

SEMI D36-1103

TERMINOLOGY FOR LCD BACKLIGHT UNIT

This standard was technically approved by the Global Flat Panel Display Committee and is the direct responsibility of the Japanese Flat Panel Display Committee. Current edition approved by the Japanese Regional Standards Committee on August 8, 2003. Initially available at www.semi.org October 2003; to be published July 2003.

1 Purpose

1.1 These terms and definitions describe various components and characteristics of backlight unit used for light source of liquid crystal display. This standard will be helpful to unify terminologies used for backlight unit, which will improve manufacturing efficiency resulting in reduction of manufacturing cost and time.

2 Scope

2.1 This standard is applicable to backlight unit. This standard includes the terminologies of components used in Backlight unit, inspection and measurement used in backlight unit field as well as panel and instrumentation field. The other area is not dealt with other SEMI standard document.

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

3 Limitations

3.1 In this standard, our target is only backlight unit and general purpose for terminology. Hence a special language used for critical purpose is not mentioned. For example, we do not define special languages depending to personal emotion and a detail or small components depending on special or general purpose.

3.2 The definition of lamp components follows the language used in lighting company or other optics related standardization.

4 Referenced Standards

4.1 CIE Standards¹

CIE 1931 — color space, CIE 1976 UCS

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

¹ CIE International Commission on Illumination, Kegelgasse 27, A-1030 Vienna, Austria

5 Terminology

5.1 General Terminology

5.1.1 Definition of Backlight Unit

5.1.1.1 Backlight unit (BLU) is a kind of light source positioned at the backside of liquid crystal panel. Since LCD is non-emissive display device it needs essentially light source.

5.1.2 Structure of BLU

5.1.2.1 As an initial light source cold cathode fluorescence lamp (CCFL) is used at bottom and/or topside of BLU. The light emitted from CCFL is entered into light guide plate (LGP). Here light is spread out two-dimensional light by scattering sites at the back surface LGP or inside of LGP. Two-dimensional light is sometimes diffused by diffusion sheet or conversed by prism sheet respectively. And final light comes into human eyes.

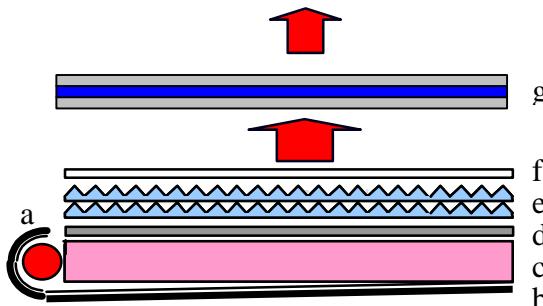


Figure 1
General structure of BLU; From a) to g), each components are indicate CCFL, reflection sheet, light guide plate, diffusion sheet, two prism sheets, protection sheet and panel respectively.

5.2 Components — The following measurement items are recommended for BLU but these items depend on user requirements if they are specified.

5.2.1 *cold cathode fluorescence lamp* — the positive column or cathode discharge lamp of the glow discharge. Namely, the ultraviolet by the releasing of the second electron of cathode stimulates the phosphor, so the lamp radiates the visible ray.

5.2.2 *reflection sheet* — the sheet that reflects the light to prevent the loss of the light toward back side of the BLU.

5.2.3 *light guide plate* — the plate, one of important components of the BLU, that changes the path of the light from line light source to face light source.

5.2.4 *diffusion sheet* — the sheet that diffuses the light to the previous surface.

5.2.5 *prism sheet* — the film to enhance the brightness.

5.2.6 *protection sheet* — a kind of sheet placed on the top of BLU for protecting other components from damage outside.

5.2.7 *frame* — the frame to make the components of the BLU stably combined.

5.2.8 *electrode* — the conductor, an electron source inside the lamp, which releases the electron by the electric current or flows the discharge current.

5.2.9 *hot (electrode)* — the electrode part receiving current from the inverter, namely anode.

5.2.10 *ground (electrode)* — the opposite part against the hot electrode of the lamp, namely cathode.

5.2.11 *inverter* — the device that transfers the voltage of the power supply from low voltage DC to high voltage AC to start the cold cathode fluorescence lamp.

5.2.12 *lamp* — the light source device mainly using the cold cathode fluorescence lamp that lights the BLU.

5.2.13 *lamp assembly* — the total light source devices to light lamp.

5.2.14 *lamp cover* — the part protecting the lamp makes the light released from the luminance factor incident over the light guide plate.

5.2.15 *lamp holder* — the part that protects the lamp from external collision or contact.

5.2.16 *lamp ring* — the part that protects lamp from the fragility.

5.2.17 *mercury* — as an ultraviolet light source, elemental name in the periodic table is Hg.

5.2.18 *mixing gas* — the gas needed to open the discharge consists of Neon and Argon gas etc maintaining the discharge.

5.2.19 *phosphor* — the phosphor with commonly red, blue and green color that transfers the ultraviolet came from the mercury to the visible ray.

5.2.20 *shrinkage tube* — the part made of insulation material to protect the wire.

5.2.21 *adhesive tape* — the tape to be used for adhering the components of the BLU to each other.

5.2.22 *back cover* — the device that complements fragile part of the lamp, etc. by means of utensil.

5.2.23 *clip* — the part fixing the sheet on the frame to prevent flux and looseness of sheet.

5.3 Characteristics

5.3.1 *luminance* — the luminance flux from the surface per unit solid angle per unit area in a given direction. Unit: nit and/or cd/m²

5.3.2 *average luminance* — average value of measured luminance.

5.3.3 *maximum luminance* — maximum value among the measured luminance.

5.3.4 *minimum luminance* — minimum value among the measured luminance.

5.3.5 *luminance uniformity* — The value that indicates the uniformity of luminance for the measured point. And the value is obtained by selecting one or multiple among below equations.

$$a) (\text{maximum}/\text{minimum}) \times 100$$

$$b) (\text{minimum}/\text{maximum}) \times 100$$

$$c) [(\text{maximum}-\text{minimum})/\text{Maximum}] \times 100$$

$$d) [(\text{maximum}-\text{minimum})/\text{Average}] \times 100$$

$$\text{where average} = \Sigma (i \rightarrow n) / N$$

5.3.6 *center point* — The position corresponding to the center of active area of BLU.

5.3.7 *chromaticity* — The indication of the suitable color for the mark of same color, namely the spectro-distribution-chart (λ) of the measured light source. The light source color is indicated by the 3 measured stimulus factors X, Y, Z (the quality of stimulus evoking the primary color sense) at the sensitivity of eye (2°).

Tri-stimulus factors: X, Y, Z

$$x = X/(X + Y + Z),$$

$$y = Y/(X + Y + Z),$$

$$z = Z/(X + Y + Z),$$

$$x + y + z = 1$$

(Referred by CIE 1931 color space)

5.3.8 *life time* — The shorter period of the time during no re-lit the lamp when lighting the lamp and during the flux of light's reaching the minimum value of regulations.

5.3.9 *correlated color temperature (CCT)* — A term used to describe the color of a light source whose chromaticity lies close to the Planckian (black body) locus on a CIE chromaticity chart. Specially, it is the temperature of a black body radiator which produces the chromaticity most similar to that of the light source evaluated. It is usually expressed in degrees Kelvin.

5.3.10 *surface temperature* — The surface temperature of the lamp and BLU.

Unit: ° C

5.3.11 *turn-on test* — The test for checking turn-on & off of the BLU.

5.3.12 *appearance test* — The test for appearance inspection of the BLU.

5.3.13 *reliability test* — The test for checking the changes of the display quality and part size during lighting of the BLU at the measurement condition (ex: temperature 60° C, humidity 75%, aging time 96hr, 250hr, 500hr).

5.3.14 *efficiency* — The ratio of total light fluxes generated by the lamp and consumed electric power at input part of the lamp.

Unit: lm / W (lumen/Watt)

5.4 *Equipment*

5.4.1 *photometer* — The equipment that measures the luminance of the object at the point of the perpendicular angle view for the surface of BLU.

5.4.2 *colorimeter* — The equipment that measures the color of the object at the point of the perpendicular angle view for the surface of BLU.

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SEMI D37-0304

SPECIFICATION FOR LIQUID CRYSTAL DISPLAY (LCD) PELLICLES

This specification was technically approved by the Global Flat Panel Display – Materials & Components Committee and is the direct responsibility of the Japanese Flat Panel Display – Materials & Components Committee. Current edition approved by the Japanese Regional Standards Committee on January 9, 2004. Initially available at www.semi.org February 2004; to be published March 2004.

1 Purpose

1.1 This specification defines standard outer and inner dimensions, stand off, minimum transmittance, and strength of pellicle film used for masks.

2 Scope

2.1 These dimensions apply to pellicle for masks that are principally used in fabricating liquid crystal displays. The mask size specifies range from 330×450 mm to 520×800 mm.

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

3 Referenced Standards

3.1 SEMI Standards

SEMI D6 — Specification for Edge Length and Thickness for Liquid Crystal Display (LCD) Mask Substrates

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

4 Terminology

4.1 Definitions

4.1.1 *stand off* — a certain distance on a mask or reticle to a membrane.

5 Ordering Information

5.1 Ordering information shall be discussed between suppliers and users.

6 Requirements

6.1 *Pellicle Size* — Pellicle Size shall be defined as shown in Table 1.

6.2 *Minimum Transmittance* — Average transmittance shall be 94.5% or more within the range of wave length of 360–440 nm.

6.3 Measurement Condition for Pellicle Film Strength

6.3.1 Not to be broken by air blow

6.3.2 Air nozzle diameter: 1 mm

6.3.3 Air pressure: 3 kg/cm²

6.3.4 Distance from air nozzle to film: 5 cm

6.3.5 Air blow time: 5 sec.

Table 1 Pellicle Size (mm)

Mask Size	Outer Diameter	Inner Diameter	Stand Off	
330×450	294	427	283	416
390×610	348	582	334	568
	353	592	340	580
520×610	474	592	460	578
500×750	436	732	418	724
520×800	474	782	456	768

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SEMI D38-0304

GUIDE FOR QUALITY AREA OF LCD MASKS

This guide was technically approved by the Global Flat Panel Display – Materials & Components Committee and is the direct responsibility of the Japanese Flat Panel Display – Materials & Components Committee. Current edition approved by the Japanese Regional Standards Committee on January 9, 2004. Initially available at www.semi.org February 2004; to be published March 2004.

1 Purpose

1.1 This guide defines the quality area of LCD masks.

2 Scope

2.1 This guide applies to masks that are principally used in fabricating liquid crystal displays. The mask size specifies range from 202.8×202.8 mm to 700×800 mm.

2.2 Substrates with an edge length less than 200 mm follow the specifications for a semiconductor mask (see SEMI P1).

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

3 Referenced Standards

3.1 SEMI Standards

SEMI D6 — Specification for Edge Length and Thickness for Liquid Crystal Display (LCD) Mask Substrates

SEMI P1 — Specification for Hard Surface Photomask Substrates

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

4 Terminology

None.

5 Quality Area of LCD Masks

5.1 For Mask Size $\leq 550 \times 650$ mm:

10 mm from (peripheral) edge of blanks

5.2 For Mask Size $> 550 \times 650$ mm, $\leq 700 \times 800$:

20 mm from (peripheral) edge of blanks

5.3 The quality area should be within the area of the writing pattern.

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SEMI D39-0704

SPECIFICATION FOR MARKERS ON FPD POLARIZING FILMS

This specification was technically approved by the Global Flat Panel Display – Color Filter & Optical Elements Committee and is the direct responsibility of the Japanese Flat Panel Display – Color Filter & Optical Elements Committee. Current edition approved by the Japanese Regional Standards Committee on April 30, 2004. Initially available at www.semi.org June 2004; to be published July 2004.

1 Purpose

1.1 Markers on FPD polarizing films, originally used for indicating optical direction of the films, have expanded the variety of products, such as identification for both sides of films, and for equipment. This also complicated the variety of shapes and colors for markers.

1.2 This standard establishes specification for unifying markers used for indicating direction of FPD polarizing films.

1.3 This specification may be applied to manufacturing process, quality control, and designing of Liquid Crystal Display (LCD) panel.

2 Scope

2.1 This document specifies markers on FPD polarizing films.

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

3 Referenced Standards

3.1 None.

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

4 Terminology

4.1 None.

5 Requirements

5.1 Markers on FPD polarizing films shall be used for indicating specific direction of the films.

5.2 *Markers Specification*

5.2.1 Makers shall be simple sign on the specific location of protective film, as follows:

Circle: 

Triangle: 

Arrow: 

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

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SEMI D40-0704

TERMINOLOGY FOR FPD SUBSTRATE DEFLECTION

This standard was technically approved by the Global Flat Panel Display - Factory Automation Committee and is the direct responsibility of the Japanese Flat Panel Display - Factory Automation Committee. Current edition approved by the Japanese Regional Standards Committee on April 30, 2004. Initially available at www.semi.org June 2004; to be published July 2004.

1 Purpose

1.1 This document provides the terms and definitions of a physical transformation when flat panel display (FPD) substrates are maintained statically.

2 Scope

2.1 These terms and definitions are applicable to both front and back substrates used in FPD fabrication.

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

3 Limitations

3.1 This standard is not applicable to sag at transportation between substrate vendors and FPD manufacturers.

4 Referenced Standards

4.1 SEMI Standards

SEMI D9 — Terminology For FPD Substrates

SEMI D25 — Specification For Flat Panel Display Substrate Shipping Case

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

5 Terminology

5.1 Definitions

5.1.1 *Displacement Terminology* — The displacement is generated by sag and lift caused by the supporting positions and/or locations of the FPD substrate. The displacement is defined by the values of two-dimensional sag and lift. Two dimensional maximum displacement is also defined by values of two-dimensional maximum sag and lift, and the thickness of the FPD substrate.

5.1.1.1 *support plain* — a support plain is defined as a horizontally ideal flat plain which is enhanced from a horizontally straight line defined from the top position of the support members, as shown in Figure 1.

5.1.1.2 *support member* — support member is a support device such as a support pin to support FPD substrates, as shown in Figure 1.

5.1.1.3 *support span* — support span is defined as at least the distance between two support members, as shown in Figure 1.

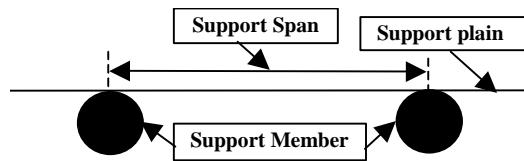


Figure 1
Definitions of Support Span, Support Member and Support Plain

5.1.1.4 *lift* — lift is defined as the displacement to the bottom surface of substrate from the support plain. It is located above position from the support plain, as shown in Figure 2.

5.1.1.5 *sag* — sag is defined as the generated distance between the bottom surface of the substrate and the support plain caused by gravity, as shown in Figure 2.

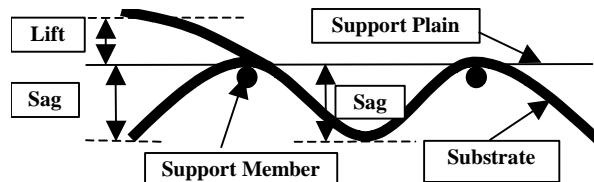


Figure 2
Definitions of Lift and Sag

NOTE 1: In case there is sag at the edge of a substrate, sag is defined by the distance to position "A" from the support plain. The position "A" is determined by the intersection point of between the edge of the bottom surface of the substrate and the edge of the beveled region, as shown in Figure 3.

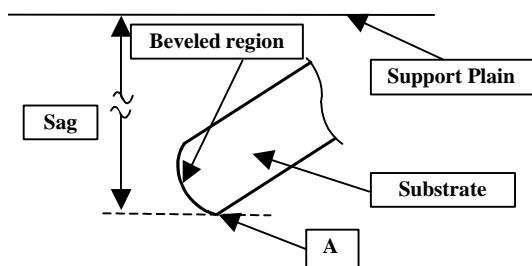


Figure 3
Definition of Sag at Edge

NOTE 2: In case there is lift at the edge of a substrate, lift is defined by the distance to position “B” from the support plain. The position “B” is determined by the intersection point of between the edge of the bottom surface of the substrate and the edge of the beveled region, as shown in Figure 4.

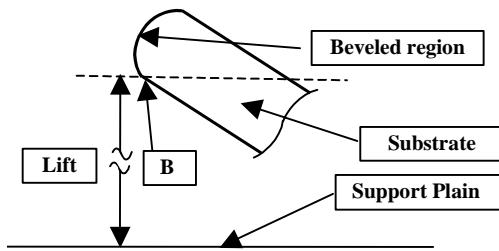


Figure 4
Definition of Lift at Edge

5.1.1.6 *maximum displacement* — two-dimensional maximum displacement (Max. Displacement) is defined by maximum lift (Max. Lift) + maximum sag (Max. Sag) + substrate thickness. Maximum displacement can have the same value at several different locations, as shown in Figure 5.

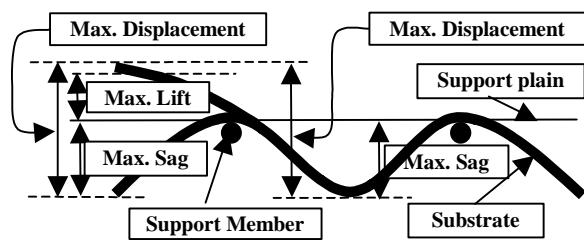


Figure 5
Maximum Displacement

5.1.2 *Displacement Mode Terminology* — displacement modes are classified into five types caused by supporting conditions, which are number of

supporting positions, distance of each supporting position and location of supporting positions.

5.1.2.1 *A Type* — substrates of this type have a convex shape. The substrate shape is named “A type”. A type mode consists of lift at center location and two sags at the left and right edges, as shown in Figure 6.

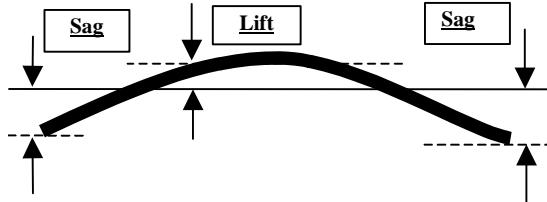


Figure 6
A Type Substrate Shape

5.1.2.2 *U Type* — the type is the concave shape of substrate. The substrate shape is named as “U type”. U type mode consists of Sag at center location and two lifts which are consist of the Lift at left and at right edge locations, as shown in Figure 7.

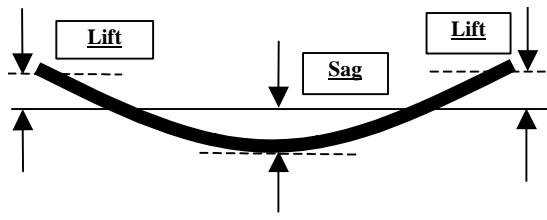


Figure 7
U Type Substrate Shape

NOTE 3: Depending on conditions, support span length may change slightly due to the substrate slipping on its support members, as shown in Figure 8. The support span is shorter when sag is relatively larger, as shown in Figure 9. On the other hand, the support span is longer when sag is relatively smaller, as shown in Figure 10. Since the change of support span is predicted to be significantly smaller than the support span, the change of support span is ignored.

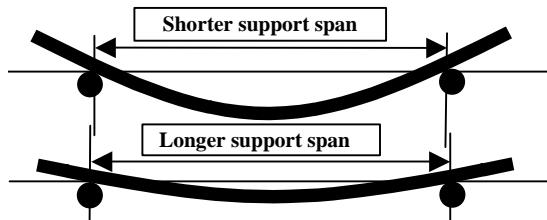


Figure 8
Change of Support Span Due to Sag Magnitude

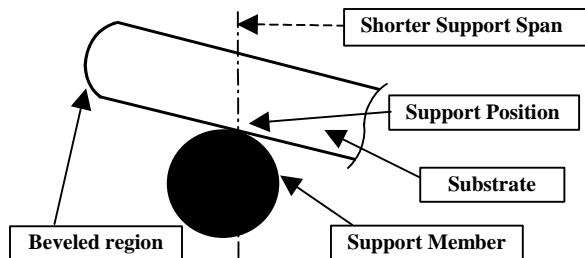


Figure 9
Position on Small Sag

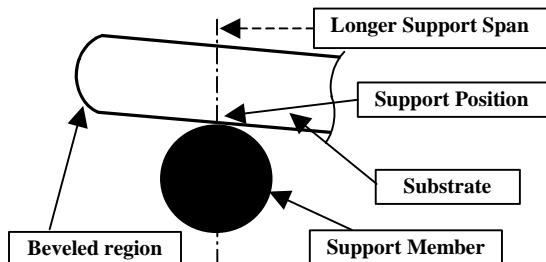


Figure 10
Position on Large sag

5.1.2.3 M Type — the type has an alphabetic “M” shape, which mixes the convex shape and the concave shape, as shown in Figure 11. This substrate shape is named “M type”. M type consists of all sag and requires sag to exist at both edge regions of the substrate, even if the substrate has a continuous wave or corrugation in its shape.

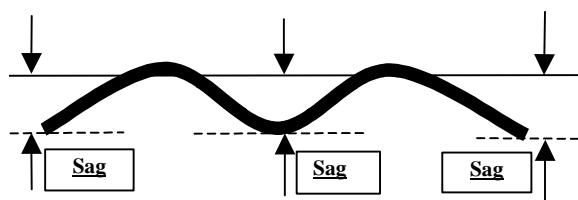


Figure 11
M Type Substrate Shape

5.1.2.4 W Type — the type has an alphabetic “W” shape, which mixes the convex shape and the concave shape, as shown in Figure 12. This substrate shape is named “W type”. W type combines sag and lift. Also W type requires lift to exist at both edge regions of the substrate, even if the substrate has a continuous wave or corrugation in its shape.

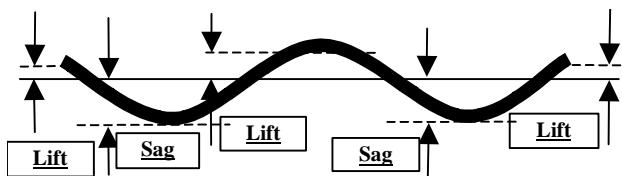


Figure 12
W Type Substrate Shape

5.1.2.5 S Type — the type has a rotated alphabetic “S” shape, which mixes the convex shape and the concave shape, as shown in Figure 13. This substrate shape is named “S” type. S type combines sag and lift. Also S type requires an asymmetrical shape, which consists of sag and lift at both edge regions, even if the substrate has a continuous wave or corrugation in its shape.

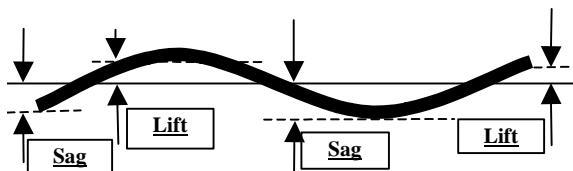


Figure 13
S Type Substrate Shape

6 Related Documents

6.1 SEAJ¹ (Semiconductor Equipment Association of Japan) Liquid Crystal Display Manufacturing Equipment Dictionary

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SEMI D41-0305

MEASUREMENT METHOD OF SEMI MURA IN FPD IMAGE QUALITY INSPECTION

This standard was technically approved by the Global Flat Panel Display - Factory Committee and is the direct responsibility of Japanese Flat Panel Display Factory Automation Committee. Current edition approved by the Japan Regional Standards Committee on November 24, 2004. Initially available at www.semi.org January 2005; to be published March 2005.

1 Purpose

- 1.1 This standard will define the application of the formula derived in SEMI D31 to various test conditions closed to visual inspection for MURA in FPD image quality inspection.
- 1.2 SEMI D31 has derived a formula to detect defects and blemishes in FPD in comparison with human eyes and CCD based instruments.
- 1.3 In general the difficulty of implementing instruments to replace human inspector lays on human factor variation with its origin and location.

2 Scope

- 2.1 This standard is applicable to FPDs. This standard mainly deals with both the measurement method of Semu and the revised definition of Semu. The target display size is typically from 8"(20.3cm) to 30"(76.2cm) diagonal.

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

3 Limitations

- 3.1 In this standard, the target of measurement, Mura, is limited as below.
- 3.2 Line defects narrower than a pixel and pixel dot defects are not the subject of this standard. In this issue, this standard follows SEMI D31.
- 3.3 In this standard, it is dealt with monochrome displays like gray scale patterns in color patterns. In this issue, this standard follows SEMI D31.

4 Reference Standards

4.1 SEMI Standards

SEMI D31 —Definition of Measurement Index (Semu) for Luminance Mura in FPD Image Quality Inspection

4.2 Other Documents

Flat Panel Display Measurement Standard, VESA FPDM 2.0, June 2001, IEC 61747-6.

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

5 Terminology

- 5.1 This standard follows SEMI D31 in terminology.

5.2 Abbreviations and Acronyms

- 5.2.1 *JND* — abbreviation for Just Noticeable Difference used in the field of Psychophysics; for a certain stimulus, the smallest change in stimulus (luminance, for example) where a difference can be perceived. Specifically, it is often used to indicate a statistical value where the probability of the difference being “perceptible” is 50% and the probability of the difference “not being perceptible” is 50%. Also expressed using lower case letters, *jnd*.



5.3 Definitions

5.3.1 *Dynamic Range* — the divided ability from a minimum detecting level to a maximum detection level of a CCD equipment.

5.3.2 *Gray Scale* — gray scale on an image display. This standard indicates level 32 out of 64 level gray scale.

5.3.3 *L0 Display* — gradation 0 out of 64. (Pitch Black)

5.3.4 *L63 Display* — gradation 63 out of 64 (Completely white)

5.3.5 *Luminance Accuracy* — the error ratio of the luminance.

5.3.6 *Measuring points* — the number of CCD sensors.

5.3.7 *Semu* — Semi Mura, Measurement index for Mura, defined in this standard. Please refer to SEMI D31.

5.3.8 *Viewing direction* — the angle between the CCD equipment and the test sample.

6 Related Definitions for Mura Measurement

6.1 Mura is a Japanese term meaning blemish and has been adopted in English to provide a name for imperfections of the display pixel matrix surfaces that are visible when the display is in operation. Inspect the display surface while displaying a white full screen, a black full screen, and a dark gray full screen (VESA FPDM 2.0). In many case, the Mura phenomena is detected by human eyes. But this is very confusing in the quality aspect. And it is very difficult to quantify the level.

6.2 The definition in SEMI D31 shows mainly that Luminance Mura is related with the *size of Mura*. Therefore this standard will follow the Cjnd concept.

6.3 Semu Definition

6.3.1 Under specific conditions, the below regression function can explain the level of Mura between the area and contrast for Human Mura JND. (Refer to SEMI D31.)

$$Cjnd = F(Sjnd) = 1.97/Sjnd^{0.33} + 0.72$$

Cjnd: Contrast of Mura at JND

(Unit: % relative to background = 100%)

Sjnd: The area of Mura at above contrast (Units: mm²)

6.3.2 In the above equation, contrast at JND is inversely proportional to the area raised to the 0.33 powers. In short, it indicates that as Mura the area gets smaller, only darker Mura can be sensed.

6.3.2.1 For the subject Mura, the Mura level, Semu, can be calculated using the below formula.

$$\begin{aligned} Semu &= |Cx| / Cjnd \\ &= |Cx| / F(Sx) \\ &= |Cx| / (1.97/Sx^{0.33} + 0.72) \end{aligned}$$

Cx: Average contrast of Mura being measured (Unit: % relative to background = 100%)

Sx: The surface area of Mura being measured (mm²)

7 Measurement Method

7.1 Environment Conditions:

- Temperature: 25 ± 2°C
- Humidity: 65 ± 20% RH
- Illumination of surrounding: in a dark room below 10 lux
- Air flow: no wind

7.2 Measurement Method

7.2.1 Warm-Up Time

7.2.1.1 Warm-up time is the period from power-on to saturation of the light intensity. The optical measurement needs 15 to 30 minutes for more accurate measurement. In case of Mura, it has a little luminance difference between the luminance of Mura and the luminance of background area. Therefore it must be measured in a saturate state.

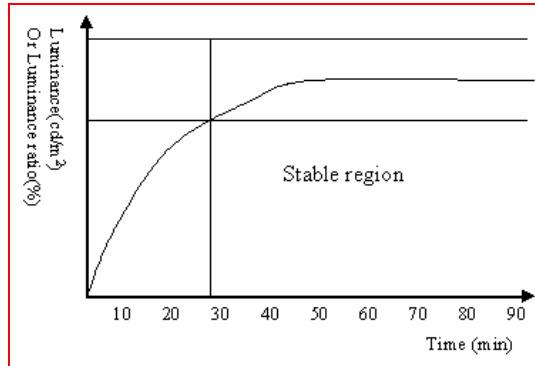


Figure 1
General Warm-Up Characteristics

7.2.2 Light Measurement Device (LMD)

7.2.2.1 When human eyes measure the FPD, it is detected in the total active area. So the measuring equipments must have similar characteristics to the human eye detecting method. In this standard, we recommend the CCD equipments to detect the Mura phenomena.

- 1) Dynamic Range — The dynamic range of the CCD equipment should be over 12 bits because the Semu value must be controlled by 0.1 Semu.
- 2) Luminance Accuracy — The Accuracy of Luminance is very important to measure Mura, because the Mura value is determined by low luminance difference. Therefore the accuracy of luminance should be $\pm 0.01 \text{ cd}/\text{m}^2$ at the $100\text{cd}/\text{m}^2$.
- 3) Measuring Points — The measuring points should be over $200(\text{H}) \times 200(\text{V})$ points. It is the same as the number of the CCD sensors.

7.2.3 Test Pattern for Semu Measurement

7.2.3.1 The test pattern should be set by $50 \text{ cd}/\text{m}^2$ in the white pattern. The permissible error is $10 \text{ cd}/\text{m}^2$.

7.2.4 Viewing Direction

7.2.4.1 The viewing direction angle should be set $90^\circ \pm 1^\circ$.

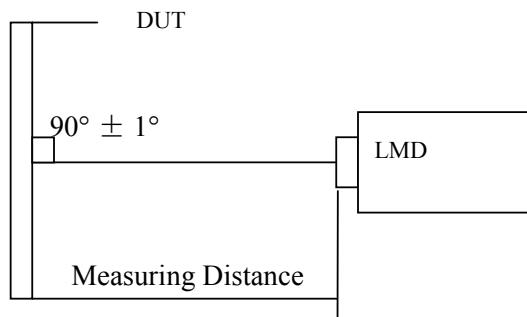


Figure 2
Viewing Direction for the Semu Measurement

7.2.5 Measuring Distance for Semu Measurement

7.2.5.1 Because we use a CCD Camera, the measuring distance is set by the same angle in all DUT. We standardize the 15" display size and testing distance is 500 mm. But the testing distance should be different from each display size.

7.2.5.2 In case of the 15" display, the angle from the center to the edge position can be calculated below.

$$\tan \theta = \frac{\text{diagonal_distance} \times 0.5}{\text{Measuring_distance}} \dots\dots\dots(4)$$

Therefore the reference angle is about 20.856° and then it can be calculated the measuring distance of all DUT.

$$\text{Measuring_distance} = \frac{\text{diagonal_distance} \times 0.5}{\tan \theta} \dots\dots\dots(5)$$

Table 1 Examples of Measuring Distance

Display Size (in.)	14.1"	15"	17"	18.1"	20.1"
Measuring Distance (mm)	470 mm	500 mm	566 mm	603 mm	670 mm

#1 This measuring distance can have the same measuring angle in each different size sample.

7.2.6 Determination of the Threshold for the Mura Area

7.2.6.1 It is very important to calculate the Semu value. If some threshold values are different from each test equipment, the Semu test results are different. Therefore we use the Cx value to determine the threshold value. In this standard, when the Cx value is over 1.01, we can consider the Mura area.

7.2.7 Area Calculation Method

7.2.7.1 There are two main parameters for calculating the Semu value. The first one is the Cx value and the other is the Sx value. The Sx value means that the surface area of Mura being measured. We can calculate the size of Mura area with counting the number of pixels under the CCD equipment test.

7.2.8 Test Sample Pattern for the Sample Test

7.2.8.1 Originally, the test pattern should be set by 50 cd/m² in the white pattern. And the permissible error is 10 cd/m². But we use some artificial Mura test patterns for checking the sensitivity of the Semu parameter. The test pattern has 10 Muras in Figure 3. Each Mura has a different gray level. In Figure 3, the left one shows darker Mura patterns, and the right one shows brighter Mura patterns.

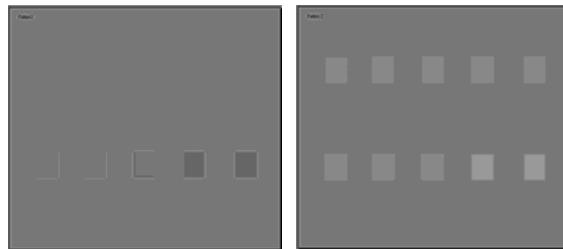


Figure 3
Test Patterns for Mura

7.2.9 Test Results for the Sample Patterns

7.2.9.1 Table 2 shows that the actual results for the darker Mura test pattern and Table 3 is for the brighter Mura pattern. Figure 4 is a graphic information result for the dark Mura and Figure 5 is a graphic information result for the bright Mura.

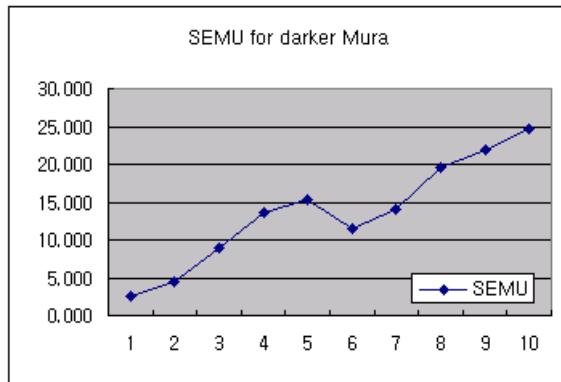


Figure 4
The Graph Information for the Darker Mura Patterns

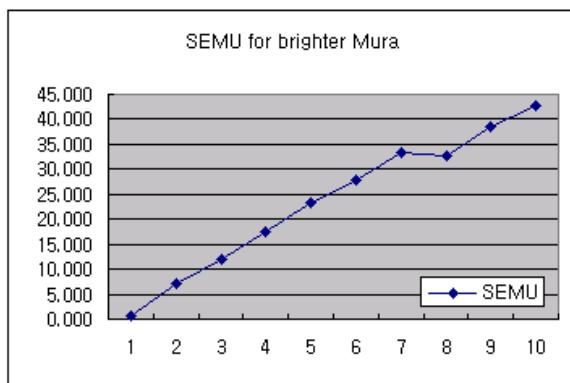


Figure 5
The Graph Information for the Brighter Mura Patterns

Table 2 The Test Result for the Darker Mura Patterns

	1	2	3	4	5	6	7	8	9	10
Mura_L	9.17	10.50	11.33	10.97	10.09	15.27	18.00	17.82	17.55	15.97
Background	9.40	10.98	12.45	12.67	11.89	17.25	20.92	22.12	22.45	21.16
Cx	2.47	4.39	8.96	13.42	15.14	11.48	13.94	19.44	21.81	24.54
Sx	400	400	400	400	400	400	400	400	400	400
Cjnd	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99
SEMU	2.491	4.425	9.025	13.515	15.249	11.562	14.039	19.581	21.968	24.715

Table 3 The Test Result for the Brighter Mura Patterns

	1	2	3	4	5	6	7	8	9	10
Mura_L	7.87	9.93	11.88	12.50	12.14	22.26	28.14	29.60	31.21	30.26
Background	7.81	9.27	10.62	10.65	9.86	17.42	21.15	22.32	22.57	21.26
Cx	0.80	7.15	11.89	17.43	23.09	27.78	33.05	32.60	38.30	42.35
Sx	400	400	400	400	400	400	400	400	400	400
Cjnd	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99
SEMU	0.806	7.201	11.977	17.553	23.261	27.987	33.291	32.839	38.575	42.658



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SEMI D42-0305

SPECIFICATION FOR ULTRA LARGE SIZE MASK SUBSTRATE CASE

This specification was technically approved by the Global Flat Panel Display Committee and is the direct responsibility of the Japanese Flat Panel Display Committee. Current edition approved by the Japanese Regional Standards Committee on January 11, 2005. Initially available at www.semi.org February 2005; to be published March 2005.

1 Purpose

1.1 This standard defines the specification for ultra large size substrate case used in LCD (Liquid Crystal Display) manufacturing process.

2 Scope

2.1 The specification defined by this standard is applicable to the case for containing substrate materials used in LCD mask manufacturing process. The objective substrate range for the mask size defined by this standard is 850 × 1,200 mm and more.

2.2 The automatic handling of the mask substrate becomes indispensable, in accordance with the larger size of the substrate. This specification defines the handling volume for mask substrate inside the case.

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

3 Referenced Standards

3.1 None.

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

4 Terminology

4.1 *Definition*

4.1.1 *case* — an enclosed container for storing one mask substrate.

5 Requirements

5.1 Handling volume for ultra large size mask substrate used in LCD manufacturing process.

5.1.1 The hatched area in Figure 1 shall be the exclusion volume for handling ultra large size mask substrate used in LCD manufacturing process.

5.1.2 Symbols used in Figure 1 are defined in Table 1.

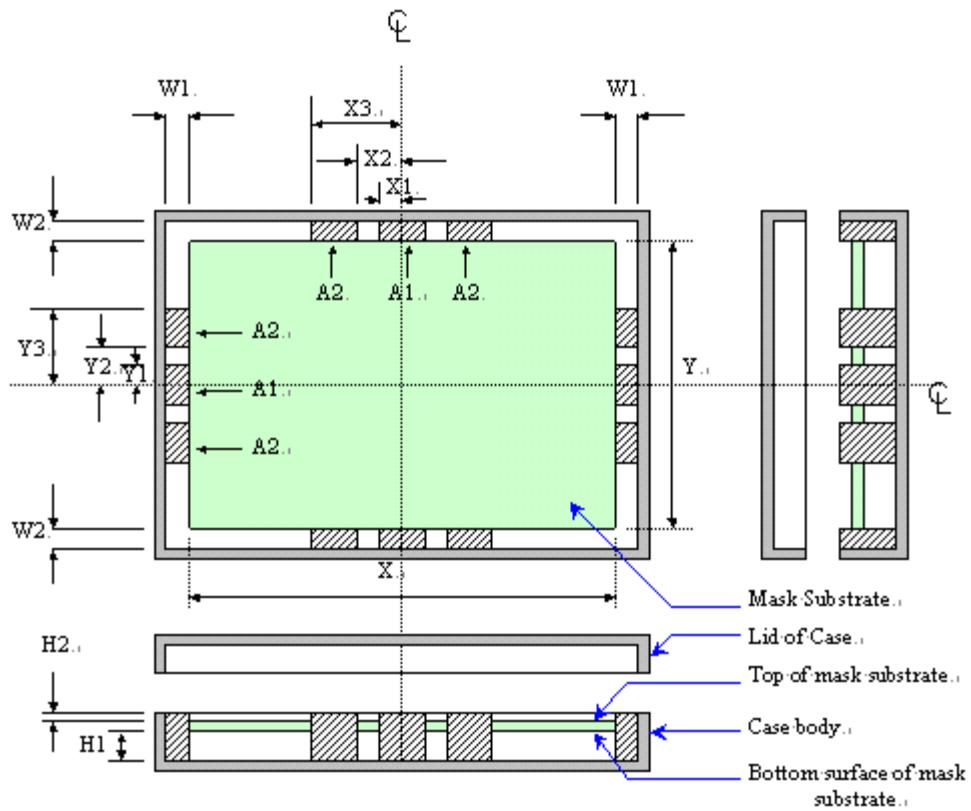


Figure 1
Handling Volume for Ultra Large Size Mask Substrate used in LCD Manufacturing Process

Table 1 Symbols Outline

Symbol Used	Definition	Specification
A1	Exclusion Volume (Handling Volume)	Defined by X1, X2, X3, W1, Y1, Y2, Y3, W2, H1, and H2
A2	Exclusion Volume (Handling Volume)	Defined by X1, X2, X3, W1, Y1, Y2, Y3, W2, H1, and H2
X	Long Side Length of Mask Substrate	Defined by the Substrate Size
Y	Short Side Length of Mask Substrate	Defined by the Substrate Size
X1	Center of Long Side Length of Mask Substrate to the edge of A1 volume	75 mm
X2	Center of Long Side Length of Mask Substrate to the inside of A2 volume	165 mm
X3	Center of Long Side Length of Mask Substrate to the outside of A2 volume	350 mm
W1	Edge of Long Side Length of Mask Substrate to the inside of the case	≥ 70 mm
Y1	Center of Short Side Length of Mask Substrate to the edge of A1 volume	75 mm
Y2	Center of Short Side Length of Mask Substrate to the inside of A2 volume	165 mm
Y3	Center of Short Side Length of Mask Substrate to the outside of A2 volume	350 mm
W2	Edge of Short Side Length of Mask Substrate to the inside of the case	≥ 80 mm



<i>Symbol Used</i>	<i>Definition</i>	<i>Specification</i>
H1	Bottom surface of mask substrate to the inside of the case body	≥ 50 mm
H2	Top of mask substrate to the top of the case body	$10 \text{ mm} \leq, \leq 15 \text{ mm}$

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SEMI INTERNATIONAL STANDARDS



GASES

Semiconductor Equipment and Materials International

SEMI C3-0699

SPECIFICATIONS FOR GASES

IMPORTANT NOTICE: This specification is incomplete. The incomplete sections are noted as "To Be Determined." The Gases Committee has agreed to publish this section prior to completion as a service to the Semiconductor Industry.

This specification 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 February 28, 1999. Initially available at www.semi.org April 1999; to be published June 1999. Originally published in 1986; previously published in 1995.

1 Preface

1.1 Recognizing the importance of impurity content of gases in the manufacture of semiconductors, suppliers responded by introducing products with improved analytical characterization, notably for trace impurities.

1.2 The SEMI Gases Committee began its efforts in the spring of 1979. With this publication, the Committee establishes the definitions, general procedures, specifications, and analytical procedures for the gases listed in the index.

1.3 Products which meet all of the requirements may be described as "meeting SEMI specifications."

1.4 Where an analytical procedure different from that provided is substituted by a supplier or user, the burden of proof is on said supplier or user to confirm the equivalency. It should be noted that the following list of criteria was considered when determining the specified analytical method.

Reliability

Ease of Use

Maintenance

Precision

Accuracy

Sensitivity

Versatility

Availability of Equipment

1.5 The specifications provided by this work are intended to serve for gases to be used in the manufacture and processing of semiconductors and advanced electronic devices and circuits. The specifications and the associated test procedures are guidelines based on the experience of suppliers and users. The function of the specifications is to establish desired standards of quality.

1.6 Where feasible, a specification of content shall be expressed as a numerical limit in units of mole per mole (mole/mole).

1.7 For a major component, the value (assay or purity) shall be expressed as a minimum permissible limit. For an impurity, the value shall be expressed as a maximum permissible limit.

1.8 A gas conforming to the specifications will commonly contain more of the major component than the minimum permissible limit or contain less of an impurity (or several impurities) than the SEMI C3 maximum permissible limit. In neither case shall the gas be considered as of higher quality than that defined by the specification.

1.9 It is not practical to consider every impurity or contaminant that might be present. For certain applications, it is recognized that more stringent or additional specifications and procedures might be required. The intent of these specifications and the associated procedures is, on one hand, to assure that a gas is suitable for the common uses to which it may be put in the manufacture and processing of semiconductor devices and, on the other hand, to be consistent with contemporary manufacturing processes for that gas.

1.10 The Committee is continuing work on improvement of specifications and procedures and addition of other gases and systems such as mixed gases. The Committee welcomes comments, suggestions, and recommendations.

2 Definitions

2.1 *Abbreviations and Symbols* — In this work, abbreviations and symbols used shall be restricted to those listed in Table 1.

Table 1 Abbreviations and Symbols

Abbreviation or Symbol	Explanation
ASTM	American Society for Testing and Materials
atm	atmosphere
BP	Boiling point
°C	temperature, degrees Celsius

<i>Abbreviation or Symbol</i>	<i>Explanation</i>
cc	cubic centimeter(s)
CGA	Compressed Gas Association
cm	centimeter
DOT	Department of Transportation (U.S.)
°F	temperature, degrees Fahrenheit
ft	foot, feet
g	gram(s)
L	liter(s)
lb	pound(s)
max	maximum
min	minimum
mL	milliliter(s)
mm	millimeter(s)
MP	melting point
mw	molecular weight (g/mole)
ohm-cm	ohm - centimeter
ppm	mole/mole x 10 ⁶
ppb	mole/mole x 10 ⁹
ppba	mole/mole x 10 ⁹ atomic
ppbw	weight/weight x 10 ⁹
psia	pounds per square inch absolute
psig	pounds per square inch gauge
SCF	standard cubic foot (feet)
temp.	temperature
vol	volume
vol/vol	volume/volume
wt.	weight
w/v	weight/volume
ww	weight/weight

2.2 *accuracy* — (To Be Determined)

2.3 *assay* — determination of the content of a specific component with no evaluation of other components.

2.4 *comparison of analytical results with specific limits* — in the comparison of an analytical result for a test with the numerical limit associated with that specification, the result shall be rounded to the number of significant figures indicated for that limit. (See Rounding Numbers, SEMI C3, Section 2.22).

2.4.1 Consequently, a specification stated as 96% minimum will be met by a result as small as 95.5%, and that stated as 96.0% minimum will be met by a result as small as 95.95%. A specification of 0.1% maximum

will be met as large as 0.14%, and that of 0.10% maximum by a result as large as 0.104%.

2.5 *cryogenic liquid* — liquid with a normal boiling point below -150°C.

2.6 *cylinder pressure* — pressure contained in a gas cylinder prior to regulation.

2.7 *cylinder tare weight* — containers which are stamped to denote the weight of the container or the weight of the container and the valve less the product. The weight does not include the weight of any protective cylinder cap.

2.8 *density* — weight per unit volume (w/v) is expressed as grams per liter for gases at zero degrees Celsius, one atmosphere.

2.9 *detection limit* — the detection limit for all the analytical methods that appear in this section of the BOSS must be established for each impurity defined. The detection limit must be stated as well as the statistical method used to establish that detection limit. The analytical method should be chosen such that the detection limit is at or below the specification.

2.10 *dewpoint* — the temperature at which liquid first condenses when vapor is cooled.

2.11 *expression of content and concentration* — unless otherwise stated, a specification limit and the analytical result related to it shall be expressed in units of mole per mole (mole/mole).

2.12 *filtration* — (To Be Determined)

2.13 *Gas Purity Guideline* — A Gas Purity Guideline is a proposed specification recommended by one or more users as needed in the future for the production of semiconductor devices. They reflect future needs in which test methods are not generally available at the time of proposal. These guidelines are approved by the Gases Committee for publication in the Standards Book. Products meeting these guidelines are not necessarily commercially available.

2.14 *heavy metals* — (To Be Determined)

2.15 *liquefied compressed gas* — a gas which under the charged pressure is partially liquid at a temperature of 21.1°C (70°F).

2.16 *metals* — (To Be Determined)

2.17 *molecular weight* — the sum of the atomic weights of all the atoms in the molecule.

2.18 *nonliquefied compressed gas* — a gas, other than a gas in solution, which under the charging pressure is entirely gaseous at a temperature of 21.1°C (70°F).

2.19 *physical properties* — physical properties shall not usually be employed for specification purposes; for information, however, representative values for a particular gas, as supplied, may be included as an item in the monograph for that gas.

2.20 *quality* — the quality is determined by subtracting the sum of the maximum acceptable gas phase impurity levels, expressed in percent, from 100. The result is truncated after the first significant figure which is not a nine. The quality does not represent an assay.

2.21 *rare gas* — any of the six gases, all noble, comprising the extreme right-hand group of the Periodic Table; namely helium, neon, argon, krypton, xenon, and radon.

2.22 *rounding numbers* — the following rules for rounding of measured or calculated values shall be employed:

2.22.1 When the figure next beyond the last place to be retained is less than 5, leave unchanged the figure in the last place retained.

2.22.2 When the figure next beyond the last place to be retained is greater than 5, increase by 1 the figure in the last place retained.

2.22.3 When the figure next beyond the last place to be retained is 5 and there are no figures beyond this 5 or only zeroes, (a) increase by 1 the figure in the last place retained if it is odd, or (b) leave the figure unchanged if it is even.

2.22.4 When the figure next beyond the last place to be retained is 5 and there are figures other than zeroes beyond this 5, increase by 1 the figure in the last place retained.

2.22.5 Obtain the rounded value in one step by direct rounding and not in two or more steps of successive rounding.

2.23 *specific gravity* — the ratio of the mass of a gas to the mass of an equal volume of air at a specified temperature. For liquids, it is the ratio of the mass of the liquid to the mass of an equal volume of water.

2.24 *specification and specification limits* — the specification limit should fall above or in the range of the result and its uncertainty.

2.25 *temperature* — temperature values shall be expressed in degrees Celsius.

2.26 *tolerances in measurements* — use the following guidelines for mixture tolerances:

2.26.1 Mixtures should be specified by the major component and the concentration of the desired minor component(s).

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

2.26.3 The impurity levels in the mixture shall not exceed the algebraic sum of the impurities specified for the designated components.

2.26.4 Mixtures shall be prepared according to the following mixing tolerances:

<i>Concentration of Minor Component</i>	<i>Preparation Tolerance Level</i>
10 - 99 ppm	$\pm A 20\%$
100 - 999 ppm	$\pm A 10\%$
0.1 - 50%	$\pm A 5\%$

2.26.5 Standards used to verify mixing tolerance (henceforward called Certified Standards) shall meet the following requirements:

<i>Range</i>	<i>Preparation Tolerance of Each Minor Component</i>	<i>Analytical Accuracy of Each Minor Component</i>
0 - 9.9 ppm	To Be Determined	To Be Determined
10 - 99 ppm	$\pm A 10\%$	$\pm A 3\%$
100 - 999 ppm	$\pm A 5\%$	$\pm A 2\%$
0.1% - 50%	$\pm A 4\%$	$\pm A 2\%$

2.27 *Calibration Standards* — Calibration standards shall be as close as practical to specification and may not exceed ten times (10 \times) the specification unless specifically excepted in procedure.

3 Samples

3.1 *Sample Size* — The quantity of gas/liquid in a single sample container shall be sufficient to perform the analysis for all the listed specifications. If a single sample does not contain a sufficient quantity of gas/liquid to perform all of the required analyses, additional samples from the same source shall be taken under similar conditions.

3.2 *Gaseous Samples* — Gaseous samples shall be representative of the gaseous supply. Sampling shall be performed in accordance with one of the following:

3.2.1 By withdrawing a sample from the supply container through a suitable connection into the sample container. (For safety reasons, the sample container and sampling system must have a rated service pressure at least equal to the pressure in the supply container.)

3.2.2 By connecting the container being sampled directly to the analytical equipment.

3.2.3 By selecting a representative cylinder from the cylinders in the lot.

3.3 *Liquid Sampling (Vaporized)* — Vaporized liquid samples shall be representative of the liquid supply. Sampling shall be in accordance with one of the following:

3.3.1 By vaporizing liquid from the supply container in the sample tubing.

3.3.2 By flowing liquid from the supply container into, or through, a suitable container in which a representative sample is collected and then vaporized.

3.4 *Liquid Samples (Liquified Compressed Gases)* — A direct connection between the liquid phase of liquified compressed gas containers and the analytical equipment can be achieved, provided suitable flash vaporization is obtained.

3.5 *Lot Acceptance Tests* — These are analyses performed on the gas/liquid in the shipping container, or a sample thereof, which is representative of the lot. (The terms "lot" and "batch" may be used interchangeably.)

3.6 *Lots* — One of the following is to be used:

3.6.1 No specific quantity or any quantity of product agreed upon between the supplier and the customer.

3.6.2 All of the product supplied during the contract period.

3.6.3 All of the product supplied or containers filled during a calendar month.

3.6.4 All of the product supplied or containers filled during seven consecutive days.

3.6.5 All of the product supplied or containers filled during a consecutive 24-hour period.

3.6.6 All of the product supplied or containers filled during one eight-hour shift.

3.6.7 All of the product supplied in one shipment.

3.6.8 All of the product supplied in one shipping container.

3.6.9 All of the product supplied in the container(s) filled on one manifold at the same time.

3.7 *Number of Samples Per Lot* — The number of samples per lot shall be in accordance with one of the following:

3.7.1 One sample per lot.

3.7.2 Any number of samples agreed upon by the supplier and the customer.

4 Sampling

4.1 For gases provided in cylinders, a sample can be taken directly for analysis. For gases provided in bulk quantities or cylinders where direct sampling is not appropriate, a sample may be taken per SEMI sampling procedures.

4.2 Sampling Procedures

4.2.1 *Cryogenic Liquid Sample* — Liquid Samples for Oxygen, Nitrogen, and Argon, using the TTU-131/E sampler.

WARNING: DO NOT USE THIS PROCEDURE FOR THE SAMPLING OF LIQUID HYDROGEN.

4.2.2 *Applicable Document* — Military Specification MIL-S-27626D 16 August 1979, Amendment 1, 24 April 1981.

4.2.3 *General Description* — The TTU-131/E cryogenic sampler is a small, portable pressure vessel used to receive, vaporize, and contain a representative sample of cryogenic liquid from a supply source. The vaporized sample is withdrawn as a gas for analytical purposes.

4.2.4 *Theory* — The sampler is used to isolate a small but representative quantity of cryogenic liquid and vaporize the major component and all volatile impurities to form a homogeneous gas sample suitable for analysis. The cryogenic liquid is used to cool a shielding space and sampling cup prior to admitting the liquid to the sampling cup. The purpose of pre-cooling the sampling cup is to prevent concentrating impurities which could result when the warm cup causes the liquid to vaporize, leaving behind impurities with higher boiling points.

4.2.4.1 When the cup is adequately cooled, the sampling valve is opened, allowing liquid to fill the cup. When the sampling valve is closed, the liquid is trapped in the cup and will vaporize as a result of atmospheric heating. Once the sample is trapped, the flow of liquid is stopped.

4.2.5 Preparation for Sampling

4.2.5.1 The sample should be kept in the same product service to avoid sample contamination. If a product change is required, always thoroughly purge the sampler or evacuate to 100 microns prior to taking the sample.

4.2.5.2 When taking the sample, the sampler is to remain secured to the bottom half of the case and MUST be in an upright position.

4.2.5.3 Inspect the sampler vessel for any obvious physical defects, such as dents, gouges, bent fittings, etc. Since the sampler is a pressurized vessel, it should be removed from service if any damage is apparent.

4.2.6 Sampling Procedure

4.2.6.1 Remove cover.

4.2.6.2 Loosen inlet and outlet fittings dust caps located on side of the vessel.

CAUTION: SAFETY GLASSES AND PROTECTIVE GLOVES ARE REQUIRED WHEN OPERATING THIS EQUIPMENT. SAMPLING SHOULD BE DONE ONLY IN WELL VENTILATED AREAS.

4.2.6.3 Relieve pressure in vessel through vents in dust caps by cautiously opening inlet sampling valve.

4.2.6.4 When gauge indicates atmospheric pressure and flow ceases, close inlet sampling valve and remove dust caps.

4.2.6.5 Connect fill hose to supply tank and inlet fittings of sampler.

4.2.6.6 Open supply tank outlet valve. Gas and liquid will begin to flow from the sampler outlet.

CAUTION: WHEN OXYGEN IS SAMPLED, SOME ADDITIONAL PRECAUTIONS MUST BE OBSERVED. IF THERE IS NO FACILITY SUCH AS A CLEAN CONCRETE PAD ON WHICH THE LIQUID OXYGEN CAN EVAPORATE SAFELY, IT WILL BE NECESSARY TO CATCH THE LIQUID OXYGEN IN A CLEAN, PREFERABLY SEAMLESS ALUMINUM BUCKET. USE A LINE WITH MINIMUM INSIDE DIAMETER OF ONE-HALF INCH TO DIRECT THE LIQUID OXYGEN INTO THE BUCKET. FURTHERMORE, IT IS IMPORTANT THAT THERE IS NO RESTRICTION IN THIS LINE BECAUSE THE BACK PRESSURE DEVELOPED MAY BE SUFFICIENT TO CAUSE THE LIQUID IN THE CUP TO OVERFLOW INTO THE LARGE CHAMBER. ON WARMUP, THE PRESSURE IN THE SAMPLER COULD BECOME EXCESSIVE, PARTICULARLY IF THE SAMPLE WERE SUBCOOLED, NECESSITATING THE REPLACEMENT OF THE SAFETY RELIEF DEVICE.

4.2.6.7 Allow sampler to cool until a steady flow of liquid appears at outlet.

CAUTION: AVOID CONTACT WITH THE FLOW OF CRYOGENIC LIQUID. THE EXTREMELY LOW TEMPERATURE CAN CAUSE PAINFUL INJURIES.

4.2.6.8 Open inlet sampling valve completely to allow liquid to enter sampling cup.

4.2.6.9 After 30 seconds, close sampling valve.

4.2.6.10 Close supply tank outlet valve.

4.2.6.11 Disconnect fill hose.

4.2.6.12 Invert sampler for five minutes to allow sampling cup to empty and provide vaporization of liquid.

4.2.6.13 At ambient temperature the sampler should be at 400-500 psig, indicating that a good sample was obtained. A lower pressure would indicate a leak in the sampler or that the cup was not filled with liquid. If there is any doubt, release gas from sampler and take another sample.

4.2.6.14 Re-install inlet and outlet fitting caps. Do not tighten.

4.2.6.15 Affix a tag identifying the product to the inlet sampling valve handwheel.

4.2.6.16 Install cover.

4.3 Gas Phase Sample — (To Be Determined)

4.4 Liquid Sample — (To Be Determined)

4.5 Delivery to Analytical Instruments — (To Be Determined)

5 Quantification (To Be Determined)

5.1 Linear Response

5.1.1 Direct Comparison

5.1.2 Calibration Curve

5.2 Non-Linear Response

5.2.1 Direct Comparison

5.2.2 Calibration Curve

6 Gas Chromatography

6.1 The analysis of many gaseous impurities in bulk and specialty gases is done by means of a gas chromatography separation of impurities from the gas matrix and quantification using a broad range of sensitive and sometimes selective detectors. This section will provide a guide for the format and representation of such a procedure. Figure 1 is designed to represent the format for submission of such methodology.

6.2 *Sample Introduction* — By its nature, gas chromatography implies the use of carrier gases into which the gaseous sample is injected and carried into the separation medium, either solid or liquid in nature.

The introduction of a gaseous sample into a carrier gas stream is accomplished by means of a sample injection valve. Valves for this purpose can be any of several types, although the most commonly used are either rotary or diaphragm valves using loops of stainless steel tubing of a known volume. It is not necessary to diagram such sample introduction systems for a procedure unless the introduction system is unique to the application by way of special conditions for safety or other reasons critical to the accuracy of the sampling technique.

6.3 Separations — It is an acceptable and common practice to quantitate several gaseous impurities with a single gas chromatographic separation. Occasionally a single injected sample volume will undergo "multidimensional" separation to achieve the necessary analysis. Rotary valves, diaphragm valves or Dean's pressure switching again are used to move impurities from one separation medium to another, or from one column to another. Where multidimensional techniques are used, they should be accompanied by flowpath diagrams which clearly indicate valving and their appropriate positions. The valving sequences used should have adequate explanation to clarify the separations.

6.4 Columns — Columns used are generally of 3.2 mm (1/8 in.) o.d. and 2.2 mm (0.085 in.) i.d. stainless steel of varying lengths and packed with one of many solid supports. Columns may be specified by length and packing material if these standard dimensions apply. If another dimension or material is used for the column itself, it should be specified by material type, and by o.d. and i.d. in mm. Packing materials should be specified by material, % coating and coating type if applicable, and mesh size. Capillary columns are assumed to be fused silica unless otherwise specified and should be specified by i.d. in mm, film thickness in μm , and length in meters. Column temperatures and applicable program rates should be given for each independently heated zone in degrees Celsius.

6.5 Carrier and Support Gases — Carrier and support gas flow rates for all separations should be specified. Carrier and support gases used should fall into the general purity requirements in Table 2 and purity need not be specified unless the analysis has specific purity requirements for safety, accuracy, or component lifetime. Flows should be specified in mL/min or L/min, or as linear velocity for open tubular capillary columns.

6.6 Detectors — Detector technology should be specified, and alternative detection may be assumed equivalent for detectors of like selectivity and sensitivity. Table 2 gives a reference of relative detector sensitivity and is provided as a guideline only.

Detector parameters are less likely to be easily formatted since detector technologies vary widely in their specific parameters. All elements critical to achieving like sensitivity must be included, such as temperature, make-up gases and their flows, flame, combustion or reaction gas ratios or flows, voltages, currents, or any additional settings as outlined by the detector manufacturer.

6.6.1 Detection limits must be specified for each impurity for the actual methodology used. Because the lower detection limit can be greatly influenced by retention time for any impurity, it is important that the detection limit be derived for the impurity within the context of the method, and not solely on the detectability of the detector. Detection limits are assumed to be calculated on a mole/mole basis.

6.7 Operating Procedures — The standard procedure assumed for the gas chromatographic analysis of most gases is as follows:

1. Inject the calibration standard onto the column using a gas sampling valve. Record the retention time(s) and peak area(s) for all impurities detected.
2. Analyze the sample to be tested in the same manner as the calibration standard.
3. Repeat 1.
4. Compare the average peak area of the calibration standard with that of the sample being tested. (See Calculation of Concentration, in Section 6.9.)

6.7.1 The method should specify any additional valve switching or special parameters which are necessary for the successful analysis of the impurities specified for the procedure. It must also specify the order of elution if multiple impurities are detected.

6.8 Calibration — Calibration for these analytical procedures is generally by external, single point calibration. Calibration gas mixtures should be specified and are assumed to be within the range specified in Section 2.26. Calibration standards are assumed to be made in a balance of gas representative of the sample unless otherwise specified. If calibration techniques other than external, point calibrations are used, they must be described.

6.9 Calculation of Concentration — The calculation of the concentration of impurity within the sample is based on a comparison of the average peak area of the impurity in the calibration standard, to the average peak area of the impurity within the sample, based on the formula:

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

It is assumed that the results of such a calculation must not exceed the specification for the product being analyzed.

6.10 *Notes* — Any special requirements for any section within gas method may be included here. Notes should reflect the minimum requirements to successfully achieve the desired detection limits with the equipment specified.

- 1.1 Impurity analyzed - description of method (e.g., gas chromatograph with thermal conductivity detector).
 - 1.1.1 Detection Limit - (mole/mole)
 - 1.1.2 Instrument Parameters
 - 1.1.2.1 Column(s): Adsorbent, mesh size, length in meters, (dimensions if other than 1/8 in. by 0.085 in.)
 - 1.1.2.2 Carrier Flow: mL/min, gas type
 - 1.1.2.3 Support Gases:
 - 1.1.2.4 Sample Volume: mL
 - 1.1.2.5 Temperatures:
 - Detector: °C
 - Columns: °C
 - Other Equipment: °C
 - 1.1.3 Calibration Standard - amount ppm (or ppb) impurity, balance if other than helium.
 - 1.1.4 Operating Procedure (Parameters)
 - 1.1.4.1 Order of elution
 - 1.1.4.2 Special instructions
 - 1.1.5 Notes
 - 1.1.6 Figures

Figure 1

Format for Gas Chromatographic Method

7 Special Analyses

7.1 *Resistivity Measurement* — The American Society of Testing and Materials (ASTM) is developing a method to measure resistivity in substrates. This method may be referenced when it is finalized.

7.2 Metal Analysis of Gaseous Silicon Compounds

7.2.1 Because of the importance of trace elemental impurities impacting silicon device characteristics, there is an increasing need for the detection and quantitative analysis of low-level impurities in the silicon materials used in device fabrication. Some of these materials are gaseous silicon-containing compounds, such as silane and the chlorosilanes. For elemental analysis by FTIR or PLS, a compound must first be converted to a solid form such as a single crystalline rod or disc. For the quantitative determination of metals and some non-metallic elements, the gases can be passed through a series of bubblers containing high-purity DI water or dilute acids. The aqueous media can be concentrated and analyzed by suitable techniques such as ICP or AA

spectroscopy. In both cases, the recovery is assumed to be 100% efficient or, in other words, there is no loss of any impurity from the silicon source.

7.2.2 FTIR (Fourier Transform Infrared) Spectroscopy, PLS (Photoluminescence Spectroscopy) and NAA (Neutron Activation Analysis) are probably the most useful tools for the detection and analysis of trace quantities of elemental impurities in solid silicon materials. NAA requires special silicon material specimens to be submitted to neutron irradiation in a nuclear reactor and the analysis facility to be approved by the Nuclear Regulatory Commission (NRC) and other government agencies. In contrast, FTIR and PLS do not require special governmental regulations, and the instrumentation is available on the open market.

7.2.3 An FTIR spectrometer is an infrared spectrometer in which a Michelson Interferometer is used in place of a grating or prism. The simplicity of the Michelson Interferometer, with only one moving part, an oscillating mirror, along with a He-Ne laser as a reference, provides nearly absolute frequency accuracy. This translates into a high spectral resolution. FTIR has greater detection efficiency since the energy-wasting slits required for dispersive spectrometers are not used. With a microcomputer to control the functions and to perform data processing, signal averaging is used to improve the signal-to-noise ratio. All these features lead to good reproducibility and rapid measurement results compared with conventional IR techniques.

7.2.3.1 As FTIR is a transmission method, it requires a single crystalline silicon specimen with flat, parallel, mirror-polished surfaces. For oxygen in silicon, the absorption peak at 9 microns is used for quantitative analyses. For carbon in silicon, the absorption band at 16 microns is used. Both measurements may be performed at room temperature. (The carbon determination has strong interference from the silicon phonon band, but can be resolved with high purity, carbon-free standards.) For the determination of shallow donors and acceptors, FTIR measurements must be made at liquid helium temperatures, but the throughput is low. Although the operation of today's FTIR instrument is relatively simple, the interpretation of the results through software and associated problems/solutions is not. Quantitative analysis is based upon the measured percent transmission at the characteristic wavelength and compared to standards. Detection limits at about 10-12°K are in the 10^{11} to 10^{13} atoms/cc for shallow donors and acceptors, 3.1×10^{14} atoms/cc for oxygen, and 2×10^{15} atoms/cc for carbon.

Table 2 Carrier Gases for GC Instruments

<i>Detector</i>	<i>Gas</i>	<i>Detection Level</i>					
		0 - 100 ppt	100 ppt - 100 ppb	100 ppb - 100 ppm	100 ppm - 1%	1% - 10%	10% - 100%
TCD/USD	Ar	N/A	N/A	99.9999%	99.999%	99.998%	99.995%
	H ₂	N/A	N/A	99.9995%	99.999%	99.99%	99.99%
	He	N/A	N/A	99.9995%	99.9995%	99.999%	99.99%
	N ₂	N/A	N/A	99.9995%	99.9995%	99.998% < 0.5 ppm O ₂	99.998%
HID/DID	He	N/A	99.9999%	99.9999%	N/A	N/A	N/A
	He*	N/A	99.995%	99.995%	N/A	N/A	N/A
FID	He	N/A	N/A	99.9995%	99.999%	99.998%	99.99%
	N ₂	N/A	N/A	99.999% < 0.05 ppm THC	99.999% < 0.05 ppm THC	99.998% < 0.5 ppm THC	99.998%
	H ₂ **	N/A	N/A	99.9995% < 0.2 ppm THC	99.999% < 0.5 ppm THC	99.99% < 0.5 ppm THC	99.99%
	Air**	N/A	N/A	HC Free < 0.1 ppm THC	Zero Air < 1 ppm THC	Zero Air < 1 ppm THC	Blended Air
FPD	He	N/A	99.9995%	99.999%	N/A	N/A	N/A
	N ₂	N/A	99.999% < 0.05 THC	99.998% < 0.5 ppm THC	N/A	N/A	N/A
	H ₂ **	N/A	99.995% < 0.2 ppm THC	99.999% < 0.5 ppm THC	N/A	N/A	N/A
	Air**	N/A	HC Free < 0.1 ppm THC	Zero Air < 1 ppm THC	N/A	N/A	N/A
RGD	Ar	99.999% < 1 ppm H ₂ /CO	N/A	N/A			
	N ₂	99.999% < 1 ppm H ₂ /CO	N/A	N/A			
	Air	Zero Air < 1 ppm H ₂ /CO	N/A	N/A			
ECD	N ₂	99.9995%	99.998% < 0.5 ppm O ₂	99.998% < 0.5 ppm O ₂	N/A	N/A	N/A
	CH ₄ /Ar	EC Grade < 1 ppb Total Halocarbons	EC Grade < 1 ppb Total Halocarbons	EC Grade < 1 ppb Total Halocarbons	N/A	N/A	N/A
PID	He	N/A	99.9995%	99.995%	N/A	N/A	N/A
	N ₂	N/A	99.9995%	99.998%	N/A	N/A	N/A
MS/MSD	H ₂	99.9995% < 0.2 ppm THC	99.999% < 0.5 ppm THC	99.999% < 0.5 ppm THC	99.99% < 0.5 ppm THC	99.99% < 0.5 ppm THC	99.99% < 0.5 ppm THC
	Ar	99.999%	99.998%	99.998%	99.998%	99.998%	99.998%
	He	99.9995%	99.999%	99.999%	99.995%	99.995%	99.995%
	N ₂	99.9995%	99.998%	99.998%	99.998%	99.998%	99.998%

*purge gas

**combustion gases

Table 3 Detectors for Gas Chromatography

<i>Detector</i>	<i>Type</i>	<i>Temp Limit</i>	<i>Analytes Analyzed*</i>	<i>Carrier Gases</i>	<i>Selectivity</i>	<i>Detectability</i>	<i>Linear Range</i>
Thermal Conductivity (TCD)	U	400	CF ₄ , PH ₃ , SF ₆ , Fixed Gases	He, H ₂ , Ar, N ₂	N/A	4 × 10 ⁻¹⁰ g/mL	>10 ⁵
Ultrasonic (USD)	U		CO ₂ , O ₂ , N ₂ , CO	He, H ₂ , Ar, O ₂ , N ₂ , Air	N/A	1 × 10 ⁻⁹ g/mL	10 ⁶
Helium Ionization (HID)	U	325	H ₂ , O ₂ , Ar, N ₂ , CO	He	N/A	4 × 10 ⁻¹⁴ g/mL	10 ⁴
Discharge Ionization (DID)	U		same as HID	He	N/A	1 × 10 ⁻¹² g/mL	10 ⁴
Flame Ionization (FID)	S	420	C ₁ -C ₅ , C _x F _y , C _x Cl _y F _z , CO, CO ₂	H ₂ /Air**, He, N ₂ , H ₂	Hydrocarbons	2 × 10 ⁻¹² g/sec	>10 ⁷
Flame Photometric (FPD)	S	420	H ₂ S	H ₂ /Air**, He, N ₂ , H ₂	S/C 10 ³ -10 ⁶ :1 P/C > 10 ⁵ :1	S < 1 × 10 ⁻¹¹ g/mL P < 1 × 10 ⁻¹² g/mL	S ≥ 10 ³ P ≥ 10 ⁴
Reduction Gas (RGD)	S	300	CO, H ₂	N ₂ , Ar, He, Air	H ₂ , CO	H ₂ 0.5 – 2 ppb CO 0.5 - 4 ppb	10 ³ -10 ⁴
Electron Capture (ECD) (Ni ⁶³)	S	420		N ₂ , P5	halogens	5 × 10 ⁻¹⁵ g	10 ⁴ (linearized)
Photoionization (PID)	S	350	PH ₃ , H ₂ S, AsH ₃	Ar, H ₂ , N ₂	By ionization energy	2 × 10 ⁻¹³ g/sec	>10 ⁷
Thermionic (TID)	S	420		H ₂ , 8% H ₂ /He	N/P 1:5 N/C 5 × 10 ⁴ :1 P/C 10 ⁵ :1	N 1 × 10 ⁻¹³ g/sec P 5 × 10 ⁻¹⁴ g/sec	10 ⁵
Mass Spectroscopy (MS)	S	320		He, H ₂	variable with mass range	EI: 10-100 pg NICI: variable as low as 25 fg	10 ⁵ -10 ⁶
Mass Density (MSD)	S						
Atmospheric Pressure Mass Spectroscopy (APIMS)							

*this does not represent detector capability; rather those analytes analyzed by current SEMI procedures

**used as combustion gases

7.2.4 Photoluminescence Spectroscopy (PLS) of single crystalline silicon specimens at liquid helium temperatures exhibits sharp emission lines. Electron hole pairs called excitons are formed by incident radiation (514.5 nm line from argon laser). The decay of these "exciton or multi-exciton" states yields the lines characteristic of the impurity atoms. For elemental detection, the observed emission lines must be assigned as being extrinsic (due to impurities) or intrinsic (due to silicon). The luminescence process itself appears too complex to attain precision or accuracy from basic calculations. The PLS emissions are analyzed as a function of wavelength, suggested to be 1077 to 1139 nm, for the elemental determination of shallow donors and acceptors in silicon. Quantitative measurements of impurities are based on the extrinsic-to-intrinsic

intensity ratio and compared to standards. The PLS emissions are analyzed using a monochromator and detected by a photomultiplier. Sensitivity in the range of 5 × 10¹⁰ to 5 × 10¹¹ atoms/cc has been claimed for B, Al, P, and As.

7.2.4.1 In order to obtain reliable measurements, the silicon specimens must be in single crystalline form with a mirrored surface. Epitaxial wafers with layers of thickness greater than 10 microns are suitable, since the sample's depth appears to be less than 5 microns. However, it is still in the development stage, and very few laboratories have such capability. A number of questions remain unanswered, including the following:

1. What is the effect of compensating impurities?

2. What is the effect of deep levels and intracenter transitions on the intensity ratios?
3. What is the effect of crystallographic defects? Of point defects? And of surface defects?
4. Interference from other shallow impurities may mask the detection of very low-level impurities. For instance, PLS signal of Al at 1078.5 nm may be affected by strong signals from As at 1079.0 nm or at 1078.2 nm.

7.2.4.2 CVD-deposited films in single crystalline form can be used for elemental detection of donors and acceptors for gaseous silicon compounds. However, PLS does not detect carbon, oxygen, or nitrogen. It is also not useful for metallic impurities. That is, in general, PLS is useful only for a very limited number of impurities.

7.2.5 Based on this analysis, it was recommended that the SEMI Gases Analytical Procedures Subcommittee should wait for further development of innovative and improved analytical techniques for the quantitative determination of low-level elemental impurities based on FTIR and PLS. Meanwhile, techniques such as ICP, AA, and Ion Chromatography may be applicable to a very large number of impurities of interest to the silicon semiconductor industry. Development and adaptation of these technologies should be encouraged.

7.2.6 AA (Atomic Absorption Spectrophotometry), ICP (Inductively Coupled Plasma Emission Spectrometry), and IC (Ion Chromatography) are probably the most valuable instrumental techniques for the detection and analysis of trace elemental impurities in aqueous media. These techniques usually require that the elements be dissolved in an aqueous medium. Due to this fact, and the fact that the elemental impurities are typically present in silane and the chlorosilanes at the ppb and sub-ppb levels (which is beyond the detection limits for these techniques for most elements) it is usually necessary to concentrate the impurities prior to analysis, such as by passing the gas through a series of impingers (i.e., gas bubblers) containing either DI water or, more commonly, acidic solutions. From analyses of the impinger solutions together with knowledge concerning impinger solution volume(s) and both the flow rate and period of flow of the gas, the concentration of the respective elements in the original gas should be readily calculated to a fairly high degree of accuracy.

7.2.6.1 Atomic Absorption Spectrophotometry can be used for elemental analyses for a multitude of elements in the periodic table at detection limits ranging from ppm to ppb, depending both on the exact configuration or setup and the element being analyzed. The principle

of the technique involves conversion of an incoming aerosol into an atomic vapor which can absorb light from a primary light source, usually a hollow cathode lamp or an electrodeless discharge lamp (EDL), the latter providing improved sensitivity and lower detection limits for certain elements. In addition, a flameless (i.e., graphite furnace) technique can be used to increase sensitivities and detection limits 50 to 100 times better.

7.2.6.2 Complementary to Atomic Absorption Spectrophotometry is Flame Emission Spectrophotometry, which differs in the absence of a primary light source. Again, a flame is utilized to convert an incoming aerosol into an atomic vapor. However, the flame also thermally elevates the atoms to an excited electronic state, and, when the atoms return to the ground state, they emit light detectable by a photomultiplier. This technique is ideal for the analysis of Li, H_a, and K at ppm and sub-ppm levels.

7.2.6.3 Inductively Coupled Plasma Emission Spectrometry (ICP) is rapidly replacing Atomic Absorption Spectrophotometry for elemental analyses of liquids, since approximately 70% of the elements in the periodic table can be determined with better precision and equal or better sensitivity with minimal sample preparation. Detection limits, in fact, for this technique range from ppm to sub-ppb levels, depending both on instrumental and/or experimental conditions and the element being analyzed. ICP involves nebulizing liquid samples either directly or in diluted form into a spray chamber where a stream of argon gas carries the smaller sample droplets into the axial channel of an argon plasma which reaches temperatures of 4000-8000°K. The common technique employs an optical system for detection. Newer techniques are being investigated using a mass spectrometer as a detection system coupled to an ICP excitation source. This is referred to as ICP/MS.

7.2.6.4 Ion Chromatography (IC) uses the principle of ion exchange to separate anionic and cationic species at detection limits ranging from ppm to ppb, depending again both on the instrumental and experimental conditions and on the element being analyzed.

7.2.6.5 Detection of ionic species in IC is most commonly accomplished by an electrical conductivity detector capable of measuring most non-transition metal cations and anions to sub-ppm levels. An electrochemical detector (for amperometric detection) is available for measuring any electroactive species having an oxidation/reduction potential near the applied electrochemical cell potential. This latter detector is capable of measuring many ionic species at ppb levels (e.g., Br⁻, CN⁻, S⁻, etc.).

7.2.6.6 Of the three aforementioned techniques, Atomic Absorption Spectrophotometry, Inductively Coupled Plasma Emission Spectrometry, and Ion Chromatography, the most sensitive method of analyzing for a multitude of elements is, without doubt, graphite furnace (i.e., flameless) Atomic Absorption Spectrophotometry. However, this instrumental technique suffers the disadvantage of being slow and tedious. Consequently, Inductively Coupled Plasma Emission Spectrometry is rapidly replacing Atomic Absorption Spectrophotometry for elemental analyses of liquids since it is a reasonably fast technique requiring minimal sample preparation with practical detection limits being superior to those of standard (i.e., flame) Atomic Absorption Spectrophotometry for most elements and approaching those of graphite furnace Atomic Absorption Spectrophotometry for many of the elements. An added attractive feature of Inductively Coupled Plasma Emission Spectrometry is that eventually it may be used to analyze for elements directly in gases. Preliminary work, in fact, is currently being conducted in this area by several research groups. Although Ion Chromatography can be used to analyze for a multitude of non-transition metal cations (e.g., Li⁺, Na⁺, K⁺, Rb⁺, Cs⁺, Ba⁺², Mg⁺², Ca⁺², Sr⁺², etc.) it is limited by the fact that currently only a few transition metals (e.g., Ni⁺², Cu⁺², Co⁺², Zn⁺², Fe⁺², Fe⁺³, Cd⁺², etc.) can be analyzed by this instrumental technique. IC, however, has the advantage in that it can be used for analyzing for complex cationic and especially anionic

species directly (e.g., NH₄⁺, NO₂⁺, NO₃⁻, SO₃⁻, SO₄⁻², PO₄⁻³, OC1⁻, SCN⁻, CN⁻, CO₃⁻², etc.) and for different oxidation states for certain elements (e.g., Fe⁺² and Fe⁺³), unlike the other techniques, which are strictly limited to elemental analyses only. Furthermore, IC is extremely valuable for analyzing ionic species, particularly anions (e.g., F⁻, Cl⁻, etc.) of many common elements which otherwise cannot be analyzed directly by either Atomic Absorption Spectrophotometry or Inductively Coupled Plasma Emission Spectrometry.

8 Standards

8.1 Certified Standards shall be made by weight traceable to the National Institute of Standards and Technology.

9 Determination of Precision

9.1 To Be Determined.

10 Safety

10.1 Because of the continuing evolution of safety precautions, it is impossible for this publication to provide definite statements related to the safe handling of individual chemicals. The user is referred to product labels, product data sheets, government regulations, and other relevant literature.

11 Gas Use Table (see Notes 1, 2)

Gas Use Table		Ammonia	Argon	Arsine	Boron Trichloride	Boron Trifluoride	Boron-11-Trifluoride	Carbon Tetrafluoride	Chlorine	Diborane	Dichlorosilane	Disilane	Helium	Hexafluoroethane	Hydrogen	Hydrogen Bromide	Hydrogen Chloride	Hydrogen Fluoride	Methyl Fluoride	Nitrogen	Nitrogen Trifluoride	Nitrous Oxide	Oxygen	Perfluoropropane	Phosphine	Silane	Silicon Tetrachloride	Sulfur Hexafluoride	Trichlorosilane	Trifluoromethane	Tungsten Hexafluoride	
		Annealing	X																													
Carrier Gas for Bubblers										X																						
Chamber Cleans					X						X				X						X											
CVD Carrier Gas	X														X																	
CVD Source		X																														
CVD B Source						X																										
CVD P Source																															X	
CVD Epitaxial								X																								
CVD Nitride	X																															
CVD Oxides																					X X			X X								
CVD Polysilicon								X X																						X		
CVD Silicides																															X	
Epitaxial Silicon																															X X	X
CVD Silicon Nitride	X							X													X			X								
CVD Tungsten/Silicide																															X	
Dopants		X																													X	
Etching	X														X		X															
Metal Etching	X	X				X									X	X X				X										X		
Nitride Etching	X				X										X			X	X											X	X	
Oxide Etching	X			X											X		X	X	X											X	X	
Silicon Etching	X															X X	X	X	X											X	X	
Ion Implant	X X	X X																														
Metal Gettering or CVD Tube Cleans																			X													
Nitridation	X																															
Oxide Gettering																			X												X	
Oxynitrides																															X	
Plasma Ashing																															X	
Pressurizing Systems															X					X												
Reactive Ion Etch	X																														X	
Reducing Gas																		X														
Sputtering	X																															
Inert Gas Blanketing/Purging	X														X					X												

NOTE 1: This table is intended to identify various gases described in the SEMI standards and the typical uses for those gases in semiconductor processes. It is not all-inclusive. As other uses arise, they should be brought to the attention of the Gases Committee so that revisions can be made to this table as needed.

NOTE 2: Performance of a gas in an application may vary depending on the impurities in the gas. It is the responsibility of the user to determine the appropriate gas quality for any specific application.

NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

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SEMI C3.12-1102

SPECIFICATION FOR AMMONIA (NH₃) IN CYLINDERS, 99.998% QUALITY

This specification 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 August 29, 2002. Initially available at www.semi.org September 2002; to be published November 2002. Originally published in 1983, previously published in 1994.

1 Description

1.1 Ammonia is a colorless, alkaline gas having a pungent odor. Ammonia dissolves readily in water. It is shipped as a liquified gas under its own vapor pressure. It is combustible.

2 Specifications

2.1 QUALITY: 99.998%

Impurities	Maximum Acceptable Level (ppm)*
Carbon monoxide (CO)	1
Hydrocarbons C ₁ – C ₃	1
Nitrogen (N ₂)	5
Oxygen (O ₂)	2
Water (H ₂ O) (v/v)	3
TOTAL LISTED IMPURITIES	12

*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.

NOTE 1: This specification applies to the gas phase of the cylinder as delivered.

3 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	17.031	17.031
Boiling point at 1 atm	-33.4°C	-28.2°F
Density of gas at 0°C (32°F) and 1 atm	0.771 kg/m ³	0.0481 lb/ft ³
Specific gravity of gas at 0°C and 1 atm (air = 1)	0.597	0.597
Density of liquid at 21.1°C (70°F)	608.7 kg/m ³	38.00 lb/ft ³

4 Analytical Procedures

NOTE 2: Observe proper safety procedures for handling and venting the ammonia sample.

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

NOTE 4: All the gases used in the analysis of the sample should contain not more than 10% of the specified value of the component of interest unless otherwise specified.

NOTE 5: All carrier lines, carrier pressure regulators and sample containers are to be constructed of stainless steel and cleaned. The systems must be assembled without pipe threads, PTFE tape or polymeric seal materials.

4.1 *Carbon Monoxide, Oxygen and Nitrogen* — This procedure is for the determination of carbon monoxide, oxygen and nitrogen in ammonia using a gas chromatograph with a helium ionization detector.

NOTE 6: Due to the extreme sensitivity of the helium ionization detector, the system is to be helium leak tested to a maximum of 10⁻⁷ atm-cc/sec.

4.1.1 *Detection Limits* — 25 ppb (mole/mole) carbon monoxide, 30 ppb (mole/mole) oxygen, 50 ppb (mole/mole) nitrogen.

4.1.2 *Instrument Parameters*

4.1.3 *Columns*:

Column 1:	HayeSep A or equivalent, 100/120 mesh, 3.28 m (10 ft) by 3.2 mm (1/8 in.) 0.085 ID.
Column 2:	HayeSep A or equivalent, 100/120 mesh, 3.28 m (10 ft) by 3.2 mm (1/8 in.) 0.085 ID.

4.1.3.1 *Carrier Flow*: 30 mL/min helium, minimum of 99.9999% purity having a maximum of 40 ppb carbon dioxide.

4.1.3.2 *Sample Volume*: 1.0 mL.

4.1.3.3 *Temperatures*:

Detector	125°C
Columns	27°C

4.1.3.4 Determine the breakdown voltage of the detector. Set operating voltage at 10 volts below breakdown.

4.1.4 *Calibration Standards* — 1–2 ppm (mole/mole) carbon monoxide, 2–3 ppm (mole/mole) oxygen, 5–6 ppm (mole/mole) nitrogen, balance helium (having a total of other impurities of less than 1 ppm).

4.1.5 *Operating Procedure*

4.1.5.1 Set timing interval on sample select valve to 10 seconds.

4.1.5.2 Set timing interval on gas sampling valve to 3:30 minutes.

4.1.5.3 Set timing interval #3 to 4:20 minutes.

4.1.5.4 Do not change the initial sample flow setting once established.

4.1.5.5 Obtain a continuous flow of the calibration standard using a clean stainless steel line (0.02 in. ID).

4.1.5.6 Inject the calibration standard onto the precolumn with the gas sampling valve. Record the retention times and peak areas. Order of elution is nitrogen, oxygen, and carbon monoxide.

4.1.5.7 Repeat Section 4.1.5.6 until reproducibility of the reading is better than 1% of full scale.

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

4.1.5.9 Repeat Section 4.1.5.8.

4.1.5.10 Compare the average peak areas of the calibration standard to that of the ammonia sample being tested. Calculate the concentrations of carbon monoxide, oxygen and nitrogen, using the formula below. The result may not exceed the specification in Section 2 of this standard.

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

4.2 Hydrocarbons C₁ – C₃ — This procedure is for the determination of hydrocarbons C₁ – C₃ in ammonia. The sample should be gas phase and analyzed using a gas chromatograph with a flame ionization detector.

NOTE 7: It is good practice to keep carrier gas flowing through the instrument. This prevents the back-diffusion of moisture and carbon dioxide in the air from contaminating the column.

NOTE 8: When not analyzing samples, the dual column switching valve should be set at the molecular sieve position to prevent the silica gel column from drying out.

NOTE 9: The silica gel column will, after repeated use, tend to dry out. This will manifest itself by the peaks of each component becoming separated farther and farther apart. Overall sensitivity will drop as the peak areas decrease due to the peaks spreading out more and more.

NOTE 10: The condition of the silica gel column is restored by allowing the carrier gas to pass through the water-saturated silica gel trap that has been provided. (Normally the carrier gas bypasses this trap.)

NOTE 11: The flame should be extinguished when not in use; also the air and hydrogen is shut off; and the helium flow reduced.

4.2.1 *Detection Limit* — 0.1 ppm (mole/mole).

4.2.2 *Instrument Parameters*

4.2.2.1 *Columns*:

Column 1: 13X molecular sieve, 3.2 mm (1/8 in.) by 3.2 mm (1/8 in.) ss, or equivalent.

Column 2: Silica gel (saturated) 4.6 m (15 ft) by 3.2 mm ss, or equivalent.

4.2.2.2 *Carrier Flow*: 50 mL/min helium.

4.2.2.3 *Sample Volume*: 5 mL.

4.2.2.4 *Column Temperature*: 40°C

4.2.2.5 *Sample Flow*: 150 mL/min.

4.2.2.6 *Air Pressure and Flow*: As specified by the instrument manufacturer's instructions.

4.2.2.7 *Hydrogen Pressure and Flow*: As specified by the instrument manufacturer's instructions.

4.2.3 *Calibration Standards* — 5–10 ppm (mole/mole) methane in nitrogen, 5–10 ppm (mole/mole) ethane, 5–10 ppm (mole/mole) propane in nitrogen.

4.2.4 *Operating Procedure*

4.2.4.1 Install the column switching as shown in Figure 1. Place the dual column switching valve in the molecular sieve column position for methane analysis. Use the silica gel column position for the analysis of C₂ – C₃ compounds.

4.2.4.2 Ignite the flame following the instrument manufacturer's instructions. Allow the system to stabilize for 15 minutes. (This is also indicated by a stable signal output.)

4.2.4.3 Inject the methane in nitrogen standard onto the molecular sieve column to determine the retention time for methane.

4.2.4.4 Inject the ethane and propane in nitrogen standard onto the silica gel column to determine the retention times for these compounds.

4.2.4.5 Inject the calibration standard onto the desired column to determine the peak area for the compound of interest. Record the retention times and peak areas.

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

4.2.4.7 Repeat Section 4.2.4.6.

4.2.4.8 Compare the average peak areas for at least three runs each of the calibration standard to that of the ammonia sample being tested. Calculate the concentration of hydrocarbons C₁ – C₃, using the formula below. The result may not exceed the specification in Section 2 of this standard.

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

4.3 Water — The method for the determination of water in ammonia is based on its decomposition by the reaction 2NH₃ → N₂ + 3H₂, which occurs completely and irreversibly when ammonia is passed over a nickel catalyst at 1000°C. Water passes through the catalyst unchanged. Therefore, the water content of ammonia may be determined by measuring the dewpoint of the stream of hydrogen and nitrogen produced by decomposition of ammonia.

NOTE 12: The system should be checked for leaks prior to use to preclude atmospheric contamination of the sample.

NOTE 13: The moisture analyzer must be calibrated according to the manufacturer's specified method prior to use.

4.3.1 Apparatus (shown in Figure 2)

4.3.1.1 The nitrogen and ammonia are connected to a tee with 3.2 mm (1/8 in.) OD stainless steel tubing, keeping the lines as short as is practical.

4.3.1.2 The nitrogen may be fed from a regulator.

4.3.1.3 The ammonia should be fed through a stainless steel needle valve.

4.3.1.4 The furnace is a Lindberg Model 54032 or equivalent, with an Inconel reaction tube. The reaction tube is 0.6 m (24 in.) long by 25 cm (1 in.) OD and terminates in 3.2 mm (1/8 in.) swaged tubing connectors.

4.3.1.5 The reaction tube is filled with nickel lumps, MCB Catalog number NX305 or equivalent.

4.3.1.6 The outlet of the furnace is connected by a 1.2 m (4 ft) by 3.2 mm (1/8 in.) OD stainless steel tubing to a moisture analyzer suitable for determining the concentration of water. The concentration of water should be a mixture of hydrogen and nitrogen at the specified concentration. This length of tubing is required to permit adequate cooling of the gas between the furnace and the moisture analyzer.

4.3.1.7 The outlet of the moisture analyzer is connected to the inlet of a 0-10 CFH flowmeter.

4.3.1.8 The outlet of the flow meter is connected to an appropriate vent for the disposal of ammonia, nitrogen and hydrogen.

4.3.2 Operating Procedure

4.3.2.1 Start a nitrogen purge flow of approximately 5 CFH.

4.3.2.2 Set furnace to 950°C and allow to equilibrate (approximately 1.5 to 2 hours).

4.3.2.3 With the ammonia cylinder valve closed, disconnect the tubing from the cylinder outlet and open the ammonia inlet needle valve to allow the ammonia sample line to purge with nitrogen. Reconnect the tubing to the cylinder outlet and close the needle valve.

4.3.2.4 Shut off the nitrogen flow.

4.3.2.5 Open the ammonia cylinder valve and adjust the needle valve to obtain a flow of approximately 5 CFH.

4.3.2.6 Gradually increase the furnace temperature, periodically testing the gas for the presence of ammonia by placing a piece of wet red litmus paper in the vent stream. If ammonia is present, the paper will turn blue. When ammonia is no longer found, the appropriate catalyst temperature has been reached.

4.3.2.7 Measure the concentration of water in the nitrogen and hydrogen stream, following the instructions of the moisture analyzer manufacturer. Periodically test the vent stream for the presence of ammonia, as described in Section 4.3.2.6, and adjust the furnace temperature as necessary.

4.3.3 Reporting of Measurement

4.3.3.1 Double the water concentration obtained in Section 4.3.2.7 to correct for the doubling of gas volume by the decomposition of the ammonia.

4.3.3.2 Subtract twice the concentration of oxygen (as measured by the procedure specified in Section 4.3) in the sample from the concentration of water calculated in Section 4.3.3.1. This is necessary because oxygen in the ammonia reacts with the hydrogen formed by decomposition to produce additional water.

4.3.3.3 The concentration of water calculated in Section 4.3.3.2 in the ammonia may not exceed the limit specified in Section 3.

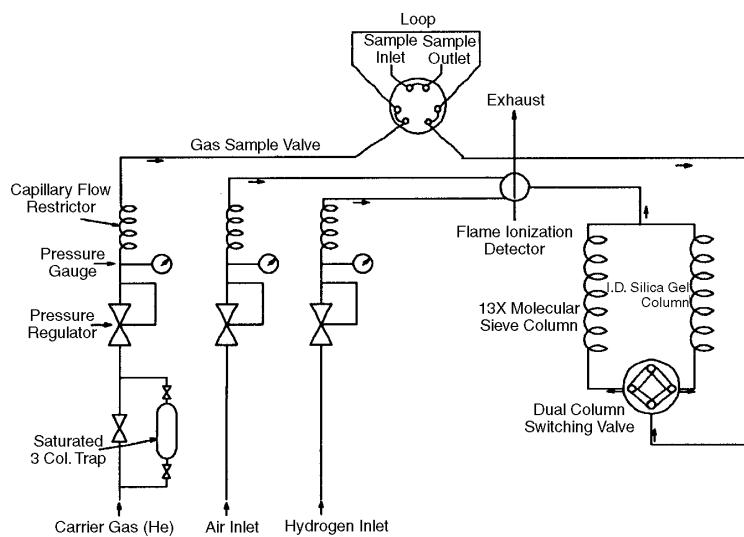


Figure 1

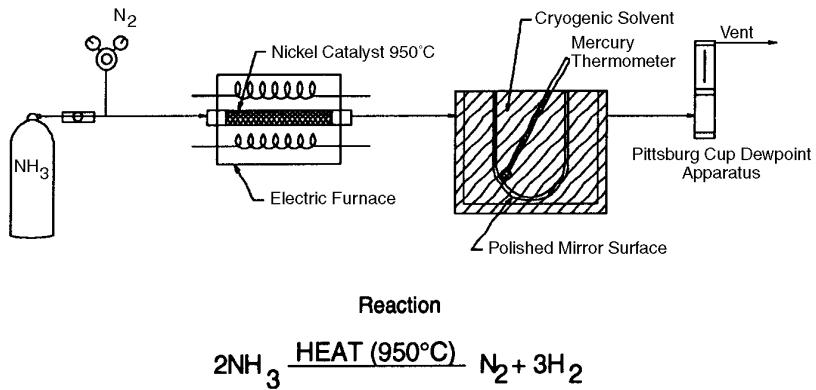


Figure 2
Electronic Ammonia Water Measurement

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SEMI C3.2-0301

SPECIFICATION FOR ARSINE (AsH₃) IN CYLINDERS, 99.94% QUALITY

This specification 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 October 17, 2000. Initially available at www.semi.org January 2001; to be published March 2001. Originally published in 1981; previously published in 1992.

1 Description

1.1 Arsine is a highly toxic, flammable, colorless gas with a disagreeable garlic-like odor. It is a hemolytic poison.

2 Specifications

QUALITY: 99.94%

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm) (See NOTE 1.)</i>
Carbon monoxide and carbon dioxide (CO + CO ₂)	2
Hydrocarbons (methane (CH ₄), ethane (C ₂ H ₆), ethylene (C ₂ H ₄), and acetylene (C ₂ H ₂))	1
Hydrogen (H ₂)	500
Hydrogen Sulfide (H ₂ S)	1
Nitrogen (N ₂)	10
Oxygen (O ₂) + Argon (Ar)	5
Phosphine (PH ₃)	10
Water (H ₂ O)	4
TOTAL LISTED IMPURITIES	533

Metals — See NOTE 2.

Particles — See NOTE 2.

NOTE 1: 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.

NOTE 2: To be determined between supplier and user.

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	77.946	77.946
Boiling point at 1 atm	-62.48°C	-80.46°F
Density of gas at 20°C (68°F) and 1 atm	3.243 kg/m ³	0.202 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	2.695	2.695
Density of liquid at B.P.	1653 kg/m ³	103.96 lb/ft ³

4 Analytical Procedures

4.1 *Carbon Monoxide, Carbon Dioxide, and Hydrocarbons (methane, ethane, ethylene, acetylene)* — This procedure is for the determination of carbon monoxide, carbon dioxide, and hydrocarbons in arsine using a gas chromatograph with a methanizer and a flame ionization detector. (See Note 1 and Figure 1.)

4.1.1 *Detection Limit* — 0.1 ppm each impurity.

4.1.2 *Instrument Parameters*

4.1.2.1 *Column:* Porapak QS, 3.5 m (12 ft) by 2 mm ID (1/8 in OD) ss or equivalent.

4.1.2.2 *Carrier Flow:* 35 mL/min helium.

4.1.2.3 *Support Gases:* Set the flow rates as specified by the instrument manufacturer.

Hydrogen:	30 mL/min added to the carrier gas between the column outlet and the methanizer inlet.
Air:	500 mL/min.

4.1.2.4 *Temperatures:*

Detector	110°C
Injector	40°C
Oven	40°C
Methanizer	370–400°C

4.1.2.5 *Sample Volume:* 3 mL.

4.1.3 *Calibration Standards* — 1–10 ppm each: carbon monoxide, carbon dioxide, methane, ethane, ethylene, and acetylene; balance helium.

4.1.4 *Operating Procedure*

4.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record retention times and peak areas. Order of elution is carbon monoxide, methane, carbon dioxide, acetylene, ethylene, ethane.

4.1.4.2 Inject the sample to be tested in same manner as the calibration standard. Vent the arsine after the ethane peak. 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 those of the arsine sample being tested. Calculate the concentration of each impurity, using the formula below. The results may not exceed specifications in Section 2 of this Standard.

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

4.2 *Phosphine* — This procedure is for the determination of phosphine in arsine using a gas chromatograph with a thermal conductivity detector.

4.2.1 *Detection Limit* — 5 ppm (mol/mol).

4.2.2 *Instrument Parameters*

4.2.2.1 Column: Porapak QS, 3.5 m (12 ft) by 2 mm ID (1/8 in OD) ss or equivalent.

4.2.2.2 Carrier Flow: 35 mL/min helium m.

4.2.2.3 Temperatures:

Detector	80°C
Injector	40°C
Oven	40°C

4.2.2.4 Sample Volume: 3 mL.

4.2.3 *Calibration Standard* — 10–50 ppm (mol/mol) phosphine, 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 time and peak area.

4.2.4.2 Inject the sample being tested in same manner as the calibration standard. Record the retention time and peak area.

4.2.4.3 Repeat 4.2.4.1.

4.2.4.4 Compare the average peak area of the calibration standard to that of the arsine sample being tested. Calculate the concentration of phosphine, using the formula below. The result may not exceed the specification in Section 2 of this Standard.

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

4.3 *Hydrogen, Nitrogen and Oxygen + Argon* — This procedure is for the determination of hydrogen, nitrogen, oxygen + argon in arsine using a gas chromatograph with a helium ionization detector.

4.3.1 *Detection Limits* — 100 ppb (mol/mol) hydrogen, 100 ppb oxygen + argon, 500 ppb 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) ID ss or equivalent
Column 2:	Molecular Sieve 5A, 3 m (10 ft) by 3.2 mm ID ss or equivalent.

4.3.2.2 Carrier Flow: 25 mL/min helium m.

4.3.2.3 Temperatures:

Detector	10°C
Injector	30°C
Oven	30°C

4.3.2.4 Sample Volume: 3 mL.

4.3.3 *Calibration Standard* — 1–10 ppm nitrogen, 1–10 ppm oxygen, 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 the same gas valve. Backflush at the time determined in 4.3.4.1 and record retention times and peak areas. Order of elution is hydrogen, oxygen + argon and nitrogen.

4.3.4.3 Inject the sample being tested in same manner as the calibration standard. 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 those of the arsine sample being tested. Calculate the concentration of each impurity, using the formula below. The results may not exceed the specifications in Section 2 of this Standard.

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

4.4 *Hydrogen Sulfide* — This procedure is for the determination of hydrogen sulfide in arsine using gas a chromatograph with a flame photometric detector.

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

4.4.2 Instrument Parameters

4.4.2.1 Column: Porapak QS 3.5 m (12 ft) by 2 mm ID (1/8 in OD) ss or equivalent.

4.4.2.2 Carrier Flow: 35 mL/min helium. Set the flow rates as specified by the instrument manufacturer.

4.4.2.3 Support Gases:

Hydrogen:	30 – 40 mL/min.
Air:	300 – 400 mL/min.

4.4.2.4 Temperatures:

Detector	75°C
Injector	40°C
Oven	40°C

4.4.2.5 Sample Volume: 1.5 mL.

4.4.3 Calibration Standard — 1–10 ppm hydrogen sulfide, balance helium.

4.4.4 Operating Procedure

4.4.4.1 Inject the calibration standard into the column using a gas sampling valve. Record retention time and peak area.

4.4.4.2 Inject the sample being tested in the same manner as the calibration standard. Vent before arsine peak is eluted using same configuration as in Figure 1.

4.4.4.3 Repeat 4.4.4.1

4.4.4.4 Compare the average peak area of the calibration standard to those of the arsine sample being tested. Calculate the concentration of the hydrogen sulfide, using the formula below. The result may not exceed the specification in Section 2 of this Standard.

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

4.5 Water — This procedure is for the determination of moisture in arsine using continuous flow electrolysis of water in a phosphorus pentoxide (P_2O_5) cell.

4.5.1 Detection Limit — 1 ppm (vol/vol).

4.5.2 Instrument Parameters

4.5.2.1 Flow Requirements — Set the sample flow rate and pressure in accordance with manufacturer's instructions.

4.5.3 Calibration Standards — Construct a calibration curve which contains at least three points covering the range of interest. Verify the standards employed

independently on a condensation dewpoint/frostpoint hygrometer.

4.5.4 Operating Procedure

4.5.4.1 Obtain representative sample of gas to be analyzed and direct to unit as with the standards.

4.5.4.2 Determine the moisture content in the sample gas by comparing the indicated concentration with the calibration curve constructed in 4.5.3. The result may not exceed the specification in Section 2 of this Standard.

NOTE 1: Carrier gases should contain less than 0.1 ppm (mol/mol) carbon monoxide and less than 0.1 ppm (mol/mol) carbon dioxide.

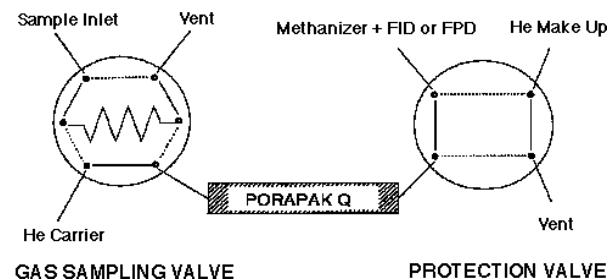


Figure 1
G. C. Configuration

NOTICE: This specification does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this specification to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the specification set forth herein for any particular application. The determination of the suitability of the specification is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials or equipment mentioned herein. This specification are subject to change without notice.

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SEMI C3.33-92 (Reapproved 0303) STANDARD FOR BORON TRICHLORIDE (BCl₃) (PROVISIONAL)

This standard was technically reapproved 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 October 25, 2002. Initially available at www.semi.org December 2002; to be published March 2003. Originally published in 1987; previously published in 1992.

1 Description

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

2 Scope

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

3 Specifications

QUALITY: 99.9995% by wt. Liquid Phase

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)*</i>
Chlorine (Cl ₂)	10 Vapor Phase
Hydrogen Chloride (HCl)	1 Vapor Phase
Phosgene (COCl ₂)	1 Vapor Phase
Aluminum (Al)	0.5 by wt. Liquid Phase
Calcium (Ca)	0.5 by wt. Liquid Phase
Copper (Cu)	0.5 by wt. Liquid Phase
Iron (Fe)	0.5 by wt. Liquid Phase
Magnesium (Mg)	0.5 by wt. Liquid Phase
Nickel (Ni)	0.5 by wt. Liquid Phase
Potassium (K)	0.5 by wt. Liquid Phase
Silicon (Si)	1 by wt. Liquid Phase
Sodium (Na)	0.5 by wt. Liquid Phase
TOTAL IMPURITIES	5 by wt. Liquud Phase

* 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.

4 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 -118° C	1372.8 kg/m ³	85.76 lb/ft ³

SEMI C3.51-1101

SPECIFICATION FOR BORON TRICHLORIDE (BCl₃), 99.98% QUALITY

This specification 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 August 27, 2001. Initially available at www.semi.org September 2001; to be published November 2001. Originally published in 1995.

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.

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

4.1 ISO Standards¹

ISO 6145-1 — Preparation of calibration gas mixtures – Dynamic volumetric methods – Part 1: Methods of calibration

NOTE 1: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Specifications

Quality: 99.98% vapor phase

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)*</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 (excluding metals)	120

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)*</i>
Iron (Fe)	0.5 wt.
Nickel (Ni)	0.5 wt.

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

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

NOTE 3: 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

¹ International Organization for Standardization, ISO Central Secretariat, 1, rue de Varembé, Case postale 56, CH-1211 Geneva 20, Switzerland. Telephone: 41.22.749.01.11; Fax: 41.22.733.34.30
Website: www.iso.ch

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 — 1–10 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 4: 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 Section 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 Section 5 of this standard.

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

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") OD SS or equivalent
Column 2	Porapak QS 60/80 mesh 2.5 m (8 ft.) by 3.2 mm (1/8") 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 — 1–5 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 Section 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.

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

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") ID Dimethyl silicone megabore capillary column (Ristek RTxi)

7.3.2.2 Carrier Gas — Helium: N6.0 25–30 mL/min (See Note 3.)

7.3.2.3 Sample Volume — 2.0 mL.

7.3.2.4 Temperatures:

Column Temperature	100°C
Detector Temperature	110°C

7.3.2.5 Bridge Current — 200 mA.

7.3.3 *Calibration Standard* — 5–10 ppm chlorine in helium.

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

7.3.4 *Operating Procedure*

7.3.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention time and peak area. A sample chromatogram is shown in Figure 3 (see Note 4).

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

7.3.4.3 Repeat Section 7.3.4.1.

7.3.4.4 Compare the average peak areas of the chlorine in the calibration standard with that obtained in the boron trichloride sample being tested. Calculate the concentration of chlorine using the formula below. The results may not exceed the specification in Section 5 of this standard.

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

7.4 Hydrogen Chloride, Phosgene, and Silicon Tetrachloride — This procedure is for the determination of hydrogen chloride, phosgene, and silicon tetrachloride using a Fourier transform infra red (FTIR) analyzer.

7.4.1 *Detection Limits*

- 10 ppm hydrogen chloride
- 1 ppm phosgene
- 0.5 ppm silicon tetrachloride

7.4.2 *Instrument Parameters*

7.4.2.1 *Cell Path Length* — 16 cm × 16 mm ID K Br windows

7.4.2.2 *Wavenumbers:*

Hydrogen Chloride	2970–2990 (cm ⁻¹)
Phosgene	855–865 (cm ⁻¹)
Silicon Tetrachloride	610–630 (cm ⁻¹)

7.4.2.3 The associated sampling system must be equipped with inert gas purging and evacuation capabilities.

7.4.3 *Calibration Standards* — Prepare calibration standards for 10–100 ppm hydrogen chloride, 1–5 ppm

phosgene, and 0.5–5 ppm silicon tetrachloride in boron trichloride.

NOTE 5: To ensure stability, the hydrogen chloride calibration standard should be prepared dynamically using a method defined in ISO 6145/part 1.1986. Phosgene and silicon tetrachloride standards may be prepared by syringe additions to liquid boron trichloride in the cooled sealed container.

7.4.4 *Operating Procedures*

7.4.4.1 Evacuate and purge the sampling system leading to the cell and purge the cell with dry nitrogen for 30 minutes. Fill the cell with calibration standard. Record the absorbance of hydrogen chloride, phosgene, and silicon tetrachloride. A sample spectrum is shown in Figures 4A, 4B, and 4C.

7.4.4.2 Fill the sample into the cell following the same procedures used in Section 7.4.4.1 above. Record the absorbance at the same wave number as the calibration standard. Calculate the concentrations of hydrogen chloride, phosgene, and silicon tetrachloride. The results should not exceed the amount specified in Section 3 of this specification.

$$\frac{\text{Measured Absorbance of the Sample}}{\text{Measured Absorbance of the Standard}} \times \frac{\text{Concentration of Standard}}{\text{Concentration of Sample}} = \frac{\text{Concentration of Sample}}{\text{Concentration of Standard}}$$

NOTE 6: The following method should be adopted for cleaning metallic parts used for sampling hydrogen chloride gas:

- a. Clean in a bath with methanol or isopropanol.
- b. Ultrasonic clean in an appropriate solvent.
- c. Rinse with isopropanol under Class 100 bench.

This cleaning method should be applied when a new cylinder has to be connected and repeated after ten samplings, or if a new cylinder has to be connected.

NOTE 7: Low-pressure plastic parts should be cleaned as follows using 18 MΩcm water:

- a. Fill sampling lines with the following solutions for 30 minutes: NH₄OH 25%: H₂O₂ 35%: H₂O = 1 to 1 to 5 parts by vol.
- b. Wash sampling lines 5 times with water.
- c. Fill sampling lines with the following solutions for 30 minutes: HCl 37%: H₂O₂ 35%; H₂O = 1 to 1 to 5 parts by vol.
- d. Wash sampling lines a minimum of 5 times with water. Terminate if the resistivity of the wash water from the lines is more than 18 MΩcm.

Cleaning of lines, flow meters and valves shall be completed before a new cylinder has to be analyzed and shall be repeated after 10 samplings, or if a new cylinder has to be analyzed. The impingers must be rinsed with water after each sampling. Before a new cylinder has to be analyzed and after five samplings, the impingers must be cleaned using the above described procedures. Drying of the impingers is not recommended (contamination risks).

NOTE 8: Cleaning of cylinder valve.

Clean outside (visible parts, threads) with wipe. Clean the interior parts of the valve-outlet with methanol or isopropanol using a syringe, followed by a purge with filtered gas.

7.5 Iron and Nickel — This procedure is for the determination of iron and nickel in gas phase boron trichloride using atomic absorption or inductive coupled plasma methods.

7.5.1 *Detection Limits*

7.5.2 *Instrument Parameters* — Operate the designated analytical instruments according to the manufacturer's instructions.

7.5.3 *Sampling Procedure*

7.5.3.1 Clean all metallic and plastic components according to the procedures described in Notes 6 and 7. Use 18 MΩcm water.

7.5.3.2 Connect all components of the sampling system.

NOTE 9: The apparatus is charged with sufficient water to just cover the rim of the funnel (approximately 100 ml). A magnetic stirrer is introduced and the apparatus closed.

7.5.3.3 Connect the sampling system to the sample boron trichloride cylinder (see Note 8).

7.5.3.4 Purge with filtered argon (< 0.01 μ) at approximately 1 L/min. for 15 minutes prior to sampling. During this period, the apparatus is inclined so as to allow a higher flow rate without excessive agitation of the water.

NOTE 10: Due to the design of the apparatus, suck-back should be prevented, but this must be guarded against. A bubble rate of about one per second should be set.

7.5.3.5 While observing the sampling apparatus, operate the needle valve to admit sample slowly to the sampling apparatus. Initial absorption will be fast.

7.5.3.6 The sample flow is halted when no further absorption seems to be taking place (i.e., when bubble rate increases at the vent).

7.5.3.7 The argon purge is re-established for a further 15 minutes.

7.5.3.8 The apparatus is allowed to cool and the contents quantitatively transferred to a 250 ml polypropylene flask.

NOTE 11: The solution prepared above will contain some crystals of boric acid which have come out of solution during cooling. For the purpose of analysis, the concentration of boric acid must be reduced. Also the amount of BCl₃ samples need to be determined.

7.5.3.9 A 5 ml aliquot of the solution is titrated against 1 M NaOH using methyl red as an indicator. The quantity of sample boron trichloride taken is then calculated.

7.5.3.10 A sample of the solution is diluted × 10 with water. The aliquot is taken from the supernatant liquid.

7.5.3.11 Analysis is carried out in a suitable analyzer capable of meeting the required detection limits. A blank solution of 1% hydrochloric acid is used. Yttrium is used as an internal standard at a level of 0.2 ppm added to all analysis solutions.

7.5.3.12 Calibration curves are prepared using standard solutions covering the elements required. Traceable standards can be obtained from national and international bodies.

7.5.3.13 Calculate the concentration of iron and nickel content using the calibration curve and the volume of boron trichloride sampled.

H₂: 200 ppm (mole/mole)

O₂+ Ar: 7 ppm "

N₂: 10 ppm "

CH₄: 7 ppm "

CO: 7 ppm "

S: Switching Peak

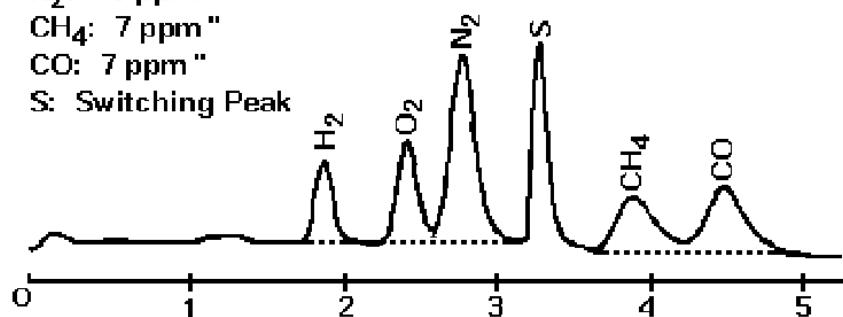


Figure 1

Chromatogram Showing the Determination of Nitrogen in Boron Trichloride

CO₂: 10 ppm (mole/mole)

S: Switching Peak

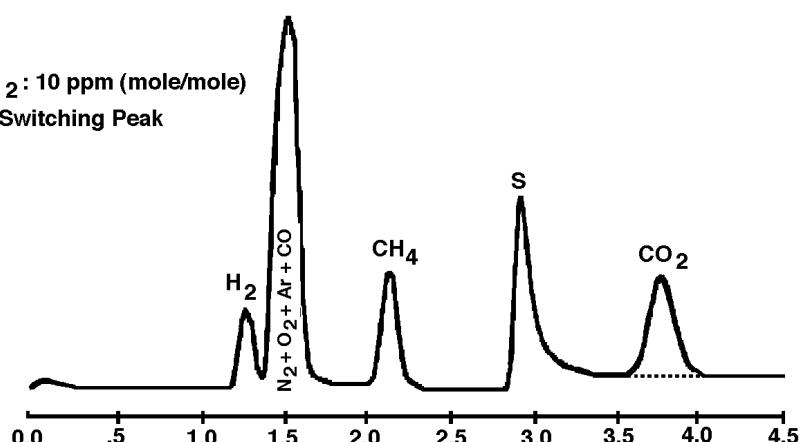


Figure 2

Chromatogram Showing the Determination of Carbon Dioxide in Boron Trichloride

CHANNEL A INJECT 08/12/92 15:55:21

1.55

HYDROGEN CHLORIDE

2.49 2.25

CHLORINE

1.88

2.07

BORON CHLORIDE

3.19

Figure 3

Chromatogram Showing the Determination of Chlorine in Boron Trichloride

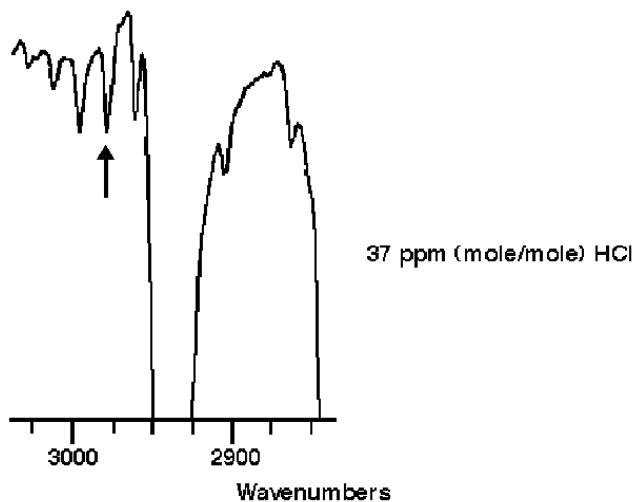


Figure 4A
Infra Red Absorption Spectra of Hydrogen Chloride in Boron Trichloride

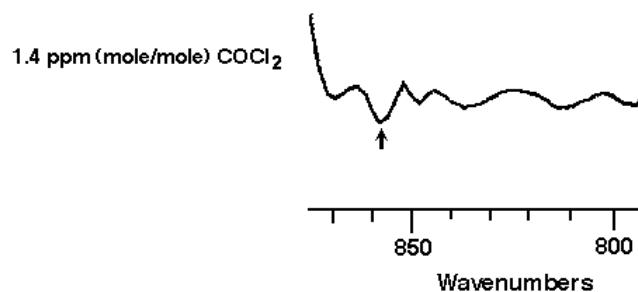


Figure 4B
Infra Red Absorption of Phosgene in Boron Trichloride

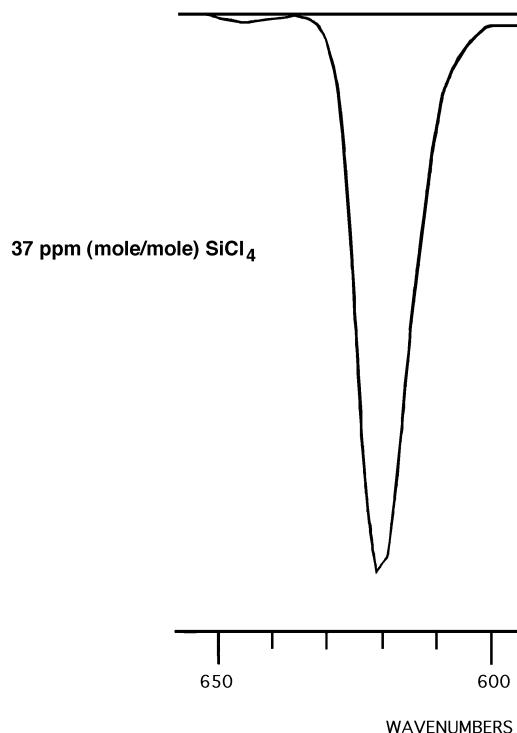


Figure 4C
Infra Red Absorption Spectra of Silicon Tetrachloride in Boron Trichloride

NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

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SEMI C3.27-1102

SPECIFICATION FOR BORON TRIFLUORIDE (BF₃) IN CYLINDERS, 99.0% QUALITY

This specification 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 July 21, 2002. Initially available at www.semi.org October 2002; to be published November 2002. Originally published in 1984; previously published in 1994.

1 Purpose

1.1 The purpose of this document is to provide a specification for Boron Trifluoride (BF₃) used in the semiconductor industry.

2 Scope

2.1 This document covers requirements for Boron Trifluoride (BF₃) used in the semiconductor industry.

2.2 If analytical methods are not complete, the requirements are presented as a guideline.

2.3 This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

3 Description

3.1 Boron trifluoride is a colorless gas. It fumes in moist air and has a pungent odor. Boron trifluoride is nonflammable and does not support combustion.

4 Referenced Standards

4.1 SEMI Standards

SEMI C1 — Specifications for Reagents

SEMI C3 — Specifications for Gases

5 Terminology

5.1 Terminology appropriate to this standard is defined in SEMI C3.

6 Specifications

QUALITY: 99.0%

Impurities	Maximum Acceptable Level (ppm)*
Gases not soluble in water	0.94%
Particles	**
Silicon Tetrafluoride (SiF ₄)	200
Sulfur Dioxide (SO ₂)	21
Total Sulfates (SO ₄ ⁻²)	7
TOTAL IMPURITIES	9628

* 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.

7 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	67.81	67.81
Boiling point at 1 atm	-100.4°C	-149.0°F
Density of gas at 70°C (21.1°F) and 1 atm	3.076 g/L	0.192 lb/ft ³
Specific gravity of gas	2.7	2.7
Critical pressure	49.2 atm	723 psia
Critical Temperature	-12.25°C	9.95°F

8 Analytical Procedures

8.1 *Sampling Procedures* — Boron trifluoride is sampled separately for water-soluble gases and water-insoluble gases. The water-soluble components are collected in a plastic bottle which contains chopped ice. After the ice melts, weighed aliquots of the solution are taken for individual determinations. The water-insoluble components are collected in a modified gas collection tube.

8.1.1 Apparatus

8.1.1.1 Balance, 1000-g capacity, capable of weighing to 0.01 g.

8.1.1.2 Polyethylene bottles (wide mouth, 1-L capacity) with caps.

8.1.1.3 Tygon^R tubing, 6 mm (1/4 in).

8.1.1.4 PTFE tubing, 6 mm (1/4 in).

8.1.1.5 Valve assembly consisting of a needle valve with a stainless steel needle, a steel bushing and steel coupling with a steel gas cylinder connection; a gas take-off consisting of a steel sleeve from the valve with steel pipe leading to a 61 cm Tygon delivery tube. The delivery tube is a temporary connection and is replaced as needed (see Figure 1).

8.1.1.6 Trap: 1-liter polyethylene bottle fitted with a 2-hole Neoprene stopper and two 4-inch pieces of 1/4" PTFE tubing which do not extend below the stopper by more than 1 inch. The Tygon delivery tube is attached to one of the PTFE tubes in the stopper (Inlet to Trap). A 61 cm length of Tygon tubing is attached to the second piece of PTFE tubing in the stopper (Outlet from Trap). The trap is supported with a clamp and ring stand.

8.1.1.7 Modified gas collecting tube (see Figure 2); 250-mL Pyrex sampling tube (modified with a graduated scale on one end).

8.1.2 Reagents

8.1.2.1 Distilled-water ice, cracked.

8.1.2.2 Nitrogen, dry; 35 ppm water, or less.

8.1.3 Operating Procedure - Water-Insoluble Components

CAUTION: Perform all work in a hood.

8.1.3.1 Prepare the gas-collecting tube by washing with distilled water, rinsing with acetone, then drying in an air oven at 100–125°C. Flush with dry nitrogen for about one minute. Leave filled with dry nitrogen; close the stopcocks.

8.1.3.2 Connect the needle valve assembly apparatus to the cylinder to be tested.

8.1.3.3 Attach the gas-collecting tube to the valve nipple with a short piece of dry Tygon tubing.

8.1.3.4 Open the cylinder valve, open the gas collecting tube stopcocks, and very carefully crack open the needle valve.

8.1.3.5 Purge the tube for 5 minutes with a slow stream of BF₃ gas.

8.1.3.6 Close the needle valve, close the gas-collecting tube stopcocks, and then close the cylinder valve.

8.1.3.7 Proceed with the analysis, after removing the gas-collecting tube from the valve nipple.

8.1.4 Operating Procedure - Water Soluble Components

CAUTION: Perform all work in a hood.

8.1.4.1 Before attaching the Tygon delivery tube, purge the sampling valve system for 1 minute with a slow stream of boron trifluoride from the cylinder. Use a separate piece of tubing when purging.

8.1.4.2 Weigh an empty 1-L polyethylene bottle with cap to the nearest 0.01 g. Record this weight as A.

8.1.4.3 Fill the bottle with about 400 g of cracked, distilled-water ice. Re-weigh the bottle with cap to the nearest 0.01 g; record as weight B.

8.1.4.4 A sampling line is used to transfer BF₃ gas from the cylinder to the sample bottle.

8.1.4.5 Connect the Tygon delivery tube from the sampling valve apparatus to the trap. Support the Tygon tubing from the trap outlet; then open the needle valve and adjust until a steady flow of BF₃ is obtained. Insert this Tygon tubing (from the trap outlet) into the polyethylene sample bottle, extending down to the bottom of the bottle. Carefully introduce the BF₃ into the ice until most of the ice is melted, then close the needle valve and remove the Tygon tubing. Remove the sample bottle and cap it. Re-weigh the bottle, cap and contents to the nearest 0.01 g. Record this weight as C.

8.1.4.6 Mix thoroughly by careful inversion until all of the ice melts, being certain to keep the plastic bottle tightly capped so that none of the liquid is lost.

8.1.4.7 Proceed with the determination of water-soluble components, being sure to do the sulfur dioxide first, since opening the bottle repeatedly may result in a considerable loss of sulfur dioxide.

8.1.4.8 Calculations for sample size:

$$B - A = \text{Grams of Ice (H}_2\text{O)}$$

$$C - A = \text{Weight of Solution (BF}_3\text{ Solution)}$$

$$C - B = \text{Weight of Water-soluble Material (grams of BF}_3\text{ gas)}$$

$$\frac{\text{Grams of BF}_3\text{ Gas}}{\text{Grams of Solution}} = \frac{C - B}{C - A} = F$$

$$\frac{\text{Grams BF}_3\text{ needed for Method}}{F} = \frac{\text{Grams Solution}}{\text{Needed for Method}}$$

8.2 Air — This procedure describes the determination of insoluble gases in boron trifluoride gas. A known volume of boron trifluoride gas is absorbed in sodium chloride solution, then any undissolved gases remaining are measured and calculated as % air.

8.2.1 Method Capabilities — The range of this method is 0.05% to 2.3%.

8.2.2 Apparatus

8.2.2.1 The modified gas collecting tube shown in Section 8.1.1.7, Figure 2.

8.2.2.2 Separatory funnel, 2000-mL Pyrex (with 61-cm Tygon^R tubing and pinch clamp attached to stopcock end).

8.2.2.3 Tygon tubing, 1/4 inch.

8.2.2.4 Balance, capable of weighing 1000 grams to 0.01 gram.

8.2.3 Reagents — All reagents used are reagent grade unless otherwise indicated.

8.2.3.1 Water — All water used in the preparation of reagents and in the procedure is either distilled or deionized.

8.2.3.2 Sodium chloride solution — Dissolve 300 g of sodium chloride in 1 liter of water.

8.2.4 Calibration - Gas Collecting Tube

8.2.4.1 Weigh the gas collecting tube to the nearest 0.1 g; weight = A, then clamp the tube securely on a ringstand with the calibrated stem up.

8.2.4.2 Fill the tube with distilled water and allow it to stand with the top stopcock open until it has reached room temperature.

8.2.4.3 Record the room temperature to the nearest 0.01°C; temperature = °C.

8.2.4.4 Close the top stopcock. Drain and dry both of the outer stems. Weigh the water-filled collecting tube to the nearest 0.1 g; weight = B.

8.2.4.5 The net weight of the water in the tube (B - A) in grams is equal to the volume of the tube in milliliters, corrected for the density of water at temperature °C. Determine this volume as follows:

$$\text{Volume in mL} = \frac{(B - A)}{D - E}$$

where D = Density (g/mL) of water at temperature °C (from Table 1).

Table 1 Density vs T°C for Water

Temp. °C	Density g/mL
20	0.99823
21	0.99802
22	780
23	756
24	732
25	0.99707

Temp. °C	Density g/mL
26	681
27	654
28	626
29	597
30	567

8.2.5 Procedure

8.2.5.1 Obtain the sample of BF₃ gas in the gas collecting tube. See Section 8.1.3 for sampling details.

8.2.5.2 Fill the separatory funnel with sodium chloride solution. Attach Tygon tubing and a pinch clamp to the funnel.

8.2.5.3 With a small dropper, fill the glass tubing leading to the stopcock opposite the calibrated end of the gas collecting tube with the salt solution. (Note: the gas collecting tube is inverted at this point.)

8.2.5.4 Open the separatory funnel stopcock and the pinch clamp on the Tygon tubing. Hold the tubing upright and when it completely fills with NaCl solution, slip the tubing onto the end of the gas collecting tube which was previously filled in step 8.2.5.3.

NOTE 1: There should be no air bubbles in the connections between the gas collecting tube and the separatory funnel. A trace of a wetting agent such as Ultrawet (Atlantic Refining Co.) in the salt solution assists in freeing small air bubbles from the walls of connecting tubes.

8.2.5.5 Hold the gas collecting tube vertical, with the calibrated end up and the entire collecting tube below the liquid level of the separatory funnel.

8.2.5.6 Open the lower stopcock of the gas collecting tube carefully and shake the tube to aid in dissolving of the BF₃ gas in the salt solution.

8.2.5.7 Bring the liquid level in the gas collecting tube equal to the liquid level in the separatory funnel.

8.2.5.8 Read and record the volume of undissolved gas in the upper calibrated end of the gas collecting tube and record this volume, to the nearest one-tenth of a milliliter; mL = F. Record the room temperature.

8.2.6 Calculations

8.2.6.1 F = Total milliliters of undissolved gas (from Section 8.2.5.8).

8.2.6.2 E = Total volume of gas collecting tube in mL; see Calibration - Gas Collecting Tube.