Designation: C1441 – 13

Standard Test Method for The Analysis of Refrigerant 114, Plus Other Carbon-Containing and Fluorine-Containing Compounds in Uranium Hexafluoride via Fourier-Transform Infrared (FTIR) Spectroscopy[1]

This standard is issued under the fixed designation C1441; 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 determining the concentrations of refrigerant-114, some other carbon-containing and fluorine-containing compounds, hydrocarbons, and partially or completely substituted halohydrocarbons that may be impurities in uranium hexafluoride when looked for specifically. The two options are outlined for this test method. They are designated as Part A and Part B.

1.1.1 To provide instructions for performing Fourier-Transform Infrared (FTIR) spectroscopic analysis for the possible presence of Refrigerant-114 impurity in a gaseous sample of uranium hexafluoride, collected in a "2S" container or equivalent at room temperature. The all gas procedure applies to the analysis of possible Refrigerant-114 impurity in uranium hexafluoride, and to the gas manifold system used for FTIR applications. The pressure and temperatures must be controlled to maintain a gaseous sample. The concentration units are in mole percent. This is Part A.

1.2 The method discribed in part B is more efficient because there isn't matrix effect. FTIR spectroscopy identifies bonds as C-H, C-F, C-Cl. To quantify HCH compounds, these compounds must be known and the standards available to do the calibration.

After a screening, if the spectrum is the UF_6 spectrum or if the other absorption peaks allow the HCH quantification, this test method can be used to check the compliance of UF_6 as specified in Specifications C787 and C996. The limits of detection are in units of mole percent concentration.

1.3 Part A pertains to Sections 7 – 10and Part B pertains to Sections 12 – 16.

1.4 These test options are applicable to the determination of hydrocarbons, chlorocarbons, and partially or completely substituted halohydrocarbons contained as impurities in uranium hexafluoride (UF_6). Gases such as carbon tetrafluoride (CF_4), which absorb infrared radiation in a region where uranium hexafluoride also absorbs infrared radiation, cannot be analyzed in low concentration via these methods due to spectral overlap/interference.

1.5 These test options are quantitative and applicable in the concentration ranges from 0.003 to 0.100 mole percent, depending on the analyte.

1.6 These test methods can also be used for the determination of non-metallic fluorides such as silicon tetrafluoride (SiF_4), phosphorus pentafluoride (PF_5), boron trifluoride (BF_3), and hydrofluoric acid (HF), plus metal-containing fluorides such as molybdenum hexafluoride (MoF_6). The availability of high quality standards for these gases is necessary for quantitative analysis.

1.7 These methods can be extended to other carbon-containing and inorganic gases as long as:

1.7.1 There are not any spectral interferences from uranium hexafluoride's infrared absorbances.

1.7.2 There shall be a known calibration or known "K" (value[s]) for these other gases.

1.8 The values stated in SI units are to be regarded as the standard.

1.9 *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

[1] This test method is under the jurisdiction of ASTM Committee C26 on Methods of Test.

Current edition approved April 1, 2013. Published July 2013 Originally approved in 1999. Last previous edition approved in 2004 as C1441–04, which was withdrawn in January 2013 and reinstated in April 2013. DOI: 10.1520/C1441-13.

[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.

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 % ^{235}U
C1052 Practice for Bulk Sampling of Liquid Uranium Hexafluoride
2.2 *USEC Document*
USEC-651 Uranium Hexafluoride: A Manual of Good Handling Practices[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *detection limit, n*—based on the minimum absorbance obtainable at a given pressure to yield a meaningful result in accordance with Eq 2. In accordance with Terminology C859, a low concentration level that can be achieved with these methods is 0.003 mol percent at the 95 % confidence level.

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

3.1.3 *K, n*—infrared absorbance constant in pressure units, where:

$$K = \frac{mole\ percent\ concentration\ standard\ (pressure)}{absorbance} \tag{1}$$

3.1.4 *"2S" container, n*—a nickel container with a 1.0 L capacity.

4. Summary of Test Methods

4.1 Part A is based on the collection of an all gas sample of UF_6. The gas sample is then analyzed at room temperature via FTIR to determine the percent Refrigerant-114 in uranium hexafluoride.

4.2 Part B is based on the collection of an all gas sample of UF_6. There are two differences with the Part A:

—the calibration is performed in UF_6.

—the path length used is 5 meters equipped with zinc selenide (ZnSe) optics.

4.3 In Parts A and B, the pressure is kept low enough so that the manifold and sample cell are filled only with gaseous UF_6.

5. Significance and Use

5.1 This test method (Part A) utilizes FTIR spectroscopy to determine the percent Refrigerant-114 impurity in uranium hexafluoride. Refrigerant-114 is an example of an impurity gas in uranium hexafluoride.

6. Hazards

6.1 Uranium hexafluoride is considered to be 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 nickel containers and well-conditioned nickel manifolds to ensure safety. Suitable handling procedures are described in USEC-651.

7. Apparatus (Part A)

7.1 *Fourier-Transform Infrared Spectrophotometer,* or dispersive infrared spectrophotometer set up to collect data in the range 4000 to 400 cm^{-1} with ± 2 cm^{-1} resolution or better.

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. (See Annex A2.)

7.3 *A Nickel Sample Cell* equipped with silver chloride windows. The pathlength used in these experiments is 10 cm (0.1m).

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

7.5 *Absorbance Data,* can be determined to 0.001 units.

8. Calibration (Part A)

8.1 The infrared spectrophotometer is calibration checked daily with a traceable standard of Refrigerant-114. The response of the instrument and the sensitivity of the pressure manometers can be evaluated based on the mole percent concentration Refrigerant-114 calculated. See Table 1 for absorbance maxima and corresponding "K" values.

8.2 The operating experience of each laboratory for precision calculations of the mole percent concentrations of uranium hexafluoride and impurities are critical to the success of the method. Total pressure should be maintained at 100 mm HgA (13.3 kPa) or less. Each laboratory shall determine the "K" values specific to its instrumentation.

8.3 The "K" values used for calibrations are good well beyond the 60 to 75 mm HgA (8 to 10 kPa) in a typical all gas sample.

8.4 The "K" values require that the mole percent concentration of a traceable standard, pressure, and absorbance of a pure gas are known. 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.

9. Procedure (Part A)

9.1 *Collecting the Sample*—An all gas sample is collected from the apparatus described in Test Method C761. See Annex A1 or Fig. 1 in Test Method C761. The isotope abundance sample tube is replaced by a "2S" container. The valve on the inverted liquid uranium hexafluoride container is closed when the pressure on the manometer reads 75 mm HgA (10 kPa). A

TABLE 1 Typical Infrared Active Gas Molecules, Their Approximate Infrared Frequencies in cm^{-1}, and Their Infrared Absorbance Constants (K) in mm Part A, Determined at Room Temperature (25°C=77°F=298K)

Infrared Active Gas Molecule	Approximate Infrared Frequency in cm^{-1}	K in kPa
Uranium Hexafluoride = UF_6	625	11
Uranium Hexafluoride = UF_6	676	1.6
Refrigerant-114 = $C_2F_4Cl_2$	922	93.2
Refrigerant-114 = $C_2F_4Cl_2$	1052	70.1
Refrigerant-114 = $C_2F_4Cl_2$	1185	48.4
Refrigerant-114a = $C_2F_4Cl_2$	1231	32.1

[3] Available from USEC Inc., 6903 Rockledge Drive, Bethesda, MD 20817.