SEMI C3.32-0819
SPECIFICATION FOR CHLORINE (Cl_2), 99.996% QUALITY

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1 Purpose

1.1 The purpose of this Document is to provide specification for chlorine (Cl_2) that is used in the semiconductor industry.

2 Scope

2.1 This Document covers requirements for chlorine 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 None.

4 Description

4.1 Gaseous chlorine is greenish-yellow and about 2.5 times as heavy as air. Chlorine has a disagreeable and suffocating odor.

5 Requirements

5.1 Quality: 99.996%

Table 1 Impurity Requirements for Chlorine

Purity	*99.996%*
Impurities	*Maximum Acceptable Level (ppm)*[#1]
Carbon dioxide (CO2)	10
Carbon monoxide (CO)	1
Hydrocarbons (C1 – C2)	1
Nitrogen (N2)	20
Oxygen (O2)	4
Water (H2O) (v/v)	See [#2]
TOTAL LISTED IMPURITIES (excluding chromium, iron, nickel, and sodium)	36
Chromium (Cr)	0.2 by wt. Liquid Phase
Iron (Fe)	0.2 by wt. Liquid Phase
Nickel	0.2 by wt. Liquid Phase
Sodium	1 by wt. Liquid Phase

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#1 An analysis of significant figures has not been considered. The number of significant figures will be based on analytical accuracy and on the precision of the provided procedure.

#2 It is not known whether H_2O remains as H_2O in Cl_2 or reacts to other species. For this reason, interpretation of H_2O measurement data is questionable. Test and acceptance criteria shall be determined between user and supplier. Possible methods include: a) determination of HCl by Fourier transform infrared spectrometry (FTIR), mass spectrometry, or gas chromatography; b) measurement of water by electrolysis in a P_2O_5 cell (reported in terms of H_2O equivalents); c) determination of water by FTIR.

6 Properties of Chlorine (for information only)

Table 2 Properties of Chlorine

	Metric Units	*US Units*
Chemical Formula	Cl_2	Cl_2
CAS Number	7782-50-5	7782-50-5
Molecular weight	70.9	70.9
Boiling point at 1 atm	−34.05°C	−29.3°F
Density gas at 20°C (68°F) and 1 atm	2.980 kg/m^3	0.1860 lb/ft^3
Specific gravity	2.473	2.473
Density liquid at −118°C (−180.8°F)	1574.8 kg/m^3	98.26 lb/ft^3

7 Test Method for Determining Impurity Limits

7.1 *Carbon Monoxide, Carbon Dioxide, and Hydrocarbons (CH_4, C_2H_2, C_2H_4, C_2H_6)* — This method is for the determination of carbon monoxide, carbon dioxide, and C_1–C_2 hydrocarbons in chlorine. The sample shall be vapor phase and analyzed using a gas chromatograph with a flame ionization detector/methanizer combination. (See Figure 1 and Notes 1, 2, 3 and 9.)

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

7.1.2 *Instrument Parameters*

7.1.2.1 *Injection Valve* — 10 port corrosion-resistant.

7.1.2.2 Sample Volume — 2 mL.

7.1.2.3 *Columns*

- *Column 1* — Porapak™ P, 3.1 m (10 ft.) by 3.2 mm (1/8 in.) OD ss or equivalent.
- *Column 2* — Porapak™ P, 3.1 m (10 ft.) by 3.2 mm (1/8 in.) OD ss or equivalent.

7.1.2.4 *Carrier Flow* — 20 mL/min nitrogen.

7.1.2.5 *Column Temperature* — 40°C.

7.1.2.6 *Air and Hydrogen Pressure and Flow* — As specified by the instrument manufacturer.

7.1.3 *Calibration Standard* — 1 to 5 ppm (mol/mol) each component in nitrogen.

7.1.4 *Operating Procedure*

7.1.4.1 Place the 10 port valve in the sample load position.

7.1.4.2 Turn on the methanizer heater and establish hydrogen flow. Allow unit to heat to operating temperature (as specified by the manufacturer).

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7.1.4.3 Establish air flow and ignite burner following the instrument manufacturer's instructions. Allow the system to stabilize for 15 minutes.

7.1.4.4 Determine the time at which ethane elutes from the first column and absorbs onto the second column so one can vent the chlorine. Inject the calibration ethane standard and record the retention time of ethane obtained without returning the valve to the load position. Multiply this time by 0.6. The result will be the time during the analysis at which the injection valve should be returned to the load position, backflushing the chlorine to vent. Repeat the injection of the ethane calibration standard, including the backflush, to ensure that enough time has passed to allow elution of ethane onto the second column.

7.1.4.5 Inject the remaining standards using the backflush technique. Record the retention times and peak areas.

7.1.4.6 Analyze the chlorine sample to be tested in the same manner as in ¶ 7.1.4.5. Repeat the sample injection until peak areas of the impurity of interest agree within 5%.

7.1.4.7 Analyze each of the calibration standards again as in ¶ 7.1.4.5.

7.1.4.8 Compare the average peak areas of the calibration standards to those of the chlorine sample being tested. Calculate the concentration of each impurity, using the formula below. The results may not exceed the specifications in § 5 of this Standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \begin{matrix}\text{Concentration}\\ \text{of Standard}\end{matrix} = \begin{matrix}\text{Concentration}\\ \text{of Sample}\end{matrix} \tag{1}$$

7.2 *Oxygen and Nitrogen* — This method is for the determination of oxygen and nitrogen in chlorine. The sample shall be vapor phase and analyzed using a gas chromatograph with a thermal conductivity detector. (See Figure 2 and Notes 1 and 2.)

7.2.1 *Detection Limits* — 2 ppm (v/v) oxygen, 5 ppm (v/v) nitrogen.

7.2.2 *Instrument Parameters*

7.2.2.1 *Injection Valve* — 10 port corrosion-resistant.

7.2.2.2 Sample Volume — 2 mL.

7.2.2.3 *Columns*

- *Column 1* — Porapak™ Q, 1.8 m (6 ft.) by 3.2 mm (1/8 in.) OD ss or equivalent.
- *Column 2* — Molecular Sieve 13X, 3.1 m (10ft.) by 3.2 mm (1/8 in.) OD ss or equivalent.

7.2.2.4 *Carrier Flow* — 20 mL/min helium.

7.2.2.5 *Temperature*

- *Column* — 30°C
- *Detector* — 50°C

7.2.3 *Calibration Standard* — 10 to 40 ppm (v/v) each component in helium.

7.2.4 *Operating Procedure*

7.2.4.1 Place the 10 port valve in the sample load position.

7.2.4.2 Inject the standard, wait one minute, then return the valve to the sample load position to backflush the principal gas to vent while allowing the oxygen and nitrogen to pass to the analytical column. Record the retention times and peak areas.

7.2.4.3 Inject the standard at least three times. All peak areas for a specific impurity should agree within 5%.

7.2.4.4 Inject the chlorine sample to be tested in the same manner as in ¶¶ 7.2.4.2 and 7.2.4.3. Record the retention times and peak areas.

7.2.4.5 Repeat ¶¶ 7.2.4.2 and 7.2.4.3.

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