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# Standard Test Method for Isotopic Analysis of Uranium Hexafluoride by Single–Standard Gas Source Multiple Collector Mass Spectrometer Method[1]

This standard is issued under the fixed designation C1428; 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 applicable to the isotopic analysis of uranium hexafluoride ($UF_6$) with $^{235}U$ concentrations less than or equal to 5 % and $^{234}U$, $^{236}U$ concentrations of 0.0002 to 0.1 %.

1.2 This test method may be applicable to the analysis of the entire range of $^{235}U$ isotopic compositions providing that adequate Certified Reference Materials (CRMs or traceable standards) are available.

1.3 *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, health, and 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]

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 *Other Document:*

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

## 3. Terminology

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

## 4. Summary of Test Method

4.1 The unknown sample and a CRM or traceable standard whose isotopic composition is close to that of the sample are introduced in sequence into the mass spectrometer, and $^{234}U$, $^{235}U$, $^{236}U$, and $^{238}U$ ions are focused through corresponding collector slits to the four separate collectors. Measurements are made that are proportional to the ratios of $^{234}U$, $^{235}U$, or $^{236}U$ to $^{238}U$. With the known composition of the CRM or traceable standard, these ratios of molar ratios permit calculation of the $^{234}U$, $^{235}U$, and $^{236}U$ contents. Memory corrections are applied based on the periodic measurement of two CRMs or traceable standards.

## 5. Significance and Use

5.1 Uranium hexafluoride is a basic material used to produce nuclear reactor fuel. To be suitable for this purpose, the material must meet criteria for isotopic composition. This test method is designed to determine whether the material meets the requirements described in Specifications C787 and C996.

## 6. Apparatus

6.1 A gas source multiple collector mass spectrometer with the following attributes:

6.1.1 The resolving power of the mass spectrometer is not less than 500. The resolving power (R) is calculated from the registered mass spectrum of both the $^{235}UF_5^+$ and $^{238}UF_5^+$ isotopes as follows:

$$R = \frac{a \cdot M}{b \cdot \Delta M} \quad (1)$$

[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.

[3] Available from United States Enrichment Corporation, 6903 Rockledge Drive, Bethesda, MD 20817.

where:

$a$ = distance between centers of the $^{235}UF_5^+$ and $^{238}UF_5^+$ peaks,
$b$ = peak width of the $^{238}UF_5^+$ isotope (10 % valley),
$M$ = 333 − mass(u) $^{238}UF_5^+$, and
$\Delta M$ = 3 = 333 − 330, 330 − mass(u) $^{235}UF_5^+$.

6.1.2 The abundance sensitivity of the mass spectrometer is specified as less than $1\times10^{-5}$ as contribution of mass 333 ($^{238}UF_5^+$) to mass 331 ($^{236}UF_5^+$).

6.1.3 The four collectors have collector slits adjusted for ions of masses 329, 330, 331, and 333. Ion currents are amplified by four amplifiers, having noise level less than 0.5 mV.

6.1.4 The ion beams are kept within the slits by an automatic beam positioner circuit.

6.1.5 The pumping system of the mass spectrometer analyser tube shall maintain a pressure less than $5\times10^{-8}$ torr with a sample flowing into the ion source.

6.1.6 The memory correction factor of the mass spectrometer as defined in 10.1 shall be consistent with the required accuracy and precision, and shall not exceed 1.005.

6.1.7 The sample inlet system shall be equipped with a manifold, including adjustable leak, and valves for introducing the sample and CRM or traceable standard in sequence and for evacuating corresponding lines. The pumping system of the inlet system must maintain a pressure less than $2\times10^{-2}$ torr by evacuating.

## 7. Hazards

7.1 Uranium hexafluoride ($UF_6$) is radioactive, toxic, and highly reactive especially with reducing substances and moisture. Appropriate laboratory facilities, materials of construction, and techniques shall be utilized when handling $UF_6$ (see for example USEC-651).

7.2 When released to atmosphere, gaseous $UF_6$ reacts with moisture to produce HF gas and toxic $UO_2F_2$ particulates. Use sufficient ventilation or respiratory protection to avoid breathing fumes. Use appropriate personal protective equipment such as gloves, eye, and face protection. Consult the Safety Data Sheet (SDS) for additional information.

## 8. Calibration and Standardization

8.1 *Uranium Hexafluoride Isotopic:*

8.1.1 Two CRMs or traceable standards are used to determine the memory correction factor. The $^{235}U$ concentration ratio of upper CRM or traceable standard ($C_{235}^{SH}$) to $^{235}U$ of lower CRM or traceable standard ($C_{235}^{SL}$) shall not be more than three ($C_{235}^{SH}/C_{235}^{SL} \leq 3$).

8.1.2 For memory correction factor determination for $^{234}U$ and $^{236}U$ isotopes, two CRMs or traceable standards are used which differ in $^{234}U$ ($^{236}U$) concentration. In concentration range 0.0002 to 0.01, $C_{234\,(236)}^{SH}/C_{234\,(236)}^{SL} \leq 8$; in concentration range 0.01 to 0.1, $C_{234\,(236)}^{SH}/C_{234\,(236)}^{SL} \leq 6$.

8.1.3 The CRMs or traceable standards used for measurements may differ in $^{235}U$ concentration from a sample. ($C_{235}^{X}$) on condition that $C_{235}^{SH}/C_{235}^{X} \leq 1.5$ and $C_{235}^{X}/C_{235}^{SL} \leq 1.5$. For $^{234}U$ ($^{236}U$) isotopes, the following range limitations shall be used: 0.0002 to 0.01 $C_{234(236)}^{SH}/C_{234(236)}^{X} \leq 4, C_{234(236)}^{X}/C_{234(236)}^{SL} \leq 4$; in concentration range 0.01 to 0.1 $C_{234(236)}^{SH}/C_{234(236)}^{X} \leq 3, C_{234(236)}^{X}/C_{234(236)}^{SL} \leq 3$.

## 9. Procedure

A typical sequence for the analytical determination is X, S, X, S, where X and S mean the introduction of the sample and the CRM or traceable standard, respectively. Each introduction is followed by ion source evacuation before the next introduction. During each introduction, a simultaneous measurement of the four uranium isotopes occurs.

The intensities of the $^{238}UF_5^+$ ion beam for both sample and CRM or traceable standard introduction shall not differ more than 3 %. Adjustment is performed by pressure equalization of the sample and standard in the inlet system.

The number of introductions per analytical sequence is dependent on the precision required.

9.1 Attach sample containers containing the appropriate sample, X, and standard, S, to the inlet system, and prepare both materials for introduction into the ion source as follows:

9.1.1 Operate the appropriate valves to remove air entrapped in the connectors and to check that there are no leaks in inlet system.

9.1.2 Freeze the $UF_6$ by immersing the sample container (the unknown sample) into a mixture of water and ice.

9.1.3 Open the valve on the container to permit evacuation of volatile impurities, and then close the valve.

9.1.4 Remove the coolant from around the container and allow the $UF_6$ to return to room temperature.

9.1.5 Repeat 9.1.2 – 9.1.4 for the CRM or traceable standard.

9.2 *Operation of the Mass Spectrometer:*

9.2.1 Operate appropriate valves to admit the CRM or traceable standard into the ion source.

9.2.2 Adjust the accelerating voltage or magnet current to focus the ion beams $^{234}UF_5^+$, $^{235}UF_5^+$, $^{236}UF_5^+$, and $^{238}UF_5^+$ to their corresponding collectors. Adjust the mass spectrometer parameters to obtain the maximum $^{238}UF_5^+$ ion current and maximum resolution.

9.2.3 Regulate the adjustable leak to obtain a $^{238}UF_5^+$ ion current of about $10^{-9}$ A.

9.2.4 Measure the ion current ratio of $^{234}U$, $^{235}U$, and $^{236}U$ to $^{238}U$.

9.2.5 Terminate the flow of the CRM or traceable standard and evacuate the ion source.

9.2.6 Repeat 9.2.1, 9.2.4, and 9.2.5 for the sample.

## 10. Calculation

10.1 The memory correction factor $M$ is calculated by the formula:

$$M_i = \frac{\dfrac{E_i^{S1} - E_i^{S2}}{E_i^{S2}}}{\dfrac{r_i^{S1} - r_i^{S2}}{r_i^{S2}}} \quad (2)$$

**TABLE 1 Uranium Isotopic CRMs Results**

| | Mass-spectrometer A | | | Mass-spectrometer B | | |
|---|---|---|---|---|---|---|
| CRMs used for determination of the memory correction factor Reg. No. RM, Lot number, Certified values, Isotope Mass Fraction, % | CRM 7533-99, 207-05 (Working CRM) $C^S_{235}$=4.9512±0.0024 $C^S_{234}$=0.04612±0.00015 $C^S_{236}$=0.01521±0.00010 | CRM 7523-99, 210-06 $C^S_{235}$=1.2047±0.0005 $C^S_{234}$=0.00747±0.00002 $C^S_{236}$=0.00231±0.00002 | | CRM 7533-99, 207-05 (Working CRM) $C^S_{235}$=4.9512±0.0024 $C^S_{234}$=0.04612±0.00015 $C^S_{236}$=0.01521±0.00010 | CRM 7520-99, 3-79 $C^S_{235}$=0.5040±0.0003 $C^S_{234}$=0.0035±0.0001 $C^S_{236}$=0.0060±0.0001 | |
| CRMs used as a sample Reg. No. RM, Lot number, Certified values, Isotope Mass Fraction, % | CRM 7530-99, 186-01 $C^X_{235}$ 4.0032 ± 0.0019 | $C^X_{234}$ 0.0360 ± 0.0002 | $C^X_{236}$ 0.0133 ± 0.0002 | CRM 7530-99, 186-01 $C^X_{235}$ 4.0032 ± 0.0019 | $C^X_{234}$ 0.0360 ± 0.0002 | $C^X_{236}$ 0.0133 ± 0.0002 |
| Mean $\left(\bar{C}^X_i\right)$ | 4.0024 | 0.03590 | 0.01324 | 4.0035 | 0.03595 | 0.01328 |
| Quantity of determinations under repeatability ($n$) | 9 | 9 | 9 | 10 | 10 | 10 |
| Relative standard uncertainty, % | 0.006 | 0.12 | 0.43 | 0.006 | 0.09 | 0.33 |
| Bias $\left(\bar{C}^X_i - C^X_i\right)$ | 0.0008 | 0.00010 | 0.00006 | 0.0003 | 0.00005 | 0.00005 |
| CRMs used for determination of the memory correction factor Reg. No. RM, Lot number, Certified values, Isotope Mass Fraction, % | CRM 7533-99, 207-05 (Working CRM) $C^S_{235}$=4.9512±0.0024 $C^S_{234}$=0.04612±0.00015 $C^S_{236}$=0.01521±0.00010 | CRM 7523-99, 210-06 $C^S_{235}$=1.2047±0.0005 $C^S_{234}$=0.00747±0.00002 $C^S_{236}$=0.00231±0.00002 | | CRM 7533-99, 207-05 $C^S_{235}$=4.9512±0.0024 $C^S_{234}$=0.04612±0.00015 $C^S_{236}$ 0.01521±0.00010 | CRM 7520-99, 3-79 (Working CRM) $C^S_{235}$=0.5040±0.0003 $C^S_{234}$=0.0035±0.0001 $C^S_{236}$=0.0060±0.0001 | |
| CRMs used as a sample Reg. No. RM, Lot number, Certified values, Isotope Mass Fraction, % | CRM 7527-99, 219-08 $C^X_{235}$ 2.8250 ± 0.0011 | $C^X_{234}$ 0.02545 ± 0.0001 | $C^X_{236}$ 0.00070 ± 0.00003 | CRM 7527-99, 219-08 $C^X_{235}$ 2.8250 ± 0.0011 | $C^X_{234}$ 0.02545 ± 0.0001 | $C^X_{236}$ 0.00070 ± 0.00003 |
| Mean $\left(\bar{C}^X_i\right)$ | 2.8248 | 0.02542 | 0.00072 | 2.8243 | 0.02544 | 0.00067 |
| Quantity of determinations under repeatability ($n$) | 10 | 10 | 10 | 10 | 10 | 10 |
| Relative standard uncertainty, % | 0.005 | 0.13 | 5.2 | 0.009 | 0.14 | 7.2 |
| Bias $\left(\bar{C}^X_i - C^X_i\right)$ | 0.0002 | 0.00003 | 0.00002 | 0.0007 | 0.00001 | 0.00003 |
| CRMs used for determination of the memory correction factor Reg. No. RM, Lot number, Certified values, Isotope Mass Fraction, % | CRM 7520-99, 3-79 $C^S_{235}$=0.5040±0.0003 $C^S_{234}$=0.0035±0.0001 $C^S_{236}$=0.0060±0.0001 | CRM 7523-99, 210-06 (Working CRM) $C^S_{235}$=1.2047±0.0005 $C^S_{234}$=0.00747±0.00002 $C^S_{236}$=0.00231±0.00002 | | CRM 7520-99, 3-79 $C^S_{235}$=0.5040±0.0003 $C^S_{234}$=0.0035±0.0001 $C^S_{236}$=0.0060±0.0001 | CRM 7523-99, 210-06 (Working CRM) $C^S_{235}$=1.2047±0.0005 $C^S_{234}$=0.00747±0.00002 $C^S_{236}$=0.00231±0.00002 | |
| CRM used as a sample Reg. No. RM, Lot number, Certified values, Isotope Mass Fraction, % | CRM 7521-99, 184-00 $C^X_{235}$ 0.71049 ± 0.00029 | $C^X_{234}$ 0.00531 ± 0.00007 | $C^X_{236}$ < 0.00002 | CRM 7521-99, 184-00 $C^X_{235}$ 0.71049 ± 0.00029 | $C^X_{234}$ 0.00531 ± 0.00007 | $C^X_{236}$ < 0.00002 |
| Mean $\left(\bar{C}^X_i\right)$ | 0.710496 | 0.00533 | . . . | 0.710463 | 0.005312 | . . . |
| Quantity of determinations under repeatability ($n$) | 10 | 10 | . . . | 10 | 10 | . . . |
| Relative standard uncertainty, % | 0.03 | 0.7 | . . . | 0.02 | 0.8 | . . . |
| Bias $\left(\bar{C}^X_i - C^X_i\right)$ | 0.000006 | 0.00002 | . . . | 0.000027 | 0.000002 | . . . |