

SEMI C3.34-92

STANDARD FOR DISILANE (Si₂H₆)

1 Description

Disilane is a pyrophoric, highly flammable, noncorrosive, colorless gas. It is toxic and a powerful irritant.

2 Specifications

QUALITY: 97% (See Note 1.)

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)*</i>
Argon (Ar) + Oxygen (O ₂)	1
Carbon dioxide (CO ₂)	1
Carbon monoxide (CO)	1
Chlorosilanes (Total hydrolyzable chlorides, reported as (Cl-))	5
Chromium (Cr)	**
Disiloxane (H ₃ SiOSiH ₃)	5
Hydrogen (H ₂)	500
Iron (Fe)	**
Monoethylsilane	50
Nickel (Ni)	**
Nitrogen (N ₂)	10
Silane (SiH ₄)	2.5%
Total methane, ethane, propane	10
Total trisilane and tetrasilane	500
Water (H ₂ O)	1
TOTAL SPECIFIED IMPURITIES (excluding chloride, chromium, iron and nickel)	2.6082%

* 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 determined between supplier and user.

Note 1: This specification applies to the gas phase of the cylinder as received.

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	62.22	62.22
Boiling point at 1 atm	-14.8°C	5.4°C
Freezing point	-132.5°C	-206.5°C
Vapor density (air = 1)	2.38	2.38

4 Analytical Procedures

4.1 *Carbon Monoxide and Carbon Dioxide* — This procedure is for the determination of carbon monoxide and carbon dioxide in disilane using a gas chromatograph with a methanizer and a flame ionization detector. (See Figure 1, Notes 1 and 2.)

4.1.1 *Detection Limit* — 0.1 ppm for each impurity.

4.1.2 *Instrument Parameters*

4.1.2.1 Column: Porapak QS 3 m (10 ft) by 1.6 mm (1/16 in) ID ss or equivalent.

4.1.2.2 Carrier Flow: 30 mL/min helium.

4.1.2.3 Sample Volume: 1.5 mL.

4.1.2.4 Support Gases: As specified by the instrument manufacturer.

Hydrogen: 20–30 mL per min added to the carrier gas between the column outlet and the methanizer inlet.

Air: 500 mL/min.

4.1.2.5 Temperatures:

Detector	110°C
Injector	60°C
Oven	60°C
Methanizer	370° – 400°C

4.1.3 *Calibration Standards* — 1–10 ppm carbon monoxide, 1–10 ppm carbon dioxide, balance helium.

4.1.4 *Operating Procedure*

4.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak areas. The order of elution is carbon monoxide, carbon dioxide. Repeat this operation.

4.1.4.2 Inject the sample to be tested in same manner as the calibration standard and vent after elution of carbon dioxide is completed, in case any silane is present. 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 that of the disilane sample being tested. Calculate the concentrations of carbon monoxide and carbon dioxide using the formula below. The results may not exceed the specifications in Section 2 of this Standard.

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

4.2 *Methane, Ethane, Propane, and Silane* — This procedure is for the determination of hydrocarbons (methane, ethane, and propane) and silane in disilane using a gas chromatograph with a thermal conductivity detector.

4.2.1 *Detection Limit* — 3 ppm for each impurity.

4.2.2 *Instrument Parameters*

4.2.2.1 Column: Porapak QS 3 m (10 ft) by 1.6 mm (1/16 in) ID ss or equivalent.

4.2.2.2 Carrier Flow: 25 mL/min. Helium.

4.2.2.3 Sample Volume: 1 mL.

4.2.2.4 Temperatures:

Detector	80°C
Injector	60°C
Oven	60°C

4.2.3 *Calibration Standards* — 10–50 ppm methane, 5–10 ppm ethane, 5–10 ppm propane, 0.1 – 1% silane, 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 times and peak areas. Order of elution is methane, silane, ethane, propane.

4.2.4.2 Inject the sample to be tested in same manner as the calibration standard. Record the retention times and peak areas.

4.2.4.3 Repeat 4.2.4.1.

4.2.4.4 Compare the average peak areas of the calibration standard to that of the disilane sample being tested. Calculate the concentrations of methane, silane, ethane, and propane, using the formula below. The results 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 Argon* — This procedure is for the determination hydrogen, nitrogen, and argon in disilane using a gas chromatograph with a helium ionization detector.

4.3.1 *Detection Limit* — 100 ppb for hydrogen and argon, 500 ppb for 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) OD ss or equivalent.
Column 2:	5A Molecular sieve, 3 m (10 ft) by 3.2 mm OD ss or equivalent.

4.3.2.2 Carrier Flow: 25 mL/min helium.

4.3.2.3 Sample Volume: 3 mL.

4.3.2.4 Temperatures:

Detector	100°C
Injector	30°C
Oven	30°C

4.3.3 *Calibration Standards* — 1–10 ppm nitrogen, 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 a gas sampling valve. Backflush at the time determined in 4.3.4.1. Record retention times and peak areas. Order of elution is hydrogen, argon, nitrogen.

4.3.4.3 Inject the sample to be tested in same manner as the calibration standard. Backflush at the time determined in 4.3.4.1. 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 that of the disilane sample being tested. Calculate the concentrations of hydrogen, nitrogen and argon 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 *Disiloxane, Monoethylsilane, Trisilane, and Tetrasilanes* — This procedure is for the determination of disiloxane, monoethylsilane, trisilane, and tetrasilanes in disilane using a gas chromatograph with a thermal conductivity detector.

4.4.1 *Detection Limits* — 5 ppm for disiloxane and 10 ppm for each of the other impurities.