



SEMI C3-95 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.

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
cc	cubic centimeter(s)
CGA	Compressed Gas Association
cm	centimeter



Table 1. Abbreviations and Symbols

<i>Abbreviation or Symbol</i>	<i>Explanation</i>
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 2.20).

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 degrees Celsius.

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 *Dewpoint* — The temperature at which liquid first condenses when vapor is cooled.

2.10 *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).

2.11 *Filtration* — (To Be Determined)

2.12 *Heavy Metals* — (To Be Determined)

2.13 *Liquified Compressed Gas* — A gas which under the charged pressure is partially liquid at a temperature of 21.1°C (70°F).

2.14 *Metals* — (To Be Determined)

2.15 *Molecular Weight* — The sum of the atomic weights of all the atoms in the molecule.

2.16 *Nonliquified 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).

2.17 *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.

2.18 *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.



2.19 *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.

2.20 *Rounding Numbers* — The following rules for rounding of measured or calculated values shall be employed:

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

2.20.2 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.

2.20.3 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.

2.20.4 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.

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

2.21 *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.

2.22 *Specification and Specification Limits* — The specification limit should fall above or in the range of the result and its uncertainty.

2.23 *Temperature* — Temperature values shall be expressed in degrees Celsius.

2.24 *Tolerances in Measurements* — Use the following guidelines for mixture tolerances:

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

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

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

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

Concentration of Minor Component	Preparation Tolerance Level
10 - 99 ppm	± A 20%
100 - 999 ppm	± A 10%
0.1 - 50%	± A 5%

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

Range	Preparation Tolerance of Each Minor Component	Analytical Accuracy of Each Minor Component
0 - 9.9 ppm	To Be Determined	To Be Determined
10 - 99 ppm	± A 10%	± A 3%
100 - 999 ppm	± A 5%	± A 2%
0.1% - 50%	± A 4%	± A 2%

2.25 *Calibration Standards* — Calibration standards shall be as close as practical to specification and may not exceed ten times (10×) 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