SEMI C54-1103 (Reapproved 0211)
SPECIFICATIONS AND GUIDELINES FOR OXYGEN

This Standard was technically approved by the global Gases Committee. This edition was approved for publication by the global Audits & Reviews Subcommittee on December 21, 2010. Available at www.semiviews.org and www.semi.org in February 2011; originally published November 2003.

NOTICE: This document was reapproved with minor editorial changes.

1 Purpose

1.1 The purpose of this document is to provide a series of specifications for different grades of Oxygen (O_2) that are used in the semiconductor industry.

2 Scope

2.1 This document covers requirements for all standard grades of oxygen used in the semiconductor industry.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Description

3.1 Oxygen is an odorless, tasteless, noncombustible diatomic gas comprising approximately 22% of the earth's atmosphere; at cryogenic temperatures it is a light blue liquid. Noncombustible; a cryogenic gas derived from liquid air by fractional distillation. Even though O_2 is non-combustible, it is a strong oxidizer that can make other materials combustible depending on its concentration.

4 Limitations

4.1 None.

5 Referenced Standards

5.1 *SEMI Standards*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

SEMI C3 — Specifications for Gases

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

6 Terminology

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

7 Requirements

7.1 Purity and other requirements for the various grades of oxygen are given in Table 1.

Table 1 Impurity and Other Requirements for Various Grades of Oxygen

Previous SEMI Reference #	*C3.22-1000 (Specification)*	*C3.23-1000 (Specification)*	*C3.41-0697 (Specification)*
Grade	2.5	3.8	5.8
Purity	99.5%	99.98%	99.9998%[#1]
Impurities	Maximum Acceptable Level (ppm)[#2]		
Carbon Dioxide and Carbon Monoxide (CO_2 + CO)	5	N/A	N/A
Carbon Dioxide (CO_2)	N/A	1	0.1

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Previous SEMI Reference #	*C3.22-1000 (Specification)*	*C3.23-1000 (Specification)*	*C3.41-0697 (Specification)*
Carbon Monoxide (CO)	N/A	1	0.1
Hydrogen (H_2)	N/A	N/A	0.1
Nitrogen (N_2)	100	30	0.5
Nitrous Oxide (N_2O)	2	1	N/A
Argon (Ar)	N/A	100	1.0
Krypton (Kr)	N/A	10	N/A
Water (H_2O) (ppmv)	1	1	0.1
Total Hydrocarbons expressed as Methane (THC)	25	1	0.1
TOTAL IMPURITIES INCLUDING RARE GASES	5000	N/A	N/A
TOTAL IMPURITIES	N/A	145	2.0
Particles	#3	#3	#3

#1 A purifier is allowed to be used to meet this specification.

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

#3 To be determined between supplier and user.

8 Physical Constants

8.1 The physical constants of oxygen are given in Table 2 (for information only).

Table 2 Physical Constants of Oxygen (for information only)

	Metric Units	*US Units*
Molecular weight	31.999	31.999
CAS Number	7782-44-7	7782-44-7
Boiling point at 1 atm	−183°C	−297.4°F
Density of gas at 21.1°C (70°F) and 1 atm	1.309 kg/m^3	0.082 lb/ft^3
Specific gravity of gas at 21.1°C and 1 atm (air = 1)	1.1049	1.1049
Density of liquid at boiling point	1142 kg/m^3	71.27 lb/ft^3

9 Analytical Procedures for Grade 2.5 Oxygen

9.1 *Carbon Monoxide, Carbon Dioxide, and Nitrous Oxide* — This procedure is for the determination of carbon monoxide, carbon dioxide and nitrous oxide concentration in oxygen using infrared spectrophotometry.

9.1.1 *Detection Limits* — 0.5 ppm carbon monoxide, 0.1 ppm carbon dioxide, and 0.2 ppm nitrous oxide.

9.1.2 *Instrument Parameters*

9.1.2.1 10 meter variable path infrared gas cell.

9.1.2.2 Grating infrared spectrophotometer.

9.1.2.3 Bourdon Vacuum Gauge.

9.1.3 *Calibration Standards* — 10 ppm carbon monoxide, 10 ppm carbon dioxide and 10 ppm nitrous oxide, balance oxygen.

9.1.4 *Operating Procedure*

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9.1.4.1 Pressurize the evacuated gas cell to 50 psia with the calibration standard. Scan the following wave numbers for absorbance: carbon monoxide 2172 cm^{-1}, nitrous oxide 2235 cm^{-1}, and carbon dioxide 2360 cm^{-1}.

9.1.4.2 Evacuate the cell and pressurize to 50 psia with the oxygen sample. Scan the appropriate wave numbers as in ¶ 9.1.4.1.

9.1.4.3 Compare the absorbance of the calibration standard to that of the oxygen sample being tested. Calculate the concentrations of carbon monoxide, carbon dioxide and nitrous oxide, using the formula below. The results may not exceed the specifications in § 7 of this standard.

$$\frac{\text{Sample Absorbance}}{\text{Standard Absorbance}} \times \begin{matrix}\text{Concentration}\\ \text{of Standard}\end{matrix} = \begin{matrix}\text{Concentration}\\ \text{of Sample}\end{matrix} \tag{1}$$

9.2 *Nitrogen* — This procedure is for the determination of nitrogen in oxygen using a gas chromatograph with a thermal conductivity detector.

9.2.1 *Detection Limit* — 10 ppm.

9.2.2 *Instrument Parameters*

9.2.2.1 *Column* — 5A Molecular Sieve, 4.6 m (15 ft.) by 3.2 mm (1/8 in.) stainless steel or equivalent.

9.2.2.2 *Carrier Flow* — 30 mL/min helium.

9.2.2.3 *Sample Volume* — 2.0 mL.

9.2.2.4 *Temperatures*

- *Detector* — 200°C
- *Column Oven* — 21°C

9.2.3 *Calibration Standard* — 100 ppm nitrogen in oxygen.

9.2.4 *Operating Procedure*

9.2.4.1 Inject the calibration standard into the column using a gas-sampling valve. Record the retention time and peak area (see Note 1).

NOTE 1: Introduce the calibration standard as many times as necessary to achieve the desired precision.

9.2.4.2 Inject the sample to be tested in same manner as the calibration standard. Record the retention time and peak area.

9.2.4.3 Repeat ¶ 9.2.4.2.

9.2.4.4 Compare the average peak area of the calibration standard to that of the oxygen sample being tested. Calculate the concentration of nitrogen, using the formula below. The result may not exceed the specification in § 7 of this standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \begin{matrix}\text{Concentration}\\ \text{of Standard}\end{matrix} = \begin{matrix}\text{Concentration}\\ \text{of Sample}\end{matrix} \tag{2}$$

9.3 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons (THC) in oxygen using a continuous flow flame ionization detector equipped total hydrocarbon analyzer (see Notes 2, 3, and 4).

NOTE 2: The 0 to 1 range can be used provided that zero and span gas standards in oxygen with known levels of hydrocarbons between 0 to 1 ppm are used in the calibration of the analyzer.

NOTE 3: As the flow rate and heat capacity of the matrix gas affect the instrument output, the zero and span gas matrices must coincide with that of the sample gas.

NOTE 4: The effective response of a flame ionization detector-equipped total hydrocarbon analyzer to different hydrocarbons can vary and must be approximated. However, the response of the most common hydrocarbon impurities in oxygen can be accurately totaled and compared to methane.

9.3.1 *Detection Limit* — 0.1 ppm

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9.3.2 *Flow Requirements*

9.3.2.1 High purity, hydrocarbon-free (less than 1.0 ppm) hydrogen: 35 to 40 mL/min or 40% hydrogen in either helium or nitrogen matrix at 75 to 80 mL/min.

9.3.2.2 Dry, hydrocarbon-free (less than 1.0 ppm) air: 350 to 400 mL/min.

9.3.2.3 Set sample flow rates in accordance with the instrument manufacturer's instructions.

9.3.3 *Calibration Standards*

9.3.3.1 Zero oxygen with known quantity of hydrocarbons at less than 0.5 ppm level.

9.3.3.2 The upper level span gas must be in oxygen and the CH_4 level must not exceed four times the concentration of the specification.

9.3.4 *Operating Procedure*

9.3.4.1 Do not change the flow settings for hydrogen, air, and sample once established.

9.3.4.2 Introduce the zero oxygen with known quantity of hydrocarbons and, using the 0 to 10 ppm range, set the needle (or output) to read the correct level using the zero adjust knob.

9.3.4.3 Introduce the span gas standard in oxygen and, using the span adjust knob, set the needle (or output reading) to match the level of hydrocarbons in the span gas.

9.3.4.4 Introduce oxygen sample into the analyzer and read the quantity of hydrocarbons on the analyzer meter. The result may not exceed the specification in § 7 of this standard.

9.4 *Water* — This procedure is for the determination of trace moisture (water) in oxygen using a continuous flowing, cooled-surface condensation, dewpoint/frost-point hygrometer (see Notes 5, 6, and 7).

NOTE 5: The sampling system and hygrometer must be designed to operate under the sample pressure, or the sample pressure must be reduced (by a regulator with a diaphragm of stainless steel or other suitable material) to accommodate the pressure restrictions of the analytical hygrometer.

NOTE 6: The National Institute of Standards and Technology (NIST) provides calibration services for the thermometers used in dewpoint/frostpoint hygrometers.

NOTE 7: This method is not applicable if other constituents in the gas will condense before water vapor, for example, carbon dioxide and/or oil contamination.

9.4.1 *Detection Limit* — 0.6 ppm (vol/vol) at −79°C (−110°F).

9.4.2 *Flow Requirements*

9.4.2.1 Set sample flow rate and pressure in accordance with the instrument manufacturer's instructions.

9.4.2.2 Gas must flow past the chilled mirror where optic means are provided to detect the deposit (or frost) and to read the thermometer measuring the temperature of the mirror.

9.4.3 *Calibration Standard* — A calibration thermometer designed to indicate temperatures around the −79°C (−110°F) is required.

9.4.4 *Operating Procedure*

9.4.4.1 Obtain a continuous flow of sample gas from the source using a clean stainless steel sampling line which has been purged dry after exposure to ambient moisture.

9.4.4.2 After pre-purging with a dry gas, allow the sample gas to flow through the sampling system and the dewpoint/frostpoint hygrometer for one hour to 24 hours to allow the entire system to reach equilibrium with regard to moisture content.

9.4.4.3 After equilibrium has been reached, cool down the mirror to determine the actual dewpoint/frostpoint of the sample gas. Follow the manufacturer's recommendations to create the temperatures needed.

9.4.4.4 Continue to verify the dewpoint/frostpoint for at least 30 minutes after a stable reading has been confirmed.

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9.4.4.5 Correct the dewpoint reading from the measured pressure to 1 atm of pressure. The result may not exceed the specification in § 7 of this standard.

9.5 *Assay of Oxygen* — This procedure describes the assay of oxygen using an "Orsat" device. This is a volumetric determination of nonabsorbable/reactable gas.

9.5.1 *Method Capability* — 99.9% Oxygen.

9.5.2 *Instrument Parameters* — See Figure 1.

9.5.2.1 Equipment:

- 1 to 100 mL calibrated certified burette, calibrated every 0.1 mL for the top 5 mL equipped with a 3-way stopcock on the top.
- 3 to 250 mL aspirator bottles.
- 1 one-hole rubber stop.
- One 1/16 in. OD soft copper wire made into hollow coils 3/8 in. OD by 3/4 in. long.
- Sufficient 3/8 in. OD tygon tubing.
- Sufficient surgical rubber tubing.
- 1 gallon distilled water.
- 2 lbs technical grade ammonium chloride.
- 1 gallon 27% ammonium hydroxide.

9.5.2.2 *Test Solution Preparation*

9.5.2.2.1 Two pounds of technical grade ammonium chloride are dissolved in one gallon of distilled water and stored in a glass jar. One-half gallon of this solution shall be combined with one-half gallon of 27% ammonium hydroxide.

9.5.3 *Equipment Assembly* — The equipment is to be assembled, as per Figure 1, in a suitable wood or metal frame so arranged that the aspirator bottle connected to the burette can be raised and lowered, as required, to transfer the gas being analyzed to and from the center aspirator bottle which is filled with copper coils. The two leveling bottles should be filled half full of testing solution and the buret and aspirator bottle, which contains the copper coils, should be completely filled.

9.5.4 *Operating Procedure*

9.5.4.1 Before analyzing the sample, perform a series of analyses using a source of oxygen of which the purity has been previously determined. This procedure is necessary to age the test solution properly and eliminate any air bubbles which may become trapped in the apparatus. Only after three consecutive analyses have been run, indicating the known purity, should you proceed with testing (see Note 8).

NOTE 8: This procedure will be required only immediately after changing the test solution or contaminating the apparatus with air.

9.5.4.2 Attach rubber tubing from the regulator of the cylinder being tested to the stopcock of the burette. Rotate the stopcock so as to draw the sample into the gas measuring burette. Collect a little more than 100 mL (i.e., below the zero mark) in the burette, and then rotate the stopcock so as to shut off the oxygen flow and remove the rubber tubing attached to the intake of the burette. If the gas sample in the burette is below the zero mark raise the leveling bottle #1 so that the "water" level in the burette is exactly zero (see Figure 2a).

9.5.4.3 Rotate the stopcock to connect the aspirator bottle #3 containing the copper coils, and transfer the oxygen into this aspirator bottle by raising the leveling bottle #1 (see Figure 2b). Invert bottle #3 containing the copper coils so that both the inlet stopper and the sided outlet are in a downward position and shake gently for 1 to 2 minutes. Stand test bottle #3 on its base and lower the leveling bottle so as to withdraw any residual gases into the gas measuring burette (see Figure 2c). Transfer at least 25 to 30 mL of test solution from the left-hand leveling bottle #2 into the right-hand leveling bottle #l, through the copper coil bottle #3, and the gas burette while, at the same time,

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gently rocking and tapping the center bottle (see Figure 2d). This will move any bubbles that might cling to the copper coil into the gas measuring burette.

9.5.4.4 Turn the stopcock off and raise the leveling bottle #l so that its liquid level is the same as the liquid level inside the upper portion of the gas burette (see Figure 2e). When the levels of the leveling bottle and the gas burette are the same, read the gas purity markings on the burette at the liquid level at this point.

9.5.5 *Maintenance*

9.5.5.1 Copper coils should be added to the test bottle as required to keep the bottles completely full. The gas burette should be kept clean with a strong detergent solution to eliminate drops of liquid that might hang up in the gas space and give incorrect purity readings. No readings should be taken when the space above the liquid level of the gas burette has any liquid drops hanging in it, as this will give an erroneous purity reading.

9.5.5.2 The solution must be replaced occasionally as it becomes exhausted. The necessity of replacement may be determined when the color begins to turn green, and by the increased length of shaking time required to get a minimum acceptable purity from the oxygen in a cylinder of known purity. Suspect an exhausted solution if analysis of a cylinder of known purity does not read the correct purity.

9.5.6 *Standardization*

9.5.6.1 The method of analysis stated above can be used as a primary standard, meaning cylinders analyzed by this method can be used as standards of measurement on electronic analyzers used to measure oxygen.

9.5.6.2 This method is specific to oxygen when carbon dioxide is not present in the sample gas. When carbon dioxide is one of the components of the sample gas, the carbon dioxide must first be scrubbed from the sample.

10 Analytical Procedures for Grade 3.8 Oxygen (see Note 1 in § 9.2.1.1)

NOTE 9: All gases used in the analysis of the sample should contain not more than 10% of the sample value of the component of interest unless otherwise specified.

10.1 *Argon* — This procedure is for the determination of argon using a gas chromatograph with a thermal conductivity detector.

10.1.1 *Detection Limit* — 25 ppm.

10.1.2 *Instrument Parameters*

10.1.2.1 *Column* — 3.6 m (12 ft.) by 3.2 mm (1/8 in.) stainless steel tubing packed with Molecular Sieve 5A, 60/80 mesh, washed to remove fines and activated at 300°C for 24 hours or equivalent.

10.1.2.2 *Carrier Flow* — 45 mL/min helium.

10.1.2.3 *Sample Volume* — 1 to 3 mL.

10.1.2.4 *Temperatures*

- *Detector* — 40°C
- *Column* — −50°C

10.1.3 *Calibration Standard* — 50 to 150 ppm argon, balance helium.

10.1.4 *Operating Procedure (refer to Figure 3)*

10.1.4.1 With the valve in Position A, purge the sample loop with the calibration standard. Switch the valve to Position B to inject the sample into the column. After the argon has been detected, switch the valve back to Position A to backflush the oxygen from the column. Record the peak area and retention time.

10.1.4.2 Inject oxygen sample to be tested in the same manner as in ¶ 10.1.4.1. Record the retention times and peak areas.

10.1.4.3 Repeat ¶ 10.1.4.2.

10.1.4.4 Calculate the concentration of argon in the sample, using the formula below. The result may not exceed the specification § 7 of this standard.

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