

SEMI C3.39-0304

STANDARD FOR NITROGEN TRIFLUORIDE (NF₃)

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1 Description

1.1 Nitrogen trifluoride is a toxic, odorless, colorless, nonflammable gas. It is a strong oxidizer and supports combustion.

2 Specifications

2.1 QUALITY: 99.98%

Impurities	Maximum Acceptable Level (ppm) (see NOTE 1)
Carbon Dioxide (CO ₂)	10
Carbon Monoxide (CO)	10
Carbon Tetrafluoride (CF ₄)	100
Nitrogen (N ₂)	10
Nitrous Oxide (N ₂ O)	10
Argon/Oxygen	10
Sulfur Hexafluoride (SF ₆)	10
Water (H ₂ O) (v/v)	1
TOTAL IMPURITIES	161 (see NOTE 1)

NOTE 1: An analysis of significant figures has not been considered. The number of significant figures will be based on analytical accuracy and the precision of the provided procedure.

3 Chemical Specification

Hydrolyzable Fluorides as HF	1 ppm
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4 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	71.00	71.00
Density of gas at 21.1°C (70°F) and 1 atm	2.94 kg/m ³	0.1838 lb./ft. ³

5 Analytical Procedures

5.1 *Carbon Tetrafluoride* — This procedure is for the determination of carbon tetrafluoride in nitrogen trifluoride using a gas chromatograph with a thermal conductivity detector. (See Figure 1 and Note 1.)

5.1.1 *Detection Limit* — 1.0 ppm.

5.1.2 *Instrument Parameters*

5.1.2.1 Column: Super Q (80/100 mesh), 4.9 m (16 ft.) by 1.75 mm (1/16 in.) ID, 3.2 mm (1/8 in.) OD ss or equivalent.

5.1.2.2 Carrier Flow: 19 mL/min helium.

5.1.2.3 Sample Volume: 0.1 mL.

5.1.2.4 Temperatures:

Detector	100°C
Oven	42°C
Gas sampling valve	42°C

5.1.2.5 Valve material: Nitronic-60 or equivalent.

5.1.3 *Calibration Standard* — 100 ppm carbon tetrafluoride, balance helium.

5.1.4 *Operating Procedure*

5.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record retention time and peak area.

5.1.4.2 Inject nitrogen trifluoride sample to be tested in the same manner as the calibration standard. Record the retention times and peak areas.

5.1.4.3 Repeat Section 5.1.4.1.

5.1.4.4 Compare the average peak area of the calibration standard to that of the nitrogen trifluoride sample being tested. Calculate the concentration of carbon tetrafluoride, using the formula below. The result may not exceed the specification in Section 2 of this standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \frac{\text{Concentration of Standard}}{\text{Concentration of Sample}} = \text{Concentration of Sample}$$

5.2 *Carbon Dioxide, Nitrous Oxide, and Sulfur Hexafluoride* — This procedure is for the determination of carbon dioxide, nitrous oxide, and sulfur hexafluoride in nitrogen trifluoride using a gas chromatograph with a thermal conductivity detector. (See Figure 1.)

5.2.1 *Detection Limit* — 1.0 ppm for carbon dioxide, nitrous oxide, and sulfur hexafluoride.

5.2.2 *Instrument Parameters*

5.2.2.1 Column: Super Q (80/100 mesh), 4.9 m (16 ft.) by 1.75 mm (1/16 in.) ID, 3.2 mm (1/8 in.) OD ss or equivalent.

5.2.2.2 Carrier Flow: 19 mL/min helium.

5.2.2.3 Sample Volume: 1.0 mL.

5.2.2.4 Temperatures:

Detector	100°C
Oven	42°C
Gas sampling valve	42°C

5.2.2.5 Valve Material: Nitronic-60 or equivalent.

5.2.3 *Calibration Standard* — 10 ppm carbon dioxide, 10 ppm nitrous oxide, 10 ppm sulfur hexafluoride, balance helium.

5.2.4 *Operating Procedure*

5.2.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak areas. The order of elution is carbon dioxide, nitrous oxide, sulfur hexafluoride. (See Figure 1 for valve configuration.)

5.2.4.2 Inject the sample to be tested in the same manner as the calibration standard. Record the retention times and peak areas.

5.2.4.3 Repeat Section 5.2.4.1.

5.2.4.4 Compare the average peak area of the calibration standard to that of the nitrogen trifluoride sample being tested. Calculate the concentration of each impurity using the formula below. The results may not exceed the specifications in Section 2 of this standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \text{Concentration of Standard} = \text{Concentration of Sample}$$

5.3 *Carbon Monoxide* — This procedure is for the determination of carbon monoxide in nitrogen trifluoride using a gas chromatograph with a methanizer and a flame ionization detector. (See Figure 1 and Note 2.)

5.3.1 Detection Limit — 0.1 ppm.

5.3.2 *Instrument Parameters*

5.3.2.1 Column: Carbosphere (80/100 mesh), 0.74 m (2.4 ft) by 1.75 mm (1/16 in) ID, 3.2 mm (1/8 in) OD ss or equivalent.

5.3.2.2 Carrier Flow: 25 mL/min helium.

5.3.2.3 Sample Volume: 0.5 mL.

5.3.2.4 Fuel gases:

Hydrogen:	Zero grade or better hydrogen at a flow rate of 45 mL/min is added to the carrier gas between the column outlet and the methanizer inlet.
Air:	Zero grade or better air is used at a flow rate of 300 mL/min.

5.3.2.5 Temperatures:

Detector	150°C
Oven	42°C
Methanizer	340°C
Gas sampling valve	70°C

5.3.2.6 Valve Material: Nitronic-60 or equivalent.

5.3.3 *Calibration Standard* — 10 ppm carbon monoxide, balance helium.

5.3.4 *Operating Procedure*

5.3.4.1 Inject the the calibration standard into the column using a gas sampling valve. Record the retention time and peak area.

5.3.4.2 Inject the sample to be tested in the same manner as the calibration standard. Record the retention time and peak area.

5.3.4.3 Repeat Section 5.3.4.1.

5.3.4.4 Compare the average peak area of the calibration standard to that of the nitrogen trifluoride sample being tested. Calculate the concentration of carbon monoxide, using the formula below. The result may not exceed the specification in Section 2 of this standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \text{Concentration of Standard} = \text{Concentration of Sample}$$

5.4 *Oxygen + Argon, Nitrogen* — This procedure is for the determination of oxygen + argon and nitrogen in nitrogen trifluoride using a gas chromatograph with a thermal conductivity detector. (See Figure 2 and Note 1.)

5.4.1 *Detection Limit* — 5 ppm of oxygen + argon and nitrogen.

5.4.2 *Instrument Parameters*

5.4.2.1 Column: Molecular sieve 13X (80/100 mesh), 6.1 m (20 ft) by 1.75 mm (1/16 in) ID, 3.2 mm (1/8 in) OD ss or equivalent.

5.4.2.2 Carrier Flow: 17 mL/min helium.

5.4.2.3 Sample Volume: 1.0 mL.

5.4.2.4 Temperatures:

Detector	140°C
Filament	240°C
Oven	100°C
Gas sampling valve	60°C

5.4.2.5 Valve Material: Nitronic-60 or equivalent.

5.4.3 *Calibration Standard* — 10 ppm oxygen, 10 ppm nitrogen, balance helium.