



Designation: C1842 – 16

Standard Test Method for The Analysis of Boron and Silicon in Uranium Hexfluoride via Fourier-Transform Infrared (FTIR) Spectroscopy¹

This standard is issued under the fixed designation C1842; 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 suitable for determining boron and silicon impurities as BF_3 and SiF_4 in uranium hexafluoride. This test method is an alternative to those described in Test Methods C761 and C1771.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 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

C859 Terminology Relating to Nuclear Materials

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

C1052 Practice for Bulk Sampling of Liquid Uranium Hexafluoride

C1703 Practice for Sampling of Gaseous Uranium Hexafluoride

C1771 Test Method for Determination of Boron, Silicon, and Technetium in Hydrolyzed Uranium Hexafluoride by Inductively Coupled Plasma—Mass Spectrometer After Removal of Uranium by Solid Phase Extraction

2.2 *Other Documents:*

ANSI N14.1 Nuclear Materials – Uranium Hexfluoride – Packaging for Transport³

ISO 7195 Nuclear Energy – Packaging of Uranium Hexafluoride (UF_6) for Transport⁴

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminology C859.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *detection limit, n*—based on the minimum absorbance obtainable at a given pressure to yield a meaningful result. In accordance with Terminology C859, a low concentration level could be achieved with these methods.

3.2.2 *FTIR, n*—Fourier-transform infrared spectroscopy.

3.2.3 *K, n*—infrared absorbance constant in pressure units [1/Pa], $K = \text{OD/Pressure}$.

3.2.4 *“IS” container, n*—a nickel or Monel container as described in ANSI N14.1.

4. Summary of Test Method

4.1 To perform the Fourier-Transform Infrared (FTIR) spectroscopic analysis of boron and silicon impurities in uranium hexafluoride, a sample must be collected in a “IS” container or equivalent with the methods described in Practices C1052 or C1703.

4.2 The bottle is kept at room temperature. The manifold and the sample cell are maintained at 50°C. In these conditions, UF_6 is mainly in solid phase in the bottle and boron and silicon are present in the gaseous phase of manifold. In this medium, the boron and silicon chemical forms are respectively BF_3 and SiF_4 .

4.3 The test method is based on the analysis in the gas phase. The gas phase is analyzed at 50°C by FTIR spectrometry to determine the B and Si concentration in uranium hexafluoride.

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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

4.4 The manifold and sample cell are filled at the vapor pressure of UF₆ at room temperature (near 12 kPa).

4.5 After a screening, if the spectrum is the UF₆ spectrum, this test method can be used to check the compliance of UF₆ as specified in Specifications C787 and C996.

4.6 The boron and silicon determinations are done on the gaseous phase. The concentration and the limits of detection are in units of µg/g U.

4.7 There are no spectral interferences from uranium hexafluoride's infrared absorbances.

5. Significance and Use

5.1 This test method utilizes FTIR spectroscopy to determine the boron and silicon concentration in uranium hexafluoride.

5.2 These detection limits are low and very effective to check the compliance of UF₆ with Specifications C787 and C996.

6. Hazards

6.1 Uranium hexafluoride is a hazardous material. It is a highly reactive and toxic substance in addition to its radioactive properties. It must be handled as a gas in containers and manifolds using materials of construction that are inert to fluorine-bearing gases, such as nickel, Monel™, copper, or aluminum.

7. Apparatus

7.1 *Fourier-Transform Infrared Spectrophotometer*, with a resolution of ±0.5 cm⁻¹ or better. The scanning range depends on the equipment being used, but at minimum shall be 600 to 1550 cm⁻¹.

7.2 *A Manifold System*, built with materials of construction inert to fluorine-bearing gases. The manifold system shall be conditioned and passivated with an appropriate fluorinating agent.

7.3 *A Sample Cell*, windows are made of material(s) inert to fluorine-bearing gases, for example, zinc selenide (ZnSe). A cell path length of more than 150 mm was found to be sufficient for the required LOD. The cell is heated at 50°C.

7.4 *A Pressure Gauge*, which can be read to 1 Pa is necessary.

7.5 *Absorbance Data*, or OD optical density, can be determined to 0.001 units.

8. Calibration and Standardization

8.1 Calibration:

8.1.1 BF₃ and SiF₄ are calibrated between 15 and 150 Pa. The cell temperature is maintained at 50°C. Different pressures of pure BF₃ or pure SiF₄ are introduced between 15 and 150 Pa. The maximum of absorbance and the scans are recorded. The response of absorbance as a function of pressure is linear. The slope of this line is K. The slope is constant from near zero absorbance to about 0.8 absorbance units.

8.1.2 The K value are measured at 1441 cm⁻¹ for BF₃ and 1029 cm⁻¹ for SiF₄ (see Fig. 1 and Fig. 2).

8.1.3 The operating experience of each laboratory for precision calculations of impurities are critical to the success of the method. Each laboratory shall determine the “K” values specific to its instrumentation: $K = \Sigma OD / \Sigma \text{Pressure}$.

8.2 *Calibration of Pressure Gauge and FTIR Instrument*—Pressure gauges and the FTIR instruments are very stable over time. Annual calibration is recommended.

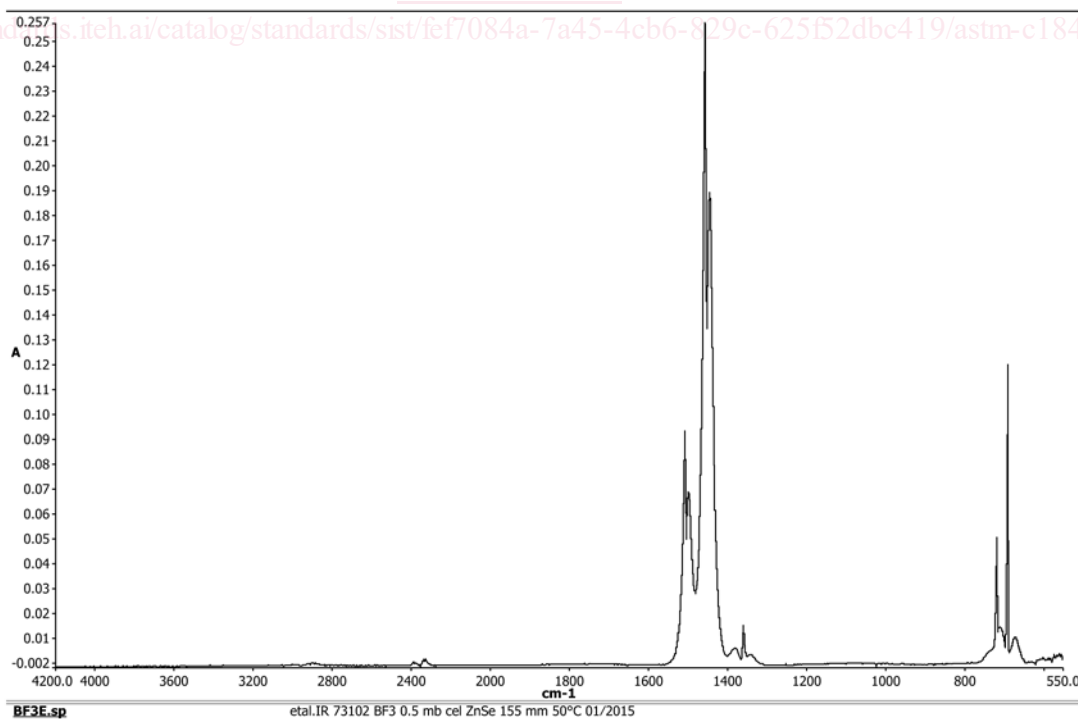


FIG. 1 BF₃ Spectrum in Pure Medium

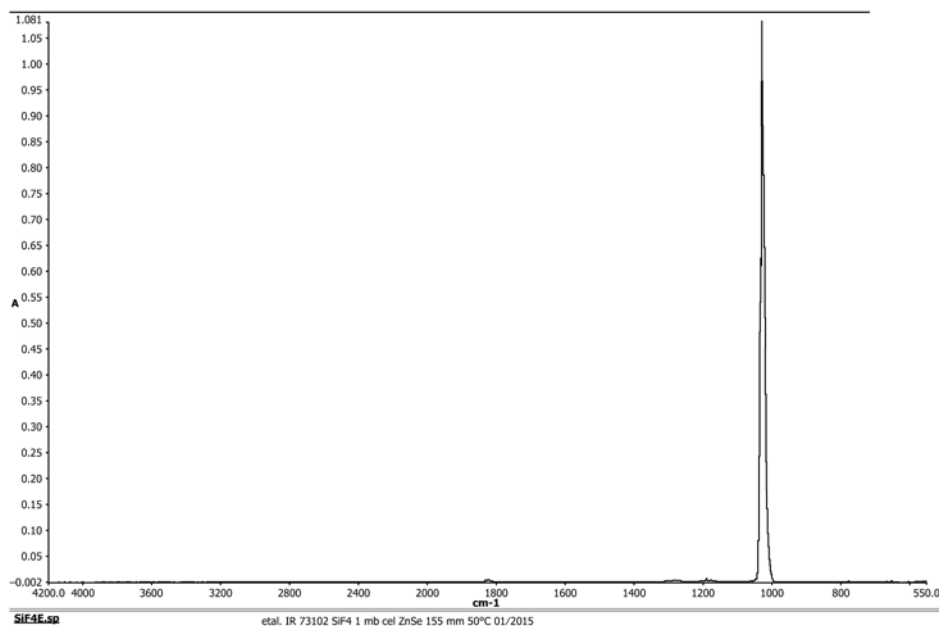


FIG. 2 SiF₄ Spectrum in Pure Medium

8.3 *Calibration Checks*—The calibration of the gauge should be checked before analyzing a UF₆ standard. After the check of the gauge calibration, 10 kPa of UF₆ standard are introduced and the maximum of absorbance at 625 cm⁻¹ is recorded. These calibration checks should be performed each day that the instrument is used. If the difference on the OD is above 1 %, the pressure gauge should be recalibrated. If the wavelength difference is greater than 0.5 cm⁻¹, then the FTIR instrument should be recalibrated

9. Procedure

9.1 *Acquire a Sample Scan:*

9.1.1 Weigh the empty bottle (M1).

9.1.2 Withdraw a sample in a 1S bottle with the process defined in Practices C1052 or C1703.

9.1.3 Weigh the bottle to determine the UF₆ mass in the bottle (M2).

9.1.4 Evacuate manifold system until readout on gauge displays a value of less than 10 Pa.

9.1.5 Verify the digital manometer for zero and full scale readings.

9.1.6 Obtain an infrared background spectrum on the FTIR to check that the manifold is clean.

9.1.7 Connect the sample 1S bottle on the manifold and control the tightness.

9.1.8 Open the bottle valve on the manifold. Wait until the pressure is stabilized and close the valve. Record the sample pressure (P).

9.1.9 Obtain the infrared spectrum. The spectrum will be the result of ten scans.

9.2 *Interpret Spectrum:*

9.2.1 Record the absorbance maxima (OD BF₃ and OD SiF₄) and the infrared spectrum (see Fig. 3).

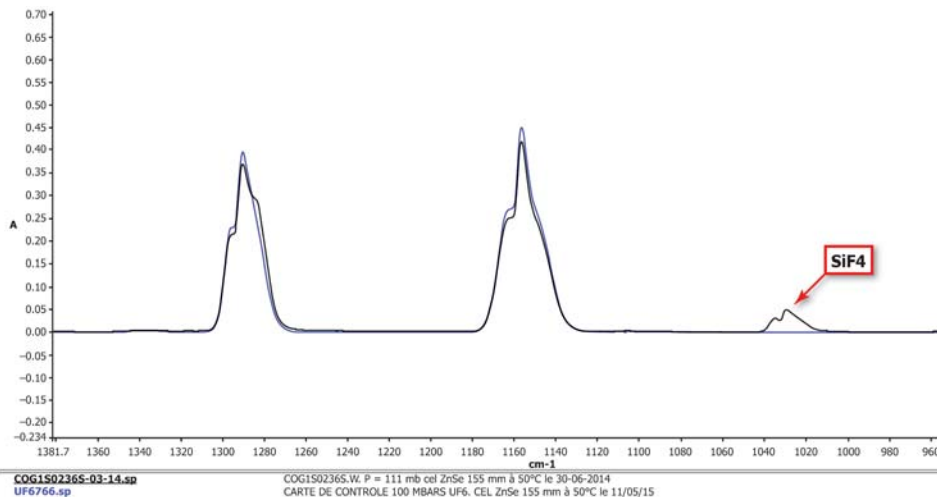


FIG. 3 SiF₄ Spectrum in UF₆ Medium