

SEMI C3.49-94 (Withdrawn 0303) STANDARD FOR BULK NITROGEN (N₂), 99.99999% QUALITY

This Standard was technically approved by the Gases Global Technical Committee. Available at www.semi.org in March 2003; originally published in 1994.

NOTICE: This Document was balloted and approved for withdrawal in 2003; it has been replaced by SEMI C59.

1 Description

1.1 Nitrogen is an odorless, tasteless, diatomic gas comprising approximately 78% of the earth's atmosphere; colorless liquid. Noncombustible; a cryogenic gas derived from liquid air by fractional distillation.

2 Specifications

Quality: 99.99999%, including rare gases

<i>Impurities</i>	<i>Maximum Acceptable Level (ppb)^{#1}</i>
Water (H ₂ O)	50
Oxygen (O ₂)	10
Carbon dioxide (CO ₂)	10
Carbon monoxide (CO)	10
Total Hydrocarbons expressed as Methane (THC)	10
Hydrogen (H ₂)	10
Particles ^{#2}	max 20 per/ft ³ >0.02 μm
TOTAL SPECIFIED IMPURITIES	100 ppb

#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.

#2 May be determined after purifier/filter. Sampling point to be agreed between supplier and user.

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	28.013	28.013
Boiling point at 1 atm	−195.8°C	−320.4°F
Density of gas at 21.1°C and 1 atm (air=1)	1.1605 kg/m ³	0.07245 lb/ft ³
Specific gravity of gas at 21.1°C and 1 atm (air=1)	0.0967	0.0967
Density of liquid at boiling point	808.8 kg/m ³	50.49 lb/ft ³

4 Analytical Procedures (see Notes 1 and 2)

4.1 *Carbon Monoxide and Hydrogen* — This procedure is for the determination of carbon monoxide and carbon dioxide in nitrogen using a gas chromatograph fitted with a reduction gas detector. (See Note 3.)

4.1.1 *Detection Limit* — Hydrogen 3 ppb, CO, 0.3 ppb (mole/mole).

4.1.2 *Instrument Parameters*

4.1.2.1 *Column 1* — 1/8 in. OD 77 in. length Unibead 1S 60-80 mesh; *Column 2* — 1/8 in. OD 30 3/4 in. length MS5A

4.1.2.2 *Carrier Flow* — 20 mL/min at 105°C nitrogen.

4.1.2.3 *Sample Volume* — 100 µL.

4.1.2.4 *Temperatures*

- *Detector* — 270°C.
- *Column Oven* — 105°C.

4.1.2.5 *Calibration Standards* 0.1 ppm to 0.5 ppm (mole/mole) carbon monoxide, 0.1 ppm to 0.5 ppm (mole/mole) hydrogen, balance nitrogen.

4.1.2.6 *Calibration Standards* — Construct a calibration curve in the range of interest. Verify the standards employed independently by established traceability to recognized national or international standards.

4.1.3 *Operating Procedure*

4.1.3.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak areas. Order of elution is hydrogen, carbon monoxide.

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

4.1.3.3 Repeat ¶ 4.1.4.1.

4.1.3.4 Compare the average peak area of the calibration standard to that of the nitrogen sample being tested. Calculate the concentrations of carbon monoxide and hydrogen using the formula below. The results may not exceed the specification in § 2 of this Standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \text{Concentration of Standard} = \text{Concentration of Sample} \quad (1)$$

4.2 *Carbon Dioxide* — This procedure is for the determination of carbon dioxide in nitrogen using a gas chromatograph with a flame ionization detector, fitted with a methanizer.

4.2.1 *Detection Limit* — 2 ppb (mole/mole).

4.2.2 *Instrument Parameters*

4.2.2.1 *Column* — 1/8 in. OD 77 in. Length Hayesep D 100 to 120 mesh.

- *Carrier Flow* — 30 mL/min nitrogen
- *Air Flow* — 250 mL/min.
- *Hydrogen Flow* — 30 mL/min.

4.2.2.2 *Sample Volume* — 5 mL.

4.2.3 *Temperatures*

- *Methanizer* — 315°C.
- *Column Temperature* — 40°C

4.2.3.1 *Calibration Standard* — 0.1 ppm (mole/mole) carbon dioxide in nitrogen.

4.2.3.2 *Calibration Standards* — Construct a calibration curve in the range of interest. Verify the standards employed independently by established traceability to recognized national or international standards.