

SEMI C3.47-1101

SPECIFICATION FOR HYDROGEN BROMIDE (HBr), 99.98% QUALITY

This specification was technically approved by the Global Gases Committee and is the direct responsibility of the North American Gases Committee. Current edition approved by the North American Regional Standards Committee on August 27, 2001. Initially available at www.semi.org September 2001; to be published November 2001. Originally published in 1993; previously published in 1995.

1 Description

1.1 Hydrogen Bromide is an irritating and corrosive gas with a sharp, penetrating, suffocating odor. It is colorless and nonflammable, but fumes in moist air.

2 Specifications

QUALITY: 99.98%, ASSAY (Total Acidity): 98% min

Impurities	Maximum Impurities Acceptable (ppm)*
Carbon Dioxide (CO ₂)	50
Carbon Monoxide (CO)	5
Hydrogen Chloride (HCl)	**
Methane (CH ₄)	15
Oxygen (O ₂) + Nitrogen (N ₂)	50
Water (H ₂ O) (v/v)	5
TOTAL LISTED IMPURITIES (excluding metals)	125

Impurities	Maximum Impurities Acceptable (ppm)*
Iron (Fe)	1 (by weight, vapor phase)

* 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.

** To be analyzed and reported (typically 500 ppm).

3 Physical Constants (for information only)

	Metric Units	U.S. Units
Molecular Weight	80.92	80.92
Boiling Point at 1 atm	-66.7°C	-88.1°F
Density of gas at 70°F (21.1°C) and 1 atm	3.333 g/L	0.208 lb/ft ³
Specific gravity of gas	2.7	2.7
Critical Pressure	85.16 bar	1234.8 psia
Critical Temperature	90°C	193.6°F

4 Analytical Procedures

4.1 *Nitrogen, Oxygen, Carbon Monoxide, Methane, and Carbon Dioxide* — This procedure is for the determination of nitrogen, oxygen + argon, carbon monoxide, methane, and carbon dioxide using a gas chromatograph with a discharge ionization detector.

4.1.1 Detection Limits

- N₂ < 10 ppb
- O₂ + Ar < 20 ppb
- CO < 20 ppb
- CH₄ < 10 ppb
- CO₂ < 10 ppb

4.1.2 *Instrument Parameters* — All components are analyzed using two columns and two detectors with a single injection. (See Figure 1.)

4.1.2.1 Columns:

- HayeSep Db 2.0 m (6 ft.) by 3.2 mm (1/8") O.D. by 2.2 mm (0.085") I.D.
- Molecular Sieve 5A 2.0 m (6 ft.) by 3.2 mm (1/8") O.D. by 2.2 mm (0.085") I.D. ss or equivalent (60/80 mesh)

4.1.2.2 *Carrier Flow* — 40 mL/min He

4.1.2.3 *Sample Volume* — 1.0 mL

4.1.2.4 *Temperatures:*

Detector	100°C
Column 1	40°C
Column 2	100°C

4.1.3 *Calibration Standards* — 5 ppm of each compound of interest including nitrogen, oxygen, carbon monoxide, methane, and carbon dioxide in helium.

4.1.4 Operating Procedures

4.1.4.1 Inject a sample of the calibration standard into the HayeSep column using a gas sample valve. Allow the effluent of HayeSep column to enter the Molecular Sieve column until after CO has eluted. Switch the switching valve to allow the methane and CO₂ to elute directly into detector A while oxygen, nitrogen, and CO separate on the molecular sieve column and elute into detector B. Record retention times and peak areas for all peaks. The order of elution from the HayeSep column to detector A is methane and CO₂. The order of elution on the molecular sieve column to detector B is oxygen, nitrogen, and CO.

4.1.4.2 Analyze the HBr sample to be tested in the same manner.

4.1.4.3 Analyze the calibration standard again.

4.1.4.4 Compare the average peak area of the calibration standard to that of the HBr sample being tested. Calculate the concentration of impurities, 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 *Water* — This procedure is for the determination of trace moisture (water) in hydrogen bromide gas using a continuous flowing, cooled surface condensation point hygrometer.

NOTE 1: The National Institute of Standards and Technology (NIST) provides calibration services for the thermometer used in condensation point hygrometers.

NOTE 2: This method is not applicable if other constituents in the gas will condense before the moisture condensation i.e., excessive carbon dioxide and/or oil contamination.

NOTE 3: The sampling system and hygrometer must be designed to operate under the sample pressure, or the sample pressure must be reduced to accommodate the pressure restrictions of the analytical hygrometer using a regulator with a stainless steel diaphragm.

4.2.1 *Detection Limit* — 2.0 ppm.

4.2.2 *Flow Requirements:*

4.2.2.1 Sample flow rate and pressure will be set in accordance with the instrument manufacturer's instructions.

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

4.2.3 *Calibration*

4.2.3.1 A calibration thermometer designed to indicate temperatures in the -89°C (-121°F) range is required.

4.2.3.2 A calibration curve representing water added to dried hydrogen bromide vs condensation point is required. This can be obtained by preparing a standard of water balance nitrogen and mixing it with dry hydrogen bromide using a dynamic gas blender with mass flow controllers or calibrated flow meters. The hydrogen bromide gas can be dried by passing it over an activated molecular sieve (5A) or magnesium perchlorate.

4.2.4 *Operating Procedures*

4.2.4.1 Obtain a continuous flow of sample gas from the source using a clean stainless steel sampling line

which has been purged dry after exposure to ambient moisture.

4.2.4.2 After prepurging with a dry gas, allow the sample gas to flow through the sampling system and the condensation point hygrometer for one-quarter hour to two hours to allow the entire system to reach equilibrium with regard to moisture content.

4.2.4.3 After equilibrium has been reached, cool the mirror to determine the actual condensation point of the sample. Follow the manufacturer's recommendations to create the temperatures needed.

4.2.4.4 Continue to verify the condensation point for at least 30 minutes after a stable reading has been confirmed.

4.2.4.5 Correct the condensation point reading from the measured pressure to 1 atm of pressure. The result may not exceed the specification in Section 2 of this standard.

4.3 *Iron* — This procedure is for the determination of iron in gaseous hydrogen bromide and analyzed by inductively coupled plasma.

4.3.1 *Sampling Apparatus*

- 3 Teflon diaphragm valves (1/4" FNPT) (Fluoroware 201-10 or equivalent)
- 2 Teflon tees (1/4") (Fluoroware UT4FN-1 or equivalent)
- 50 ft. Teflon tubing (1/4") (Fluoroware AT250-047 or equivalent)
- Teflon flow meter (1/4" FNPT calibrated for HBr flow) (Fluoroware FM-4N-1P or equivalent)
- 8 Teflon connectors (1/4" tube to 1/4" MNPT) (Fluoroware C4-CFN-1 or equivalent)
- 9 gas washing bottles (375 mL) (Saville #507 or equivalent with 501-4-2 cap)
- Tee purge assembly (Matheson 4756-330 or equivalent)
- Manual control valve (Matheson 55A-330 or equivalent)
- Cylinder scale that can measure the mass of the cylinder to ± 1 g accuracy.

4.3.2 *Water* — 18 megaohm-cm DI water is used for all dilution blanks and washings.

4.3.3 *Reagents* — All reagents are of the highest purity available minimizing background metal contamination.