



Standard Test Method for Isotopic Abundance Analysis of Uranium Hexafluoride by Multi-Collector, Inductively Coupled Plasma-Mass Spectrometry¹

This standard is issued under the fixed designation C 1477; 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 covers the isotopic abundance analysis of ^{234}U , ^{235}U , ^{236}U , and ^{238}U in samples of hydrolysed uranium hexafluoride (UF_6) by inductively coupled plasma source, multi-collector, mass spectrometry (ICP-MC-MS). This test method is also described in ASTM STP 1344.

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 761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride²

C 787 Specification for Uranium Hexafluoride for Enrichment²

C 996 Specification for Uranium Hexafluoride Enriched to less than 5 % ^{235}U ²

D 1193 Specification for Reagent Water³

2.2 Other Document:

STP 1344 Applications of Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) to Radionuclide Determinations⁴

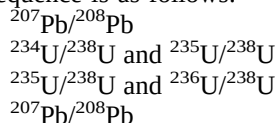
3. Summary of Test Method

3.1 Samples are received either in the form of uranium hexafluoride (UF_6) or aqueous uranic solution. The UF_6 samples are hydrolysed, diluted, and acidified with nitric acid. Uranic solution samples are diluted and acidified with nitric acid. Subsequently, an internal reference of either thorium or lead isotopes⁵ is added to each diluted sample.

3.2 The samples are contained in polythene tubes that are inserted into the auto-sampler rack of the mass spectrometer. Sample details are input to the computer and the instrument is prepared for measurement. The automatic measuring sequence is initiated.

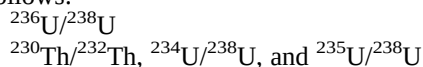
3.3 Uranium isotopic reference materials (UIRMs) are used to calibrate the instrument. Each UIRM is prepared in aqueous solution (acidified with nitric acid) and spiked with the same internal reference as the samples. This calibration solution is measured and a mass bias parameter is calculated that is stored and subsequently imported into each of the sample measurements⁶ to correct the measured uranium isotopic ratios.

3.4 Measurement of isotopic ratios in the calibration solution and the subsequent samples is initiated by customized software. The measurement sequence is determined by the internal reference used. Using the lead internal reference, the sequence is as follows:



Using the mean of the two lead ratios and the mass bias parameter imported from the calibration, the current mass bias factor is computed. The mass bias factor is then used to correct the measured uranium isotopic ratios. These corrected ratios are used to calculate the abundances of ^{234}U , ^{235}U , and ^{236}U .

3.5 Using the thorium internal reference, the sequence is as follows:



Using the $^{230}\text{Th}/{}^{232}\text{Th}$ ratios (that are acquired simultaneously to the $^{234}\text{U}/{}^{238}\text{U}$ and $^{235}\text{U}/{}^{238}\text{U}$ ratios) and the mass bias parameter imported from the calibration, the mass bias factor is computed. The mass bias factor is then used to correct the the measured $^{234}\text{U}/{}^{238}\text{U}$ and $^{235}\text{U}/{}^{238}\text{U}$ ratios in “real time.” The $^{236}\text{U}/{}^{238}\text{U}$ is retrospectively corrected for mass bias. The abundances are expressed as % atomic. A printout of results and mass spectrometer parameters is obtained for each sample. Details of the mass bias correction are presented in Appendix X1.

⁶ The uranium isotopic reporting limits and reporting errors are listed in section 16 and Appendix X1 respectively.

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 June 10, 2000. Published August 2000.

² Annual Book of ASTM Standards, Vol 12.01.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Available from ASTM Headquarters.

⁵ Thorium is preferred as the internal reference because thorium data can be acquired simultaneously to uranium data.