



Standard Test Method for Isotopic Analysis of Uranium Hexafluoride by Single- Standard Gas Source Mass Spectrometer Method¹

This standard is issued under the fixed designation C 1344; 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 analysis of uranium hexafluoride (UF_6) and may be used for the entire range of ^{235}U isotopic compositions for which standards are available.

1.2 This test method is applicable to the determination of the isotopic relationship between two UF_6 samples. If the abundance of a specific isotope of one sample (the standard) is known, its abundance in the other can be determined. This test method is flexible in that the number of times a given material is admitted to the ion source may be adjusted to the minimum required for a specified precision level.

1.3 The sensitivity with which differences between two materials can be detected depends on the measuring system used, but ratio-measuring devices can generally read ratio-of-mol ratio differences as small as 0.0001.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:

C 787 Specification for Uranium Hexafluoride for Enrichment²

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

2.2 Other Document:

USEC-651, *Uranium Hexafluoride: A Manual of Good Handling Practices*³

¹ This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved Aug. 10, 1997. Published May 1997.

² *Annual Book of ASTM Standards*, Vol 12.01.

³ Available from U.S. Enrichment Corporation, 6903 Rockledge Dr., Bethesda, MD 20817.

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *drop through, n*—a measurement of the amount of the $^{238}\text{UF}_5^+$ ion beam that can be passed through the $^{235}\text{UF}_5^+$ collector slit and measured on the $^{235}\text{UF}_5^+$ collector, stated as a percentage of the total $^{238}\text{UF}_5^+$ signal.

3.1.2 *memory corrections, n*—corrections applied to the sample analysis results for memory effects.

3.1.3 *memory effect, n*—the inability of the mass spectrometer to omit completely the isotopic composition of the sample analyzed previously from attributing to the results of further samples analyzed.

3.1.4 *normal isotopic abundance material, n*— UF_6 having a value of 0.711 weight percent (wt %) ^{235}U .

3.1.5 *ratio-of-mol-ratios, n*—the mol ratio ($^{235}\text{U}/^{238}\text{U}$) of the sample divided by the mol ratio of the standard, or the inverse condition of the mol ratio of the standard divided by the mol ratio of the sample.

4. Summary of Test Method

4.1 *Test Method*—The unknown sample and a standard with an isotopic composition close to that of the sample are introduced in sequence into the Neir mass spectrometer. UF_5^+ ions of the isotopes are focused through a mass-resolving collector slit and onto a faraday cup collector. Measurements are made of $^{235}\text{UF}_5^+$ to the total of the other UF_5^+ isotopes. With the known composition of the standard, calculation of the ^{235}U composition of the sample can be determined.

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 isotopic composition. This test method is designed to determine whether the material meets the requirements described in Specifications C 787 and C 996.

5.2 ASTM Committee C-26 Safeguards Statement:

5.2.1 The material (uranium hexafluoride) to which this test method applies is subject to the nuclear safeguards regulations governing its possession and use. The analytical procedure in this test method has been designated as technically acceptable for generating safeguards accountability data.

5.2.2 When used in conjunction with appropriate certified

reference materials (CRMs), this procedure can demonstrate traceability to the national measurement base. However, adherence to this procedure does not automatically guarantee regulatory acceptance of the regulatory safeguards measurements. It remains the sole responsibility of the user of this test method to ensure that its application to safeguards has the approval of the proper regulatory authorities.

6. Apparatus

6.1 *Neir Mass Spectrometer*, with the following features and capabilities:

6.1.1 A single-focusing spectrometer, with a 127-mm minimum deflection radius, is satisfactory when equipped and focused as follows:

6.1.1.1 The sample inlet system must have two sample holders, to which UF_6 containers can be attached, and the necessary valves to evacuate the sample lines through which the sample and standard are introduced. The sample inlet system should be nickel or Monel for use with corrosive gases, and should have minimum volume.

6.1.1.2 A single adjustable leak, operated by an automatic leak control mechanism for admitting the sample into the spectrometer ion source, is preferred.

6.1.1.3 The pumping system of the spectrometer analyzer tube must maintain a pressure below 5×10^{-8} torr with sample flowing into the ion source.

6.1.1.4 Focus the instrument for resolution consistent with precision requirements. A high-current ion beam of 5×10^{-10} to 1×10^{-9} amps is necessary, with a signal-to-noise ratio greater than 3000 in the low-current amplifier system.

6.1.1.5 A dual collector must be used, so that ions from one isotope are passed through a resolving slit and focused on a low-current collector, and ions from all other isotopes are focused on a high-current collector. The preferred method of maintaining the low-current ion beam within the collector slit is by an automatic beam positioner circuit. A resolving slit with adjustable width features enhances the measurement of all isotopes but is not mandatory for isotopic measurements.

6.1.1.6 The amplified high- and low-current signals are fed into a multimeter or other device capable of ratioing high- and low-current signals. If a multimeter is used, the multimeter must have a minimum of 5.5 digits of resolution, a means of ratioing the high- and low-current signals, and interactive communication capability with the controller.

6.1.1.7 The memory effect of the spectrometer must be consistent with the precision required since a high memory level is usually more variable than a low one. Memory values of 2 to 3 % are typical, but up to 10 % memory can be tolerated. The memory characteristics of a spectrometer must be established from periodic measurement of the effect. Current memory values usually will apply until the ion source is replaced, repairs are made on the sample inlet system, or the instrument is refocused so the flow rate of UF_6 is altered significantly.

6.1.1.8 The computer control of the mass spectrometer must allow the operator to monitor parameters of the spectrometer and check other operating conditions. The development of an interactive program allows input of sample information, performs necessary calculations, makes memory corrections, and

records data. Flexibility of the interactive program allows pausing of the instrument for adjustment or restart capability, or both. Suggested methods of analysis checks include the standard deviation (SD) on individual data points, linearity of the data set, and a check of source pressure differences between the standard and sample that can be monitored by the computer program. Manifold valve actuation, conditioning time, and pump-out time are features of the computer control program.

7. Hazards

7.1 Since UF_6 is radioactive, toxic, and highly reactive, especially with reducing substances and moisture (see USEC-651), appropriate facilities and practices for analysis must be provided.

8. Procedure

8.1 *Calibration of Isotopic Standards:*

8.1.1 One working standard is required for the analysis of a sample at any specific concentration of any isotope. Two working standards are required to determine memory corrections. Memory can be measured more precisely with a large difference between two working standards, but the adverse effect of introducing wide concentration ranges into the mass spectrometer must be considered. Ideally, the values obtained from the high- and low-memory standards should symmetrically bracket those of the sample to be corrected. Working standards approximately 5 % apart (having a ratio of ratios of 1.05) are suitable for most applications.

8.1.2 A reasonable limit for the relative e between the unknown sample and the working standard to which it is compared is 2.5 %. A series of working standards prepared at 5 % intervals and used for sample comparisons thus enables this 2.5 % limit.

8.1.3 Prepare a working standard, and standardize against an oxide blend of CRM standards that is within 0.02 % of the value of the working standard.

8.2 *Sample Preparation:*

8.2.1 Attach tubes containing the appropriate working standard, S , and the sample, X , to the spectrometer inlet system, and prepare the materials for introduction into the ion source, as follows:

8.2.1.1 If adequate sample and working standard are available, open all valves between the sample and working standard containers and the pumping system, except the valves on the sample and working standard containers. If the amount of sample or working standard is limited, proceed to 8.2.2.

8.2.1.2 Open the valve on the sample container, and then close it quickly to vent gases to the pumping system.

8.2.1.3 After the pumping system has evacuated the vented gases, repeat the steps given in 8.2.1.2 a second time.

8.2.1.4 Repeat the steps given in 8.2.1.2 and 8.2.1.3 for the working standard.

8.2.2 Use the following alternative method of sample purification if the amount of the sample or working standard is limited:

8.2.2.1 Operate the appropriate valves to remove air entrapped in the connectors and to determine that there are no leaks into the inlet system.

8.2.2.2 Freeze the UF_6 by immersing the container in a