

# Background Statement for SEMI Draft Document 6002A REVISION TO SEMI C59-1104 (Reapproved 0211), SPECIFICATIONS ~~AND GUIDELINES~~ FOR NITROGEN

**Notice:** This background statement is not part of the balloted item. It is provided solely to assist the recipient in reaching an informed decision based on the rationale of the activity that preceded the creation of this Document.

**Notice:** Recipients of this Document are invited to submit, with their comments, notification of any relevant patented technology or copyrighted items of which they are aware and to provide supporting documentation. In this context, “patented technology” is defined as technology for which a patent has issued or has been applied for. In the latter case, only publicly available information on the contents of the patent application is to be provided.

## Background

Document 6002, line item ballot, was approved for ballot to correct the nonconforming title in Cycle 4-16. Unrelated comments were received from the LI ballot, which required failing of the Document and sending it for full Letter Ballot after re-work. This major revision incorporates and addresses the negatives and comments along with the changes required to correct the nonconforming title.

**Notice:** Additions are indicated by underline and deletions are indicated by ~~strikethrough~~.

## Review and Adjudication Information

	Task Force Review	Committee Adjudication
<b>Group:</b>	Gases Specification Task Force	Facilities & Gases NA TC Chapter
<b>Date:</b>	November 7, 2016	November 8, 2016
<b>Time &amp; Timezone:</b>	13:00 – 14:00 PDT	9:00 – 12:00 Noon PDT
<b>Location:</b>	SEMI Headquarters 3081 Zanker Road	SEMI Headquarters 3081 Zanker Road
<b>City, State/Country:</b>	San Jose, CA/USA	San Jose, CA/USA
<b>Leader(s)/Authors:</b>	Mohamed Saleem (Fujikin) Matthew Milburn (UCT) Thomas Fritz (WIKI)	Mohamed Saleem (Fujikin) Steve Lewis (LPCiminelli)
<b>Standards Staff:</b>	Laura Nguyen ( <a href="mailto:lnguyen@semi.org">lnguyen@semi.org</a> )	Laura Nguyen ( <a href="mailto:lnguyen@semi.org">lnguyen@semi.org</a> )

This meeting’s details are subject to change, and additional review sessions may be scheduled if necessary. Contact the task force leaders or Standards staff for confirmation.

Telephone and web information will be distributed to interested parties as the meeting date approaches. If you will not be able to attend these meetings in person but would like to participate by telephone/web, please contact Standards staff.

Check [www.semi.org/en/standards](http://www.semi.org/en/standards) on calendar of event for the latest meeting schedule.

# SEMI Draft Document 6002A REVISION TO SEMI C59-1104 (Reapproved 0211), SPECIFICATIONS ~~AND GUIDELINES~~ FOR NITROGEN

This Standard was technically approved by the global Gases Committee. This edition was approved for publication by the global Audits & Reviews Subcommittee on December 21, 2010. Available at [www.semiviews.org](http://www.semiviews.org) and [www.semi.org](http://www.semi.org) in February 2011; originally published November 2002; previously published November 2004.

## 1 Purpose

1.1 The purpose of this Document is to provide a series of specifications for different grades of Nitrogen (N<sub>2</sub>) 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.~~

**NOTICE:** SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other 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 and Documents

5.1 *SEMI Standards and Safety Guidelines*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

SEMI C3 — Specifications for Gases

**NOTICE:** Unless otherwise indicated, all documents cited shall be the latest published versions.

## 6 Terminology

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

**Table 1 Impurity and Other Requirements for Various Grades of Nitrogen**

<i>Previous SEMI Reference #</i>	<i>C3.5-93 (Specification)</i>	<i>C3.15-93 (Specification)</i>	<i>C3.48-0200 (Specification)</i>	<i>C3.29-96 (Specification)</i>	<i>C3.28-0200 (Guideline)</i>	<i>C3.49-94 (Specification)</i>
Grade	4.8	5.2	5.4	5.5	5.6	7.0
Purity	99.998%	99.9992%	99.9994%	99.9995% <sup>#1</sup>	99.9996%	99.99999%
Impurities	Maximum Acceptable Level (ppm) <sup>#2</sup>					
Carbon Dioxide (CO <sub>2</sub> )	1	1	0.5	0.2	0.5	0.010
Carbon Monoxide (CO)	5	2	2	2	0.5	0.010
Hydrogen (H <sub>2</sub> )	2	2	2	2	1	0.010
Oxygen (O <sub>2</sub> )	3	1	0.5	0.2	0.5	0.010

<i>Previous SEMI Reference #</i>	<i>C3.5-93 (Specification)</i>	<i>C3.15-93 (Specification)</i>	<i>C3.48-0200 (Specification)</i>	<i>C3.29-96 (Specification)</i>	<i>C3.28-0200 (Guideline)</i>	<i>C3.49-94 (Specification)</i>
Water (H <sub>2</sub> O) (ppmv)	1	1	0.5	0.2	0.5	0.050
Total Hydrocarbons expressed as Methane (THC)	1	1	0.5	0.2	0.5	0.010
TOTAL SPECIFIED IMPURITIES	13	8	6	4.8	3.5	0.100
Particles	#3	#3	#3	#3	#3	Max. 20/ft <sup>3</sup> > 0.02 μm <sup>#4</sup>

#1 A purifier is allowed to be used to meet this Specification.

#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.

#3 To be determined between supplier and user.

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

## 7 Requirements

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

## 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)**

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	28.013	28.013
CAS Number	7727-37-9	7727-37-9
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 <sup>3</sup>	0.07245 lb/ft <sup>3</sup>
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 <sup>3</sup>	50.49 lb/ft <sup>3</sup>

## 9 Analytical Procedures for Grade 4.8 Nitrogen

NOTE 1: Introduce the calibration standard as many times as necessary to achieve the desired precision.

NOTE 2: All gases used in the analysis of the sample should contain no 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 3: 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 stainless steel; or Chromosorb® 102, 2 m (6.6 ft.) by 3.2 mm (1/8 in.) OD by 2.2 mm (0.085 in.) ID stainless steel; 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 to 5 ppm carbon monoxide, 1 to 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 ¶ 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:

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

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 to 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 ¶ 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:

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \begin{matrix} \text{Concentration} \\ \text{of Standard} \end{matrix} = \begin{matrix} \text{Concentration} \\ \text{of Sample} \end{matrix} \quad (2)$$

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 to 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 4: The 0 to 1 range can be used provided that zero and span gas standards in nitrogen with known levels of hydrocarbons between 0 to 1 ppm are used in the calibration of the analyzer.

NOTE 5: 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 6: 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 to 40 mL/min or 40% hydrogen in either helium or nitrogen matrix at 75 to 80 mL/min.

9.4.2.2 Dry, hydrocarbon-free (less than 0.1 ppm) air: 350 to 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 to 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 7: 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  $-90^{\circ}\text{C}$  ( $-130^{\circ}\text{F}$ ).

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 pre-purging 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.

9.5.5 Determine the moisture content of the nitrogen sample by comparing the reading to calibration curve. The result may not exceed the specification in Table 1.

**10 Analytical Procedures for Grade 5.2 Nitrogen (see Note 1 and Note 2 in § 9)**

10.1 *Carbon Monoxide and Carbon Dioxide* — Use the procedure in ¶ 9.1 for the analysis of carbon monoxide and carbon dioxide.

10.1.1 *Hydrogen* — Use the procedure in ¶ 9.2 for the analysis of hydrogen.

10.1.2 *Oxygen* — Use the procedure in ¶ 9.3 for the analysis of oxygen except that the standard should be between 1 and 5 ppm oxygen in nitrogen.

10.1.3 *Total Hydrocarbons* — Use procedure ¶ 9.4 for the determination of total hydrocarbons except that the calibration standard should be at the 0.1 ppm level.

10.1.4 *Water* — This procedure is for the determination of moisture (water) in nitrogen using a continuous flowing, cooled-surface condensation, dewpoint/ frostpoint hygrometer (see Note 7 in § 9.).

NOTE 8: The National Institute of Standards and Technology (NIST) provides calibration services for the thermometers used in dewpoint/frostpoint hygrometers.

NOTE 9: This method is not applicable if other constituents in the gas will condense before water vapor, for example, carbon dioxide and/or oil contamination.

10.1.4.1 *Detection Limit* — 0.6 ppm (vol/vol) or  $-79^{\circ}\text{C}$  ( $-110^{\circ}\text{F}$ ).

10.1.4.2 *Flow Requirements*

10.1.4.2.1 Set sample flow rate and pressure in accordance with the instrument manufacturer's instructions.

10.1.4.2.2 Gas must flow past the chilled mirror where optic means are provided to detect the deposit (or frost) and to read the thermometer measuring the temperature of the mirror.

10.1.4.3 *Calibration Standard* — A calibration thermometer designed to indicate temperatures in the  $-79^{\circ}\text{C}$  ( $-110^{\circ}\text{F}$ ) range required.

10.1.4.4 *Operating Procedure*

10.1.4.4.1 Obtain a continuous flow of sample gas from the source using a clean stainless steel (or copper) sampling line which has been purged dry after exposure to ambient moisture.

10.1.4.4.2 After pre-purging with a dry gas, allow the sample gas to flow through the sampling system and the dewpoint/frostpoint hygrometer for one hour to 24 hours to allow the entire system to reach equilibrium with regard to moisture content.

10.1.4.4.3 After equilibrium has been reached, cool down the mirror to determine the actual dewpoint/frostpoint of the sample gas. Follow the manufacturer's recommendations to create the temperatures needed.

10.1.4.4.4 Continue to verify the dewpoint/frostpoint for at least 30 minutes after a stable reading has been confirmed.

10.1.4.4.5 Correct the dewpoint reading from the measured pressure to 1 atm of pressure. The result may not exceed the specification in Table 1.

## **11 Analytical Procedures for Grade 5.4 Nitrogen (see Note 1 and Note 2 in § 9)**

11.1 *Carbon Monoxide and Carbon Dioxide* — Use the procedure in ¶ 9.1 for the analysis of carbon monoxide and carbon dioxide.

11.2 *Hydrogen* — Use the procedure in ¶ 9.2 for the analysis of hydrogen.

11.3 *Oxygen* — Use the procedure in ¶ 9.3 for the analysis of oxygen except that the standard should be between 0.5 and 2.5 ppm oxygen in nitrogen.

11.4 *Total Hydrocarbons* — Use procedure ¶ 9.4 for the determination of total hydrocarbons except that the calibration standard should be at the 0.1 ppm level.

11.5 *Water* — Use procedure in ¶ 9.5 for the determination of water.

## **12 Analytical Procedures for Grade 5.5 Nitrogen (see Note 1 and Note 2 in § 9)**

12.1 *Carbon Monoxide and Hydrogen* — This procedure is for the determination of carbon monoxide and hydrogen in nitrogen using a gas chromatograph with a reduction gas detector (see Note 3 in ¶ 9.1).

12.1.1 *Detection Limits* — 2 ppb carbon monoxide, 20 ppb hydrogen.

12.1.2 *Instrument Parameters*

12.1.2.1 *Column* — 5A molecular sieve, 0.9 m (3 ft.) by 3 mm (1/4 in.) stainless steel, or equivalent.

12.1.2.2 *Carrier Flow* — 40 mL/min air or nitrogen.

12.1.2.3 *Sample Volume* — 2.0 mL.

12.1.2.4 *Temperatures*

- *Detector* — 280°C
- *Column Oven* — 250°C

12.1.3 *Calibration Standard* — 1 to 5 ppm carbon monoxide, 1 to 5 ppm hydrogen in nitrogen.

12.1.4 *Operating Procedure*

12.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak areas. Order of elution is hydrogen, then carbon monoxide. Run standard analysis in triplicate for stability determination relative to purging of calibration system components.

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

12.1.4.3 Repeat ¶ 12.1.4.1.

12.1.4.4 Compare the average peak areas of the calibration standard to that of the nitrogen sample being tested. Calculate the concentrations of carbon monoxide and hydrogen, using the formula:

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \frac{\text{Concentration of Standard}}{\text{Concentration of Sample}} = \text{Concentration of Sample} \quad (2)$$

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

12.2 *Carbon Dioxide* — This procedure is for the determination of carbon dioxide in nitrogen using a continuous flow, non-dispersive infrared analyzer.

12.2.1 *Detection Limit* — 50 ppb.

12.2.2 Set sample flow rate and pressure in accordance with the instrument manufacturer's instructions.

12.2.3 *Calibration Standard* — Zero nitrogen with a known quantity of carbon dioxide at not more than 0.05 ppm. Span gas nitrogen with a known concentration of 1 to 10 ppm carbon dioxide.

12.2.4 *Operating Procedure*

12.2.4.1 Do not change the initial sample flow setting once established.

12.2.4.2 Introduce the zero nitrogen with known quantity of carbon dioxide and set the needle (or output reading) to read the correct level using the zero adjustment knob.

12.2.4.3 Introduce the span gas standard in nitrogen and using the span adjust knob, set the needle (or output reading) to match the levels of carbon dioxide in the span gas.

12.2.4.4 Repeat ¶ 12.2.4.2 and ¶ 12.2.4.3 until reproducibility of readings is better than 1% of full scale.

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

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

12.3.1 *Detection Limit* — 50 ppb.

12.3.2 Set sample pressure and flow rates in accordance with the instrument manufacturer's instructions.

12.3.3 *Calibration Standard* — 1 to 10 ppm oxygen in nitrogen or accordance with the instrument manufacturer's instructions.

12.3.4 *Operating Procedure*

12.3.4.1 Do not change the initial sample flow setting once established.

12.3.4.2 Introduce nitrogen containing less than 2 ppm oxygen through a de-oxygenation catalyst to verify that there is no leakage of air into the system and to demonstrate that the detection limit can be achieved.

12.3.4.3 Introduce the calibration standard. Using the span adjust knob, set the needle (or output reading) to match the level of oxygen in the calibration gas.

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

12.4 *Water* — This procedure is for the determination of trace moisture (water) in nitrogen using a continuous flowing piezoelectric hygrometer (see Note 7 in ¶ 9.5).

12.4.1 *Detection Limit* — 0.1 ppmv or  $-90^{\circ}\text{C}$  ( $-130^{\circ}\text{F}$ ).

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

12.4.3 *Calibration Standard* — Construct a calibration curve which contains at least three points covering the range of interest. The standards employed will be verified independently by another analytical method.

12.4.4 *Operating Procedure*

12.4.4.1 Obtain a continuous flow of sample gas from the source using a clean and passivated 316 stainless steel line which has been purged dry after exposure to ambient moisture.

12.4.4.2 After pre-purging 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. The result may not exceed the specification in Table 1.

12.5 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons in nitrogen using a continuous flow flame ionization detector-equipped total hydrocarbon analyzer (See Notes 4, 5, and 6 in ¶ 9.4).

12.5.1 *Detection Limit* — 0.1 ppm.

12.5.2 *Flow Requirements*

12.5.3 High-purity, hydrocarbon-free (less than 1.0 ppm) hydrogen: 35 to 40 mL/min or 40% hydrogen in either helium or nitrogen matrix at 75 to 80 mL/min.

12.5.4 Dry, hydrocarbon-free (less than 1.0 ppm) air: 350 to 400 mL/min.



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

12.5.6 *Calibration Standards*

12.5.6.1 Zero nitrogen with known quantity of hydrocarbons at 0.1 ppm level.

12.5.6.2 *Span Gas* — Nitrogen with known quantity (1 to 10 ppm) hydrocarbons.

12.5.7 *Operating Procedure*

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

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

12.5.7.3 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.

12.5.7.4 Repeat ¶ 12.5.7.2 and ¶ 12.5.7.3 until reproducibility of readings is better than 1% of full scale.

12.5.8 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.

**13 Analytical Procedures for Grade 5.6 Nitrogen (see Note 1 and Note 2 in § 9)**

13.1 *Carbon Monoxide and Hydrogen* — Use the procedure in ¶ 12.1 for the determination of carbon monoxide and hydrogen.

13.2 *Carbon Dioxide* — Use procedure ¶ 12.2 for the determination of carbon dioxide.

13.3 *Oxygen* — Use procedure ¶ 12.3 for the analysis of oxygen.

13.4 *Water* — Use procedure ¶ 9.5 for the determination of water.

13.5 *Total Hydrocarbons* — Use procedure ¶ 9.4 for the determination of hydrocarbons, except that the calibration standard should be at 0.1 ppm hydrocarbons.

**14 Analytical Procedures for Grade 6.0 Nitrogen (see Note 1 and Note 2 in § 9)**

14.1 *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 in ¶ 9.1).

14.1.1 *Detection Limit* — Hydrogen 3 ppb, CO, 0.3 ppb.

14.1.2 *Instrument Parameters*

- *Column 1* — 3.2 mm (1/8 in.) OD 77 in. length Unibead 1S 60/80 mesh.
- *Column 2* — 3.2 mm (1/8 in.) OD 30 ¾ in. length Molecular Sieve 5A.

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

14.1.2.2 *Sample Volume* — 100 µL.

14.1.2.3 *Temperatures*

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

14.1.3 *Calibration Standards* — 0.1 to 0.5 ppm carbon monoxide, 0.1 to 0.5 ppm hydrogen, balance nitrogen.

14.1.3.1 Construct a calibration curve in the range of interest. Verify the standards employed independently by established traceability to recognized national or international standards.

14.1.4 *Operating Procedure*

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

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

14.1.4.3 Repeat ¶ 14.1.4.1.

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

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \frac{\text{Concentration of Standard}}{\text{Concentration of Sample}} = \text{Concentration of Sample} \quad (3)$$

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

14.2 *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.

14.2.1 *Detection Limit* — 2 ppb.

14.2.2 *Instrument Parameters*

14.2.2.1 *Column* — 3.2 mm (1/8 in.) OD 77 in. length Hayesep® D 100/120 mesh.

14.2.2.2 *Flow Rates*

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

14.2.2.3 *Sample Volume* — 5 mL.

14.2.2.4 *Temperatures*

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

14.2.3 *Calibration Standard* — 0.1 ppm carbon dioxide in nitrogen.

14.2.3.1 Construct a calibration curve in the range of interest. Verify the standards employed independently by established traceability to recognized national or international standards.

14.2.4 *Operating Procedure*

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

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

14.2.4.3 Repeat ¶ 14.2.4.1.

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

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \frac{\text{Concentration of Standard}}{\text{Concentration of Sample}} = \text{Concentration of Sample} \quad (4)$$

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

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

14.3.1 *Detection Limit* — 2 ppb.

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

14.3.3 *Calibration Standards* — 50 ppb oxygen in nitrogen or in accordance with the instrument manufacturer's instructions.

14.3.4 *Operating Procedure*

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

14.4 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons in nitrogen using a gas chromatograph fitted with a flame ionization detector and a back flush valve.

14.4.1 *Detection Limit* — 2 ppb.

14.4.2 *Instrument Parameters*

14.4.2.1 *Column* — 3.2 mm (1/8 in.) OD 77 in. length Hayesep® D 100/120 mesh.

14.4.2.2 *Flow Rates*

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

14.4.2.3 *Sample Volume* — 5 mL.

14.4.2.4 *Temperatures* — Column Temperature 40°C.

14.4.3 *Calibration Standard* — 0.1 ppm methane in nitrogen.

14.4.3.1 *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.

14.5 *Water* — This procedure is for the determination of trace moisture (water) in nitrogen using a continuous flowing phosphorous pentoxide hygrometer (see Note 7 in § 9.5).

14.5.1 *Detection Limit* — 10 ppbv.

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

14.5.3 *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.

14.5.4 *Operating Procedure*

14.5.4.1 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.

14.5.4.2 After pre-purging with a dry gas, allow the sample gas to flow through the sampling system and the P<sub>2</sub>O<sub>5</sub> moisture hygrometer until a stable reading is obtained.

14.5.4.3 Determine the moisture content of the nitrogen sample by comparing the reading to calibration curve. The result may not exceed the specification in Table 1.

**NOTICE:** SEMI makes no warranties or representations as to the suitability of the Standards and Safety Guidelines set forth herein for any particular application. The determination of the suitability of the Standard or Safety Guideline is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. Standards and Safety Guidelines are subject to change without notice.

By publication of this Standard or Safety Guideline, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this Standard or Safety Guideline. Users of this Standard or Safety Guideline are expressly advised that determination of any such patent rights or copyrights and the risk of infringement of such rights are entirely their own responsibility.