

SEMI C3.2-0301

SPECIFICATION FOR ARSINE (AsH₃) IN CYLINDERS, 99.94% QUALITY

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1 Description

1.1 Arsine is a highly toxic, flammable, colorless gas with a disagreeable garlic-like odor. It is a hemolytic poison.

2 Specifications

QUALITY: 99.94%

Impurities	Maximum Acceptable Level (ppm) (See NOTE 1.)
Carbon monoxide and carbon dioxide (CO + CO ₂)	2
Hydrocarbons (methane (CH ₄), ethane (C ₂ H ₆), ethylene (C ₂ H ₄), and acetylene (C ₂ H ₂))	1
Hydrogen (H ₂)	500
Hydrogen Sulfide (H ₂ S)	1
Nitrogen (N ₂)	10
Oxygen (O ₂) + Argon (Ar)	5
Phosphine (PH ₃)	10
Water (H ₂ O)	4
TOTAL LISTED IMPURITIES	533

Metals — See NOTE 2.

Particles — See NOTE 2.

NOTE 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 procedure.

NOTE 2: To be determined between supplier and user.

3 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	77.946	77.946
Boiling point at 1 atm	-62.48°C	-80.46°F
Density of gas at 20°C (68°F) and 1 atm	3.243 kg/m ³	0.202 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	2.695	2.695
Density of liquid at B.P.	1653 kg/m ³	103.96 lb/ft ³

4 Analytical Procedures

4.1 Carbon Monoxide, Carbon Dioxide, and Hydrocarbons (methane, ethane, ethylene, acetylene) — This procedure is for the determination of carbon monoxide, carbon dioxide, and hydrocarbons in arsine using a gas chromatograph with a methanizer and a flame ionization detector. (See Note 1 and Figure 1.)

4.1.1 Detection Limit — 0.1 ppm each impurity.

4.1.2 Instrument Parameters

4.1.2.1 Column: Porapak QS, 3.5 m (12 ft) by 2 mm ID (1/8 in OD) ss or equivalent.

4.1.2.2 Carrier Flow: 35 mL/min helium.

4.1.2.3 Support Gases: Set the flow rates as specified by the instrument manufacturer.

Hydrogen:	30 mL/min added to the carrier gas between the column outlet and the methanizer inlet.
Air:	500 mL/min.

4.1.2.4 Temperatures:

Detector	110°C
Injector	40°C
Oven	40°C
Methanizer	370–400°C

4.1.2.5 Sample Volume: 3 mL.

4.1.3 Calibration Standards — 1–10 ppm each: carbon monoxide, carbon dioxide, methane, ethane, ethylene, and acetylene; balance helium.

4.1.4 Operating Procedure

4.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record retention times and peak areas. Order of elution is carbon monoxide, methane, carbon dioxide, acetylene, ethylene, ethane.

4.1.4.2 Inject the sample to be tested in same manner as the calibration standard. Vent the arsine after the ethane peak. Record the retention times and peak areas.

4.1.4.3 Repeat 4.1.4.1.

4.1.4.4 Compare the average peak areas of the calibration standard to those of the arsine sample being tested. Calculate the concentration of each impurity, using the formula below. The results may not exceed specifications 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 *Phosphine* — This procedure is for the determination of phosphine in arsine using a gas chromatograph with a thermal conductivity detector.

4.2.1 *Detection Limit* — 5 ppm (mol/mol).

4.2.2 *Instrument Parameters*

4.2.2.1 Column: Porapak QS, 3.5 m (12 ft) by 2 mm ID (1/8 in OD) ss or equivalent.

4.2.2.2 Carrier Flow: 35 mL/min helium.

4.2.2.3 Temperatures:

Detector	80°C
Injector	40°C
Oven	40°C

4.2.2.4 Sample Volume: 3 mL.

4.2.3 *Calibration Standard* — 10–50 ppm (mol/mol) phosphine, balance helium.

4.2.4 *Operating Procedure*

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

4.2.4.2 Inject the sample being tested in same manner as the calibration standard. Record the retention time and peak area.

4.2.4.3 Repeat 4.2.4.1.

4.2.4.4 Compare the average peak area of the calibration standard to that of the arsine sample being tested. Calculate the concentration of phosphine, using the formula below. The result may not exceed the specification in Section 2 of this Standard.

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

4.3 *Hydrogen, Nitrogen and Oxygen + Argon* — This procedure is for the determination of hydrogen, nitrogen, oxygen + argon in arsine using a gas chromatograph with a helium ionization detector.

4.3.1 *Detection Limits* — 100 ppb (mol/mol) hydrogen, 100 ppb oxygen + argon, 500 ppb nitrogen.

4.3.2 *Instrument Parameters*

4.3.2.1 Columns:

Column 1:	Porapak QS, 2.5 m (8 ft) by 3.2 mm (1/8 in) ID ss or equivalent
Column 2:	Molecular Sieve 5A, 3 m (10 ft) by 3.2 mm ID ss or equivalent.

4.3.2.2 Carrier Flow: 25 mL/min helium.

4.3.2.3 Temperatures:

Detector	10°C
Injector	30°C
Oven	30°C

4.3.2.4 Sample Volume: 3 mL.

4.3.3 *Calibration Standard* — 1–10 ppm nitrogen, 1–10 ppm oxygen, 1–10 ppm argon, 10–100 ppm hydrogen, balance helium.

4.3.4 *Operating Procedure*

4.3.4.1 Determination of the backflush time: Inject a methane sample (1–1000 ppm, balance helium) using a 10 port gas valve and backflush at different times. Select the backflush time so that the methane peak is split by the backflush.

4.3.4.2 Inject the calibration standard into the column using the same gas valve. Backflush at the time determined in 4.3.4.1 and record retention times and peak areas. Order of elution is hydrogen, oxygen + argon and nitrogen.

4.3.4.3 Inject the sample being tested in same manner as the calibration standard. Record the retention times and peak areas.

4.3.4.4 Repeat 4.3.4.2.

4.3.4.5 Compare the average peak areas of the calibration standard to those of the arsine sample being tested. Calculate the concentration of each impurity, using the formula below. The results may not exceed the specifications in Section 2 of this Standard.

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

4.4 *Hydrogen Sulfide* — This procedure is for the determination of hydrogen sulfide in arsine using gas chromatograph with a flame photometric detector.

4.4.1 *Detection Limit* — 0.1 ppm (mol/mol).