

SEMI C3.35-1109

SPECIFICATION FOR HYDROGEN CHLORIDE (HCl), 99.997% QUALITY

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1 Purpose

1.1 The purpose of this document is to provide specification for hydrogen chloride (HCl) that is used in the semiconductor industry.

2 Scope

2.1 This document covers requirements for hydrogen chloride used in the semiconductor industry.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard 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 None.

4 Description

4.1 Hydrogen chloride is a colorless, pungent, corrosive gas having a suffocating odor. Hydrogen chloride is heavier than air and fumes strongly in moist air. It is very soluble in water and alcohol, and also soluble in ether. While in the cylinder under pressure, hydrogen chloride is in the form of a gas over liquid. As long as liquid is present in the cylinder, the pressure will remain fairly constant. When the liquid phase is exhausted, the cylinder pressure will drop rather rapidly.

5 Specifications

5.1 Quality: 99.997% (see Notes 1, 2, and 3)

Impurities	Maximum Acceptable Level (ppm) ^{#1}
Carbon dioxide (CO ₂)	10
Hydrogen (H ₂)	5
Nitrogen (N ₂)	10
Oxygen (O ₂) + Argon (Ar)	1
Methane and Acetylene	1
Water (H ₂ O) (v/v)	1
TOTAL LISTED IMPURITIES (excluding metals)	28
Iron (Fe)	1 wt. vapor phase

#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 procedure provided.

NOTE 1: Due to the reactive nature of the material and the impurities, this product should be used within a period which may be specified by the supplier.

NOTE 2: It is recommended that the user discontinue the use of a cylinder prior to complete consumption of the liquid phase. The content of cylinders should be determined by weight, not pressure.

NOTE 3: This specification applies to the gas phase of the cylinder as received.

6 Physical Constants (for information only)

	Metric Units	US Units
Molecular Formula	HCl	HCl
CAS Number	7647-01-0	7647-01-0
Molecular weight	36.465	36.465
Boiling point at 1 atm	-85.0°C	-121.0°F
Density of gas at 21.1°C (70°F) and 1 atm	1.522 kg/m ³	0.095 lb/ft ³
Specific gravity of gas at 0°C (32°F) and 1 atm	1.268	1.268

7 Analytical Procedures

7.1 *Carbon Dioxide* — This procedure is for the determination of carbon dioxide in hydrogen chloride using a gas chromatograph with a thermal conductivity detector. A backflush is used to keep the main component from reaching the detector.

7.1.1 *Detection Limit* — 2 ppm.

7.1.2 *Instrument Parameters*

7.1.2.1 *Column* — Porapak™ Q, 2.4 m (8 ft.) by 6.4 mm (1/4 in.) OD ss or equivalent.

7.1.2.2 *Carrier Flow* — 40 mL/min helium.

7.1.2.3 *Temperatures*

- *Detector* — 50°C
- *Column Oven* — 40°C

7.1.2.4 *Sample Volume* — 10 mL.

7.1.3 *Calibration Standard* — 100 ppm carbon dioxide in helium.

7.1.4 *Operating Procedure*

7.1.4.1 To determine the backflush time, inject the calibration standard, using a gas sampling valve. Record the retention time for carbon dioxide. Choose the backflush time so that the complete carbon dioxide peak is eluted.

7.1.4.2 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak areas. Backflush at the time determined in ¶ 7.1.4.1.

7.1.4.3 Inject the sample to be tested in same manner as the calibration standard. Record the retention times and peak areas. Backflush the main component at the time determined in ¶ 7.1.4.1.

7.1.4.4 Repeat ¶ 7.1.4.2.

7.1.4.5 Compare the average peak area of the calibration standard to that of the hydrogen chloride sample being tested. Calculate the concentration of carbon dioxide, using the formula below. The result may not exceed the specification in § 5 of this Standard.

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

7.2 *Hydrogen* — This procedure is for the determination of hydrogen in hydrogen chloride using a gas chromatograph with a thermal conductivity detector.

7.2.1 *Detection Limit* — 3 ppm.

7.2.2 Instrument Parameters

7.2.2.1 Columns

- Column 1 — Ascarite®, 0.3 m (1 ft.) by 9.6 mm (3/8 in.) OD ss or equivalent.
- Column 2 — Molecular Sieve 5A, 2.4 m (8 ft.) by 6.4 mm (1/4 in.) OD ss or equivalent.

7.2.2.2 Carrier Flow — 40 mL/min nitrogen.

7.2.2.3 Temperatures

- Detector — 50°C
- Column Oven — 40°C

7.2.2.4 Sample Volume — 10 mL.

7.2.3 Calibration Standard — 50 ppm hydrogen in nitrogen.

7.2.4 Operating Procedure

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

7.2.4.2 Inject the sample to be tested in same manner as the calibration standard. Record the retention time and peak area.

7.2.4.3 Repeat ¶ 7.2.4.1.

7.2.4.4 Compare the average peak area of the calibration standard with that of the hydrogen chloride sample being tested. Calculate the concentration of hydrogen using the formula below. The result may not exceed the specification in § 5 of this standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \text{Concentration of Standard} = \text{Concentration of Sample} \quad (2)$$

7.3 Iron — This procedure is for the determination of iron sampled from gaseous hydrogen chloride and analyzed by inductively coupled plasma.

7.3.1 Sampling Apparatus

- 3 Teflon® diaphragm valves (1/4 in. FNPT) (Fluoroware 201-10 or equivalent).
- 2 Teflon® tees (1/4 in.) (Fluoroware UT4FN-1 or equivalent).
- 50 ft. Teflon® tubing (1/4 in.) (Fluoroware AT250-047 or equivalent).
- Teflon® flow meter (1/4 in. FNPT calibrated for HCl flow) (Fluoroware FM-4N-1P or equivalent).
- 8 Teflon® connectors (1/4 in. tube to 1/4 in. 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.

7.3.2 Water — 18 megohm-cm DI water is used for all dilution blanks and washings.

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

7.3.4 Sampling Gaseous Hydrogen Chloride

7.3.4.1 Assemble entire sampling apparatus (see Figure 1) in laboratory hood for safety.

7.3.4.2 Fill gas washing bottles (scrubbers) with 75 mL 18 mega-ohm DI water.

7.3.4.3 Start nitrogen purge to remove air from system and to check for leaks.

7.3.4.4 Close V4 and close nitrogen purge.

7.3.4.5 Close V2.