Designation: D 2685 – 95 (Reapproved 1998)

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for
Air and Carbon Tetrafluoride in Sulfur Hexafluoride by Gas Chromatography [1]

This standard is issued under the fixed designation D 2685; 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 determination of air (Note 1) and carbon tetrafluoride as impurities in sulfur hexafluoride.

NOTE 1—Nitrogen, oxygen, or any of their mixtures is considered to be air. Commercial grade air or nitrogen is used for standardization.

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:*

D 2472 Specification for Sulfur Hexafluoride [2]

3. Summary of Test Method

3.1 Air and carbon tetrafluoride are separated physically by gas chromatography and compared to corresponding components separated under similar conditions from a reference standard mixture of known composition. The individual compounds of air are not separated. The composition of the sample is calculated from its chromatogram by comparing the area of the peak of each component with the area of the peak of the corresponding component on the reference standard chromatogram.

4. Significance and Use

4.1 Air and carbon tetrafluoride (CF_4) are two contaminants of interest in sulfur hexafluoride (SF_6). Both of these contaminants adversely affect the performance of SF_6 when used as an electrical insulating gas. Specification for maximum levels of these contaminants are given in Specification D 2472.

4.2 Gas chromatography is used to separate these contaminants from a sample of SF_6 and to determine their concentration.

5. Apparatus

5.1 *Gas Chromatograph,* consisting of a sample inlet system, adsorption column, flow meter, detector, and data handling system. Ensure that the column material of construction and sample components are compatible. The apparatus must completely separate air, carbon tetrafluoride, and sulfur hexafluoride as indicated by return of the recorded peak to the base line between each successive peak. Chromatograms must be reproducible so that successive runs of a reference standard agree on each component peak area or height within 5 %.

6. Reagents and Materials

6.1 *Cylinder of Helium Gas.*

6.2 *Reference Standard Mixture*—A gas mixture that contains known percentages of air and carbon tetrafluoride in helium or air and carbon tetrafluoride in sulfur hexafluoride is required. The concentration of a component in the reference sample should not be less than 50 % nor more than 300 % of the concentration of the corresponding component in the unknown.

7. Calibration and Standardization

7.1 *Apparatus Preparation*—Prepare the gas chromatograph for use as directed by the manufacturer. The following operating conditions have been found satisfactory. However, any combination of conditions that result in complete separations as indicated in the apparatus section will be satisfactory.

Carrier gas	helium, 40 to 50 mL/min
Column	Porapak Q-80/100 mesh or Porapak R 50/80 mesh
Column size	6 to 10 Ft (2–3.5 m) by ¼ in. (6.4 mm) nominal
Column temperature	40 to 50°C
Detector temperature	70 to 80°C
Sample volume	2 to 5 mL approximately
Attenuation	lowest which will keep peaks on a scale may be varied during run
Detector	thermal conductivity

7.2 *Example of Chromatography Conditions*—Specific conditions used by one laboratory which produces acceptable chromatograms are as follows:

Carrier gas	helium, 50 mL/min
Column packing	Porapak R 50/80 mesh
Column size and material	0.25 in. (6.4 mm) nominal ID, 10 ft (3.5 m) long, copper
Column temperature	48°C
Detector	thermal conductivity

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[1] This test method is under the jurisdiction of ASTM Committee D-27 Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.03 on Analytical Tests.

Current edition approved Sept. 10, 1995. Published November 1995. Originally published as D 2685 – 68. Last previous edition D 2685 – 94.

[2] *Annual Book of ASTM Standards*, Vol 10.03.

Detector temperature	75°C
Sample volume	5 mL

7.3 *Reference Standard Introduction and Separation*—Connect the reference standard cylinder to the needle valve and proceed in accordance with Section 8. Repeat the standardization in accordance with the laboratory's quality assurance procedures.

8. Procedure

8.1 *Sample Introduction and Separation*—Connect the sample cylinder to the inlet of the gas sampling valve with an intervening valve. Support the sample cylinder in an inverted position so the sample will be taken from the liquid phase and open the sample cylinder valve. Open the needle valve so that the vaporized sample purges the sampling valve at a rate of 20 to 100 mL/min. Inject a 2 to 5-mL sample of the gas into the column and record the chromatogram.

8.2 Under the conditions given in Section 7, the approximate elution times are air, 1.2 min; CF_4, 2 min; and SF_6, 4 min. Elution times will vary in accordance with column size and sample size.

8.3 Close the sample cylinder valve and then the needle valve and disconnect the sample cylinder.

9. Calculation

9.1 Determine the concentration of sample components by comparison of the areas or heights of the corresponding peaks. The calculations shown are for peak areas. Calculations using peak heights are analogous.

9.2 Measure the height and width at half height of each component peak in the standard sample chromatograms in millimetres, and calculate the average area of each component peak as follows:

NOTE 2—The area of the component peaks may be determined directly with an integrator, if available.

$$A = \Sigma(b \times h \times a)/n \tag{1}$$

where:

A = average area of each component peak,
b = peak width at half height,
h = peak height above base line.
a = attenuation,
n = number of peaks included in average, and
Σ = summation of the individual peak areas.

9.3 Calculate the concentration of each component in the sample as follows:

$$C = (S \times A_s)/A_r \tag{2}$$

where:

C = mole percent of each component in the sample
S = mole percent of the corresponding component in the reference standard (see Appendixes),
A_s = average area of the corresponding component peaks in the sample, and
A_r = average area of the corresponding component peaks in the reference standard.

9.4 The composition of the sample can be converted to a weight percent basis as follows (see Note 3):

$$\begin{aligned} CW_{\text{air}} &= 2898\ C_{\text{air}}/(28.98\ C_{\text{air}} + 88\ C_{CF_4} + 146\ C_{SF_6}) \\ CW_{CF_4} &= 8800\ C_{CF_4}/(28.98\ C_{\text{air}} + 88\ C_{CF_4} + 146\ C_{SF_6}) \\ CW_{SF_6} &= 100 - (CW_{\text{air}} + CW_{CF_4}) \end{aligned} \tag{3}$$

where:

CW_{air} = weight percent air,
CW_{CF_4} = weight percent carbon tetrafluoride, and
CW_{SF_6} = weight percent sulfur hexafluoride.

NOTE 3—If nitrogen is used instead of air, molecular weights should be adjusted accordingly.

10. Precision and Bias

10.1 *Repeatability*—Three measurements made in the same laboratory using a single reference sample should be considered suspect if the difference between the highest value and the lowest value exceeds 0.004 weight % (40 ppm by weight). See Appendix X1 for typical results from one laboratory.

10.2 *Reproducibility*—Reproducibility between laboratories has not been determined for this test method, as there are not a sufficient number of laboratories performing this test method.

10.3 *Bias*—Bias for this test method has not been determined, as there is not a sufficient number of laboratories performing this test method.

11. Keywords

11.1 air; carbon tetrafluoride; sulfur hexafluoride

APPENDIX

(Nonmandatory Information)

X1. SINGLE LABORATORY TYPICAL RESULTS

X1.1 On the basis of data taken during the method development of the liquid phase analysis of SF_6, a coefficient of variation of ±5 % relative to the mean values of the air and CF_4 concentrations may be achieved. This standard development is applicable to both electronic peak area and manual measurements of the peak areas as described in this test method.

X1.2 Table X1.1 shows the results of six runs of the same sample.

TABLE X1.1 Repeatability Data

	Air, ppm (wt)	CF_4, ppm (wt)
	101	255
	105	265
	101	255
	95	242
	99	251
	97	247
Mean	100	253
Standard Deviation	3.2	7.2
Coefficient of Variation	3.2 %	2.9 %