

SEMI C3.51-1101 (Withdrawn 0312)

SPECIFICATION FOR BORON TRICHLORIDE (BCl₃), 99.98% QUALITY

This Standard was technically approved by the global Gases Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on December 24, 2011. Available at www.semiviews.org and www.semi.org in March 2012; originally published in 1995; previously published November 2011.

NOTICE: This Document was balloted and approved for withdrawal in 2012; replaced by SEMI C71.

1 Purpose

1.1 To define the specification and analytical methods and validate the specifications for BCl₃.

2 Scope

2.1 This Specification relates to a geometry of 0.5 μ or less (0.35 to 0.5 range) and describes analytical techniques using gas phase analysis.

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 Description

3.1 Boron trichloride is a colorless toxic and corrosive gas at room temperature and atmospheric pressure which fumes in the presence of moist air. It has a choking odor.

4 Referenced Standards and Documents

4.1 ISO Standard¹

ISO 6145-1 — Preparation of Calibration Gas Mixtures – Dynamic Volumetric Methods – Part 1: Methods of Calibration

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Specifications

5.1 Quality: 99.98% vapor phase

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)^{#1}</i>
Nitrogen (N ₂)	5
Carbon Dioxide (CO ₂)	2
Chlorine (Cl ₂)	10
Hydrogen Chloride (HCl)	100
Phosgene (COCl ₂)	1
Silicon Tetrachloride (SiCl ₄)	2
TOTAL LISTED IMPURITIES ^{#1} (excluding metals)	120
Iron (Fe)	0.5 wt.
Nickel (Ni)	0.5 wt.

¹ International Organization for Standardization, ISO Central Secretariat, 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland; Telephone: +41.22.749.01.11, Fax: +41.22.733.34.30, <http://www.iso.org>

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

6 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	117.17	117.17
Boiling point at atm	12.4°C	54.3°F
Density of gas at 20°C and 1 atm	5.326 kg/m ³	0.3325 lb/ft ³
Specific gravity	4.12	4.12
Density of liquid at -18°C	1372.8 kg/m ³	85.76 lb/ft ³

7 Analytical Procedures

NOTE 1: The boiling point of boron trichloride is 12.4°C. It is recommended that, prior to the sampling operation, the cylinder should be allowed to reach room temperature to prevent the possibility of any suck-back.

7.1 *Nitrogen* — This procedure is for the determination of nitrogen in boron trichloride using a gas chromatograph fitted with a backflush valve and a thermal conductivity detector.

7.1.1 *Detection Limits* — 0.5 ppm nitrogen.

7.1.2 *Instrument Parameters*

7.1.2.1 *Columns*

- Column 1: Porapak QS 60/80 mesh 2 m (6 ft.) × 3.2 mm (1/8 in.) OD SS or equivalent.
- Column 2: Molecular sieve 5A 60/80 mesh 2 m (6 ft.) × 3.2 mm (1/8 in.) OD SS or equivalent.

7.1.2.2 *Carrier Gas Flow* — Helium N6.0 Flow 25 mL/min.

NOTE 2: The helium carrier gas should contain less than 10 ppb of the impurities it is desired to measure. This is best achieved by using a commercial helium gas purifier.

7.1.2.3 *Sample Volume* — 2.5 mL.

7.1.2.4 *Temperatures*

- *Column Temperature* — 100°C.
- *Detector Temperature* — 100°C.

7.1.2.5 *Bridge Current* — 200 mA.

7.1.3 *Calibration Standard* — 1 ppm to 10 ppm nitrogen in helium.

7.1.3.1 *Calibration* — Construct a calibration curve in the range of interest. Verify the standards employed independently by established traceability to recognized national or international standards.

7.1.4 *Operating Procedure*

7.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Adjust the backflush valve operation sequence to present the nitrogen. Record the retention time and peak area. A sample chromatogram is shown in Figure 1.

NOTE 3: Introduce the calibration standard as many times as necessary to achieve the desired precision.

7.1.4.2 Inject the sample to be tested in the same manner as the calibration standard. Record the retention time and peak areas (see Note 2).

7.1.4.3 Repeat ¶ 7.1.4.1.

7.1.4.4 Compare the average peak areas of the nitrogen in the calibration standard to that of the boron trichloride sample being tested. Calculate the concentration of nitrogen using the formula below. The results may not exceed the specification in § 5 of this Standard.

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

7.2 *Carbon Dioxide* — This procedure is for the determination of carbon dioxide in boron trichloride using a gas chromatograph fitted with a backflush and a thermal conductivity detector.

7.2.1 *Detection Limits* — 0.5 ppm carbon dioxide.

7.2.2 *Instrument Parameters*

7.2.2.1 *Columns*

- *Column 1* — Porapak QS 60/80 mesh 3m (10 ft.) by 3.2 mm (1/8 in.) OD SS or equivalent.
- *Column 2* — Porapak QS 60/80 mesh 2.5 m (8 ft.) by 3.2 mm (1/8 in.) OD SS or equivalent.

7.2.2.2 *Carrier Gas Flow* — Helium N6.0 flow 25 mL/mm (see Note 3).

7.2.3 *Sample Volume* — 2.5 mL.

7.2.4 *Temperatures*

- *Column Temperature* — 60°C.
- *Detector Temperature* — 110°C.

7.2.5 *Bridge Current* — 200 mA.

7.2.6 *Calibration Standard* — 1 ppm to 5 ppm carbon dioxide in helium.

7.2.6.1 *Calibration* — Construct a calibration curve in the range of interest. Verify the standards employed independently by established traceability to recognized national or international standards.

7.2.7 *Operating Procedure*

7.2.7.1 Inject the calibration standard into the column using a gas sampling valve. Adjust the backflush valve operation sequence to present the carbon dioxide peak. Record the retention time and peak area. A sample chromatogram is shown in Figure 2 (see Note 4).

7.2.7.2 Inject the sample to be tested in the same manner as the calibration standard (see Note 2). Record the retention time and peak areas.

7.2.7.3 Repeat ¶ 7.2.7.1.

7.2.7.4 Compare the average peak areas of the carbon dioxide in the calibration standard to that in the boron trichloride sample being tested. Calculate the concentration of carbon dioxide using the formula below.

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

7.3 *Chlorine* — This procedure is for the analysis of chlorine in boron trichloride using a gas chromatograph with a thermal conductivity detector.

7.3.1 *Detection Limits* — 5 ppm chlorine.

7.3.2 *Instrument Parameters*

7.3.2.1 *Column* — 60 m (200 ft.) 0.53 mm (1/32 in.) ID Dimethyl silicone megabore capillary column (Ristek RTxi)

7.3.2.2 *Carrier Gas* — Helium: N 6.0 25 mL to 30 mL/min (see Note 3).

7.3.2.3 *Sample Volume* — 2.0 mL.