

SEMI C3.55-0200

STANDARD FOR SILANE (SiH₄), BULK

This standard 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 December 15, 1999. Initially available at www.semi.org January 2000; to be published February 2000.

1 Description

1.1 Silane is a pyrophoric, flammable colorless gas.

2 Specifications

QUALITY: 99.994%

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)</i>
<i>Gas Phase</i>	
Carbon Monoxide (CO)	0.1
Carbon Dioxide (CO ₂)	0.1
Chlorosilanes (ionizable chlorides) including HCl, reported as chloride (Cl ⁻)	1
Non-methane Hydrocarbons (C ₂ -C ₄)	0.1 total
Hydrogen (H ₂)	50
Methane (CH ₄)	0.1
Nitrogen (N ₂)	1
Argon (Ar)	1
Disiloxane (H ₃ SiOSiH ₃)	1
Methyl Silane (SiH ₃ -CH ₃)	1
Disilane (Si ₂ H ₆)	1
Water (H ₂ O)(vol/vol)	1
Particles	(See NOTE 1.)
Total Specified Impurities	57.4
<i>Deposited Layer</i>	
Carbon (C)	0.5 ppma (See NOTE 2.)
Oxygen (O)	1 ppma (See NOTE 2.)
Aluminum (Al)	0.2 ppba (See NOTE 2.)
Antimony (Sb)	0.2 ppba (See NOTE 3.)
Arsenic (As)	0.2 ppba (See NOTE 3.)
Boron (B)	0.2 ppba (See NOTE 3.)
Gallium (Ga)	0.2 ppba (See NOTE 3.)
Phosphorus (P)	0.2 ppba (See NOTE 3.)
Chromium (Cr)+ Copper (Cu)+ Iron (Fe)+ Nickel (Ni)+ Zinc (Zn)	1.0 ppba total (See NOTE 3.)

NOTE 1: To be determined between supplier and user.

NOTE 2: ppma is defined to be atoms of impurity per 10⁶ atoms of silicon.

NOTE 3: ppba is defined to be atoms of impurity per 10⁹ atoms of silicon.

3 Electrical Specification

Resistivity	greater than 2,000 ohm-cm (n-type)
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4 Physical Constants

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	32.112	32.112
Boiling point at 1 atm.	-112°C	-169°F
Density of gas at 21.1°C (70°F) and 1 atm.	1.342 kg/m ³	0.0839 lb/ft ³
Specific gravity of gas at 21.1°C and 1 atm (air = 1)	1.114	1.114
Density of liquid at boiling point	711 kg/m ³	44.39 lb/ft ³

5 Analytical Procedures (see NOTE 1)

5.1 *Carbon Monoxide, Carbon Dioxide, Argon, Nitrogen and Methane* — This procedure is for the determination of carbon monoxide, carbon dioxide, argon, nitrogen and methane in silane using a gas chromatograph with a discharge ionization detector.

5.1.1 *Detection Limit* — 30 ppb (mole/mole)

5.1.2 *Instrument Parameters*

5.1.2.1 *Column:*

HAYESEP D 60/80, 6.5 m (21.3 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID stainless steel; or equivalent.

5.1.2.2 *Carrier Flow* — 25 mL/min helium

5.1.2.3 *Sample Volume* — 1 mL

5.1.2.4 *Temperatures:*

Detector	140°C
Column Oven	40°C for 4 min., to 120°C at 10°C/min., hold 120°C for 8 min.
Injection Temperature	45°C

5.1.3 *Calibration Standards* — 1 ppm (mole/mole) for all components to be tested, balance helium.

5.1.4 Operating Procedures

5.1.4.1 Inject the calibration standard and sample as described in Section 6.6 of SEMI C3. The order of elution is nitrogen, argon, carbon monoxide, methane, and carbon dioxide. (See NOTE 2.)

5.1.4.2 Compare the average peak area of the calibration standard to that of the silane sample being tested. Calculate the concentrations of the analytes as described in Section 6.8 of SEMI C3. The result shall be reported in the unit of concentration needed and to the number of significant digits of the variable with the least number of significant digits. This value may not exceed that specified in Section 2.

5.2 *Total Chlorides* — This procedure is for the determination of total chlorides in silane by titration of a hydrolyzed sample of silane. (See NOTE 1.)

5.2.1 *Detection Limit* — 0.5 ppm (mole/mole)

5.2.2 *Instrument Parameters* — See Figure 1.

5.2.2.1 Equipment

5.2.2.1.1 Safety purge regulator with proper fittings.

5.2.2.1.2 Flow meter capable of measuring 0.25 standard liters per minute (0.5 SCF/hour) (Brooks or equivalent), calibrated for silane.

5.2.2.1.3 One cylinder of nitrogen with regulator.

5.2.2.1.4 One ice bath.

5.2.2.1.5 Three magnetic stirring bars.

5.2.2.1.6 Two 2000 mL heavy-duty sidearm flasks (Fisher Cat. No. 10-181G or equivalent).

5.2.2.1.7 One #9 one-hole rubber stopper.

5.2.2.1.8 One #9 two-hole rubber stopper.

5.2.2.1.9 Two Pyrex-brand tubes with fritted cylinders (Fisher Cat. No. 11-138B or equivalent).

5.2.2.1.10 One gas washing bottle with fritted cylinder, 125 mL cap. (Fisher Cat. No. 03-040A or equivalent).

5.2.2.1.11 One buret, 50 mL capacity with Teflon stopcock (Fisher Cat. No. 03-700-22C or equivalent).

5.2.2.1.12 One buret stand (Fisher Cat. No. 14-688 or equivalent).

5.2.2.1.13 Miscellaneous clamps, support stands, and rubber hose.

5.2.2.1.14 Two 250 mL Erlenmeyer flasks.

5.2.2.2 Reagents

5.2.2.2.1 Potassium hydroxide flakes, technical (Fisher Cat. No. P-246 or equivalent).

5.2.2.2.2 Mercuric nitrate crystal (Fisher Cat. No. M-168 or equivalent).

5.2.2.2.3 Sodium chloride crystal (Fisher Cat. No. S-271 or equivalent).

5.2.2.2.4 Diphenyl carbazone (Fisher Cat. No. D-86 or equivalent).

5.2.2.2.5 Bromophenol blue (Fisher Cat. No. B-392 or equivalent).

5.2.2.2.6 Nitric acid (Fisher Cat. No. AA-200 or equivalent).

5.2.2.2.7 Ethanol, denatured (Fisher Cat. No. AA-407 or equivalent).

5.2.3 Operating Procedure

5.2.3.1 Assemble apparatus as shown in Figure 1 and purge entire system with nitrogen for 30 minutes.

5.2.3.2 Fill each 2000 mL sidearm flask with about 1700 mL of 15% potassium hydroxide solution (15 g KOH per each 100 mL water).

5.2.3.3 Fill the gas washing bottle with about 75 mL of deionized water that has been degassed.

5.2.3.4 Pass exactly one cubic foot of silane through the system at a rate of 0.5 cubic feet per hour.

5.2.3.5 Stop silane flow and purge system for 30 minutes.

5.2.3.6 Remove gas washing bottle from system and transfer contents quantitatively and with the aid of three 25 mL deionized water washings, to a 250 mL Erlenmeyer flask.

5.2.3.7 Add a few drops of indicator solution, 5 g diphenyl carbazone plus 0.5 g bromophenol blue dissolved in 750 mL ethanol, plus 250 mL deionized water.

5.2.3.8 Add, in a drop-wise fashion, sufficient 0.2 N HNO_3 (13 mL concentrated. HNO_3 , diluted to 1 L in deionized water) in order to just turn the solution to yellow from purple.

5.2.3.9 Titrate with a solution of mercuric nitrate $\text{Hg}(\text{NO}_3)_2$, (diluted to 1000 mL in deionized water; then 50 mL diluted to 500 mL in deionized water) which has been previously standardized to 5.0 mL of a sodium chloride solution (approximately 165 ng NaCl, accurately weighed, dissolved in 100 mL deionized water).