

SEMI C3.56-0600

SPECIFICATION FOR DIBORANE MIXTURES

This specification was technically approved by the Global Gases Committee and is the direct responsibility of the European Gases Committee. Current edition approved by the European Regional Standards Committee on April 4, 2000. Initially available at www.semi.org May 2000; to be published June 2000.

1 Purpose

1.1 To define the specification and analytical methods and validate the specifications for diborane mixtures.

2 Scope

2.1 Diborane is not used as a pure gas but as a mixture with balance gases of N₂, H₂, Ar, He, or SiH₄.

2.2 The quality of the mixture is affected by a number of factors which include:

- the purity of the gases used for mixture preparation;
- component filling precision;
- the cylinder preparation process and *in-situ* reactions of diborane with the internal surface of the cylinder.

2.2.1 Furthermore, the elapsed time after filling is important due to the chemical instability, and hence decomposition, of the diborane (see Appendix 1).

2.2.2 All component gases shall adhere to the appropriate SEMI specification, if available.

2.3 The quality of the final mixture can be checked after manufacturing by analyzing each cylinder for the concentration of diborane and for key impurities (tracers), namely H₂, N₂, O₂.

2.4 This specification does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this specification to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

3 Referenced Standards

3.1 DIN Standard¹

DIN 50457-1

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

4 Specifications

4.1 Mixing Tolerances

Concentration of Diborane	Preparation Tolerance Level
10–999 ppm	± 20%
0.1–10%	± 5%

4.2 Analytical precision: Determination of Diborane Content

Concentration of Diborane	Analytical Precision
10–99 ppm	± 3%
100–999 ppm	± 2%
0.1–10%	± 2%

N.B. Analytical precision is based on at least 6 replicate determinations

4.3 Impurities

4.3.1 In the final mixture the concentration of impurities should fulfill the following condition:

$$C_i(\text{mix}) = \left[\frac{X_{(\text{diborane})}}{100} \cdot c_i(\text{diborane}) \right] + \left[\frac{X_{(\text{main})}}{100} \cdot c_i(\text{main}) \right]$$

$c_i(\text{mix})$ — concentration of impurity i in the mixture (ppm)

$c_i(\text{diborane})$ — maximum concentration of impurity i in diborane (ppm)

$c_i(\text{main})$ — maximum concentration of impurity i in the balance gas (ppm)

$X_{(\text{diborane})}$ — concentration of diborane in the mixture (%)

$X_{(\text{main})}$ — concentration of balance gas in the mixture (%)

5 Analytical Procedure for the Determination of Diborane Concentration

NOTE 2: This procedure is for the determination of the diborane content using a Fourier Transform Infrared analyzer.

5.1 Detection limit — 2 ppm (mole/mole)

¹ Deutsches Institut für Normung e.V., Beuth Verlag GmbH, burggrafenstrasse 4-10, D10787 Berlin, Germany, www.din.de

5.2 Instrument parameters

5.2.1 Cell path length

5.2.1.1 10 cm (mixtures 100 ppm to 10%)

5.2.1.2 10 m (mixtures up to 100 ppm)

Wavenumber — 2444–2739 cm^{-1}

Resolution — 4 cm^{-1}

5.2.2 Sample Flow

5.2.2.1 20 l/h at 10 cm path length

5.2.2.2 40 l/h at 10 m path length

5.3 Calibration standard — diborane in hydrogen

NOTE 3: Depending on the concentration of diborane and the storage temperature the standard can be unstable (Section 5). It is recommended to use an appropriate standard with a diborane concentration as low as possible and to store it at low temperature. If there are doubts about the stability of the standard the diborane concentration can be verified by wet chemical methods. (See DIN 50457-1.)

5.4 Operating Procedures

5.4.1 Purge the sampling system leading to the cell and the cell with dry nitrogen for 15 minutes and until a zero absorbance is obtained between 2444–2739 cm^{-1} . Then purge the cell with the calibration standard and record the absorption of diborane. When a steady absorbance reading is obtained, after approximately 5–10 minutes, the true concentration of the standard shall be recorded. Record the pressure in the gas cell using a calibrated pressure-measuring device.

5.4.2 To determine the concentration of diborane in the mixture set the sample flow through the cell following the same procedures as detailed above. Record the absorption at the same wave number as the calibration standard. Ensure that gas cell pressures are equal during calibration and the determination. Calculate the concentration of diborane. The result must conform to the specification in Section 4 of this standard.