

SEMI C59-1103

SPECIFICATIONS AND GUIDELINES FOR NITROGEN

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1 Purpose

1.1 The purpose of this document is to provide a series of specifications for different grades of Nitrogen (N₂) that are used in the semiconductor industry.

2 Scope

2.1 This document covers requirements for all grades of nitrogen used in the semiconductor industry.

2.2 If analytical methods are not complete, the requirements are presented as a guideline.

2.3 This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

3 Description

3.1 Nitrogen is an odorless, tasteless, noncombustible diatomic gas comprising approximately 78% of the earth's atmosphere; at cryogenic temperatures it is a

colorless liquid. Noncombustible; a cryogenic gas derived from liquid air by fractional distillation.

4 Limitations

4.1 None.

5 Referenced Standards

5.1 *SEMI Standards*

SEMI C1 — Specifications for Reagents

SEMI C3 — Specifications for Gases

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

6 Terminology

6.1 Terminology appropriate to this standard is defined in SEMI C3.

7 Requirements

7.1 Purity and other requirements for the various grades of nitrogen are given in Table 1.

Table 1 Impurity and Other Requirements for Various Grades of Nitrogen

Previous SEMI Reference #	C3.5-93 (Specification)	C3.48-0200 (Specification)	C3.29-96 (Specification)	C3.28-0200 (Guideline)	C3.49-94 (Specification)
Grade	4.8	5.4	5.5	5.6	7.0
Purity	99.998%	99.9994%	99.9995% (See Note 1.)	99.9996%	99.99999%
Impurities	Maximum Acceptable Level (ppm) (See Note 2.)				
Carbon Dioxide (CO ₂)	1	0.5	0.2	0.5	0.010
Carbon Monoxide (CO)	5	2	2	0.5	0.010
Hydrogen (H ₂)	2	2	2	1	0.010
Oxygen (O ₂)	3	0.5	0.2	0.5	0.010
Water (H ₂ O) (ppmv)	1	0.5	0.2	0.5	0.050
Total Hydrocarbons expressed as Methane (THC)	1	0.5	0.2	0.5	0.010
TOTAL SPECIFIED IMPURITIES	13	6	4.8	3.5	0.100
Particles	(See Note 3.)	(See Note 3.)	(See Note 3.)	(See Note 3.)	Max. 20/ft ³ > 0.02 µm (See Note 4.)

Note 1: A purifier is allowed to be used to meet this specification.

Note 2: An analysis of significant figures has not been considered. The number of significant figures is based on analytical accuracy and the precision of the provided procedure.

Note 3: To be determined between supplier and user.

Note 4: May be determined after purifier/filter. Sampling point to be agreed between supplier and user.

8 Physical Constants

8.1 The physical constants of nitrogen are given in Table 2 (for information only).

Table 2 Physical Constants of Nitrogen (for information only)

	Metric Units	US Units
Molecular weight	28.013	28.013
Boiling point at 1 atm	-195.8°C	-320.4°F
Density of gas at 21.1°C (70°F) and 1 atm	1.1605 kg/m ³	0.07245 lb/ft ³
Specific gravity of gas at 21.1°C and 1 atm (air = 1)	0.967	0.967
Density of liquid at boiling point	808.8 kg/m ³	50.49 lb/ft ³

9 Analytical Procedures for Grade 4.8 Nitrogen

NOTE 2: Introduce the calibration standard as many times as necessary to achieve the desired precision.

NOTE 3: All gases used in the analysis of the sample should contain not more than 10% of the sample value of the component of interest unless otherwise specified.

9.1 *Carbon Monoxide and Carbon Dioxide* — This procedure is for the determination of carbon monoxide and carbon dioxide in nitrogen using a gas chromatograph with a flame ionization detector and methanizer.

NOTE 4: Carrier gases should contain less than 0.1 ppm carbon monoxide and less than 0.1 ppm hydrogen.

9.1.1 *Detection Limit* — 100 ppb

9.1.2 *Instrument Parameters*

9.1.2.1 *Column*: Porapak T or Q, 3 m (9.8 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID ss; or Chromosorb 102, 2 m (6.6 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID ss; or equivalent.

9.1.2.2 *Carrier Flow*: 30 mL/min helium.

9.1.2.3 *Sample Volume*: 0.5 to 2.0 mL.

9.1.2.4 *Temperatures*:

Detector	280°C
Column Oven	60°C
Methanizer	350°C

9.1.3 *Calibration Standards* — 1–5 ppm carbon monoxide, 1–5 ppm carbon dioxide, balance nitrogen.

9.1.4 *Operating Procedure*

9.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times

and peak areas. Order of elution is carbon monoxide, carbon dioxide.

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

9.1.4.3 Repeat Section 9.1.4.1.

9.1.4.4 Compare the average peak area of the calibration standard to that of the nitrogen sample being tested. Calculate the concentrations of carbon monoxide and carbon dioxide, using the formula:

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

9.1.4.5 The result may not exceed the specification in Table 1.

9.2 *Hydrogen* — This procedure is for the determination of hydrogen in nitrogen using a gas chromatograph with a helium ionization detector.

9.2.1 *Detection Limit* — 500 ppb.

9.2.2 *Instrument Parameters*

9.2.2.1 *Column*: 5A molecular sieve, 1.9 m (6 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) stainless steel or equivalent.

9.2.2.2 *Carrier Flow*: 30 mL/min helium.

9.2.2.3 *Sample Volume*: 3.0 mL

9.2.2.4 *Temperatures*:

Detector	125°C
Column Temperature	65°C

9.2.3 *Calibration Standard* — 1–5 ppm hydrogen in nitrogen.

9.2.4 *Operating Procedure*

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

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

9.2.4.3 Repeat Section 9.2.4.1.

9.2.4.4 Compare the average peak area of the calibration standard to that of the nitrogen sample being tested. Calculate the concentration of hydrogen, using the formula:

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

9.2.4.5 The result may not exceed the specification in Table 1.

9.3 *Oxygen* — This procedure is for the determination of oxygen in nitrogen using a continuous flow analyzer using an electrochemical method.

9.3.1 *Detection Limit* — 100 ppb.

9.3.2 *Flow Rate* — Set sample flow rates in accordance with the instrument manufacturer's instructions.

9.3.3 *Calibration Standard* — 3–15 ppm oxygen in nitrogen or in accordance with the instrument manufacturer's instructions.

9.3.4 *Operating Procedure*

9.3.4.1 Do not change the sample flow setting once established.

9.3.4.2 Introduce nitrogen sample and record oxygen reading. The result may not exceed the specification in Table 1.

9.4 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons in nitrogen using a continuous flow flame ionization detector equipped total hydrocarbon analyzer.

NOTE 5: The 0–1 range can be used provided that zero and span gas standards in nitrogen with known levels of hydrocarbons between 0–1 ppm are used in the calibration of the analyzer.

NOTE 6: As the flow rate and heat capacity of the matrix gas affect the instrument output, the zero and span gas matrices must coincide with that of the sample gas.

NOTE 7: The effective response of a flame ionization detector-equipped total hydrocarbon analyzer to different hydrocarbons can vary and must be approximated. However, the response of the most common hydrocarbon impurities in nitrogen can be accurately totaled and compared to methane.

9.4.1 *Detection Limit* — 0.1 ppm.

9.4.2 *Flow Requirements*

9.4.2.1 High purity, hydrocarbon-free (less than 0.1 ppm) hydrogen: 35–40 mL/min or 40% hydrogen in either helium or nitrogen matrix at 75–80 mL/min.

9.4.2.2 Dry, hydrocarbon-free (less than 0.1 ppm) air: 350–400 mL/min.

9.4.2.3 Set sample flow rates in accordance with the instrument manufacturer's instructions.

9.4.3 *Calibration Standards*

9.4.3.1 Nitrogen with known quantity of hydrocarbons at 0.5 ppm level.

9.4.3.2 The upper level span gas not exceeding 5 times the concentration of the specification.

9.4.4 *Operating Procedure*

9.4.4.1 Do not change the flow settings for hydrogen, air and sample once established.

9.4.4.2 Introduce the nitrogen with known quantity of hydrocarbons and, using the 0–10 ppm range, set the needle (or output) to read the correct level using the zero adjust knob.

9.4.4.3 Introduce the span gas standard in nitrogen and, using the span adjust knob, set the needle (or output reading) to match the level of hydrocarbons in the span gas.

9.4.4.4 Introduce nitrogen sample into the analyzer and read the quantity of hydrocarbons on the analyzer meter. The result may not exceed the specification in Table 1.

9.5 *Water* — This procedure is for the determination of trace moisture (water) in nitrogen using a continuous flowing piezoelectric hygrometer.

NOTE 8: The sampling system and hygrometer must be designed to operate under the sample pressure, or the sample pressure must be reduced (by a regulator with a diaphragm of stainless steel or other suitable material) to accommodate the pressure restrictions of the analytical hygrometer.

9.5.1 *Detection Limit* — 0.1 ppmv or –90°C (–130°F).

9.5.2 *Flow Requirements* — Set the sample pressure and flow rate in accordance with the instrument manufacturer's instructions.

9.5.3 *Calibration Standards* — Construct a calibration curve which contains at least three points covering the range of interest. Verify the standards employed independently by another analytical method.

9.5.4 *Operating Procedure*

9.5.4.1 Obtain a continuous flow sample of gas from the source using a clean and passivated stainless steel line which has been purged dry after exposure to ambient moisture.

9.5.4.2 After prepurging with a dry gas, allow the sample gas to flow through the sampling system and the piezoelectric moisture hygrometer until a stable reading is obtained.