride used to produce nuclear-reactor fuel must meet certain criteria for its isotopic composition. This test method may be used to help determine if sample materials meet the criteria described in Specifications C787 and C996.

6. Apparatus

6.1 Mass spectrometer with the following features and capabilities:

6.1.1 An ion source with an accelerating voltage of approximately 8 kV,

6.1.2 A resolving power of greater than or equal to 500,

6.1.3 A minimum of three points of attachment for standards or samples,

6.1.4 An ion collection system consisting of four collector cups stationed to collect $^{234}UF_5^+$, $^{235}UF_5^+$, $^{236}UF_5^+$, and $^{238}UF_5^+$ ions,

6.1.5 An ion-current amplifier for each collector cup,

6.1.6 A voltage-to-frequency (V-to-F) converter for each amplifier,

6.1.7 A counter for each V-to-F converter, and

6.1.8 Computer control over opening and closing valves, the timing, and the integration of analytical sequences.

7. Procedure

7.1 *Select standards:*

7.1.1 Choose high and low standards that bracket the sample isotope(s) being evaluated. If the mass percent of ^{234}U, ^{235}U, and ^{236}U are all desired, then the two standards must bracket each of the three isotopes to permit calculation of all isotopes for every run.

7.1.2 If standards that bracket all isotopes are unavailable, analyze the isotope(s) bracketed by the originally selected standards, then select other standards to run the remaining isotope(s).

7.2 *Prepare Sample and Standards:*

7.2.1 Attach sample and standard containers to the spectrometer.

7.2.2 Open and close the appropriate valves to evacuate the air from the inlet system.

7.2.3 Open the sample and standard containers individually and vent the gas phase to the cold trap. This is to remove impurities that may bias the results or interfere with the ionization. If necessary, freeze the UF_6 with ice water or a mixture of crushed dry ice and isopropyl alcohol to permit longer venting without losing large amounts of UF_6.

7.2.4 Permit exhaust system pressure to recover.

7.2.5 Check to see if impurities have been sufficiently removed by introducing UF_6 into the ion source and observing pressure, or exhausting through the cold trap and observing pressure on the other side, or any other suitable means.

7.2.6 If necessary, repeat 7.2.3 – 7.2.5 until samples are clean.

7.3 *Prepare Instrument:*

7.3.1 Adjust instrument parameters to focus ion beams in proper collectors and maximize the $^{238}UF_5^+$ current reading.

7.3.2 Enter standard values and other information if needed for calculations performed by computer.

7.3.3 Program the spectrometer to run two of the following six-entry, symmetrical sequences: low standard, sample, high standard, high standard, sample, low standard.

7.4 *Run the Analysis:*

7.4.1 Obtain measurements from all four collectors during each entry.

8. Calculation

8.1 Perform the following operations for each of the ^{234}U, ^{235}U, and ^{236}U isotopes:

8.1.1 For each entry, obtain a ratio by dividing the UF_5^+ ion count of the desired isotope by the $^{238}UF_5^+$ ion count.

8.1.2 Find the mean of the two low standard ratios and designate this A.

8.1.3 Find the mean of the two sample ratios and designate this X.

8.1.4 Find the mean of the two high standard ratios and designate this B.

NOTE 1—In a six-entry symmetrical run sequence,

$$(r_1+r_6)/2 = A \quad (1)$$

$$(r_2+r_5)/2 = X \quad (2)$$

$$(r_3+r_4)/2 = B \quad (3)$$

where:

r_n = the ratio from the n^{th}entry.

8.1.5 Find the mass percent ratio of the low value standard (*A* standard) by dividing the mass percent of the desired isotope by the mass percent ^{238}U.

$$E_A = \text{mass \% } ^{234}U/\text{mass \% } ^{238}U \quad (4)$$

$$H_A = \text{mass \% } ^{235}U/\text{mass \% } ^{238}U \quad (5)$$

$$Y_A = \text{mass \% } ^{236}U/\text{mass \% } ^{238}U \quad (6)$$

8.1.6 Find the equivalent mass percent ratio for the high value standard (*B* standard.) Label it either E_B, H_B, or Y_B.

8.1.7 Find the difference (*D*) between the mass percent ratios of the *A* and *B* standards.

NOTE 2—$E_B - E_A = D_E$, $H_B - H_A = D_H$, and $Y_B - Y_A = D_Y$

8.1.8 Find the mass percent ratio (desired isotope/^{238}U) of the sample by calculating E_X , H_X, or Y_X as follows:

$$E_X = ((X-A)/(B-A))\cdot D_E + E_A \quad (7)$$

$$H_X = ((X-A)/(B-A))\cdot D_H + H_A \quad (8)$$

$$Y_X = ((X-A)/(B-A))\cdot D_Y + Y_A \quad (9)$$

NOTE 3—Calculations in 8.1.8 are based on an algebraic manipulation of Lagrange's interpolation formula using two known values of a function.

8.2 Calculate the mass percent as follows:

$$e = 100\%\cdot E_X/(1+E_X+H_X+Y_X) \quad (10)$$

$$x = 100\%\cdot H_X/(1+E_X+H_X+Y_X) \quad (11)$$

$$y = 100\%\cdot Y_X/(1+E_X+H_X+Y_X) \quad (12)$$