Designation: C1832 – 22

Standard Test Method for Determination of Uranium Isotopic Composition by Modified Total Evaporation (MTE) Method Using Thermal Ionization Mass Spectrometer[1]

This standard is issued under the fixed designation C1832; 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 describes the determination of the isotope amount ratios of uranium material as nitrate solutions by the modified total evaporation (MTE) method using a thermal ionization mass spectrometer (TIMS) instrument.

1.2 The analytical performance in the determination of the $^{235}U/^{238}U$ major isotope amount ratio by MTE is similar to the ("classical") total evaporation (TE) method as described in C1672. However, in the MTE method, the evaporation process is interrupted on a regular basis to allow measurements and subsequent corrections for background from peak tailing, perform internal calibration of a secondary electron multiplier (SEM) detector versus the Faraday cups, peak centering, and ion source refocusing. Performing these calibrations and corrections on a regular basis during the measurement, improves precision, and significantly reduces uncertainties for the minor isotope amount ratios $^{234}U/^{238}U$ and $^{236}U/^{238}U$ as compared to the TE method.

1.3 In principle, the MTE method may yield major isotope amount ratios without the need for mass fractionation correction. However, depending on the measurement conditions, small variations are observed between sample turrets. Therefore, a small correction based on measurements of a certified reference material is recommended to improve consistency. The uncertainty around the mass fractionation correction factor usually includes unity.

1.4 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.5 *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.*

1.6 *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]

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C776 Specification for Sintered Uranium Dioxide Pellets for Light Water Reactors

C787 Specification for Uranium Hexafluoride for Enrichment

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets for Light Water Reactors

C859 Terminology Relating to Nuclear Materials

C967 Specification for Uranium Ore Concentrate

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

C1008 Specification for Sintered (Uranium-Plutonium) DioxidePellets—Fast Reactor Fuel (Withdrawn 2014)[3]

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis

[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 Feb. 1, 2022. Published March 2022. Originally approved in 2016. Last previous edition approved in 2021 as C1832 – 21. DOI: 10.1520/C1832-22.

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

C1411 Practice for The Ion Exchange Separation of Uranium and Plutonium Prior to Isotopic Analysis
C1625 Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances by Thermal Ionization Mass Spectrometry
C1672 Test Method for Determination of Uranium or Plutonium Isotopic Composition or Concentration by the Total Evaporation Method Using a Thermal Ionization Mass Spectrometer
C1871 Test Method for Determination of Uranium Isotopic Composition by the Double Spike Method Using a Thermal Ionization Mass Spectrometer
D1193 Specification for Reagent Water
E2586 Practice for Calculating and Using Basic Statistics
E2655 Guide for Reporting Uncertainty of Test Results and Use of the Term Measurement Uncertainty in ASTM Test Methods

3. Terminology

3.1 Terminology C859 contains terms, definitions, descriptions of terms, nomenclature, and explanations of acronyms and symbols specifically associated with standards under the jurisdiction of Committee C26 on Nuclear Fuel Cycle.

3.2 *Definitions:*

3.2.1 *abundance sensitivity, n*—in isotope amount ratio measurements, the ratio of the measured intensity of an ion beam at a mass *m*, to the measured intensity from the same isotope measured at one mass unit difference (for example, $m \pm 1$).

3.2.1.1 *Discussion*—Abundance sensitivity is a measure of the magnitude of the peak tailing correction. For measuring uranium on thermal ionization mass spectrometer (TIMS) and inductively coupled plasma mass spectrometry (ICP-MS) instruments, the abundance sensitivity is typically calculated as the ratio of the measured signal intensities at masses 237 and 238 using a suitable uranium sample.

3.2.2 *dark noise, n*—observed count rate on an ion counting detector measured without incident ion beam

3.2.3 *total evaporation, TE, n*—analytical method for determination of isotope amount ratios of uranium or plutonium, as described in Test Method C1672, also called “classical” total evaporation in this test method.

3.2.4 *turret, n*—holder for sample filaments.

3.2.4.1 *Discussion*—Alternate names for turret are carousel, magazine, wheel.

3.3 *Acronyms and Abbreviations:*

3.3.1 *cpm*—counts per minute

3.3.2 *cps*—counts per second

3.3.3 *CRM*—Certified Reference Material

3.3.4 *DS*—Double Spike

3.3.5 *DU*—Depleted Uranium

3.3.6 *FAR*—Faraday Cup

3.3.7 *HEU*—High Enriched Uranium

3.3.8 *IAEA*—International Atomic Energy Agency

3.3.9 *ICPMS*—Inductively Coupled Plasma Mass Spectrometry

3.3.10 *IRMM*—Institute for Reference Materials and Measurements

3.3.11 *ITU*—Institute for Transuranium Elements

3.3.12 *JRC*—Joint Research Centre of the European Union

3.3.13 *JRC-G.2*—Unit for Standards for Nuclear Safety, Security and Safeguards of JRC (new name for the nuclear part of the former IRMM)

3.3.14 *JRC-G.II.6*—Unit for Nuclear Safeguards and Forensics of JRC (part of the former ITU)

3.3.15 *LEU*—Low Enriched Uranium

3.3.16 *MTE*—Modified Total Evaporation

3.3.17 *NBL*—New Brunswick Laboratory

3.3.18 *RSD*—Relative Standard Deviation; SD (see below) divided by the mean value of the observations in repeated sampling

3.3.19 *RSE*—Relative Standard Error; SE (see below) divided by the mean value of the observations in repeated sampling

3.3.20 *SD*—Standard Deviation; according to Practice E2586, 3.1.30, the square root of the sum of the squared deviations of the observed values in the sample divided by the sample size minus one

3.3.21 *SE*—Standard Error; according to Practice E2586, 3.1.29, standard deviation of the population of values of a sample statistic (that is, the mean value) in repeated measurements, or an estimate of it.

3.3.21.1 *Discussion*—According to Practice E2586, 3.1.30, if the standard error (SE, see above) of a statistic is estimated, it will itself be a statistic with some variance that depends on the sample size, that is, the number of observed values in the sample (Practice E2586, 3.1.26).

3.3.21.2 *Discussion*—According to Guide E2655, 5.8.4.1, from statistical theory, a 95 % confidence interval for the mean of a normal distribution, given n independent observations x_1, x_2, ..., x_n drawn from the distribution is, $x_{mean} \pm t \times \mathrm{SD} / \sqrt{n}$, where x_{mean} is the sample mean, SD is the standard deviation of the observations (see above), and t is the 0.975 percentile of the Student’s t distribution with n-1 degrees of freedom. Because Student’s t distribution approaches the Normal as n increases, the value of t approaches 1.96 as n increases. This is the basis for using the (coverage) factor 2 for expanded uncertainty. The standard error (SE) of the mean value of a series of n independent repeated measurements can be derived from that by using $t = 1$, so the standard error (SE) is given by $\mathrm{SD}/\sqrt{n}$.

3.3.22 *SEM*—Secondary Electron Multiplier

3.3.22.1 *Discussion*—In the scientific literature the acronym SEM is also used for Scanning Electron Microscope, but within this document SEM represents Secondary Electron Multiplier.

3.3.23 *SGAS*—Safeguards Analytical Services Laboratory of the IAEA