

SEMI C3.12-1102

SPECIFICATION FOR AMMONIA (NH₃) IN CYLINDERS, 99.998% QUALITY

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1 Description

1.1 Ammonia is a colorless, alkaline gas having a pungent odor. Ammonia dissolves readily in water. It is shipped as a liquified gas under its own vapor pressure. It is combustible.

2 Specifications

2.1 QUALITY: 99.998%

Impurities	Maximum Acceptable Level (ppm)*
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

*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.

NOTE 1: This specification applies to the gas phase of the cylinder as delivered.

3 Physical Constants (for information only)

	Metric Units	US Units
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 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

4 Analytical Procedures

NOTE 2: Observe proper safety procedures for handling and venting the ammonia sample.

NOTE 3: Introduce the calibration standard as many times as necessary to achieve the desired precision.

NOTE 4: All the gases used in the analysis of the sample should contain not more than 10% of the specified value of the component of interest unless otherwise specified.

NOTE 5: All carrier lines, carrier pressure regulators and sample containers are to be constructed of stainless steel and cleaned. The systems must be assembled without pipe threads, PTFE tape or polymeric seal materials.

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

NOTE 6: 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.

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

4.1.2 Instrument Parameters

4.1.3 Columns:

Column 1:	HayeSep A or equivalent, 100/120 mesh, 3.28 m (10 ft) by 3.2 mm (1/8 in.) 0.085 ID.
Column 2:	HayeSep A or equivalent, 100/120 mesh, 3.28 m (10 ft) by 3.2 mm (1/8 in.) 0.085 ID.

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

4.1.3.2 Sample Volume: 1.0 mL.

4.1.3.3 Temperatures:

Detector	125°C
Columns	27°C

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

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

4.1.5 Operating Procedure

4.1.5.1 Set timing interval on sample select valve to 10 seconds.

4.1.5.2 Set timing interval on gas sampling valve to 3:30 minutes.

4.1.5.3 Set timing interval #3 to 4:20 minutes.

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

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

4.1.5.6 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.

4.1.5.7 Repeat Section 4.1.5.6 until reproducibility of the reading is better than 1% of full scale.

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

4.1.5.9 Repeat Section 4.1.5.8.

4.1.5.10 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 Section 2 of this standard.

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

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

NOTE 7: It is good practice to keep carrier gas flowing through the instrument. This prevents the back-diffusion of moisture and carbon dioxide in the air from contaminating the column.

NOTE 8: When not analyzing samples, the dual column switching valve should be set at the molecular sieve position to prevent the silica gel column from drying out.

NOTE 9: The silica gel column will, after repeated use, tend to dry out. This will manifest itself by the peaks of each component becoming separated farther and farther apart. Overall sensitivity will drop as the peak areas decrease due to the peaks spreading out more and more.

NOTE 10: The condition of the silica gel column is restored by allowing the carrier gas to pass through the water-saturated silica gel trap that has been provided. (Normally the carrier gas bypasses this trap.)

NOTE 11: The flame should be extinguished when not in use; also the air and hydrogen is shut off; and the helium flow reduced.

4.2.1 *Detection Limit* — 0.1 ppm (mole/mole).

4.2.2 *Instrument Parameters*

4.2.2.1 *Columns:*

Column 1: 13X molecular sieve, 3.2 mm (1/8 in.) by 3.2 mm (1/8 in.) ss, or equivalent.

Column 2: Silica gel (saturated) 4.6 m (15 ft) by 3.2 mm ss, or equivalent.

4.2.2.2 *Carrier Flow:* 50 mL/min helium.

4.2.2.3 *Sample Volume:* 5 mL.

4.2.2.4 *Column Temperature:* 40°C

4.2.2.5 *Sample Flow:* 150 mL/min.

4.2.2.6 *Air Pressure and Flow:* As specified by the instrument manufacturer's instructions.

4.2.2.7 *Hydrogen Pressure and Flow:* As specified by the instrument manufacturer's instructions.

4.2.3 *Calibration Standards* — 5–10 ppm (mole/mole) methane in nitrogen, 5–10 ppm (mole/mole) ethane, 5–10 ppm (mole/mole) propane in nitrogen.

4.2.4 *Operating Procedure*

4.2.4.1 Install the column switching as shown in Figure 1. Place the dual column switching valve in the molecular sieve column position for methane analysis. Use the silica gel column position for the analysis of C₂ – C₃ compounds.

4.2.4.2 Ignite the flame following the instrument manufacturer's instructions. Allow the system to stabilize for 15 minutes. (This is also indicated by a stable signal output.)

4.2.4.3 Inject the methane in nitrogen standard onto the molecular sieve column to determine the retention time for methane.

4.2.4.4 Inject the ethane and propane in nitrogen standard onto the silica gel column to determine the retention times for these compounds.

4.2.4.5 Inject the calibration standard onto the desired column to determine the peak area for the compound of interest. Record the retention times and peak areas.

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

4.2.4.7 Repeat Section 4.2.4.6.