



Designation: C 1441 – 99

## 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<sup>1</sup>

This standard is issued under the fixed designation C 1441; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers determining the concentrations of refrigerant-114, other carbon-containing and fluorine-containing compounds, hydrocarbons, and partially or completely substituted halohydrocarbons that may be impurities in uranium hexafluoride. 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 Part B involves a high pressure liquid sample of uranium hexafluoride. This method can be applied to the limits of detection for hydrocarbons, chlorocarbons, and partially or completely substituted halohydrocarbons as specified in Method C 996. The limits of detection are in units of mole percent concentration.

1.3 Part A pertains to Sections 7-10 and 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 ( $\text{UF}_6$ ). Gases such as carbon tetrafluoride ( $\text{CF}_4$ ), which absorb infrared radiation in a region where uranium hexafluoride also absorbs infrared radiation, cannot be analyzed via these methods due to spectral overlap/interference.

1.5 These test options are quantitative and applicable in the concentration ranges from 0.0001 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 ( $\text{SiF}_4$ ), phosphorus pentafluoride ( $\text{PF}_5$ ), boron trifluoride ( $\text{BF}_3$ ), and hydrofluoric acid (HF), plus metal-containing fluorides such as molybdenum hexafluoride ( $\text{MoF}_6$ ). 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:

C 761 Test Methods for Chemical, Mass Spectrometric, Nuclear and Radiochemical Analysis of  $\text{UF}_6$ <sup>2</sup>

C 787 Specification for Uranium Hexafluoride for Enrichment<sup>2</sup>

C 859 Terminology Relating to Nuclear Materials<sup>2</sup>

C 996 Specification for Uranium Hexafluoride enriched to Less Than 5 % <sup>235</sup>U<sup>2</sup>

C 1052 Practice for Bulk Sampling of Liquid Uranium Hexafluoride<sup>2</sup>

#### 2.2 USEC Document

USEC-651 Uranium Hexafluoride: A Manual of Good Handling Practices<sup>3</sup>

#### 2.3 ANSI Standard:

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C 26.05 on Methods of Test.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 12.01.

<sup>3</sup> Available from USEC Inc., 6903 Rockledge Drive, Bethesda, MD 20817.

ANSI N14-1 Uranium Hexafluoride: Packaging for Transport<sup>4</sup>

2.4 ISO Standard:

ISO/DIS 7195 Packaging for Uranium Hexafluoride (UF<sub>6</sub>) for Transport<sup>4</sup>

### 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 C 859, a low concentration level that can be achieved with these methods is 0.01 mole 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:

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

3.1.4 *mm HgA, n*—millimetres mercury absolute.

3.1.5 “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<sub>6</sub>. 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 a liquid phase UF<sub>6</sub> sample. The sample is collected through a manifold and is introduced as a gas into a 60°C (140°F) thermostated infrared gas cell.

4.3 In order to achieve a representative sample in UF<sub>6</sub>, the analyzed sampling containers must be filled to more than 1/3 of capacity with UF<sub>6</sub>. The reason for this is to minimize the effect of the gas phase contribution. (This is for Part B.)

4.4 In Part A, the pressure is kept low enough so that the manifold and sample cell are filled only with gaseous UF<sub>6</sub>; whereas, in Part B segregation in gas-liquid phases may require, for a complete analysis, two determinations. The heated manifold and cell support higher sample pressures resulting in increased sensitivity. Both options meet the criteria set forth in Specification C 996.

### 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. The detection of hydrocarbons, chlorocarbons, and partially or completely substituted halohydrocarbons in UF<sub>6</sub> (Part B) is governed by the provisions in Specification C 996.

### 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<sup>-1</sup> with ± 2 cm<sup>-1</sup> 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 0.01 mm HgA is necessary.

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

7.6 *Windows*, are made of material(s) inert to fluorine-bearing gases, for example, silver chloride (AgCl).

### 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.

**TABLE 1 Typical Infrared Active Gas Molecules, Their Approximate Infrared Frequencies in cm<sup>-1</sup>, 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 <sup>-1</sup>	K in mm
Uranium Hexafluoride = UF <sub>6</sub>	625	82.5
Uranium Hexafluoride = UF <sub>6</sub>	676	11,950
Refrigerant-114 = C <sub>2</sub> F <sub>4</sub> Cl <sub>2</sub>	922	699
Refrigerant-114 = C <sub>2</sub> F <sub>4</sub> Cl <sub>2</sub>	1052	526
Refrigerant-114 = C <sub>2</sub> F <sub>4</sub> Cl <sub>2</sub>	1185	363
Refrigerant-114a = C <sub>2</sub> F <sub>4</sub> Cl <sub>2</sub>	1231	241

<sup>4</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

## 9. Procedure (Part A)

9.1 *Collecting the Sample*—An all gas sample is collected from the apparatus described in Test Method C 761. See Annex A1 or Fig. 1 in Test Method C 761. 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 total of three samples are obtained in this manner. If three sample containers (“2S” or equivalent) are not available, three gas charges from one sample can be substituted. However, if the full pressure in the sample container is less than 50 mm HgA (6.7 kPa), the three gas charges from one sample option is not recommended.

NOTE 1—The manifold system must be conditioned and passivated with an appropriate fluorinating agent to generate high quality analytical results.

### 9.2 Acquire Background Scan (Refer to Annex A2):

NOTE 2—The vacuum manometer Valve C must be open in order for pressure in mm to be read.

9.2.1 Ensure that the cold trap inlet valve (L) and crossover valves (MX1 and MX2) are closed.

9.2.2 Ensure that the chem trap outlet (R), chem trap inlet (Y), sample cell inlet (A), vacuum pump inlet (P), and sample port (S1, S2, or S3) valves are open.

9.2.3 Ensure that all other valves other than Valve C are closed.

9.2.4 Evacuate manifold system until readout on thermocouple gage (T2) displays a value of less than 10  $\mu\text{m}$ .

9.2.5 Verify the digital manometer for zero and full scale readings, if not adjust accordingly.

9.2.6 Obtain an infrared background spectrum on the FTIR.

### 9.3 Acquire Initial Sample Scan:

9.3.1 Close chem trap inlet valve (Y).

9.3.2 Open the sample container valve and charge the manifold with the full contents of the sample container.

NOTE 3—If the total pressure of the sample is in excess of 100 mm HgA, a resample is desirable.

9.3.3 Close the sample container valve.

9.3.4 Obtain the infrared spectrum of the gases in the sample charge.

### 9.4 Interpret Spectrum:

9.4.1 Record the absorbance maxima for the three Refrigerant-114 bands cited in Table 1, if any are present. The absorbance maximum at 1052  $\text{cm}^{-1}$  typically experiences the least amount of overlap.

9.4.2 Record the absorbance maximum for Refrigerant-114a from Table 1, if any is present.

9.4.3 Record the absorbance maximum for uranium hexafluoride at 676  $\text{cm}^{-1}$ .

9.4.4 Record the pressure (in mm) from the readout of the digital manometer (C). (If the pressure exceeds 100 mm HgA resampling is necessary due to the possibility of freeze-out of the  $\text{UF}_6$ .)

9.4.5 Monitor the absorbance of uranium hexafluoride at 625  $\text{cm}^{-1}$  of the full pressure gas charge.

9.4.5.1 If the absorbance at full pressure exceeds 0.8 units partial evacuation of the manifold is necessary in accordance with the action steps in 9.5.

9.4.5.2 If the absorbance at full pressure is less than 0.8 units, a resample is desirable.

### 9.5 Partial Evacuation of the Manifold System:

9.5.1 Close the chem trap outlet valve (R).

9.5.2 Open the chem trap inlet valve (Y).

9.5.3 Close the chem trap inlet valve (Y) when the pressure on the digital manometer is no longer decreasing.

9.5.4 Allow a minimum of 30 s residency time in the chem trap (E).

9.5.5 Open the chem trap outlet valve (R) to vent any remaining gases to the always energized vacuum pump (W).

9.5.6 Close the chem trap outlet valve (R) when the readout on the thermocouple gage (T2) is less than 10  $\mu\text{m}$ .

9.5.7 Repeat step 9.5.1-9.5.6, until the pressure on the digital manometer reads 1.00 mm HgA.

9.6 *Scanning the Sample for Uranium Hexafluoride at 625  $\text{cm}^{-1}$ :*

9.6.1 Scan the sample for uranium hexafluoride at a pressure that results in an infrared peak less than 0.80 absorbance units.

9.6.2 Record the magnitude of the absorbance maximum for the uranium hexafluoride peak at 625  $\text{cm}^{-1}$ .

9.6.3 Record the pressure (in mm) from the readout of the digital manometer for the uranium hexafluoride peak at 625  $\text{cm}^{-1}$ .

NOTE 4—If the pressure required to obtain an absorbance less than 0.8 units at 625  $\text{cm}^{-1}$  is less than 0.40 mm HgA, the values obtained at 676  $\text{cm}^{-1}$  are likely to be more reliable.

### 9.7 Total Evacuation of the Manifold System:

9.7.1 Repeat the action steps in 9.5 until the pressure on the digital manometer reads 0.20 mm HgA or less.

9.7.2 Open the cold trap inlet valve (L) and at least one of the crossover valves (MX1 or MX2).

9.7.3 Continue the total evacuation until the thermocouple gauge (T2) reads below 10  $\mu\text{m}$  and the digital manometer reads 0.00 mm HgA.

9.7.4 Rezero the digital manometer if the readout stabilizes for 2 min at a reading other than 0.00 mm HgA.

### 9.8 Replicate Experiments:

9.8.1 Proceed to Section 10 if the three gas changes from one sample was used in 9.1.

9.8.2 Repeat action steps 9.3-9.7.4 twice more, using a fresh replicate sample from the three “2S” containers received.

## 10. Calculations of Mole Percent Concentrations (Part A)

10.1 Calculate the average mole percent concentrations of Refrigerant-114 and Refrigerant-114a based on their respective absorbances, the “K” values, and the total pressure in the manifold as indicated in Eq 2:

NOTE 5—If the uranium hexafluoride concentration is high, based on the data obtained from the measurements at 625  $\text{cm}^{-1}$ , the uranium hexafluoride band at 1157  $\text{cm}^{-1}$  may interfere with the Refrigerant-114 band at 1185  $\text{cm}^{-1}$ . The Refrigerant-114 concentration may be biased high should this result be included with the data obtained at 922  $\text{cm}^{-1}$  and 1052  $\text{cm}^{-1}$ .

$$\text{mole percent concentration} = \frac{(\text{absorbance}) (K)}{\text{total pressure}} \quad (2)$$

10.2 Calculate mole percent concentration for uranium hexafluoride based on the absorbance at 625 or 676 cm<sup>-1</sup>, the appropriate “K” value, and the total pressure in the manifold as indicated in Eq 2.

10.3 Determination of the mean mole percent concentrations of Refrigerant-114 and UF<sub>6</sub> plus the percent concentration Refrigerant-114 in UF<sub>6</sub>.

10.3.1 Calculate the mean mole percent concentrations of both Refrigerant-114 and Refrigerant-114a in accordance with 10.1 if any is present, using each of the indicated absorbance frequencies listed in Table 1. This result is based on the three gas charges from one sample or three replicate samples.

10.3.2 Sum the mean mole percent concentrations of Refrigerant-114 and Refrigerant-114A and record as total Refrigerant-114.

10.3.3 Calculate the mean mole percent concentration of uranium hexafluoride in accordance with 10.2. This result is based on the three gas charges from one sample or three replicate samples.

10.3.4 Calculate the percent concentration of total Refrigerant-114 in uranium hexafluoride in accordance with Eq 3:

$$\begin{aligned} \text{Percent total REFRIGERANT-114 in UF}_6 \\ = \left( \frac{\text{mole \% total REFRIGERANT-114}}{\text{mole \% UF}_6} \right) * 100 \end{aligned} \quad (3)$$

NOTE 6—*Sample Result Criteria*—If the mole percent concentration of uranium hexafluoride in Part A is less than 50 %, a resample is desirable.

## 11. Precision and Bias (Part A)

11.1 Each sample was analyzed a minimum of three times to ensure precision. A grand mean containing a minimum of nine points (the three absorbance bands for Refrigerant-114 from the three replicate samples) for Refrigerant-114 and three points for the fundamental absorbance for Refrigerant-114a were used. Under ambient conditions, a gas charge of a known pressure to a leak-tight manifold, will yield absorbance maxima at the reference wavenumber frequencies that are both repeatable and reproducible.

11.2 A standard gas mixture of Refrigerant-114 in nitrogen with NIST certification or equivalent, is analyzed each shift to ensure that the gas handling system has been properly calibrated.

11.3 Five standards of Refrigerant-114 in nitrogen were purchased from a commercial source.<sup>5</sup> The NIST traceable standards were: 50.0 ppm (0.00500 mole percent), 100. ppm (0.0100 mole percent), 150. ppm (0.0150 mole percent), 200. ppm (0.0200 mole percent), and 500. ppm (0.0500 mole percent); respectively. In addition, a blank gas containing only nitrogen and two “unknown” mixtures of Refrigerant-114 in nitrogen were also analyzed. A round robin schedule of the five standards, the blank, and the two “unknowns” was established. The eight gas samples were analyzed six times each for a grand total of 48 experiments. These results are summarized in Table 2.

11.4 The repeatability standard deviation is a measure of the precision of the results. The repeatability standard deviation for these values is 0.004 %.

11.5 The bias is based on the difference between the mean of many points (here: *n* = 48) and an accepted reference value. Standard gas mixtures of Refrigerant-114 in nitrogen with NIST certification or equivalent are suitable for establishing the bias of the method. It is important to note that “exaltation” phenomena are not significant in an all gas sample.

11.6 The percent bias for these values is 7 %. The results are statistically significant at the 95 % confidence level.

## 12. Apparatus (Part B)

### 12.1 Infrared Spectrophotometer:

12.1.1 While the apparatus is drained, the background spectrum of the infrared system is measured. The optical density or absorbance in the 1430 to 700 cm<sup>-1</sup> range must not exceed 0.0005.

<sup>5</sup> The sole source of supply of the apparatus known to the committee at this time is Scott Specialty Gas Co. Troy Michigan. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

**TABLE 2 Approximate True Refrigerant-114 Concentration (ppm)<sup>A</sup>**

Note—St. Dev. = Classical standard deviation

Bias = Mean - True value.

% RSD = (Std. Dev./Mean) \* 100 % (RSD = Relative Standard Deviation)

% Bias = [(Mean - True Value)/True Value]\*100 %.

	% ppm	50.0 ppm	100 ppm	150 ppm	200 ppm	Unknown No. 1	Unknown No. 2	500 ppm
Values	0	0.00563	0.00977	0.0191	0.0225	0.0262	0.0320	0.0562
	0	0.00780	0.00112	0.0201	0.0196	0.0155	0.0205	0.0492
	0	0.00400	0.0137	0.0229	0.0253	0.0271	0.0225	0.0632
	0	0.00480	0.0156	0.0136	0.0189	0.0298	0.0343	0.0485
	0	0.00480	0.0144	0.0133	0.0176	0.0274	0.0395	0.0512
	0	0.00380	0.00527	0.0186	0.0226	0.0348	0.0154	0.0459
Mean	0.0	0.00514	0.01166	0.019	0.0211	0.0268	0.0274	0.0524
St. Dev.	0.0	0.00146	0.00379	0.00379	0.00286	0.00634.	0.00926	0.00632
Bias	0.0	0.00014	0.00166	0.00294	0.00108	<sup>B</sup>	<sup>B</sup>	0.00237
% RSD	0 %	28.4 %	32.5 %	21.1 %	13.6 %	23.7 %	33.8 %	12.1 %
% Bias	0 %	2.8 %	16.6 %	19.6 %	5.4 %	<sup>B</sup>	<sup>B</sup>	4.7 %

<sup>A</sup>Data collected under the experimental conditions defined in Section 9.

<sup>B</sup>These are unknown production samples; bias cannot be determined on them.

### 12.2 Liquefaction and Filling Manifold:

12.2.1 The infrared spectrometer may be connected either:

12.2.1.1 Directly into a uranium enrichment device, and the withdrawal point (for cylinder filling), or

12.2.1.2 Onto a sub-sampling bench, or

12.2.1.3 Onto a specific manifold enabling connection of one or several vessels of types 1S, 2S, ANSI-14, or CEA 23 D.

12.2.2 In all cases, connection networks allow the following operations:

12.2.2.1 Evacuating the piping to less than 0.02 torr = 20  $\mu$ m.

12.2.2.2 UF<sub>6</sub> extracting, or through recycling, either through trapping and material collecting.

12.2.2.3 Cleaning all pipe networks after operating (for example, maintenance, replacement of defective parts).

12.2.2.4 Graduated volume or pipette for liquid UF<sub>6</sub> (see Fig. 1 in Practice C 1052).

12.2.2.5 Manometer with an accuracy of  $\pm 5$  mm HgA (torr)

12.2.2.6 Pressure gauge capable of measuring 0.01 mm HgA (torr).

### 12.3 Infrared Cell:

12.3.1 The optical path is 0.200 m = 20.0 cm = 200. mm

12.3.2 The infrared cell has two valves and is inert to fluorinated materials.

12.3.3 Windows are made of silver chloride (AgCl) and metal parts are constructed from either nickel or monel.

12.3.4 The infrared cell is permanently heated at 60°C (140°F)  $\pm 1^\circ$ C (1.8°F) within a thermostatted enclosure.

## 13. Calibration (Part B)

13.1 See Tables 3 and 4 for absorbance maxima, related optical density, and detection limit in mole percent for carbon-containing compounds and fluorine - containing gases; respectively.

13.2 Total pressure in the infrared cell shall be maintained between 1000 mm HgA (133 kPa) and 1300 mm HgA (173 kPa).

**TABLE 3 Part B: Carbon-Containing Compounds @ 60°C Limits of Detection in Mole Percent of Impurities in UF<sub>6</sub> Total Pressure = 800 mBar = 600 mm HgA = 80kPa**

Refrigerant Designation	Chemical Formula	Wavenumber in cm <sup>-1</sup>	Related Optical Density	Detection Limit in Mole Percent
R-11	CFCl <sub>3</sub>	1087	0.00094	0.006
R-12	CF <sub>2</sub> Cl <sub>2</sub>	1107	0.00090	0.004
R-13	CF <sub>3</sub> Cl	1215	0.00088	0.0002
R-13B	CF <sub>3</sub> Br	1208	0.0011	0.0001
R-113	CF <sub>2</sub> ClCFCl <sub>2</sub>	1121	0.00090	0.0005
R-114	CF <sub>2</sub> ClCF <sub>2</sub> Cl	1052	0.00083	0.0004
R-115	CF <sub>3</sub> CF <sub>2</sub> Cl	1242	0.0010	0.0002
R-142	CH <sub>3</sub> CF <sub>2</sub> Cl	885	0.00093	0.0007
R-21	CHFCl <sub>2</sub>	1087	0.00085	0.0006
R-23	CHF <sub>3</sub>	1379	0.00061	0.0010
R-318	CF <sub>3</sub> CF=CFCF <sub>3</sub> cis & trans	1198	0.00096	0.0002
RC-318	$\begin{array}{c} \text{CF}_2 \text{---} \text{CF}_2 \\   \qquad \qquad   \\ \text{CF}_2 \text{---} \text{CF}_2 \end{array}$	962	0.0012	0.0002
Total	...	...	...	0.0141

**TABLE 4 Part B: Fluorine-Containing Gases @ 60°C Limits of Detection in Mole Percent of Impurities in UF<sub>6</sub> Total Pressure = 800 mBar = 600 mm HgA = 80 kPa**

GAS	WAVENUMBER in cm <sup>-1</sup>	RELATED OPTICAL DENSITY	DETECTION LIMIT in MOLE PERCENT
SiF <sub>4</sub>	1028	0.02	0.0002
PF <sub>5</sub>	955	0.02	0.0007
BF <sub>3</sub>	1443	0.02	0.0008
MoF <sub>6</sub>	741	0.01	0.0001
HF	3976	0.02	0.0001

13.3 The apparatus is calibrated in pure gas with the same infrared cell as that used for the analysis at a temperature of 60°C.

13.3.1 Particular attention shall be given to “exaltation” (energy transfer) phenomena and interferences especially when several infrared active components are simultaneously present.

13.3.2 It is wise to check such effects through repeated analyses under the same conditions (identical temperature and pressure) as will be applied to the samples, plus on pure gas mixtures used for calibration (UF<sub>6</sub> and the chloro-fluoro-halo-carbons).

## 14. Procedure (Part B)

### 14.1 Collecting Sample:

14.1.1 Refer to Practice C 1052 and Test Method C 761.

14.1.2 In order to achieve a representative sample of UF<sub>6</sub> the analyzed sample cylinders must be filled with more than 1/3 full with UF<sub>6</sub>. The reason is to minimize the effect of the gas phase contribution.

14.2 Connect the sample cylinder with the manifold system at the relevant locations. The sample cylinder must be connected upside down, the valve in the lower position.

14.3 Evacuate and test the equipment to ensure vacuum integrity.

14.4 Heat the sample cylinder and equipment to 80  $\pm 5^\circ$ C and ensure homogeneity of samples in accordance with the procedure established for the equipment.

14.5 Perform a second vacuum integrity test. Acquire background infrared spectrum in the range 4000 to 400 cm<sup>-1</sup>.

14.6 Open the sample container valve and rinse the manifold system with UF<sub>6</sub> sample at 150  $\pm 7.5$  mm HgA (20  $\pm 1.0$  kPa) vapor pressure.

14.7 Close the sample cylinder valve and evacuate the manifold system into a UF<sub>6</sub> trap through a vacuum pump.

14.8 Open the sample container valve and charge the appropriate liquid phase UF<sub>6</sub> quantity into the graduated volume (pipette).

14.9 Close the sample container valve.

14.10 Release the UF<sub>6</sub> volume inside the equipment system and infrared cell. The pressure must be between 1000 and 1300 mm HgA (133 and 173 kPa).

14.11 Acquire sample infrared spectrum in the range 4000 to 400 cm<sup>-1</sup>.

14.12 Record the absorbance maxima for the carbon compounds and fluorine compounds cited in Tables 3 and 4, if any are present, after having deducted the background.

14.13 Evacuate the entire system into a UF<sub>6</sub> trap through a vacuum pump.

14.14 When some carbon compounds are detected in liquid phase UF<sub>6</sub>, it is recommended to perform a check-analysis of the gas phase of the same UF<sub>6</sub> sample for confirmation of the infrared spectrum.

**15. Calculation of Mole Percent Concentrations (Part B).**

15.1 Final results are reported in molar percent. The Lambert-Beer law is used to determine the partial pressure of carbon-containing or fluorine-containing products that may be present as impurities in UF<sub>6</sub> in accordance with Eq 4. Partial pressure is calculated per Eq 5.

$$C = \frac{\text{(partial pressure)}}{\text{(total pressure)}} \times 100 \% \tag{4}$$

where:

C = concentration in molar percent.

$$\text{partial pressure} = \frac{(nRT)}{V} \tag{5}$$

where:

n = amount in moles,

R = gas constant = 83.2 (bar × litre/(mole × K),

T = absolute temperature in K, and

V = volume in litres.

**16. Precision and Bias (Part B)**

16.1 *Detection Limit*—The detection limits are cited in the Tables 3 and 4 for some carbon-containing compounds plus gas molecules containing fluorine. Other carbon-containing compounds could be determined provided that a suitable calibration is performed. Impurities are identified through the position and intensities of their infrared absorbance bands expressed in wavenumbers (cm<sup>-1</sup>). The concentrations are expressed in mole percent. Optical densities or absorbances are included for information only and are valid for a specific instrument and the conditions used to obtain the spectrum. The detection limits cited in Tables 3 and 4 reflect the conditions set forth in Terminology C 859.

16.2 *Precision*—The precision data will be available on or before January 1, 2004.

16.3 *Bias*—The bias data will be available on or before January 1, 2004.

**17. Keywords**

17.1 carbon compounds; chlorocarbons; fluoride compounds; Fourier-transform infrared spectroscopy; halohydrocarbons; hydrocarbons; refrigerant-114; uranium hexafluoride

**ANNEXES**

**(Mandatory Information)**

**A1. ENSURE AN ALL GAS SAMPLE**

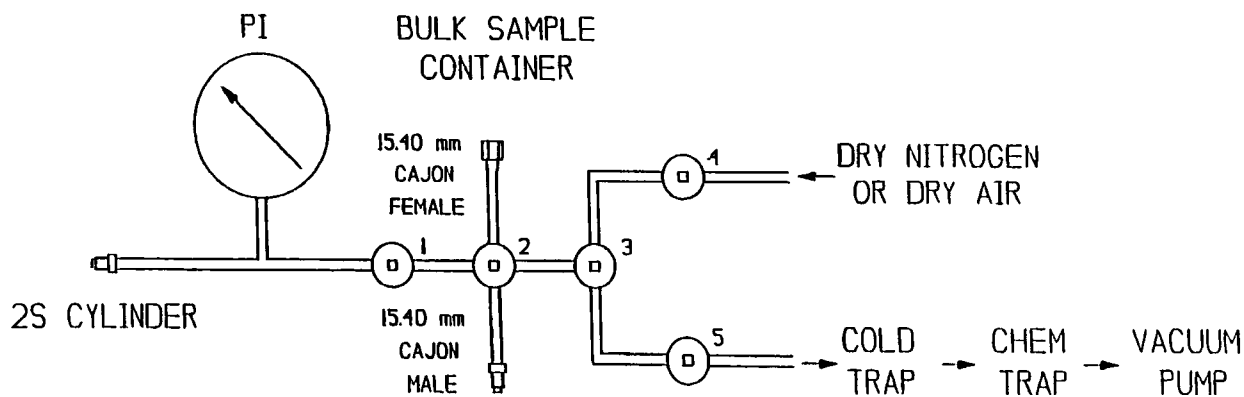
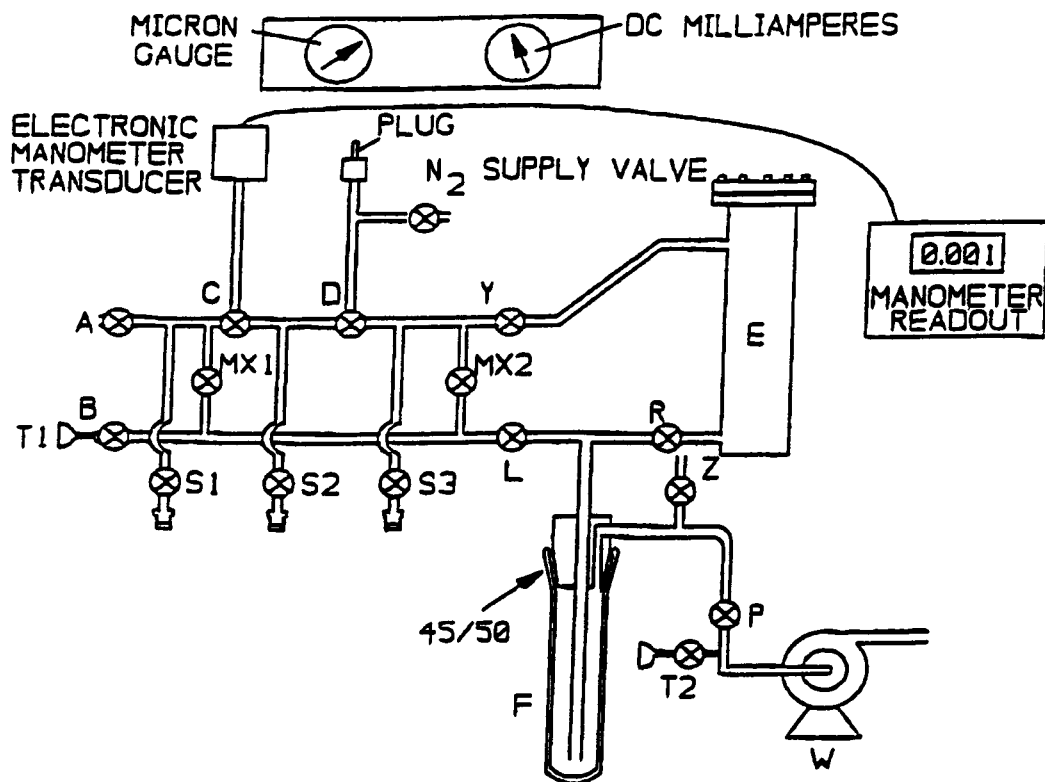


FIG. A1.1 Ensure an All Gas Sample

A2. DIAGRAM OF MANIFOLD SYSTEM IN HANDLING CORROSIVE GASES

Multi-Valve, Multi-Port Gas Manifold System For FTIR Applications



LEGEND

- |   |  |
|---|--|
| A-TO INFRARED CELL                                  | L-BOTTOM MANIFOLD COLD TRAP INLET VALVE      |
| B-T1 ISOLATION VALVE                                | R-CHEM TRAP OUTLET VALVE                     |
| C-TO MANOMETER TRANSDUCER                           | S1,2,3-SAMPLE HOSE VALVES                    |
| D-TO NITROGEN SUPPLY AND HF MANIFOLD                | P-VACUUM PUMP INLET VALVE                    |
| E-CHEM TRAP   | W-OIL VACUUM PUMP                            |
| ( 50/50 SODA LIME/ALUMINA                           | Y-TOP MANIFOLD CHEM TRAP INLET VALVE         |
| F-COLD TRAP (LIQUID NITROGEN IN CRYOSCOPIC VESSEL ) | Z-BLEED VALVE ( OPEN TO AIR, BREAKS VACUUM ) |
| MX1,2-MANIFOLD CROSSOVER VALVES                     |  |
| T1,T2-THERMOCOUPLE GAUGES                           |  |

FIG. A2.1 Diagram of Manifold System in Handling Corrosive Gases

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