

SEMI C3.12-0116 (Reapproved 0922) SPECIFICATION FOR AMMONIA (NH₃) IN CYLINDERS, 99.998% QUALITY

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NOTICE: The section symbol (§) is used to reference a numbered section, paragraph, or header and all subordinate paragraphs, headers, and embedded material (i.e., EXCEPTIONs, lists) therein. The paragraph symbol (§) is used to reference a particular numbered paragraph and embedded material therein. When duplicated (i.e., §§ and §§) the symbols are used to reference more than one section or paragraph, respectively.

NOTICE: Paragraphs entitled 'NOTE:' are not an official part of this Standard or Safety Guideline and are not intended to modify or supersede the official Standard or Safety Guideline. These have been supplied by the global technical committee to enhance the usage of the Standard or Safety Guideline.

1 Purpose

1.1 The purpose of this Standard is to provide specification for ammonia (NH₃) that is used in the semiconductor industry.

2 Scope

2.1 This Standard covers requirements for ammonia used in the semiconductor industry.

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 Referenced Standards and Documents

3.1 SEMI Standards and Safety Guidelines

SEMI C3 — Specification for Gases

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

4 Description

4.1 Ammonia is a colorless, alkaline gas having a pungent odor. Ammonia dissolves readily in water. It is shipped as a liquefied gas under its own vapor pressure. It is flammable.

5 Terminology

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

6 Specifications

6.1 Purity: 99.998%. The impurity limits of 99.998% ammonia are defined in Table 1.

Table 1 Impurity Limits for Ammonia

<i>Purity</i>	<i>99.998%</i>
<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)^{#1}</i>
Carbon monoxide (CO)	1
Hydrocarbons C ₁ – C ₃	1
Nitrogen (N ₂)	5
Oxygen (O ₂)	2
Water (H ₂ O) (v/v)	3
TOTAL LISTED IMPURITIES	12

#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 test method.

6.2 This Specification applies to the gas phase of the cylinder as delivered.

7 Properties of Ammonia (for information only)

Table 2 Properties of Ammonia

	<i>Metric Units</i>	<i>US Units</i>
Molecular Formula	NH ₃	NH ₃
CAS Number	7664-41-7	7664-41-7
Molecular weight	17.031	17.031
Boiling point at 1 atm	-33.4°C	-28.2°F
Density of gas at 0°C (32°F) and 1 atm	0.771 kg/m ³	0.0481 lb/ft ³
Specific gravity of gas at 0°C (32°F) and 1 atm (air = 1)	0.597	0.597
Density of liquid at 21.1°C (70°F)	608.7 kg/m ³	38.00 lb/ft ³

8 Test Methods for Determining Impurity Limits

NOTE 1: Observe proper safety procedures for handling and venting the ammonia sample.

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

NOTE 3: All the gases used in the analysis of the sample should contain no more than 10% of the specified value of the component of interest unless otherwise specified.

NOTE 4: All carrier lines, carrier pressure regulators and sample containers are to be constructed of stainless steel and cleaned. The systems should be assembled without pipe threads, PTFE tape or polymeric seal materials.

8.1 *Carbon Monoxide, Oxygen and Nitrogen* — This method is for the determination of carbon monoxide, oxygen and nitrogen in ammonia using a gas chromatograph with a helium ionization detector.

NOTE 5: Due to the extreme sensitivity of the helium ionization detector, the system is to be helium leak tested to a maximum of 10^{-7} atm-cc/sec.

8.1.1 *Detection Limits* — 25 ppb (mole/mole) carbon monoxide, 30 ppb (mole/mole) oxygen, 50 ppb (mole/mole) nitrogen.

8.1.2 *Instrument Parameters*

8.1.3 *Columns*

- *Column 1* — Divinylbenzene-ethyleneglycoldimethylacrylate porous polymers, 100/120 mesh, 3.28 m (10 ft.) by 3.2 mm (1/8 in.) 0.085 ID.
- *Column 2* — Divinylbenzene-ethyleneglycoldimethylacrylate porous polymers, 100/120 mesh, 3.28 m (10 ft.) by 3.2 mm (1/8 in.) 0.085 ID.

8.1.3.1 *Carrier Flow* — 30 mL/min helium, minimum of 99.9999% purity having a maximum of 40 ppb carbon dioxide.

8.1.3.2 *Sample Volume* — 1.0 mL.

8.1.3.3 *Temperatures*

- *Detector* — 125°C
- *Columns* — 27°C

8.1.3.4 Determine the breakdown voltage of the detector. Set operating voltage at 10 volts below breakdown.

8.1.4 *Calibration Standards* — 1 to 2 ppm (mole/mole) carbon monoxide, 2 to 3 ppm (mole/mole) oxygen, 5 to 6 ppm (mole/mole) nitrogen, balance helium (having a total of other impurities of less than 1 ppm).

8.1.5 *Operating Method*

8.1.5.1 Set timing interval on gas sampling valve to 3 seconds (0.05 minutes). (Refer to Figure 3.)

8.1.5.2 Set timing interval on gas sampling valve to 720 seconds (12.0 minutes) to backflush precolumn 1.

8.1.5.3 Adjust gas sampling valve timing interval as needed based on recovery on carbon monoxide peak.

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

8.1.5.5 Obtain a continuous flow of the calibration standard using a clean stainless steel line 0.508 mm (0.02 in.) ID.

8.1.5.6 Inject the calibration standard onto the precolumn with the gas sampling valve. Record the retention times and peak areas. Order of elution is nitrogen, oxygen, and carbon monoxide.

8.1.5.7 Repeat ¶ 8.1.5.6 until reproducibility of the reading is better than 1% of full scale.

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

8.1.5.9 Repeat ¶ 8.1.5.8.

8.1.5.10 Compare the average peak areas of the calibration standard to that of the ammonia sample being tested. Calculate the concentrations of carbon monoxide, oxygen and nitrogen, using the formula below. The result may not exceed the specification in § 6 of this Standard.

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

8.2 *Hydrocarbons C₁ – C₃* — This method is for the determination of hydrocarbons C₁ – C₃ in ammonia. The sample shall be gas phase and analyzed using a gas chromatograph with a flame ionization detector.