

SEMI C60-0305 (Reapproved 0211) SPECIFICATIONS AND GUIDELINES FOR NITROUS OXIDE (N₂O)

This Standard was technically approved by the Gases Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on December 21, 2010. Available at www.semiviews.org and www.semi.org in February 2011; originally published March 2005.

NOTICE: This Document was reapproved with minor editorial changes.

1 Purpose

1.1 The purpose of this Document is to provide a series of specifications for different grades of nitrous oxide (N₂O) that are used in the semiconductor industry.

2 Scope

2.1 This Document covers requirements for all grades of nitrous oxide 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 Nitrous oxide is an oxidizing, colorless, liquefied gas with a sweetish odor. It is a simple asphyxiant. Vapor pressure is about 745 psig at 70°F.

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.

7 Requirements

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

Table 1 Impurity and Other Requirements for Various Grades of Nitrous Oxide

<i>Previous SEMI Reference #</i>	<i>C3.13-1000 (Specification)</i>	<i>C3.50-1000 (Guideline)</i>
Grade	4.7	5.4
Purity	99.997% ^{#2, #3}	99.9994% ^{#2, #3}
Impurities	Maximum Acceptable Level (ppm) ^{#1}	Maximum Acceptable Level (ppm) ^{#1}
Ammonia (NH ₃)	5	0.1
Carbon Dioxide (CO ₂)	2	0.5
Carbon monoxide (CO)	1	0.1

<i>Previous SEMI Reference #</i>	<i>C3.13-1000 (Specification)</i>	<i>C3.50-1000 (Guideline)</i>
Hydrocarbons C ₁ -C ₅	1	0.1 (as C ₁)
Nitric Oxide (NO)	1	0.1
Nitrogen (N ₂)	10	3
Nitrogen dioxide (NO ₂)	1	0.1
Oxygen (O ₂)	2	N/A
Oxygen (O ₂) + Argon (Ar)	N/A	0.5
Water (H ₂ O) (v/v)	3	1.0
TOTAL SPECIFIED IMPURITIES	26	5.5

#1 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.

#2 This specification applies to the gas phase of the cylinder as delivered.

#3 It is recommended that the user discontinue use of the cylinder prior to complete consumption of the liquid phase. The contents of cylinders should be determined by weight, not pressure.

8 Physical Constants

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

Table 2 Physical Constants of Nitrous Oxide (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular Weight	44.01	44.01
CAS Number	10024-97-2	10024-97-2
Boiling point at 1 atm	-88.5°C	-127.4°F
Density of gas at 70°F (21.1°C) and 1 atm	1.947 g/L	0.1160 lb/ft ³
Specific gravity of gas	1.53	1.53
Critical pressure	71.46 atm	1052 psia
Critical temperature	36.4°C	97.6°F

9 Analytical Procedures for Grade 4.7 Nitrous Oxide (See Appendix 1)

NOTE 1: Rapid withdrawal of gaseous nitrous oxide from a liquified source (e.g., cylinder) can result in a significant cooling effect which may condense the nitrous oxide in the sampling lines.

9.1 *Ammonia* — This procedure is for the determination of ammonia in nitrous oxide using an apparatus employing a detector tube filled with a color-reactive chemical.

9.1.1 *Detection Limit* — 0.2 ppm (mol/mol).

9.1.2 *Apparatus*

9.1.2.1 *Pressure Regulator* — Outlet Pressure Range 0 to 30 psig.

9.1.2.2 *Flowmeter* — Range 0 to 200 mL/min.

9.1.2.3 *Detector Tube* — Range 2 to 30 ppm.

9.1.2.4 Stop watch.

9.1.2.5 6 mm (1/4 in.) flexible tubing.

9.1.3 Operating Procedure

9.1.3.1 Attach a suitable regulator to the cylinder outlet.

9.1.3.2 Connect the flowmeter to the regulator using flexible tubing.

9.1.3.3 Open the cylinder valve and purge the flowmeter with nitrous oxide.

9.1.3.4 After the line is thoroughly purged, stop the flow of nitrous oxide and attach the detector tube to the outlet of the flowmeter.

9.1.3.5 Start the flow of nitrous oxide and set the flow rate to 100 mL/min. The pressure and temperature of the sample must be within the tolerances specified by the manufacturer.

9.1.3.6 Pass 100 mL of nitrous oxide through the detector tube.

9.1.3.7 Determine the concentration of ammonia according to the detector tube manufacturer's instructions. The concentration may not exceed the specification in Table 1 of this Standard.

9.2 Carbon Monoxide and Carbon Dioxide — This procedure is for the determination of carbon monoxide and carbon dioxide in nitrous oxide using a gas chromatograph with a flame ionization detector and methanizer with back flush (see Figure A1-1).

NOTE 2: In order to prevent nitrous oxide interference, a helium backflush to vent is needed for 3 to 5 times the analysis time (estimated to be 3 to 5 minutes). Because nitrous oxide is converted into ammonia in the methanizer, stainless steel (ss) is required; copper tubing and fittings are not used. The backflush removes the nitrous oxide from Column No. 1 preparing the column for the next sample. Careful timing is required to identify carbon monoxide and carbon dioxide distinguishing both from the time methane would appear, if present. Carbon monoxide and carbon dioxide are converted to methane in the methanizer; therefore, both will be detected by the FID detector at the specific times determined by the analytical sequence of the standard.

9.2.1 **Detection Limit** — 1 ppm (mol/mol), (50 ppb depending on conditions).

9.2.2 Instrument Parameters

9.2.2.1 Columns

- **Column 1** — Porapak® QS, 4.6 m (15 ft.) by 5 mm (3/16 in.) or equivalent.
- **Column 2** — Molecular sieve 5A, 1.97 m (6 ft.) by 5 mm (3/16 in.), 80/100 mesh (reference only) or equivalent.

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

9.2.2.3 **Sample Volume** — 0.5 to 2.0 mL.

9.2.2.4 Temperatures

- **Detector** — 35°C.
- **Column Oven** — 35°C.
- **Methanizer** — 500°C.

9.2.3 **Calibration Standard** — 1 to 5 ppm (mol/mol) carbon monoxide, 1 to 5 ppm (mol/mol) carbon dioxide, balance nitrous oxide.

9.2.4 Operating Procedure

9.2.4.1 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 monoxide, methane, carbon dioxide, and nitrous oxide. Back flush 18 minutes.

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

9.2.4.3 Repeat ¶ 9.2.4.1.

9.2.4.4 Compare the average peak areas of the calibration standard to that of the nitrous oxide sample being tested. Calculate the concentrations of carbon monoxide and carbon dioxide, using the formula below. The result may not exceed the specification in Table 1 of this Standard.